

Universidade do Minho
Escola de Engenharia

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**An Innovative Cementitious
Geocomposite for Smart
Infrastructure Monitoring**



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An Innovative Cementitious Geocomposite for Smart Infrastructure Monitoring

Tese de Doutoramento em Engenharia Civil

Trabalho efetuado sob a orientação do

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Resumo.

Esta tese apresenta um trabalho de investigação abrangente sobre materiais geocompósitos cimentícios (areia estabilizada) com capacidade de monitorização. Estas misturas usam materiais inteligentes projetados para monitorização inteligente de infraestruturas em sistemas de transporte e ferrovias. O estudo explora a utilização de nanotubos de carbono híbridos (CNT) e nanoplaquetas de grafeno (GNP) como agentes de funcionalização em compósitos cimentícios, visando aprimorar o desempenho mecânico e permitir a monitorização em tempo real. Numa fase inicial, foram otimizadas técnicas de dispersão dos nanomateriais utilizando 10% de Pluronic F-127 e 50% de tributyl phosphate (TBP), as quais foram submetidas a um banho de ultrassons. As diferentes dispersões aplicadas em argamassas cimentícias devido à semelhança com a areia estabilizada cimentícia e à escassez de investigações neste domínio. O conceito foi validado com sucesso, levando à produção de geocompósitos cimentícios auto-sensíveis. A otimização adicional do cimento, do teor de água, das cargas e dos métodos de medição, levou à obtenção de melhorias significativas no desempenho físico, mecânico e de piezoresistividade do geocompósito, devido ao sucesso da incorporação bem-sucedida de nano materiais. Por outro lado, foi estudado o impacto da utilização de materiais fibrosos na capacidade de sensorização dos geocompósitos, resultando igualmente em melhorias nas propriedades mecânicas, mas com capacidade reduzida de auto monitorização. Dada a complexidade da capacidade de sensorização dos geocompósitos, foi realizada uma investigação abrangente dos vários parâmetros que influenciam o comportamento piezoresistivo. O estudo avaliou também o tamanho, a disposição dos elétrodos, a temperatura, a humidade e as condições de carga, levando à obtenção de resultados muito importantes para a otimização das capacidades de sensorização. O aumento do comprimento, da largura e da espessura da matriz circundante às cargas condutoras, gerou um aumento da resistividade elétrica, tendo as mudanças de comprimento revelado um efeito mais significativo. Maiores aspect ratios of conductive fillers originaram limiares de percolação reduzidos, indicando maior condutividade elétrica. Os geocompósitos apresentaram uma correlação positiva entre a temperatura e os valores do fator de medida- gauge factor (GF), mostrando potencial para aplicações de deteção de temperatura. No entanto, verificou-se que o aumento do teor de humidade afetou negativamente a compatibilidade de deteção, resultando em valores reduzidos de GF. Para além disso, a frequência de carga influenciou igualmente os valores de mudança fracional da resistência elétrica - fractional change in resistance (FCR). Frequências mais altas na região elástica resultaram num maior FCR, enquanto que, em situações para além do limite elástico, frequências mais altas reduziram o valor do FCR. Os geocompósitos demonstraram uma sensibilidade significativa a mudanças de deformação, tornando-os adequados para monitorização de tráfego e do estado de conservação em diversos cenários operacionais. A relação entre valores de GF, módulo e análise de correlação de imagens digitais - *digital image correlation* (DIC) ofereceu uma abordagem promissora para avaliar o comportamento mecânico e os níveis de danos dos geocompósitos sob diferentes condições de carga.

Palavras-chave: Geocompósitos Cimentícios Inteligentes; Monitorização de Infraestruturas Inteligentes; Nanoplaquetas de Grafeno; Nanotubos de Carbono; Piezoresistividade; Transporte e Ferrovia.

Abstract.

This thesis presents a comprehensive investigation into self-sensing cementitious geocomposites (stabilized sand), a class of smart materials designed for intelligent infrastructure monitoring in transportation and railway systems. The study focuses on the potential of hybrid carbon nanotubes (CNT) and graphene nanoplatelets (GNP) as fillers in cementitious composites, aiming to enhance structural performance and enable real-time monitoring. In the preliminary phase, optimized dispersion techniques using 10% PF-127 and 50% TBP, subjected to sonication, were developed and tested on cementitious mortar due to its resemblance to cementitious stabilized sand and the scarcity of research in this domain. The concept was successfully validated, leading to the creation of self-sensing cementitious geocomposites. Through further optimization of cement, water content, fillers, and measurement methods, significant improvements in the geocomposite's physical, mechanical, and piezoresistivity performance were achieved, thanks to the successful incorporation of nanomaterials. The impact of fibrous materials on the smart geocomposite's capabilities was examined, resulting in improved mechanical properties but reduced sensing ability. Given the complexity of the geocomposite's sensing capacity, a comprehensive investigation into various parameters influencing piezoresistive behaviour was conducted. The study assessed size, electrode layout, temperature, humidity, and loading conditions, leading to valuable insights for optimizing sensing capabilities. Increasing the length, width, and thickness of the matrix surrounding conductive fillers raised electrical resistivity, with length changes having the most significant effect. Higher aspect ratios of conductive fillers led to reduced percolation thresholds, indicating enhanced electrical conductivity. The geocomposite displayed a positive correlation between temperature and gauge factor (GF) values, showing potential for temperature-sensing applications. However, increased moisture content negatively affected sensing compatibility, resulting in decreased GF values. Loading frequency also influenced the fractional change in resistance (FCR) values. Higher frequencies in the elastic region resulted in increased FCR, while beyond the elastic threshold, higher frequencies reduced FCR. The geocomposite demonstrated exceptional sensitivity to strain changes, making it suitable for traffic and health monitoring in diverse operational scenarios. The relationship between GF, modulus, and digital image correlation (DIC) analysis provided a promising approach to assess mechanical behaviour and damage levels of the geocomposite under different loading conditions. Additionally, the composite's self-heating properties offered innovative applications in temperature monitoring, de-icing, and infrastructure maintenance. Overall, this thesis significantly contributes to the advancement of smart materials in intelligent structures. The self-sensing cementitious geocomposites hold immense potential to revolutionize infrastructure monitoring, ensuring sustainability, resilience, and enhanced performance in critical transportation and railway systems.

Keywords: Carbon nanotubes (CNT); Graphene nanoplatelets (GNP); Piezoresistivity; Self-sensing Cementitious Geocomposites; Smart Infrastructure Monitoring; Transportation and Railway.

Contents.

Chapter 1.	Introduction	1
1.1	Introduction to Smart Infrastructure Monitoring	1
1.2	Background on Existing Monitoring Techniques:	3
1.3	Problem Statement:	6
1.4	Overview of the self-sensing cementitious composites:	8
1.5	Scope of the Study:	10
1.6	Research Objectives:	11
1.7	Thesis Outline:	12
1.8	List of publications subjacent to this thesis:	16
1.9	Contributions and Originality:	16
Chapter 2.	State of The Art	20
2.1	Introduction	22
2.2	Metal-Based Self-Sensing Composites	23
2.3	Carbon Black–Based Self-Sensing Composites	25
2.4	Carbon Fibre–Based Self-Sensing Composites	26
2.4.1.	Carbon Short Fibres	26
2.4.2.	Continuous Carbon Fibres	30
2.4.2.1.	Non-polymeric, Continuous Carbon Fibre–Based, Self-Sensing Cementitious Composites	30
2.4.2.2.	Polymeric, Continuous Carbon Fibre–Based, Self-Sensing Cementitious Composites	31
2.5	Carbon Nanotubes and Carbon Nanofiber –Based, Self-Sensing Cementitious Composites	34
2.6	Graphene-Based, Self-Sensing Cementitious Composites	37
2.7	Influential Factors	38
2.7.1.	Conductive Filler Dispersion	38
2.7.2.	Electrode Configuration and Resistance Measurement	40
2.7.3.	Temperature	41
2.7.4.	Water-to-Cement Ratio	42
2.7.5.	Sand-to-Cement Ratio	42
2.7.6.	Freeze–Thaw Cycles	42

2.7.7. Dry–Wet Cycles	43
2.8 Conduction Theories and Equivalent Circuit Models in Self-Sensing Cementitious Composites ..	43
2.8.1. Conduction Theories	43
2.8.2. Equivalent Circuits	45
2.9 Applications	46
2.9.1. Structural Health Monitoring	46
2.9.2. Traffic Monitoring.....	47
2.10 Challenges	48
2.11 Concluding Remarks.....	52
Chapter 3. Dispersion Method Development	54
3.1 Introduction	56
3.2 Materials and Methods	58
3.2.1. Raw materials.....	58
3.2.2. CNT+GNP dispersion	60
3.2.3. Cementitious composite fabrication.....	60
3.2.4. Characterization of CNT+GNP Aqueous Suspensions.....	61
3.2.5. Characterization of the CNT+GNP cementitious composites.....	62
3.3 Results and Discussion	63
3.3.1. Aqueous Suspension Characterization	63
3.3.2. Cementitious Composite Characterization	73
3.4 Conclusion	81
Chapter 4. Self-sensing Cementitious Mortar Development.	85
4.1 Introduction	87
4.2 Raw Materials and Characterization	89
4.2.1. CNT+GNP Dispersion Method	90
4.2.2. Cementitious Composite Fabrication	92
4.2.3. Mechanical and Microstructural Characterization.....	93
4.2.4. Piezoresistivity Measurement	94
4.3 Results and Discussion	95
4.3.1. Mechanical Characterization of Cementitious Composite	95

4.3.2. Microstructural Investigation	100
4.3.3. Durability of CNT+GNP reinforced cementitious mortar	102
4.3.4. Piezoresistive Behavior	103
4.4 Conclusion	113
Chapter 5. Self-sensing Cementitious Geocomposite Development	115
5.1 Introduction	117
5.2 2. Materials and Methods	118
5.2.1. Raw Materials.....	118
5.2.2. CNT and GNP Dispersion Method	119
5.2.3. Specimen Fabrication	120
5.2.4. Mechanical and Microstructural Characterization.....	121
5.2.5. Piezoresistivity Measurement	122
5.3 Results	123
5.3.1. Compaction Tests Results	123
5.3.2. Microstructural Investigations.....	126
5.3.3. Cement Hydration Evaluation	128
5.3.4. Mechanical Characterizations.....	131
5.3.5. Durability Investigation	132
5.3.6. Piezoresistivity Behavior Investigation	133
5.4 4. Conclusion	145
Chapter 6. Effects of The Fibers on Physical, Mechanical, and Piezoresistivity Performances of Cementitious Geocomposite.	146
6.1 Introduction	148
6.2 Materials and Methods	150
6.2.1. Raw Materials.....	150
6.2.2. Dispersion, fabrication and characterization methods	153
6.3 Results and analysis	160
6.3.1. Fiber Surficial Characterizations	160
6.3.2. Compaction and Microstructural Investigations.....	163
6.3.3. Mechanical Properties	173

6.3.4. Durability evaluations	176
6.3.5. Piezoresistivity Investigations.....	176
6.4 Sustainable Issues	183
6.5 Conclusions.....	183

Chapter 7. Effects of Size and Electrodes Layout on Sensing Potential of Cementitious Geocomposite 186

7.1 Introduction	188
7.2 Materials and Methods	190
7.2.1. Materials	190
7.2.2. Samples Preparation	191
7.2.3. Electrical Resistivity Measurement.....	193
7.2.4. Piezoresistivity Investigations.....	195
7.2.5. Mechanical properties investigation	197
7.3 Results	197
7.3.1. Effects of Length Changes.....	197
7.3.2. Effects of Cross-Section Changes	199
7.3.3. Effects of Volume Changes.....	203
7.3.4. Effects of Fillers' Aspect Ratio	204
7.3.5. Piezoresistivity Investigations.....	206
7.3.6. Mechanical Investigations	208
7.4 Conclusions.....	210

Chapter 8. Effects of Temperature, Humidity Loading Type and Speed on Sensing Potential of Cementitious Geocomposite 212

8.1 Introduction	214
8.2 Materials and Methods	216
8.2.1. Raw materials.....	216
8.2.2. CNMs dispersion	218
8.2.3. Specimen fabrication	219
8.2.4. Mechanical and microstructural investigations.....	219
8.2.5. Self-heating assessment.....	220

8.2.6. Self-sensing investigations.....	220
8.3 Results and discussion.....	224
8.3.1. Mechanical assessment.....	224
8.3.2. Microstructural investigation.....	225
8.3.3. Self-heating performance	228
8.3.4. Self-sensing performance	230
8.4 Conclusions.....	241
Chapter 9. Conclusions and Future Research Line.	244
9.1 Introduction	245
9.2 Conclusions.....	245
9.3 Future Research Directions:	247
Bibliography.	247

List of Figures.

Chapter 1

Figure 1-1. The schematic final representation of the thesis outline and relations among the thesis' objectives, chapters and main topics.....	13
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Chapter 2

Figure 2-1. The effect of CSF aspect ratio on electrical resistivity and conductivity of cementitious composites, [81, 132, 133].	26
Figure 2-2. Correlation between strain, stress, and electrical resistance [135].....	28
Figure 2-3. (a) Continuous carbon fibre textile (b) Correlation between strains measured by strain gauges and a textile sensor [150]	31
Figure 2-4. (a) Morphology of hybrid CF–GF composite rods (b) Variation of stress (solid line) and frictional resistance (dotted line) during monotonic tensile testing of hybrid GF–CF rods with GF and CF contents of 48 and 0.2 vol%, respectively, [164]	32
Figure 2-5. Schema of hybrid carbon particle–GF self-sensing bars [165].....	33
Figure 2-6. (a) Manufacturing process of a BCR. (b) Cross-section and braided surface [170].....	34
Figure 2-7. Components of hybrid GF–CNM self-sensing rod [166]	36
Figure 2-8. Fractional change in electrical resistance and change in load, and midspan deflection state at points 1, 2, and 3, [164].....	36
Figure 2-9. Different electrode configurations for resistance / voltage measurements in cementitious composites.....	40
Figure 2-10. Setup of a non-contact electrical resistance measurement device and its equivalent circuit.	41
Figure 2-11. Typical application forms of self-sensing composites for structural health monitoring: a) bulk form, b) coating form, c) sandwich form, d) bonded form, and e) embedded form.	47

Chapter 3

Figure 3-1. CNT and GNP Raman analysis.....	58
Figure 3-2: Morphology of (a) CNTs and GNPs dry mix-(b) CNT (c) GNP	59
Figure 3-3: Chemical structure of : (a) Pluronic F-127 _ (b) Sodium dodecylbenzene sulfonate (SDBS)_ (c) Tributyl phosphate (TBP).....	59

Figure 3-4: Wavelength and maximum absorbance of CNT+GNP suspensions prepared with Pluronic_F127.....	64
Figure 3-5: Wavelength and maximum absorbance of CNT+GNP suspensions prepared with SDBS.....	64
Figure 3-6: Morphology of dry CNT+GNP extracted agglomerate from aqueous suspension: (a) Prepared by Pluronic F-127_ (b) Prepared by SDBS.....	68
Figure 3-7: Optical micrographs of CNT/GNP suspensions, (a) Pluronic: 1%CG-3H-10%P+TBP+40°C, (b) SDBS: 1%CG-3H-5%S+TBP+40° C	71
Figure 3-8: Compressive and Flexural strength of cementitious composites after 28 days of hydration period.....	74
Figure 3-9: SEM morphology of reinforced cementitious composite by (a): CNT+GNP/SDBS_ (b): CNT+GNP/Pluronic	76
Figure 3-10: SEM morphology of CNT+GNP/SDBS sample after mechanical strength measurement ...	77
Figure 3-11: Capillary water absorption of cementitious composites.....	78
Figure 3-12: Thermal analysis of cementitious composite_ (a): TGA analysis - (b): DSC analysis.....	79
Figure 3-13: Energy-dispersive X-ray spectroscopy (EDX) of cementitious composites and their normalized elemental analysis	80
Figure 3-14: Thermography of cementitious composite at different times: (a) Immediately after heating (both cross-section and side-view), (b) After one hour, (c) After two hours.	83

Chapter 4

Figure 4-1: Morphology of CNTs and GNPs dry mix.....	89
Figure 4-2: Chemical structure of: (a) Pluronic F-127_ (b) Tributyl phosphate 97%[145].....	90
Figure 4-3: Grain size curves of cement and sand.....	91
Figure 4-4: CNT and GNP Raman analysis results	91
Figure 4-5: Representation of the geometrical parameters and resistivity measurement principle.....	92
Figure 4-6: Freeze-Thaw temperature cycle	94
Figure 4-7: a) Flexural, and b) compressive strength of CNT+GNP incorporating cementitious composites at different curing ages.....	96
Figure 4-8: Normalized compressive (Comp) and flexural (Flex) strength of plain and CNMs reinforced mortar for different curing times.....	97
Figure 4-9: Plain and CNMs reinforced rupture modulus in different hydration periods: a) Flexural, b) Compressive.....	97

Figure 4-10: Images of specimens after tests: a) specimen GC(0.5%) after flexural test, b) specimen GC(0.5%) after compression test.	98
Figure 4-11: SEM morphology of CNT+GNP specimen after mechanical strength measurement	100
Figure 4-12: Apparent porosity of nano intruded cement mortars.....	100
Figure 4-13: Dry bulk density of nano intruded cement mortars	101
Figure 4-14: The weight loss rate of cementitious composites in different freeze-thaw cycles.	102
Figure 4-15: Relative dynamic elasticity modulus of CNT+GNP intruded cement mortar after 180 freeze-thaw cycles.....	103
Figure 4-16: The electrical resistance of reinforced cementitious composite by different CNT+GNP concentration and curing age.	104
Figure 4-17: Buried GNPs and CNTs among of the hydration products.....	104
Figure 4-18: SEM image of incorporated CNT+GNP into the cement mortar	106
Figure 4-19: Schematic of tunneling effect for CNT/CNT and CNT/GNP cases.....	106
Figure 4-20: The fractional change in resistivity together with cyclic compression response for reinforced cementitious composite by different CNT+GNP concentration	107
Figure 4-21: The fractional change in resistivity together with axial strain under cyclic compression loading	108
Figure 4-22: The ratio of rupture compressive modulus (E_{cr}) to compressive modulus at 10KN loading (E_{c10})	109
Figure 4-23: Variation of strain with the fractional change in resistivity for CNT+GNP reinforced specimens.....	109
Figure 4-24: The fractional change in resistivity together with the cyclic flexural response for reinforced cementitious composite by different CNT+GNP concentration	110
Figure 4-25: The fractional change in resistivity together with axial strain under cyclic compression loading	111
Figure 4-26: The ratio of rupture flexural modulus (E_{fr}) to flexural modulus at 500N loading (E_{f500}).....	112
Figure 4-27: Variation of strain with the fractional change in resistivity for CNT+GNP reinforced specimens under flexural cyclic load	112
Figure 4-28: Variation of gauge factors in flexural and compression loading	113

Chapter 5

Figure 5-1. CNT and GNP morphology (dry mix).	119
--	-----

Figure 5-2. Raman analysis results of CNTs and GNPs.	120
Figure 5-3. Representation of the specimen geometrical and electrode layout.	121
Figure 5-4. A schema of the climatic cycle protocol.	122
Figure 5-5. Compaction curves of plain and reinforced cementitious stabilized sand (CSS) by different carbon nanomaterial (CNM) concentrations.	123
Figure 5-6. Maximum dry density and degree of saturation for plain and reinforced CSS with different CNMs concentration.	124
Figure 5-7. " $\gamma_d/\gamma_{d(max)}$ " vs. " $S_r-S_{r(opt)}$ " relationship.	124
Figure 5-8. A schema of compacted: a) Plain CSS, reinforced, b) CSS by CNMs.	125
Figure 5-9. An ultrasonic wave passing time for plain and reinforced CSS with different CNM concentrations.	126
Figure 5-10. Scanning electron microscopy (SEM) images of the reinforced CSS by high CNM concentration CG (0.34%) (The areas with white markings are the areas selected for EDX analysis). ...	127
Figure 5-11. SEM morphology of CNTs and GNPs crack bridging and deviation mechanism in CNM-reinforced CSS.	128
Figure 5-12. Thermal analysis of plain and reinforced CSS by different concentrations of CNMs: a) TGA thermograms, b) DSC thermograms.	129
Figure 5-13. X-ray diffraction analysis (XRD) patterns of hardened plain and reinforced CSS by different CNM concentrations.	131
Figure 5-14. a) Unconfined compressive strengths of different CSSs, b) the amount of strain at the rupture point and $E_{(50\%)}$ after 28 days of hydration.	132
Figure 5-15. Weight loss and ultrasonic wave passing time after 12 climatic cycles.	133
Figure 5-16. Electrical resistivity of hardened plain and reinforced CSS by different CNM concentrations.	133
Figure 5-17. The electrical resistivity of the specimen CG (0.34%) at optimum, 2% more than optimum, and 2% less than optimum water content.	134
Figure 5-18. Electrical resistivity of the reinforced specimens with different concentrations of the CNM after climatic cycles.	135
Figure 5-19. CNM morphologies after climatic cycles (the areas with white markings are the areas selected for EDX analysis).	136
Figure 5-20. Raman spectra of the CNMs after climatic cycles.	137

Figure 5-21. The fractional change in resistivity and axial strain under cyclic compression loading for reinforced CSS by different CNM concentrations.....	138
Figure 5-22. Variation of strain with the FCR for reinforced CSS by different CNMs concentrations.....	138
Figure 5-23. The fractional change in resistivity together with axial strain under cyclic compression loading for specimen CG (0.34%) at optimum, 2% more than optimum, and 2% less than optimum water content.....	139
Figure 5-24. A schema of the compacted specimen composed of the CNMs: a) fresh sample at optimum water content, b) fresh sample at the higher or lower water content c) hardened specimen at the higher or lower water content d) hardened specimen at the higher or lower water content, subjected to the compression loading.	140
Figure 5-25. Variation of strain with the FCR for specimen CG (0.34%) at optimum, 2% more than optimum, and 2% less than optimum water content.....	141
Figure 5-26. The fractional change in resistivity together with axial strain under cyclic compression loading for reinforced CSS by different CNMs concentrations after climatic cycles.	142
Figure 5-27. Variation of strain with the fractional changes in electrical resistivity (FCR) for nano-intruded specimens after climatic cycles.....	143
Figure 5-28. Variation of CNM reinforced specimens' gauge factors under compression loading: a) in normal condition, b) with different water content, c) after climatic cycles.....	144

Chapter 6

Figure 6-1. The particle size distribution (PSD) curve of the sand and cement [207, 409].....	151
Figure 6-2. GNP and CNT morphology (dry mix).	152
Figure 6-3. X-ray diffraction (XRD) patterns of CNT and GNP.	152
Figure 6-4. Chemical structures of: (a) Pluronic F-127_ (b) Tributyl phosphate 97%, [145].	153
Figure 6-5. Raman spectroscopy of CNTs and GNPs after dispersion.	154
Figure 6-6. Representation of the specimen geometry and electrode layout.	156
Figure 6-7. The process of pull-out specimen fabrication: a) cross-section of the mold, b) dimensions of the specimen.....	157
Figure 6-8. Cyclic compression loading pattern.....	159
Figure 6-9. Fourier transform infrared spectroscopy (FTIR) spectra of raw and treated fibers.	160

Figure 6-10. Surface morphology of glass fibers: a) pure glass fiber, b) hydrofluoric acid treated glass fiber. (The marked areas in the scanning electron microscopy (SEM) images are the areas selected for energy-dispersive X-ray spectroscopy (EDX) analysis.)	161
Figure 6-11. Surficial morphology of Dyneema fiber: a) pure Dyneema fiber, b) sulfuric acid treated Dyneema fiber. (The marked areas in the SEM images are the areas selected for EDX analysis.)	161
Figure 6-12. Surficial morphology of PP fiber: a) pure PP fiber, b) sulfuric acid treated PP fiber. (The marked areas in the SEM images are the areas selected for EDX analysis.)	162
Figure 6-13. Compaction curves of reinforced CS with different types and concentrations of the fibers.	163
Figure 6-14. A schema of different fiber filler functions in CS: a) without fiber, b) reinforced with PP fibers, c) reinforced with glass fibers, d) reinforced with Dyneema fibers.	164
Figure 6-15. The relation between fiber types and concentration by the: a) maximum dry density, b) optimum water content.	165
Figure 6-16. The saturation degree of reinforced CS with different fiber types and concentrations.	166
Figure 6-17. $\gamma_d/\gamma_d(\max)$ vs. "Sr-Sr (opt) relations.	166
Figure 6-18. Ultrasonic wave passing times vs. the maximum dry density for reinforced CS by different fiber types and concentrations after 28 days of hydration.	167
Figure 6-19. The mechanism of crack bridging and deviation: a) Dyneema fiber, b) glass fiber.	168
Figure 6-20. Morphology of the CS reinforced by fibers: a) PP fiber (fiber extruding), b) glass fiber (fiber rupture), c) Dyneema fiber (fiber elongation).	169
Figure 6-21. Dyneema fiber surface morphology, a) low magnification, b) high magnification.	170
Figure 6-22. Glass and PP fiber surface morphology, a) low magnification of glass fiber, b) high magnification of glass fiber, c) PP fiber.	170
Figure 6-23. The XRD patterns of treated and extracted fiber from the CS: a) Dyneema fiber, b) PP fiber.	171
Figure 6-24. The single-fiber pullout test results of different fibers: a) Pull-out load/ displacement curves, b) Interfacial properties.	172
Figure 6-25. Unconfined compressive strength (UCS) of reinforced CS with different types and concentrations of the fibers after 28 days of hydration.	174
Figure 6-26. The stress–strain curves of reinforced CS with different types and concentration of the fibers after 28 days of hydration.	174

Figure 6-27. The amounts of the strain at rupture point and $E_{(50\%)}$ for reinforced CS with different types and concentrations of the fibers after 28 days of hydration.	175
Figure 6-28. Weight loss of reinforced CS with different types and concentrations of the fibers against wetting and drying cycles.	176
Figure 6-29. Electrical resistivity of reinforced CS with different types and concentrations of fibers after 72 h of oven-drying.	177
Figure 6-30. Variation of the fractional changes in the electrical resistivity (FCR) vs. the strain for reinforced CS with different types and concentrations of the fibers: a) PP fiber, b) glass fiber, c) Dyneema fiber.	179
Figure 6-31. The strain and FCR for fiber-reinforced and non-reinforced specimens: a) maximum and residual strain, b) maximum and residual FCR.	180
Figure 6-32. The relation between the FCR and strain for reinforced CS with different types and concentrations of fibers: a) PP fiber, b) glass fiber, c) Dyneema fiber.	181
Figure 6-33. Gauge factor variations for reinforced CS with different types and concentrations of the fibers: a) PP fiber, b) glass fiber, c) Dyneema fiber.	182

Chapter 7

Figure 7-1. The volume of the surrounded matrix between the electrodes.	190
Figure 7-2. Schematic diagram of the specimens' dimensions (reinforced by hybrid CNT/GNP, 1:1), L: length, W: width, T: thickness.	192
Figure 7-3. Schematic diagram of the specimens' dimensions (reinforced by different length of CF and CNT, separately).	192
Figure 7-4. Experimental test configuration for electrical resistance measurement.	194
Figure 7-5. Test setup and electrode configuration: a) compression, b) flexural.	195
Figure 7-6. Cyclic loading patterns used for piezoresistivity behaviour investigations: a) flexural mode, b) compression mode.	196
Figure 7-7. Volumetric electrical resistance of CNT/GNP-reinforced specimens with different lengths (surrounded between electrodes).	197
Figure 7-8. Volumetric electrical resistance of the specimens with different lengths and reinforced by 0.5 and 1.5% CNT/GNP.	198
Figure 7-9. Relation between electrical percolation threshold and matrix volume of CNT/GNP reinforced specimens with different lengths.	199

Figure 7-10. Volumetric electrical resistance of CNT/GNP reinforced specimens with: a) different width, b) different thickness.	201
Figure 7-11. Relation between electrical percolation threshold and matrix volume for CNT/GNP reinforced specimens with: a) different width, b) different thickness.	202
Figure 7-12. Electrical resistivity of specimens reinforced with hybrid CNT/GNP at the percolation threshold content.....	203
Figure 7-13. Relation between electrical percolation threshold and matrix volume for CNT/GNP reinforced specimens with different volumes.	203
Figure 7-14. The relative aspect ratio of the carbon-based fillers vs percolation threshold for the specimens with different volumes.	204
Figure 7-15. The percolation threshold vs matrix volume (surrounded between electrodes) for the different CF and CNT reinforced specimens.....	205
Figure 7-16 Piezoresistivity response of the beam under flexural cyclic loading.....	207
Figure 7-17 Piezoresistivity response of the cubic specimen under compression cyclic loading.	207
Figure 7-18. Mechanical properties of the 10×10×85 cm ³ beam reinforced with 3.47% of CNT/GNP (SCG 3.47%).....	208
Figure 7-19. Mechanical properties of the 15 cm cubic specimen reinforced with 2.4% of CNT/GNP (SCG 2.7%).....	210

Chapter 8

Figure 8-1. The X-Ray spectra of carbon nanomaterials: a) GNPs, b) MWCNTs.	216
Figure 8-2. The morphology of carbon nanomaterials: a) GNPs, b) MWCNTs, c) the mix of CNT/GNPs.	217
Figure 8-3. Particle size distribution of the used cement and sand.	218
Figure 8-4. CNMs dispersion process used in this study.	219
Figure 8-5. The specimen configuration and electrode layout.	219
Figure 8-6. The test setup used for self-heating assessment.....	220
Figure 8-7. Specimen and electrical circuit configuration in piezoresistivity investigations: a) setup used for evaluation of different loading patterns, b) hybrid specimen, c) setup used for evaluation of temperature and humidity effects.....	221
Figure 8-8. The loading patterns used for the piezoresistivity investigation: a) 0.1 Hz, b) 0.5Hz, c) 1.0Hz, d) creep loading, e) multiple loading, d) fatigue loading, e) pattern used for hybrid specimen.	222

Figure 8-9. a): Specimen configuration and b): compressive cyclic loading pattern used for damage detection.	223
Figure 8-10. a) Loading pattern used for assessment of temperature and humidity effects, b) specimen configuration.	224
Figure 8-11. Main mechanical characteristics of the smart and plain cementitious geocomposite: a) compressive and flexural strength, b) compressive failure strain and compressive elastic modulus (E50%), c) compressive elastic threshold strain and stress.....	225
Figure 8-12. SEM photographs of the smart cementitious composite: a), b), and c) crack bridging and divination by CNTs and GNPs, d) CNTs and GNPs agglomerations.	226
Figure 8-13. Thermography analysis of smart and plain composite: a) TGA, b) DSC, c) DTG.....	227
Figure 8-14. XRD spectra of the smart and plain composite.....	228
Figure 8-15. Self-heating performance of the smart cementitious geocomposite: a) the heating rate, b) self-heating power.....	229
Figure 8-16. Thermography images of smart cementitious geocomposite: a) 3A and 4V, b) 6A and 8V, c) 9A and 12V.	229
Figure 8-17. Effects of temperature on the electrical performance of the cementitious geocomposite: a) variations of electrical resistance against temperature, b) temperature coefficient of resistance, c) effects of temperature on piezoresistivity behaviour, d) variations of FCR against the strains in different temperatures.....	231
Figure 8-18. Effects of water content on the sensitivity performance of the cementitious geocomposite: a) piezoresistivity behaviour, d) variations of FCR against the strains in different water contents.	232
Figure 8-19. piezoresistivity behaviour of the smart geocomposite in different frequencies and load amplitude: a) 0.1 Hz, b) 0.5Hz, c)1.0 Hz.	234
Figure 8-20. Rectangular loading cycle: a) 0.625 MPa, b) 1.25 MPa, c) 3.125 MPa.....	235
Figure 8-21. The piezoresistive response of the smart cementitious geocomposite under the continuous cyclic loading: a) continuous incremental compressive cyclic loading, b) long-term continuous compressive cyclic loading (250 cycles, 0.5Hz).	236
Figure 8-22. The trend of FCR-strain together with stress-strain during the monotonic load increasing.....	237
Figure 8-23. The typical strain distribution at different stages of monotonic loading: a) after 65% loading, b) after 93% loading c) after 100% loading (a moment before the failure).	238

Figure 8-24. The trend of FCR and strain changes during the cyclic compressive loading: a) stress-strain curve, b) FCR together with strain over time.	239
Figure 8-25. The typical strain distribution at different stages of cyclic compressive loading: a) after 63% loading, b) after 91% loading c) after 100% loading (a moment before the failure).	240
Figure 8-26. Piezoresistive response of the smart cementitious geocomposite in hybrid section.....	241

List of Tables.

Chapter 2

Table 2-1. Summary of major infrastructure monitoring methods and their advantages and limitations	24
Table 2-2. Summary of studies investigation carbon short fibres in cementitious matrices	28
Table 2-3. Summary of studies investigating the damage monitoring performance achieved using CNTs and CNFs	37
Table 2-4. Advantages and limitations of different conductive-filler dispersion methods.....	40
Table 2-5. Characteristics of different conduction theories	45
Table 2-6. Characteristics of different equivalent circuit models	50
Table 2-7. Summary of previous studies on cementitious self-sensing composites in different forms for structural health monitoring	51
Table 2-8. Previous research on self-sensing composites for traffic monitoring applications.	52

Chapter 3

Table 3-1: GNPs and CNTs characteristics	59
Table 3-2: Particle size distribution of the sand [322]	60
Table 3-3: Chemical composition and properties of Ordinary Portland Cement.....	60
Table 3-4: Agglomerate total area of various CNT+GNP aqueous suspensions.....	70
Table 3-5: Zeta Potential of CNT+GNP suspensions	72
Table 3-6: CNM hydrodynamic diameter in different suspensions characterized by DLS	73
Table 3-7: Test results of cementitious composites according standard procedures.....	75
Table 3-8: Electrical resistance and humidity of cementitious composites specimens	80
Table 3-9: Comparison of electrical resistance for carbon nanoparticles reinforced cementitious composites.....	82

Chapter 4

Table 4-1: GNPs and CNTs characteristics	90
Table 4-2: Particle size distribution of the sand [145].	90
Table 4-3: Chemical composition and properties of Ordinary Portland Cement.....	90
Table 4-4: Comparison of mechanical properties with previous studies for CNMs reinforced cementitious mortar after 28 days of hydration.	99

Table 4-5: Ultrasonic wave passing times of nano intruded cement mortars	102
Table 4-6: Comparison of electrical resistance for CNM reinforced cementitious composites after 28 days of curing.....	105
Table 4-7: Comparison of gauge factors for cementitious composite under compressive loading.	113

Chapter 5

Table 5-1. Characteristics of graphene nanoplatelets (GNPs) and carbon nanotubes (CNTs).....	118
Table 5-2. Sand particle size distribution.	119
Table 5-3. Cement hydration crystal chemical composites.	127
Table 5-4. CNMs' chemical composition.....	136

Chapter 6

Table 6-1. Physical properties of the sand [207].....	151
Table 6-2. Multi-wall carbon nanotube (MWCNT) and graphene nanoplatelets (GNP) specifications [145].....	151
Table 6-3. Physical properties of glass, polypropylene, and Dyneema fibers.	153
Table 6-4. Mix proportions of the reinforced cemented sand (CS). Carbon nanomaterial (CNM), polypropylene (PP).....	156
Table 6-5. Chemical analysis of pure and treated fibers.	162

Chapter 7

Table 7-1. Characterization of Multi-wall carbon nanotube (MWCNT), graphene nanoplatelets (GNP), and short carbon fibre (CF).....	191
Table 7-2. Sand particle size distribution.	191

Chapter 8

Table 8-1. The main characteristics of GNP and MWCNT used in this study.	217
Table 8-2. The main physical characteristics of the sand.	218
Table 8-3. Self-heating performance of the different cementitious composite.....	230

List of acronyms

AC	Alternating Current
ACIS	Alternating Current Impedance Spectroscopy
AR	Augmented Reality
BCRs	Braided Composite Rods
CB	Carbon Black
CBP	Carbon Black Powder
CF	Carbon Fibre
CH	Calcium Hydroxide
CNF	Carbon Nanofibers
CNMs	Carbon Nanomaterials
CNT	Carbon Nanotube
CPyCl	Cetylpyridinium Chloride
CSF	Carbon Short Fibre
CSS	Cementitious Stabilized Sand
DC	Direct Current
DIC	Digital Image Correlation
DLS	Dynamic Light Scattering
DSC	Differential Scanning Calorimetry
EDX	Energy Dispersive X-Ray Analysis
ESD	Electrostatic Dissipative
FCR	Fractional Change In Resistivity
FTIR	Fourier Transform Infrared Spectroscopy
GF	Gauge Factor
GFs	Glass Fibres
GNPs	Graphene Nanoplatelets
GO	Graphene Oxide
IoT	Internet Of Things
MWCNT	Multiwall Carbon Nanotube
NCB	Nanocarbon Black

NT	TiO ₂
NT	Nano Titanium
OPC	Ordinary Portland Cement
PDI	Polydispersity Index
PEO	Poly-Oxyethylene
PF-127	Pluronic F-127
PP	Polypropylene
PPO	Poly-Oxypropylene
PZT	Lead Zirconate Titanate (piezoelectric ceramic
SDS	Sodium Dodecyl Sulfate
SEM	Scanning Electron Microscope
SF	Steel Fibre
SSG	Self-sensing Geocomposite
TBP	Tributyl Phosphate
TGA	Thermogravimetric Analysis
UAVs	Unmanned Aerial Vehicles
UCS	Unconfined Compression Strength
UHMWPE	Ultra-High-Molecular-Weight Polyethylene
USC	Unified Soil Classification System
VR	Virtual Reality
WSNs	Wireless Sensor Networks

Chapter 1. Introduction

1.1 Introduction to Smart Infrastructure Monitoring

In today's rapidly advancing technological landscape, the concept of Smart Infrastructure Monitoring has emerged as a critical facet of modern civil engineering [1]. This innovative approach leverages cutting-edge technologies and data-driven methodologies to enhance the safety, sustainability, and operational efficiency of essential civil structures, such as bridges, roads, buildings, and more [2]. By continuously monitoring these infrastructures including transportation and railway, engineers and stakeholders can gain real-time insights, proactively identify potential issues, and implement timely interventions, ensuring the longevity and optimal performance of these vital assets [3]. Monitoring civil engineering structures is of utmost significance for several reasons: **Enhanced Safety:** The safety of civil engineering structures is paramount, as they support transportation systems, provide shelter, and facilitate essential services. Smart Infrastructure Monitoring enables engineers to detect structural vulnerabilities or signs of deterioration early on, preventing potential disasters and ensuring the safety of the public and users of these structures [4]. **Optimized Maintenance and Resource Allocation:** By closely monitoring the health of infrastructures, engineers can schedule maintenance and repairs proactively, optimizing the use of resources. This minimizes downtime, reduces costs, and extends the lifespan of the structures, leading to more sustainable and economically viable projects [5]. **Resilience to Natural Disasters:** Civil structures often face the challenge of withstanding natural disasters such as earthquakes, floods, and hurricanes. Through advanced monitoring systems, engineers can monitor real-time stress, strain, and deformation data, enabling them to implement necessary reinforcements and fortifications to enhance the resilience of these structures [6]. **Efficient Operation and Traffic Management:** Smart Infrastructure Monitoring plays a pivotal role in optimizing the efficiency of transportation networks. Real-time data on road conditions, traffic flow, and bridge capacity can be analyzed to improve traffic management, reduce congestion, and enhance overall mobility [7]. **Data-Driven Decision Making:** The integration of various monitoring technologies, such as sensors, IoT devices, and remote sensing, generates vast amounts of data. By employing advanced analytics and machine learning, this data can be transformed into valuable insights, guiding engineers in making data-driven decisions regarding

maintenance strategies, design improvements, and future infrastructure development [8]. **Sustainability and Environmental Impact:** Monitoring allows engineers to assess the environmental impact of civil structures throughout their lifecycle. By identifying areas of high energy consumption, emissions, or material waste, smart infrastructure monitoring enables the implementation of eco-friendly practices and promotes sustainable development in the construction industry [9]. **Predictive Maintenance:** Traditional maintenance approaches often rely on scheduled inspections, which may not capture developing issues. Smart Infrastructure Monitoring facilitates predictive maintenance, where anomalies and potential failures are detected before they escalate into significant problems, leading to more cost-effective and efficient maintenance practices [10]. **Public Trust and Stakeholder Confidence:** Continuous monitoring and proactive maintenance efforts improve public trust and confidence in civil infrastructure projects. This reassures citizens, investors, and stakeholders that these structures are reliable, safe, and well-maintained, fostering a positive image for both engineering organizations and the community at large [11]. Smart transportation and railway infrastructure monitoring involves the application of advanced technologies and data-driven solutions to improve the efficiency, safety, and sustainability of transportation systems, including railways. Here are some key aspects and components of smart transportation and railway infrastructure monitoring: **Intelligent Traffic Management Systems:** These systems use real-time data from various sources, such as sensors, cameras, and GPS devices, to monitor traffic conditions. They can analyze traffic flow, identify congested areas, and optimize signal timings to reduce congestion and improve the overall traffic management [12]. **Connected Vehicles and V2X Communication:** Smart transportation incorporates connected vehicle technologies that allow vehicles to communicate with each other and with infrastructure (V2X communication). This communication enables the exchange of critical information, such as traffic conditions, road hazards, and upcoming signals, to enhance safety and efficiency [13]. **Real-time Passenger Information:** In the context of public transportation, real-time passenger information systems provide travelers with live updates on arrival times, delays, and alternative routes. This information helps passengers make informed decisions and reduces waiting times at transit stops [14]. **Railway Track Monitoring:** Sensors placed along railway tracks can monitor various parameters, including track condition, temperature, and stress. This data helps detect anomalies and potential issues with the tracks, enabling proactive maintenance and reducing the risk of derailments [15]. **Predictive Maintenance for Rolling Stock:** Smart railway infrastructure monitoring involves using data analytics and machine learning to predict maintenance needs for trains and rolling stock. By analyzing real-time data from onboard sensors, maintenance can be scheduled based on actual wear and tear, leading to cost savings and increased reliability [16].

Automated Fare Collection and Ticketing: Smart transportation systems often incorporate automated fare collection and ticketing systems, reducing the need for physical tickets and enabling seamless payment methods, such as contactless cards and mobile apps [17]. **Remote Monitoring and Control Centres:** Control centres equipped with advanced monitoring tools enable real-time oversight of transportation and railway infrastructure. Operators can quickly respond to incidents, manage traffic flow, and coordinate emergency services when needed [18]. **Traffic and Demand Analytics:** By analysing historical and real-time data, transportation authorities can gain insights into traffic patterns, peak hours, and demand trends. This information helps in optimizing routes, schedules, and resource allocation [19]. **Smart Parking Solutions:** Smart transportation systems may include smart parking solutions that use sensors and data analytics to guide drivers to available parking spaces, reducing traffic congestion in search of parking spots [20]. **Energy Efficiency and Sustainability:** Smart transportation and railway infrastructure monitoring aim to optimize energy consumption and reduce the environmental impact. This includes the use of electric or hybrid vehicles, energy-efficient lighting, and sustainable materials in infrastructure construction [21].

By integrating these technologies and data-driven approaches, smart transportation and railway infrastructure monitoring systems can make transportation networks more efficient, safer, and user-friendly, contributing to improved overall urban mobility and transportation experiences. In conclusion, Smart Infrastructure Monitoring represents a paradigm shift in civil engineering, revolutionizing how we design, construct, and manage essential infrastructure. The adoption of this approach brings about a range of benefits, encompassing enhanced safety, optimized maintenance, resilience to disasters, efficient operation, data-driven decision-making, sustainability, and increased stakeholder confidence. Embracing smart infrastructure monitoring is crucial to building a more resilient, sustainable, and technologically advanced society that can thrive in the face of future challenges.

1.2 Background on Existing Monitoring Techniques:

Civil engineering has relied on traditional monitoring methods for decades to assess the health and performance of critical infrastructure [22]. These methods typically involve the use of various sensors and devices, such as strain gauges, accelerometers, and displacement sensors, to gather data on structural behavior and environmental conditions [23]. While these techniques have been valuable in providing essential insights, they also come with inherent limitations that necessitate the exploration of innovative approaches to overcome these challenges effectively. Strain gauges are widely used to measure the strain or deformation experienced by civil structures [24]. These devices are typically bonded to the surface of

a structure and change their electrical resistance in response to mechanical strain. Strain data are then collected and analyzed to assess the structural integrity and potential points of failure [25]. Strain gauges are generally suitable for localized measurements, making it challenging to monitor large structures comprehensively. Additionally, installing and maintaining strain gauges can be time-consuming and costly. Moreover, these gauges do not provide real-time data, limiting their ability to detect sudden changes or dynamic loading events [26]. Accelerometers are utilized to measure acceleration forces experienced by structures, particularly in response to vibrations and seismic activities. These devices are crucial for assessing the dynamic behavior and stability of buildings, bridges, and other civil infrastructure under varying load conditions [27]. Accelerometers are primarily focused on measuring acceleration, which requires additional integration steps to determine displacement or velocity. Additionally, accelerometers are sensitive to noise and temperature variations, making accurate measurements challenging in some environments. Moreover, accelerometers often lack the ability to provide continuous, long-term monitoring without significant power consumption [28]. Displacement sensors, such as linear variable differential transformers (LVDTs) and extensometers, are used to measure the relative movement between two points on a structure. These sensors play a crucial role in assessing structural deflection, settlement, and overall deformation [29]. Displacement sensors are generally suitable for localized measurements, similar to strain gauges. Scaling up to monitor entire structures can be cost-prohibitive and impractical. Moreover, the installation of displacement sensors may require physical alterations to the structure, which can be disruptive and challenging to implement in existing infrastructures [30]. Inclinometers are sensors used to measure the angle of tilt or inclination of a structure with respect to the vertical axis. They are particularly useful in monitoring the stability of slopes, embankments, and retaining walls, helping detect potential sliding or settlement issues [31]. Tiltmeters measure small changes in the tilt or inclination of a structure. They are commonly used to monitor the stability of tall buildings, towers, and other vertical structures [32]. Load cells are sensors designed to measure the forces applied to a structure. They are used to assess the load-carrying capacity of bridges, floors, and other components, ensuring that they operate within safe limits [33]. Ground-Penetrating Radar is a non-destructive testing method used to assess subsurface conditions. It can detect voids, cracks, and other defects in concrete structures, pavements, and underground utilities [34]. Remote sensing techniques, such as LiDAR (Light Detection and Ranging) and aerial imaging, are used to monitor large areas and inaccessible locations. These methods provide valuable data for infrastructure assessment and environmental monitoring. Environmental sensors measure parameters like temperature, humidity, wind speed, and air quality in the vicinity of civil structures [35]. Understanding the environmental conditions is crucial for assessing

the impact on infrastructure performance and durability. While traditional monitoring techniques have been instrumental in civil engineering, they have their limitations that impede comprehensive and real-time assessments of infrastructure [36]. The advancement of technology has led to the emergence of smart and novel monitoring techniques in civil engineering, revolutionizing the way infrastructure is assessed and managed. These innovative approaches leverage cutting-edge technologies and data-driven methodologies to provide real-time, comprehensive, and precise insights into the health and performance of civil structures [37]. Some of the prominent smart and novel monitoring techniques include:

Fiber Optic Sensors: Fiber optic sensors use optical fibers to measure strain, temperature, and other physical parameters in civil structures. They offer high accuracy, immunity to electromagnetic interference, and the ability to cover long distances, making them ideal for monitoring large-scale infrastructure like bridges and pipelines [38].

Wireless Sensor Networks (WSNs): WSNs consist of a network of interconnected wireless sensors deployed throughout a structure. These sensors communicate with a central system to collect and analyze data on structural behavior, environmental conditions, and other relevant parameters. WSNs provide real-time monitoring without the need for extensive cabling [39].

Unmanned Aerial Vehicles (UAVs): UAVs or drones are used for aerial inspections and surveys of civil infrastructure. They can capture high-resolution images, LiDAR data, and thermal imagery, providing valuable information for structural assessment and maintenance planning [40].

Remote Sensing and Satellite Imagery: Satellite-based remote sensing techniques enable monitoring large geographical areas and infrastructure networks. This data can be used to detect ground subsidence, assess land use changes, and identify potential hazards to infrastructure [41].

Internet of Things (IoT) Sensors: IoT sensors are integrated into civil structures to monitor various parameters, such as structural integrity, temperature, humidity, and vibrations. These sensors continuously transmit data to centralized databases for real-time analysis [42].

Augmented Reality (AR) and Virtual Reality (VR): AR and VR technologies are utilized for immersive visualization and simulations of civil structures. Engineers can use these technologies to explore virtual models, assess potential scenarios, and identify areas that require attention [43].

Energy Harvesting Systems: Energy harvesting systems capture and store energy from the environment, such as vibrations or temperature gradients, to power monitoring sensors and devices. This approach reduces the reliance on external power sources, enhancing the sustainability and autonomy of monitoring systems [44].

1.3 Problem Statement:

In the context of transportation and railway systems, monitoring the condition of tracks, embankments, and other critical components is essential to ensure passenger safety, efficient operations, and cost-effective maintenance [45]. Conventional monitoring methods face challenges when dealing with expansive areas and difficult-to-access locations, making comprehensive monitoring a complex and resource-intensive task [1]. Cementitious stabilized sand is a construction material used in civil infrastructure projects, including transportation and railway applications [1, 46]. It is a mixture of sand, cement, and water, which undergoes a stabilization process to improve its engineering properties. The resulting material offers several advantages, making it suitable for various construction purposes [47]. Here's how cementitious stabilized sand is used in civil infrastructure projects: **Subgrade Stabilization:**

In transportation and railway projects, the subgrade is the natural soil foundation upon which the pavement or tracks are constructed. Stabilizing the subgrade with cementitious stabilized sand improves its strength and load-bearing capacity, preventing settlement and deformation under traffic loads [48].

Road Base and Pavement Construction: Cementitious stabilized sand can be used as a base material for road construction. It provides a strong and stable foundation for the upper layers of the road, enhancing its durability and reducing the risk of cracking or rutting [49]. **Railroad Track Bed**

Stabilization: For railway projects, the track bed or ballast is critical for ensuring the stability and proper alignment of the tracks. Cementitious stabilized sand can be used to improve the ballast's properties, reducing the need for frequent maintenance and improving the overall performance of the railway track [50]. **Erosion Control:** In areas prone to erosion, cementitious stabilized sand can be used to stabilize embankments, slopes, and retaining walls, preventing soil erosion and protecting the infrastructure from damage [51].

Sustainable Construction: Cementitious stabilized sand can be an environmentally friendly alternative to traditional construction methods. It reduces the need for using natural aggregates, which helps conserve natural resources and reduces the environmental impact [52]. **Rapid**

Construction: The use of cementitious stabilized sand can speed up construction processes as it can be prepared and placed quickly. This can be particularly advantageous for projects with tight schedules or those requiring minimal disruptions to traffic or railway operations [53]. **Cost-Effectiveness:**

Cementitious stabilized sand can be more cost-effective than some other stabilization methods. It can often use locally available materials, reducing transportation costs, and the stabilization process can be accomplished with less specialized equipment [54]. **Versatility:** Cementitious stabilized sand can be

tailored to meet specific project requirements by adjusting the mix proportions. This versatility allows it to be used in various soil conditions and for different construction applications [55].

Smart infrastructure including transportation and railway monitoring stands as a transformative force in modern civil engineering, revolutionizing how we design, construct, and manage essential infrastructure [1]. By integrating advanced technologies, data analytics, and real-time monitoring, engineers can ensure the safety, sustainability, and efficiency of civil structures, thereby bolstering public trust and confidence in these vital assets [56]. Embracing smart infrastructure monitoring is not just a prudent engineering decision; it is a commitment to building a resilient, sustainable, and technologically advanced society that can thrive in the face of evolving challenges [57]. As technology continues to evolve, the potential for smart infrastructure monitoring to shape the future of civil engineering remains boundless, making the world a safer and better-connected place for generations to come. Modern civil infrastructure, including transportation networks and railway systems, plays a crucial role in supporting societal needs and economic growth [3]. Ensuring the safety, sustainability, and optimal performance of these critical assets is of paramount importance. Traditional monitoring techniques have provided valuable insights into the health of civil structures. However, they often have limitations that hinder real-time and comprehensive assessments, leading to potential risks and inefficiencies [56]. Even with recent progress, the majority of monitoring instruments like fibre Bragg grating sensors, electric strain gauges, and PZT-based piezoelectric sensors still rely on a limited number of sensors placed at specific locations on the relevant infrastructure. Moreover, these instruments are often characterized by low durability and accuracy, complex and costly production processes, and their detection mechanisms may not be compatible with the behavior of geomaterials [46]. As a result, many of these methods are deemed inadequate. Indeed, current monitoring techniques for civil infrastructure typically rely on external sensors and devices to gather data on structural health and performance. These methods often require expensive and time-consuming installation, and they may not provide the desired level of accuracy, coverage, and real-time insights needed for proactive decision-making [1]. Additionally, the deployment of external sensors can disrupt the integrity of the structure and introduce vulnerabilities. All the monitoring techniques available, self-sensing composites that utilize piezoresistive behavior and possess intrinsic sensing capabilities are the most comprehensive, immediate, and effective solution for infrastructure monitoring and damage detection, considering the properties and characteristics of geomaterials [1]. These intelligent geomaterials enable the creation of smart infrastructure with built-in sensing and structural health monitoring capabilities, leading to improved safety, durability, functionality, and reliability of the infrastructure. Self-sensing stabilized sand or cementitious geocomposites present a novel approach to enhancing the sustainability of geomaterials [58].

1.4 Overview of the self-sensing cementitious composites:

Self-sensing composites are a revolutionary class of smart materials that possess the remarkable ability to sense and respond to changes in their environment without the need for external sensors or monitoring systems [59]. These cutting-edge composites integrate sensing capabilities directly into their structure, enabling real-time monitoring and data collection. This comprehensive overview will delve into the key features, working principles, applications, benefits, and future prospects of self-sensing composites, highlighting their potential to transform various industries and pave the way for a more intelligent and sustainable future [60]. The defining characteristic of self-sensing composites lies in their intrinsic ability to detect and quantify changes in physical properties, such as strain, stress, temperature, or humidity, and transform these changes into measurable electrical signals [61]. The sensing capability is often achieved by incorporating conductive materials, such as carbon fibrous or nanomaterials and/or metallic particles, into the composite matrix. The conductive phase generates an extensive network of conductive pathways based on percolation and quantum tunnelling mechanisms [62]. When subjected to external stimuli, such as mechanical deformation or changes in temperature, the conductive elements within the composite experience alterations in their electrical conductivity. These changes are then captured and interpreted as meaningful data, providing valuable insights into the material's condition and its interaction with its surroundings [63]. While numerous investigations have examined self-sensing applications in diverse composites like concrete, cement paste, polymer, and asphalt [64, 65], based on the authors knowledge there is no research concerning self-sensing geomaterials.

Self-sensing cementitious composites have emerged as a groundbreaking technology with the potential to revolutionize the construction industry. By integrating inherent sensing capabilities into cement-based materials, these smart composites offer a wide array of benefits and advantages that can significantly enhance the performance, durability, and safety of infrastructure [66]. In this comprehensive overview, we will delve into the key benefits and advantages of self-sensing cementitious composites, highlighting their transformative impact on various applications in civil engineering and beyond: **Real-Time Structural Health Monitoring:** One of the most significant advantages of self-sensing cementitious composites is their ability to provide real-time structural health monitoring. With embedded sensors directly within the material, engineers can continuously monitor changes in strain, stress, and deformation in concrete structures. This instantaneous feedback enables early detection of potential damage or deterioration, allowing for prompt intervention and maintenance, thereby enhancing the overall safety and longevity of the infrastructure [67]. **Proactive Maintenance and Cost Savings:** By facilitating real-

time monitoring, self-sensing cementitious composites enable a proactive approach to maintenance. Identifying structural issues at their nascent stages allows for timely repair and rehabilitation, preventing the escalation of damage and the need for costly and extensive repairs later on. This proactive maintenance approach can lead to substantial cost savings over the lifecycle of the infrastructure [68].

Improved Structural Safety and Reliability: Self-sensing cementitious composites contribute significantly to improving the safety and reliability of civil structures. By continuously monitoring critical components, such as bridges, dams, and buildings, these composites can detect structural anomalies that may compromise their integrity. This capability reduces the risk of catastrophic failures, enhances public safety, and instills confidence in the long-term performance of the infrastructure [69].

Enhanced Durability and Service Life: Self-sensing cementitious composites are designed to be highly durable and resistant to environmental stressors. Their ability to detect early signs of degradation enables timely repairs and preventive measures, preventing the spread of damage and extending the service life of the infrastructure. As a result, the need for frequent replacements and costly overhauls is significantly reduced [70].

Sustainable Construction Practices: Incorporating self-sensing capabilities directly into the material eliminates the requirement for external sensors and monitoring systems. This streamlined approach reduces material waste, construction complexity, and energy consumption associated with the installation and maintenance of external sensors. Consequently, self-sensing cementitious composites align with sustainable construction practices, promoting resource efficiency and reducing the environmental impact of infrastructure projects [71].

Tailored Sensing Properties: Self-sensing cementitious composites offer flexibility in tailoring their sensing properties to suit specific applications and engineering requirements. By adjusting the type and concentration of conductive additives, the sensitivity and range of sensing can be customized for different structural elements and monitoring purposes [1].

Remote Monitoring and Data Analytics: With self-sensing capabilities, data can be collected remotely from embedded sensors, reducing the need for manual inspections and on-site visits. This remote monitoring enables continuous data acquisition, which can be analyzed using advanced data analytics and machine learning algorithms. The insights derived from these analyses can inform predictive maintenance strategies and optimize asset management, enhancing the overall efficiency of infrastructure operations [72].

Adaptability and Versatility: Self-sensing cementitious composites are compatible with various construction methods and can be seamlessly integrated into traditional concrete mixes. They can be used for a wide range of civil engineering applications, including bridges, tunnels, pavements, and high-rise buildings, making them versatile and adaptable to diverse project requirements [73].

Facilitating Smart Infrastructure: The incorporation of self-sensing cementitious composites lays the

foundation for the development of smart infrastructure. These materials enable structures to self-monitor, adapt to changing conditions, and communicate critical information in real-time. This transition towards smart infrastructure improves asset management, optimizes resource allocation, and supports the creation of resilient and intelligent urban environments [74]. **Research and Technological**

Advancement: The pursuit of self-sensing cementitious composites has fostered extensive research and technological advancements in the fields of materials science, nanotechnology, and sensor technology [75]. This ongoing progress is not only beneficial for the development of self-sensing composites but also contributes to broader advancements in material engineering and the adoption of smart materials in various industries [1]. The development of self-sensing cementitious geocomposites represents a remarkable advancement in the field of smart materials and geotechnical engineering. Combining the benefits of self-sensing technology with the versatility of geocomposites, this innovative approach offers a transformative solution for monitoring and enhancing the performance of geotechnical structures, infrastructures (including transportation and railway), and subsurface applications [56, 58]. However, due to the novelty of the subject, developing self-sensing cementitious geocomposite still faces several challenges including: **Type of Conductive Phase and their Dispersion:** The type of conductive phase

and their dispersion method used in developing self-sensing cementitious geocomposites plays a critical role in determining the material's sensing capabilities, sensitivity, and overall performance. The choice of conductive phase and dispersion technique can significantly influence the efficiency and effectiveness of the self-sensing properties such as: mechanical properties, electrical conductivity, sensitivity and responsiveness, compatibility with cementitious matrix, stability and durability, fabrication ease [76].

Effects of Water and Cement Content: Stabilized sand, also known as cementitious stabilized sand, is a mixture of sand and a small amount of cement with optimum moisture that is usually compacted using the compaction method to the maximum dry density at the optimum moisture. Hence the effects of the cement and water reduction as well as the compaction method on the physical and mechanical and sensing performance of the composite should be considered [1]. **Effective factors:** Since the piezoresistive properties and sensing capabilities of the self-sensing cementitious geocomposite are affected by various parameters such as electrode layout, measurement circuit, humidity and temperature, the effects of these factors should be considered [77, 78].

1.5 Scope of the Study:

The present study aims to investigate the development, characterization, and performance evaluation of an innovative cementitious geocomposite with self-sensing capabilities for smart infrastructure (including

transportation and railway) monitoring. The proposed geocomposite represents a cutting-edge advancement in the field of smart materials and civil engineering, with the potential to revolutionize the monitoring and management of transportation and railway infrastructure. While this study aims to contribute valuable insights into the development and application of the self-sensing cementitious geocomposite, certain scope need to be acknowledged: **Laboratory-Scale Testing:** The performance evaluation (including mechanical, microstructural, durability and piezoresistivity) will primarily be conducted at the laboratory scale, and as such, the real-world behavior of the geocomposite under various environmental conditions may differ. **Long-Term Performance:** As a research study, the long-term performance of the self-sensing geocomposite in actual infrastructure projects may not be fully explored. Real-world durability and response to aging processes will require further investigation. **Environmental and Economics Issues, and Practicality:** The practical implementation of self-sensing geocomposites in large-scale transportation and railway projects may have cost and environmental implications and require feasibility assessments. **Limitations of Conductive Additives:** The selection and availability of suitable conductive additives may influence the geocomposite's properties and self-sensing performance.

1.6 Research Objectives:

By addressing the research questions and objectives using appropriate methodologies, the study aims to contribute to the advancement of smart materials and the enhancement of transportation and railway infrastructure monitoring. Acknowledging the limitations, this research lays the foundation. The primary objectives of this research are as follows:

- **Guiding methodology, choosing an appropriate conductive phase and dispersion method:** The conductive phase in self-sensing cementitious geocomposites serves as a pathway for electrical charges. It allows the geocomposite to detect mechanical changes and display self-sensing capabilities. The primary purpose of incorporating a conductive phase is to enable real-time monitoring of structural health, which is crucial for proactive maintenance and safety enhancement in civil engineering infrastructures. To achieve optimum performance, several key factors should be considered when selecting a conductive phase for self-sensing cementitious geocomposites: electrical conductivity, compatibility with cementitious matrix, durability, cost-effectiveness. Achieving a homogeneous dispersion of the conductive phase within the cementitious matrix is critical for uniform sensing performance. Improper dispersion can lead to localized regions with compromised self-sensing capabilities, affecting the overall reliability of the geocomposite. Some key features and

characteristics of an effective dispersion method for self-sensing cementitious geocomposite development are including: homogeneous dispersion, compatibility, agglomerate disruption, minimization of air voids, time and cost efficiency, consistency and reproducibility, scalability, minimal environmental impact.

- **Proof of the concept:** The efficiency of the conductive filler and dispersion method for the development of a sensitive affordable cementitious geocomposite needs to be evaluated. In this route, it might be better that the potential of the conductive fillers and their dispersion method in terms of mechanical, microstructural, durability and piezoresistivity performances are examined and optimized in cementitious mortar due to the lack of knowledge about self-sensing cementitious geocomposite and its similarity to the cementitious mortar.
- **Self-sensing cementitious geocomposite development:** Based on the above proof of concept self-sensing cementitious geocomposite can be developed by reduction of the cement concentration and water content. However, in this route the utilized factors may need to be optimized. Furthermore, geotechnical, microstructural, durability and piezoresistivity of the smart geocomposite needs to be evaluated in details and improve to specific operation level using different methods such as incorporation of the fibers and etc.

Addressing the affecting factors: Since the sensing capacity of the composite can be affected by various factors, the effects of major parameters on the piezoresistivity behaviour of the composite need to be investigated in order to enhance its sensing capacity to the operational level. In this route base on the scope of the research work and application of the self-sensing cementitious geocomposite for smart transportation and railway infrastructure monitoring the effects of, size, electrode layout, temperature, moisture, and load (speed and shape) on the sensing potential of the composite needs to be addressed.

1.7 Thesis Outline:

The following sections provide an outline of the thesis titled "An Innovative Cementitious Geocomposite for Smart Infrastructure Monitoring." This thesis aims to address the growing need for sustainable and intelligent solutions in infrastructure monitoring, specifically focusing on transportation and railway networks. Figure 1-1 illustrates the relations existing among the thesis' objectives, chapters and main subtopics.

Chapter 1. Introduction

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Figure 1-1. The schematic final representation of the thesis outline and relations among the thesis' objectives, chapters and main topics.

Since this is a paper-based thesis, the main body of work consists of multiple distinct research papers published in reputable academic journals. In this route, the structure of this thesis is organized as follows based on the scope and objectives of the research work:

Chapter 2 (State of the art): This chapter provided a comprehensive, critical, and up-to-date review of the existing literature and technologies related to infrastructure monitoring, with a specific focus on self-sensing cementitious composites and their application in smart transportation and railway systems. This comprehensive literature survey furnished us with all the indispensable elements necessary to initiate the establishment and advance the progression of an ingenious self-sensing cementitious geocomposite tailored for the astute monitoring of smart infrastructures including transportation and railway. The survey encompasses pivotal aspects, including the characterization of the conductive phase and non-conductive matrix in terms of their composition and properties, as well as the intricate methodologies pertaining to their dispersion and fabrication. Moreover, it delves into the various loading schemes, measurement techniques, and electrical circuits employed, while also considering the influential factors, such as humidity, temperature, aggregate composition, water-cement ratio, and curing processes, which inherently impact the composite's performance. Additionally, this survey sheds light on the associated theoretical explorations, providing invaluable insights into the underlying principles and challenges encountered in this domain. By assimilating this wealth of knowledge, stakeholders in the field of smart

infrastructure development can foster enhanced innovation and address the prevailing complexities that lie ahead.

Chapter 3 (Dispersion method development): In this chapter, drawing upon the insightful findings extracted from the thorough literature survey, an innovative, congruent, and efficacious dispersion methodology was meticulously devised.

Chapter 4 (Self-sensing cementitious mortar development): In this chapter based on the previous finding, a comprehensive investigation was conducted to assess the efficacy of the proposed dispersion technique and the influence of diverse dosages of conductive filler on the mechanical, microstructural, durability and piezoresistivity attributes of the cementitious mortar. This empirical inquiry in the mortar was undertaken owing to the profound resemblance between the cementitious mortar and the cementitious geocomposite, as well as the novelty inherent to the subject matter of self-sensing cementitious geocomposites. Furthermore, the paucity of existing knowledge concerning self-sensing cementitious geocomposites, juxtaposed with the wealth of information available on self-sensing mortar in the literature, accentuates the significance and timeliness of this investigation.

Chapter 5 (Self-sensing cementitious geocomposite development): In this chapter, building upon the insights gleaned from the preceding section, wherein the development of self-sensing cementitious mortar and the comprehensive examination of the conductive filler, dispersion methodology, and measurement electrical circuit were expounded, we embarked on the creation of a self-sensing cementitious geocomposite. This was achieved through the strategic reduction of cement concentration and water content to achieve the desired outcome. The selection and calibration of pertinent influencing factors were once again fine-tuned to achieve optimal performance. In addition to the primary formulation, a meticulous evaluation of the geotechnical, microstructural, durability, and piezoresistive properties of this intelligent geocomposite was undertaken. This endeavour is geared towards a comprehensive understanding of its multifaceted characteristics and behaviour.

Chapter 6 (Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite): In this, we explored the impact of incorporating fibrous materials in the composite's matrix, with a specific focus on augmenting performance levels for targeted operational scenarios. The goal was to uncover the synergistic effects of these fibrous elements on enhancing the overall performance of the smart geocomposite. Through this comprehensive investigation, we aimed to bolster the efficacy and applicability of the self-sensing cementitious geocomposite in the realm of smart infrastructure monitoring such as transportation and railway.

Chapter 7 (Effects of size and electrodes layout on sensing potential of cementitious geocomposite):

Given the inherent complexity of the composite's sensing capacity, it became imperative to conduct a comprehensive investigation into the influence of various key parameters on the piezoresistive behaviour of the material. This endeavour is crucial in optimizing the composite's sensing capabilities to meet operational requirements effectively. Within the context of this research, and considering the intended application of the self-sensing cementitious geocomposite for smart transportation and railway infrastructure monitoring, a thorough analysis of the impact of certain critical factors was performed. Specifically, the effects of size, electrode layout.

Chapter 8 (Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite):

In this chapter, the effects of temperature, moisture, and applied load (including speed and shape) on the sensing potential of the composite demanded meticulous scrutiny. A systematic assessment of these parameters will enable us to discern their individual and collective contributions to the sensing performance of the geocomposite. Furthermore, it was imperative to explore the additional applications of this composite beyond its primary function. In this regard, the potential use of the material in damage diagnosis and self-heating performances was also evaluated. Understanding the composite's behaviour in these contexts will unveil its versatility and expand the scope of its practical implementation. By addressing these aspects systematically, we endeavoured to bolster the overall efficacy and applicability of the self-sensing cementitious geocomposite, establishing it as an advanced solution for monitoring and enhancing the integrity of transportation and railway infrastructure.

Chapter 9 (Conclusions and future research line): In the final section, the chapter concluded with a cohesive summary, reiterating the thesis's significance and contributions to the field of smart infrastructure monitoring. The chapter emphasized the relevance and potential impact of the self-sensing cementitious geocomposite in ensuring the sustainability and resilience of transportation and railway systems. Additionally, the chapter reinforced the importance of future research endeavours to enhance the material's capabilities and broaden its implementation in diverse infrastructure contexts. By culminating with a forward-looking perspective, Chapter Six successfully concluded the thesis, leaving the door open for further advancements and innovations in the domain of smart infrastructure monitoring using the innovative cementitious geocomposite.

1.8 List of publications subjacent to this thesis:

Chapter 2: Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "A review of intrinsic self-sensing cementitious composites and prospects for their application in transport infrastructures." *Construction and Building Materials* 310 (2021): 125139.

<https://doi.org/10.1016/j.conbuildmat.2021.125139>

Chapter 3: Abedi, Mohammadmahdi, Raul Fanguero, and Antonio Gomes Correia. "An effective method for hybrid CNT/GNP dispersion and its effects on the mechanical, microstructural, thermal, and electrical properties of multifunctional cementitious composites." *Journal of Nanomaterials* 2020 (2020).

<https://doi.org/10.1155/2020/6749150>

Chapter 4: Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "Ultra-sensitive affordable cementitious composite with high mechanical and microstructural performances by hybrid CNT/GNP." *Materials* 13, no. 16 (2020): 3484. <https://doi.org/10.3390/ma13163484>

Chapter 5: Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "Development of a novel multifunctional cementitious-based geocomposite by the contribution of CNT and GNP." *Nanomaterials* 11, no. 4 (2021): 961. <https://doi.org/10.3390/nano11040961>

Chapter 6: Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "Innovative self-sensing fiber-reinforced cemented sand with hybrid CNT/GNP." *Smart Materials and Structures* 30, no. 10 (2021): 105034. <https://doi.org/10.1088/1361-665x/ac2108>

Chapter 7: Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "Effects of electrodes layout and filler scale on percolation threshold and piezoresistivity performances of a cementitious-based geocomposite." *Nanomaterials* 12, no. 10 (2022): 1734. <https://doi.org/10.3390/nano12101734>

Chapter 8: Abedi, Mohammadmahdi, Mohammad Jawed Roshan, Federico Gulisano, Javad Shayanfar, Mostafa Adresi, Raul Fanguero, and António Gomes Correia. "An Advanced Cement-Based Geocomposite with Autonomous Sensing and Heating Capabilities for Enhanced Intelligent Transportation Infrastructure." *Construction and Building Materials*. No. 411 (2024): 134577. <https://doi.org/10.1016/j.conbuildmat.2023.134577>

1.9 Contributions and Originality:

The present section aims to elucidate the substantial contributions and originality of this research endeavour within the domain of smart infrastructure monitoring, specifically focusing on transportation

and railway applications, through the development and exploration of an innovative cementitious geocomposite. **Novelty of the Smart Cementitious Geocomposite:** The primary contribution of this thesis lies in the conception and development of an innovative cementitious geocomposite with self-sensing capabilities. The novel amalgamation of conductive fillers and cementitious geomaterials has given rise to a multifunctional material that exhibits high mechanical, microstructural, durability, piezoresistive and joule behaviour, enabling it to function as a smart and multifunctional material within critical infrastructure elements such as transportation and railway. **Advancements in Smart Infrastructure Monitoring:** The research work presented here represents a significant advancement in the realm of smart infrastructure monitoring. By leveraging the self-sensing attributes of the cementitious geocomposite, transportation and railway systems can be endowed with real-time monitoring capabilities. This development holds the potential to revolutionize the traditional approaches to infrastructure management by enabling proactive, data-driven decision-making for improved performance and longevity. **Comprehensive Investigation of Key Parameters:** A noteworthy aspect of this research is the systematic investigation of critical parameters that influence the sensing capacity of the cementitious geocomposite. By conducting a detailed study on size, electrode layout, temperature, moisture, and applied load, this work has provided crucial insights into optimizing the composite's sensing potential, making it adaptable to a wide range of operational scenarios. **Integration of Fibrous Materials for Enhanced Performance:** The incorporation of fibrous materials to enhance the composite's performance represents an original approach within the field. The meticulous exploration of the synergistic effects of these fibrous elements has yielded valuable insights, highlighting the composite's versatility and potential to improve specific operational aspects. **Applicability to Damage Diagnosis and Self-Heating:** Another significant contribution is the assessment of the cementitious geocomposite's application beyond traditional monitoring. By evaluating its effectiveness in damage diagnosis and self-heating performances, this research opens up new avenues for the composite's utilization in diverse scenarios, enhancing its overall practicality and relevance. **Potential for Sustainable and Resilient Infrastructure:** The outcomes of this research contribute to the broader objective of fostering sustainable and resilient infrastructure development. By enabling real-time monitoring and proactive maintenance, the self-sensing cementitious geocomposite can enhance the longevity and performance of transportation and railway systems, while reducing the environmental impact and life-cycle costs.

In conclusion, the outcomes of this thesis highlight the ground-breaking work in the development of the innovative cementitious geocomposite and its application to smart infrastructure monitoring in

transportation and railway sectors. The research addresses critical parameters, explores novel integration of self-sensing materials, and expands the composite's applicability to damage diagnosis and self-heating performances. By paving the way for enhanced infrastructure resilience and sustainability, this research represents a pioneering advancement in the field of smart infrastructure development.

1.10 List of cumulative publications within the scope of the thesis topic during Ph.D. period:

1. Abedi M, Roshan MJ, Gulisano F, Shayanfar J, Adresi M, Figueiro R, et al. An advanced cement-based geocomposite with autonomous sensing and heating capabilities for enhanced intelligent transportation infrastructure. *Construction and Building Materials*. 2024; 411:134577. <https://doi.org/10.1016/j.conbuildmat.2023.134577>
2. Abedi M, Shayanfar J, Al-Jabri K. Damage assessment via machine learning approaches: a systematic review. *Asian Journal of Civil Engineering*. 2023;1-30. <https://doi.org/10.1007/s42107-023-00748-5>
3. Abedi M, Sanivada UK, Mirian SA, Hassanshahi O, Al-Jabri K, Correia AG, et al. A self-sensing and self-heating planar braided composite for smart civil infrastructures reinforcement. *Construction and Building Materials*. 2023; 387:131617. <https://doi.org/10.1016/j.conbuildmat.2023.131617>
4. Abedi M, Hassanshahi O, Rashidell A, Ashtari H, Meddah MS, Dias D, et al. A sustainable cementitious composite reinforced with natural fibers: An experimental and numerical study. *Construction and Building Materials*. 2023; 378:131093. <https://doi.org/10.1016/j.conbuildmat.2023.131093>
5. Abedi M, Figueiro R, Correia AG, Shayanfar J. Smart Geosynthetics and Prospects for Civil Infrastructure Monitoring: A Comprehensive and Critical Review. *Sustainability*. 2023;15(12):9258. <https://doi.org/10.3390/su15129258>
6. Abedi M, Hassanshahi O, Barros JA, Correia AG, Figueiro R. Three-dimensional braided composites as innovative smart structural reinforcements. *Composite Structures*. 2022; 297:115912. <https://doi.org/10.1016/j.compstruct.2022.115912>
7. Abedi M, Figueiro R, Correia AG. Effects of electrodes layout and filler scale on percolation threshold and piezoresistivity performances of a cementitious-based geocomposite. *Nanomaterials*. 2022;12(10):1734. <https://doi.org/10.3390/nano12101734>

8. Abedi M, Fangueiro R, Correia AG. A review of intrinsic self-sensing cementitious composites and prospects for their application in transport infrastructures. *Construction and Building Materials*. 2021; 310:125139. <https://doi.org/10.1016/j.conbuildmat.2021.125139>
9. Abedi M, Fangueiro R, Correia AG. Effects of multiscale carbon-based conductive fillers on the performances of a self-sensing cementitious geocomposite. *Journal of Building Engineering*. 2021; 43:103171. <https://doi.org/10.1016/j.jobe.2021.103171>
10. Abedi M, Fangueiro R, Correia AG. Innovative self-sensing fiber-reinforced cemented sand with hybrid CNT/GNP. *Smart Materials and Structures*. 2021;30(10):105034. <https://iopscience.iop.org/article/10.1088/1361-665X/ac2108/meta>
11. Abedi M, Fangueiro R, Correia AG. Development of a novel multifunctional cementitious-based geocomposite by the contribution of CNT and GNP. *Nanomaterials*. 2021;11(4):961. <https://doi.org/10.3390/nano11040961>
12. Abedi M, Correia AG, Fangueiro R. Geotechnical and piezoresistivity properties of sustainable cementitious stabilized sand reinforced with recycled fibres. *Transportation Engineering*. 2021; 6:100096. <https://doi.org/10.1016/j.treng.2021.100096>
13. Abedi M, Fangueiro R, Gomes Correia A. Ultra-sensitive affordable cementitious composite with high mechanical and microstructural performances by hybrid CNT/GNP. *Materials*. 2020;13(16):3484. <https://doi.org/10.3390/ma13163484>
14. Abedi M, Fangueiro R, Correia AG. An effective method for hybrid CNT/GNP dispersion and its effects on the mechanical, microstructural, thermal, and electrical properties of multifunctional cementitious composites. *Journal of Nanomaterials*. 2020;2020. <https://doi.org/10.1155/2020/6749150>
15. Abedi M, Fangueiro R, Camões A, Correia AG. Evaluation of CNT/GNP's synergic effects on the Mechanical, Microstructural, and durability properties of a cementitious composite by the novel dispersion method. *Construction and Building Materials*. 2020; 260:120486. <https://doi.org/10.1016/j.conbuildmat.2020.120486>

Chapter 2. State of The Art.

« In spite of everything, I still believe people are really good at heart. »

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

- Abedi, Mohammadmahdi, Raul Figueiro, and António Gomes Correia. "A review of intrinsic self-sensing cementitious composites and prospects for their application in transport infrastructures." *Construction and Building Materials* 310 (2022): 125139.
<https://doi.org/10.1016/j.conbuildmat.2021.125139>

Abstract

Monitoring of transport infrastructures, in terms of early damage detection, can prevent the loss of life and economic damage associated with sudden infrastructure collapse and inform timely intervention, such as repair, to increase the sustainability and service life of infrastructures. Self-sensing cementitious geocomposites with the ability to detect stress, strain, and damage based on a piezoresistive mechanism enable the development of more integrated and viable geomaterial monitoring solutions than existing monitoring technologies. Self-sensing cementitious geocomposites are composed of conductive phases embedded in cementitious geomaterials that exhibit both sensing ability and superior mechanical properties. The states of stress, strain, displacement, and damage in infrastructures can be investigated by analysing the change in their electrical resistance. In this review, different types of self-sensing composites, their preparation, influential parameters, and associated theoretical investigations are discussed in detail to inform future advances in the development of self-sensing geocomposites. The challenges of this technology have also been summarised. This review is expected to stimulate and inform research that explores the development and application prospects of self-sensing cementitious geocomposites.

2.1 Introduction

Geomaterials, as an integral part of transport infrastructures, play a crucial role in the stability and safety of these structures. Structural monitoring enables the early detection of damage that, if addressed, reduces the risk of sudden collapses and associated economic damage and optimises the service life of structures. Furthermore, infrastructure monitoring systems are useful platforms for extracting current data at an operational level which have many benefits in various areas of smart cities management such as transportation, and traffic flow monitoring. Both of these aspects support the economic sustainability of societies and quality of life of members. In this regard, the integrated and real-time monitoring of stress, strain, and displacement in cementitious geocomposites, a widely used geomaterial, is the key factor. Internationally, significant research efforts are expended on developing innovative and smart infrastructure monitoring techniques [79, 80] to meet ever-growing engineering demands and take advantage of relevant technological advances. Despite recent advances, most monitoring instrumentation or equipment, such as fibre Bragg grating sensors, electric strain gauges, and PZT-based piezoelectric sensors, involve the placement of a limited number of sensors in spot locations along applicable infrastructure. In addition, owing to the low durability and accuracy of these instruments, complexity and high cost of their production, and incompatibility of their detection mechanisms with geomaterial behaviour, many of these methods are considered insufficient. Among all the monitoring methods summarised in Table 2-1, considering the properties and nature of geomaterials, self-sensing composites with intrinsic sensing capabilities based on piezoresistive behaviour provide the most integrated, real-time, and practical solution for infrastructure monitoring and damage detection. This smart geomaterial can facilitate the development of intelligent infrastructure with sensing and structural health monitoring abilities, thus enhancing infrastructure safety, durability, serviceability, and reliability [3]. Self-sensing geocomposites (SSGs) offer a new approach to enhancing the sustainability of geomaterials. Self-sensing composites are formed by dispersing conductive phases in conventional cementitious composites that impart the ability to sense internal strain, stress, cracks, or damage while maintaining or even enhancing the physical properties and durability of the composite. The conductive phase generates an extensive network of conductive pathways based on percolation and quantum tunnelling mechanisms which is disturbed or changed if the composite is stressed or deformed under external force or environmental action, altering its electrical behaviour. Hence, stress (or external force), strain (or deformation), damage, and cracking under dynamic and static conditions can be detected by evaluating the electrical properties of the self-sensing composite. SSGs can act as structural and sensing components, eliminating the need for additional sensors. Several studies have evaluated self-sensing materials featuring various composites

such as concrete, cement paste, polymers, and asphalt [81, 82]. However, to the best of the authors' knowledge, no reported study has assessed SSGs. Notably, the piezoresistive behaviour and sensitivity of composites are easily affected by multiple external and internal parameters such as the type and content of the conductive phase and non-conductive matrix, manufacturing methods (dispersion and curing methods), loading type, measurement method, and environmental conditions (humidity and temperature). This paper provides a systematic review of the main advances in self-sensing composite technology to inform the development of SSGs.

2.2 Metal-Based Self-Sensing Composites

Initial investigations of cementitious self-sensing composites featured a cement matrix and metallic conductive phases such as steel fibres, steel shavings, and steel slag [83-85]. Steel fibres have been proven to be more effective than the other two steel forms in reducing the electrical resistivity of concrete, while enhancing the flexural and tensile strength owing to a pull-out effect [86]. The sensitivity of a cementitious composite reinforced by steel fibres reportedly improves with increasing fibre concentration up to, approximately, the percolation threshold. The performance of both macro steel fibres (diameter $\approx$ 0.25 mm to 0.64 mm and length $\approx$ 13 mm to 32 mm) and super-fine steel fibres (diameter $\approx$ 8 μ m to 20 μ m and length $\approx$ 10 mm) have been investigated in self-sensing composites. However, composites featuring super-fine steel fibres exhibit superior piezoresistive behaviour under cyclic loading. Moreover, the application of macroscale metallic conductive phases can promote microcrack formation in the composite and consequently impair its mechanical properties [87]. Notably, the piezoresistive behaviour of cementitious composites can be affected by the corrosion of metallic conductive phases. With the advancement of nanotechnology, metal nanomaterials such as nano TiO_2 (NT) and nano Fe_2O_3 are garnering significant attention as low-cost conductive fillers owing to their remarkable mechanical, electrical, and chemical features [88].

Table 2-1. Summary of major infrastructure monitoring methods and their advantages and limitations

Sensors and technologies	Application method	Features and advantages	Limitations	Reference
Strain gauges	Attached to the surface, Embedded	Wide application, high flexibility, low cost, wide measurement range, non-destructive testing, soft robotics, low transverse effect, high permissible current.	Low durability, complex production, non-intrinsic mechanism, point sensors, sensitive to humidity and temperature, low resolution of strain, limited reliability and stability in highly explosive monitoring atmospheres, low measurement frequency.	[89, 90]
Fibre optics	Embedded	High accuracy and sensitivity, non-conductive testing, easy installation, small size, highly resistant to electromagnetic interference (EMI), lightweight, long-distance monitoring, wide range of measurement, high elasticity.	High production costs, vulnerable, non-intrinsic mechanism, limited measurement resolution, and low measurement frequency, pretension required for measurement, loop optical fibre measurement, limited bending diameter.	[91-93]
Fibre Bragg gratings	Embedded	Multiplexed specifications, the ability of real-time and wireless monitoring, the capability of dynamic monitoring, small size and lightweight, wide range of measurement, resistant to EMI.	Point strain measurement, complex production and encapsulation, high production costs, pretension required for measurement.	[92]
Piezoelectric sensors	Embedded, Composite	Energy harvesting, easy fabrication, lightweight, low power consumption.	Point measurement, environmentally hazardous, low durability.	[94]
Piezoceramics	Embedded	Wide application, high sensitivity, appropriate durability, functional at high temperatures.	Point strain measurement, high cost, non-intrinsic mechanism, cannot be used on a curved or irregular surface (e.g. a weld or a corner).	[95-97]
Electrochemical and biosensors	Composite	Measuring chemical reactions, short response time, low energy consumption.	Low durability, environmentally hazardous, complicated encapsulation method, medium selectivity, signal sensitive to operation temperature and humidity.	[98]
Self-sensing composites	Composite	High sensitivity and durability, easy to manufacture, wide measurement range, appropriate sensitivity to dynamic load, high resolution, improves mechanical and physical properties of the composite matrix.	High production costs and pore formation in the case of high conductive-phase concentrations.	[81]
Shape memory alloy	Embedded	High sensitivity and accuracy, appropriate damping behaviour.	Point strain measurement, high cost, non-intrinsic.	[99]
X-ray or C-scan	Non-contact	Wide application, non-destructive testing.	High cost of production, extra analysis, and non-real time, radiative.	[100]
Camera	Non-contact	Convenient, simple operation.	High cost, vulnerable.	[101]

Owing to the small size of NT particles (approximately 20 nm), their addition improves the physical properties of cementitious composites by a functional filler mechanism and increases the initial hydration rate [102, 103]. Although the electrical conductivity of metal nanomaterials is not as high as that of carbon nanomaterials (CNMs), previous studies have shown that nanometal fillers can significantly reduce

the electrical resistivity of cementitious matrices [104-106]. However, their low durability, vulnerability to corrosion, and reactivity with environmental compounds have limited their application in geomaterials.

2.3 Carbon Black–Based Self-Sensing Composites

Informed by earlier studies of self-sensing composites and motivated by their potential benefits, researchers have attempted to improve their sensitivity and electrical behaviour through the use of inexpensive fillers. Carbon black (CB), which is produced by the thermal decomposition of heavy petroleum products, is such a filler. CB or nanocarbon black (NCB) are types of paracrystalline carbon that are characterised by good conductivity, cost efficiency, and high specific surface area, albeit lower than those of carbon nanotubes (CNTs) and carbon nanofibers (CNFs) [107]. The incorporation of CB into cementitious composites enable quasi-static and dynamic monitoring based on piezoresistive behaviours [108, 109]. However, the inclusion of CB does not enhance the mechanical performance of the composite, unlike the inclusion of other carbon-based materials [110, 111].

Although the chemical structure and geometric properties of CB, as a filler, increase its dispersion potential in aqueous suspensions, large amounts are required to form conductive paths in composites [107]. In addition, CB-based self-sensing cementitious composites typically demonstrate lower strain and crack sensitivity than carbon nanotubes (CNT) and carbon nanofibers–based (CNF) self-sensing cementitious composites [107, 112-116].

Monteiro et al. reported that the percolation threshold of a cementitious mortar containing CB is approximately 10%. Moreover, the composite achieves a gauge factor of approximately 23.14, at this concentration [108]. Another study lowered the percolation threshold to 6.5% by reducing the temperature to 15 °C, resulting in a gauge factor of approximately 50 at this temperature [109].

However, high concentrations of CB can impair the physical and mechanical properties, and durability of cementitious composites owing to the formation of agglomerates and pores [117, 118]. This is especially important in cement-stabilised geomaterials in which low amounts of cement and water are used since the consequent low amounts of cement hydration products are not sufficient to envelop all the CB particles, resulting in an incoherent microstructure.

2.4 Carbon Fibre–Based Self-Sensing Composites

2.4.1. Carbon Short Fibres

Carbon short fibres (CSFs) are fibrous materials that are highly suitable fillers for self-sensing composites owing to characteristics such as corrosion resistance, high-temperature tolerance, low electrical resistance, and high thermal conductivity. In addition, carbon fibres (CFs) have a high tensile modulus and long-axis strength that improve the mechanical properties of composites to a certain extent; hence, they have been widely used as conductive phases in cementitious, polymer, and asphalt composites [107, 110, 119-122]. However, the relatively high price of these fibres limits their extensive application [123].

Studies have found that the volume percentage of CSFs required to achieve self-sensing cementitious composites with appropriate sensitivity ranges from approximately 0.5 to 3% [81, 110, 124, 125]. However, increasing the CSF volume percentage beyond 1.0–1.5%, usually causes some agglomeration [126, 127]. Furthermore, the incorporation of high amounts of CSFs reduces the workability of cementitious matrices necessitating the use of superplasticisers [128]. Studies have shown that the geometrical dimensions and aspect ratios of CSFs significantly affect the percolation threshold, sensitivity, and physical properties of composites [129-131].

As demonstrated in Figure 2-1, the electrical resistivity of a cementitious composite reinforced with CSFs decreases from 8×10^6 to $6 \times 10^6 \Omega \cdot \text{cm}$ with increasing CSF aspect ratio from 2:1 to 12:1.

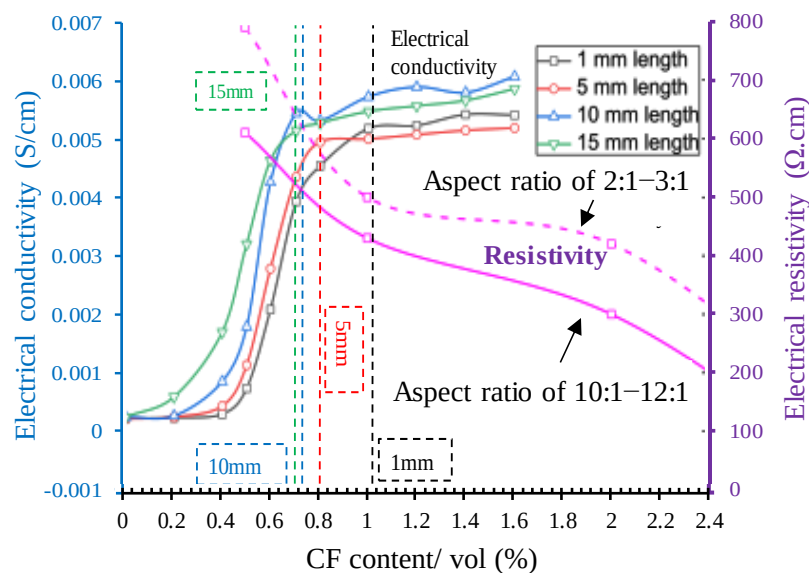


Figure 2-1. The effect of CSF aspect ratio on electrical resistivity and conductivity of cementitious composites, [81, 132, 133].

The effects of the aspect ratio on the piezoresistive behaviour of CSF-reinforced, cement-based mortar under different cyclic compression loads were evaluated by Baeza et al. [129]. They prepared composite

specimens (160×40×40 mm in size) incorporating 0.5–1 wt% of CSFs (7.2 μm in diameter and 3, 6, and 12 mm in length) and measured their mechanical and strain sensitivity.

They found that the percolation threshold and piezoresistive response of the composite decrease with increasing aspect ratio of the fibres, while relatively high fibre lengths reduce the compressive strength of cementitious mortar. A maximum sensitivity (gauge factor) of approximately 32 is achieved by a composite containing 0.5 wt% of CSFs, 12 mm in length. In general, composites incorporating fibres with high aspect ratios demonstrate lower electrical resistance than composites incorporating fibres with low aspect ratios that realise an equivalent total fibre length [81, 129, 134]. However, CSFs are hydrophobic and their dispersion in water becomes more difficult with increasing fibre length and aspect ratio.

The piezoresistive behaviour and mechanical properties of cementitious mortar containing 2, 3, and 4 wt% of CSFs (relative to the weight of the cement) with length and diameter of 6 mm and 7.5 μm , respectively, were evaluated by Donnini et al. [124]. Although the incorporation of CSFs into cementitious mortar at these weight percentages generally decreases the compressive strength of the mortar due to an increase in its porosity, the electrical conductivity of the composite reduces significantly with increasing CSF content such that the percolation threshold achieved is approximately 3%. They showed that the electrical conductivity of a specimen containing 1 wt% of CSFs decreases with increasing cement hydration time, while the electrical resistance of specimens containing 3 and 4 wt% of CSFs reaches a constant value after 60 days of hydration. In addition, optimum sensitivity under cyclic compression loading is achieved by a specimen reinforced with 3 wt% of CSFs, determined by measuring the fractional change in its electrical resistivity using a two-probe method and alternating current (AC). In this study, the cement:sand and water:sand ratios were approximately 66.7:100 and 42.7:100, respectively.

A correlation between the fractional change in the electrical resistivity and the change in the strain of concrete reinforced with 0.5, 1, 1.5, and 2 wt% CSFs (relative to the weight of the cement) has been reported by Cholker et al. [135]. They used CSFs with a diameter and length of 7 μm and 6 mm, respectively. Although the compression strength of the composites decreases with increasing CSF content, the composites containing 1.5 and 2 wt% of CSFs demonstrate appropriate piezoresistive behaviour, as shown in Figure 2-2.

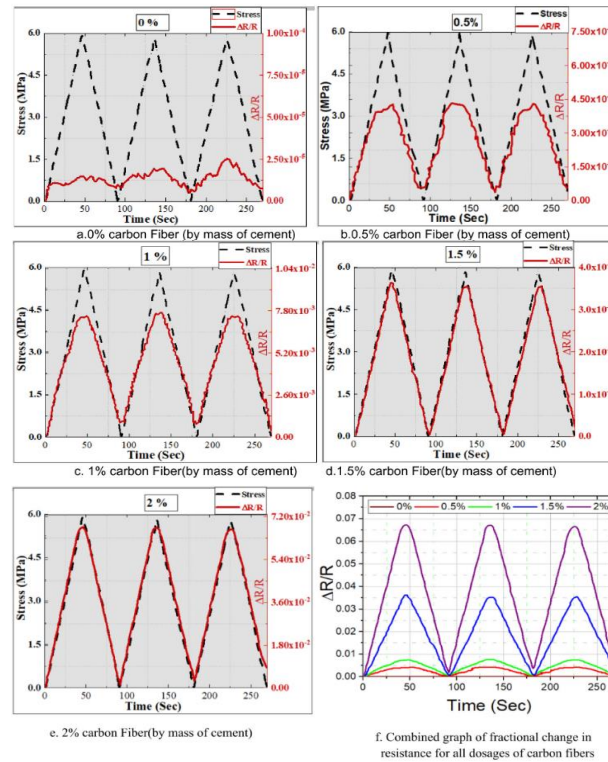


Figure 2-2. Correlation between strain, stress, and electrical resistance [135].

Combining CSFs with other conductive fillers such as steel fibres or carbon nanomaterials (CNMs) is also an effective way to increase the sensitivity of a composite and reduce its percolation threshold [125, 136-138]. A summary of studies that have investigated the damage-monitoring performance of CSF-reinforced cementitious matrices is presented in Table 2-2.

Table 2-2. Summary of studies investigation carbon short fibres in cementitious matrices

Composite type	Conductor type	CSF length (mm)	Concentration (%) (by volume (vol.) or weight (wt.))	Electrical factors			References
				$\Delta R/R_0$ (%)	Gauge factor	Resistivity ($\Omega \cdot \text{cm}$)	
Concrete	CSF	5	0.5 of vol.	0.37	-	-	[139]
			2.0 of vol.	1.01	-	-	
			3.0 of vol.	1.32	-	-	
Cement paste	CSF	5	15.0 of wt.	-	55.3	-	[133]
			20.0 of wt.	-	3707	-	
			25.0 wt.	-	51.9	-	
Cement paste	CSF	7	1.0 of vol.	-	-	675	[140]
			3.0 of vol.	-	-	66	
			5.0 of vol.	-	-	17	
Cement paste	CSF + CNT	10	15 of vol (CSF)	26	-	-	[138]
			15 of vol. (CSF) + 1 of vol. (CNT)	23	-	-	
Cement paste	SF + CSF	10	0.36 of vol.	-	-	57	[141]
			0.72 of vol.	-	-	16	
Mortar	CB + SF + CSF	5	15 of wt. (SF) + 0.5 of wt. (CB)	-	1.6	-	[142]
			15 of wt. (SF) + 0.5 of wt. (CSF)	-	327	-	
			15 of wt. (SF) + 1 of wt. (CSF)	-	332	-	
			15 of wt. (SF) + 0.5 of wt. (CB) + 0.5 of wt. (CSF)	-	17	-	
			50 of vol. (clay) + 0.6 of vol. (CF)	13	-	-	
Mortar	Conductive clay + CSF	5	30 of vol. (clay) + 0.6 of vol. (CF)	17	-	-	[143]
			30 of vol. (clay) + 0.9 of vol. (CF)	16	-	-	

The combination of different conductive material with different geometrical shape and scale not only increase specific surface area and caused an increment in the conductive paths but also facilitate their dispersal process. This is especially important in geotechnical construction that often use optimally low amounts of water and cement, since the dispersion of carbon conductive fillers in aqueous suspensions is one of the most common dispersion methods in cementitious composites [144-146]. Although existing research on the effect of cement content on the piezoresistive behaviour of cementitious composites is limited, available reports of studies suggest that a coherent and stable structure is required to maintain the microscale conductive network formed by CFs. Reducing the cement content increases the percolation threshold necessitating the use of greater amounts of fibres [133]. Besides, by reducing the amount of cement, the possibility of porosity formation due to the presence of micro fillers increases.

Therefore, the amount of cement, a determining factor of the strength of cementitious composites, plays a crucial role in the piezoresistive performance of a CF- based composite. However, composites with greater ductility show better piezoresistive behaviour due to premature nanoscale cracks that form during the early stages of loading. These cracks might cause residual strain and/or fractional change in electrical resistivity and disturbance. Notably, the use of CSFs reduces the sensitivity of the microscale conductive networks to such cracks and increases composite ductility, which is one of the advantages of CFs. In cementitious geocomposites, the conventional amount of cement used for stabilisation is approximately 5–12% of the soil mass [147, 148]. It is, accordingly, not feasible to incorporate high amounts of CSFs or CSFs with long lengths in stabilised soils. Generally, interactions between fibres and soil grains are strongly dependent on the size and geometrical shape of the grain [149]. The presence of fibre agglomerates among soil grains increases the gaps between them and consequently decreases the density of the specimen, which can greatly reduce the cohesion and physical characteristics of cementitious geocomposites. Therefore, the application of a conductive phase that combines CSFs and CNMs in cementitious geocomposites with low cement and water content might mitigate the disadvantages associated with high CSF content.

4.2. Continuous Carbon Fibres

4.2.1. Non-polymeric, Continuous Carbon Fibre–Based, Self-Sensing Cementitious Composites

Owing to the unique mechanical and electrical properties of continuous CF, they can be used in the production of multifunctional cementitious composites as woven or non-woven elements. However, the absence of polymeric support increases the effect of moisture on the sensing ability of the composite. Self-sensing concretes with strain-sensing ability have recently been developed by incorporating continuous CF–based textiles, as shown in Figure 2-3. The difference between the strains measured by strain gauges and the textile sensor is approximately 5%, which indicates a good correlation. In addition, the gauge factor for this smart concrete was reported to be approximately 10. A hybrid continuous carbon and glass fibre (GF) textile was developed by Goldfeld et al. [150] for moisture and damage sensing in a concrete beam. They achieved an electromechanical sensing gauge factor of approximately 1 and detected a humid environment by fractional changes in electrical resistance in the order of 10^{-5} , applying the Wheatstone bridge principle. Non-polymeric continuous CF composites are also suitable for the development of multifunctional cementitious geocomposites, especially fine-grain geomaterials, owing to

the higher interaction and consolidation ability of these types of composites. However, this type of composite is sensitive to environmental factors.

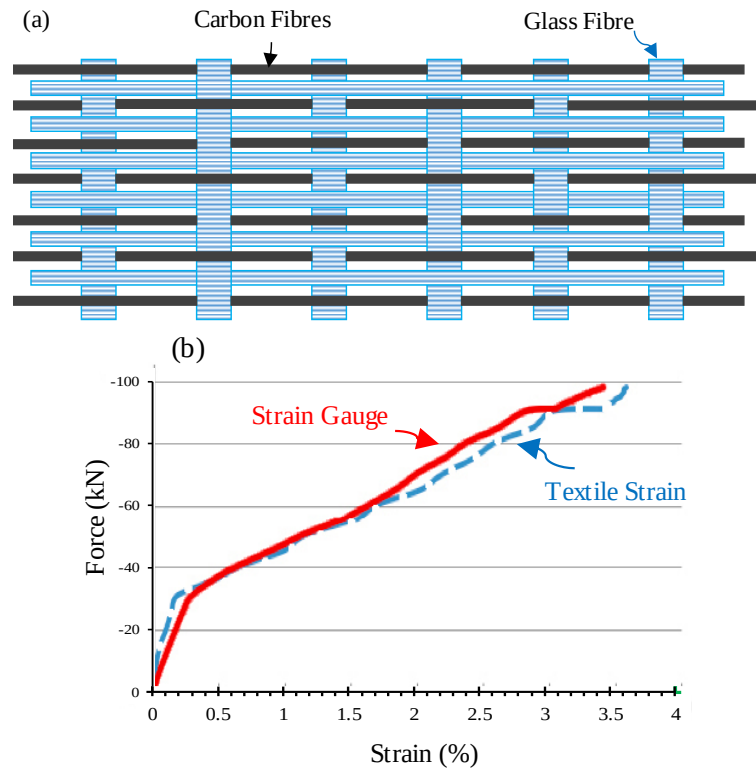


Figure 2-3. (a) Continuous carbon fibre textile (b) Correlation between strains measured by strain gauges and a textile sensor [150]

4.2.2. Polymeric, Continuous Carbon Fibre–Based, Self-Sensing Cementitious Composites

CF-reinforced polymers are composites produced by combining different forms of the carbon fibres, including textile fabrics or unidirectional tows (either knitted or woven), and polymeric resin under appropriate conditions [119]. These composites are characterised by low density, high rigidity, high strength, excellent damping or energy dissipation, high resistance to impacting and corrosion, and modifiable thermal expansion [151]. In addition, because of the presence of conductive CFs in their structures and their piezoresistive behaviour which is affected by strain, stress, displacement, and damage, these composites have attracted the attention of many researchers engaged in the development of self-sensing composites [152-154]. Furthermore, hybrid composites of carbon and other fibres (e.g., aramid, polypropylene, and glass) have also been developed with enhanced sensing capability and strength. The strain-sensing capability and properties of CF-reinforced polymers depend on the arrangement of the composite structure components [155]. The strain sensitivity of unidirectional, CF-reinforced epoxy composites is reportedly in the fibre direction. Tensile loading reversibly reduces the longitudinal electrical resistance, whereas the transverse electrical resistance increases with increasing

strain. This can be attributed to changes in the electrical contacts of the CFs owing to changes in fibre alignment. The degree of alignment of fibres in a composite subjected to tensile loading increases in the loading direction which increases the electrical contacts of the fibres and causes a reduction in resistance [156]. However, an excessive increase in the load can lead to a decrease in the fibre cross sections and an increase in the electrical resistance. Generally, the gauge factors of these types of CF-reinforced polymers in the longitudinal and transverse directions range from -35.7 to -37.6 and $+34.2$ to $+48.7$, respectively. Hence, they are highly suitable for sensing applications in geomaterials in transport infrastructures, especially railway foundations. Plastic laminates reinforced with CFs are reportedly effective at detecting cracks, delamination, and different types of damage that occur within their structures [157-159]. However, CF-reinforced polymers are brittle and exhibit low ductility. Recently, researchers have attempted to engineer lightweight ductile multifunctional reinforcements as replacements for steel to avoid its characteristic corrosion and associated defects. Hybrid continuous CF–epoxy composites were developed with high breaking strains, ductility, and tailorable mechanical properties. Subsequent studies have explored the modification of hybrid continuous CF–epoxy composites with other materials to improve their strength and self-sensing performance [160, 161]. The proportion of constituent fibres, their properties, and their arrangement within composites are crucial factors affecting the ductility and detection capabilities of the composite. GFs, as one of the most conventional and widely available type of fibre, have attracted the attention of many researchers for the development of hybrid polymeric CF–GF composites with superior ductility and sensitivity [162, 163]. As illustrated in Figure 2-4 (a), these composites are composed of CF cores that are enclosed in GFs.

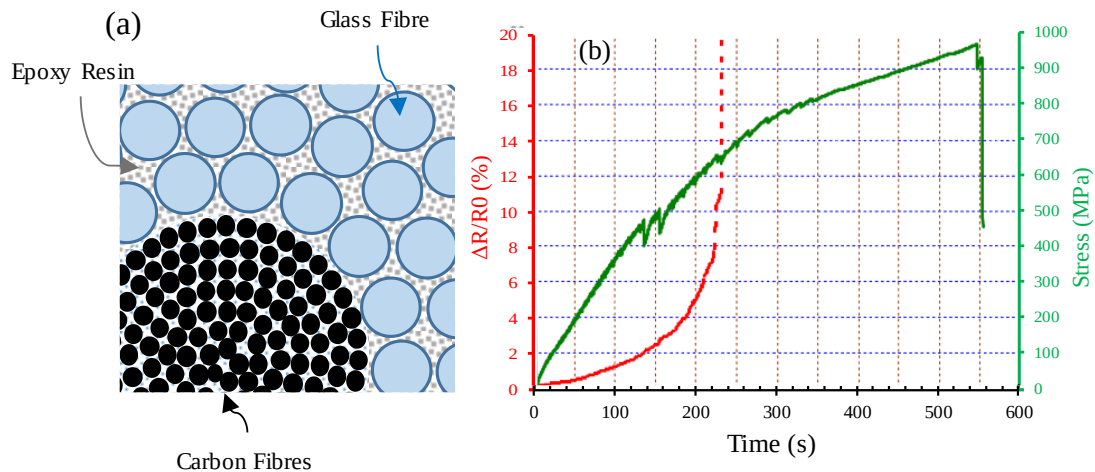


Figure 2-4. (a) Morphology of hybrid CF–GF composite rods (b) Variation of stress (solid line) and frictional resistance (dotted line) during monotonic tensile testing of hybrid GF–CF rods with GF and CF contents of 48 and 0.2 vol%, respectively, [164]

The composites exhibit appropriate piezoresistive behaviour under cyclic and monotonic tensile loading.

Figure 2-4 (b) shows that the electrical resistance of the composite increases sharply during the breakage of CFs, this behaviour can be used as an alarm signal. Such an alarm signal is obtained near the

composite failure load of a composite with a CF and GF content of 2.4 and 49 vol%, respectively. However, the electrical resistance of composites with lower CF contents (i.e. 0.2 vol% (GF: 48 vol%) or 0.6 vol% (GF: 48 vol%), rises sharply under loads considerably less than their failure load. Hence, suitably designed, hybrid CF–GF–epoxy self-sensing composites are viable candidates for monitoring the structural health of civil infrastructure. However, hybrid CF–GF–epoxy composites are not able to detect damage at an early stage, the fractional changes in their electrical resistance under strain of less than 0.6% are approximately 1%, limiting their application. Appropriate strain detection under low strain is only achieved under pre-stressed conditions by calculating the residual resistance of the CF–GF composites [165]. Hence, to increase the strain sensitivity of CF–GF self-sensing composites, continuous CFs were replaced with carbon particles (Figure 2-5) which increased the fractional change in electrical resistance by approximately 6.2% under 0.6% strain and enhanced the ability of the composite to detect damage at early stages [166].

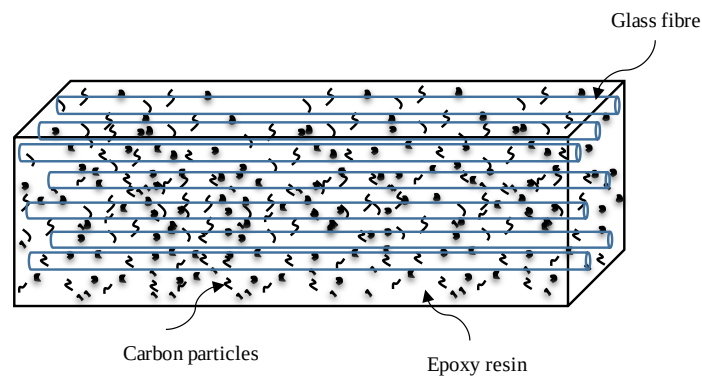


Figure 2-5. Schema of hybrid carbon particle–GF self-sensing bars [165].

Braided composite rods (BCRs) are self-sensing, hybrid continuous CF–GF composites that show high sensitivity under low strain [167-170]. In BCRs, a specific combination of CFs and GFs are impregnated with a polymeric resin and axially over-braided using polyester filaments (Figure 2-6). The resulting structures are cured to manufacture composite rods.

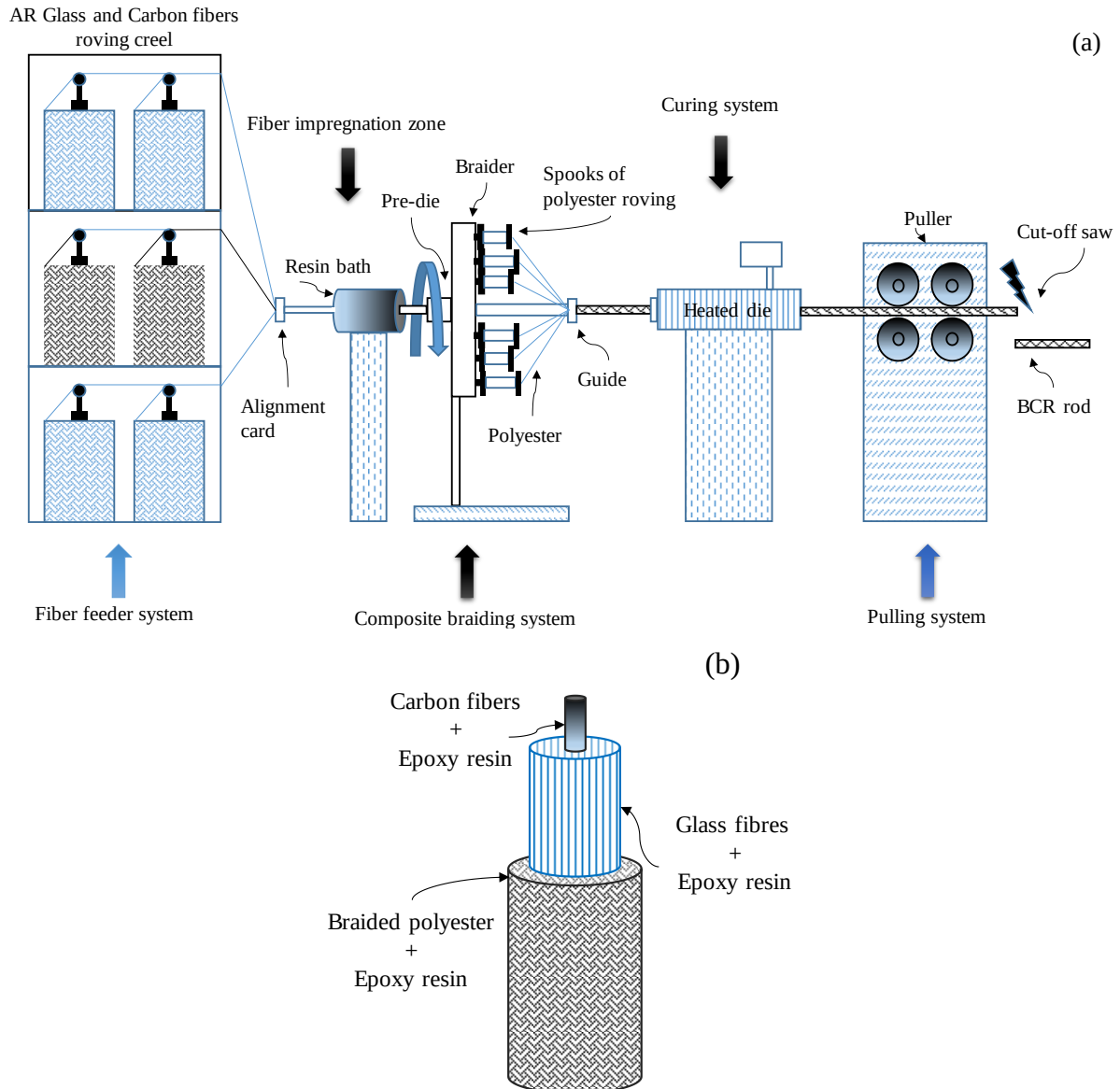


Figure 2-6. (a) Manufacturing process of a BCR. (b) Cross-section and braided surface [170].

In this technique, the introduction of a certain degree of axial CF misalignment, through the adjustment of the braiding parameters of the polyester filaments (including tension and speed), changes the electrical contacts of the CFs in the BCR and, concomitantly, the electrical resistance under low strain. In these composites, a reduction in the CF content increases the strain-sensing capability of the BCRs, and a maximum gauge factor of approximately 24 is achieved under a flexural strain of 0.5%.

2.5 Carbon Nanotubes and Carbon Nanofiber –Based, Self-Sensing Cementitious Composites

CNTs and CNFs are one-dimensional nanomaterials that, along with advances in nanotechnology, enable the development of a wide range of multifunctional cement-based materials. CNTs are composed of single or multiple layers of graphene that are wrapped in the shape of a cylinder, whereas in CNFs, the graphene

layers are arranged similar to stacks of cups, plates, or cones to create a nanoscale rod. These nanostructures are responsible for the remarkable physical, electrical, and thermal properties of CNTs and CNFs that have attracted the attention of researchers [79, 80, 171-175].

These conductive CNMs, once incorporated into non-conductive cementitious composites, form numerous nanoscale conductive paths owing to their high aspect ratios and specific areas. The sensitivity of these nanoscale conductive networks to low stress and strain increase the detection performance of a composites such that it is able to detect nanoscale cracks in the early stages of damage. In addition, reinforcing composites with certain amounts of CNTs and CNFs improves their physical and mechanical characteristics. However, the high price of CNTs and CNFs and their dispersion challenges limit the use of these nanomaterials.

Strong Van der Waals forces between the nanocarbon particles and the high specific surface areas of these nanomaterials greatly increase their tendency to agglomerate. In addition, the hydrophobicity of CNTs and CNFs retards their dispersion in aqueous suspensions.

Although techniques have been developed to promote the dispersion of CNMs in solutions, many of these adversely affect the intrinsic physical and/or electrical properties of the matrix and/or CNMs. Hence, an affordable and compatible dispersion technique is required to effectively impart the CNM properties to the composites. This is especially important when relatively high amounts of CNMs are used to achieve high sensitivity. High amounts of CNMs promote the formation of agglomerates which impair the physical properties of the composite matrix. However, the combination of these CNMs with conductive materials with different geometrical properties could resolve the dispersion difficulties and reduce production costs. To date, multiple multifunctional composites featuring CNTs, CNFs, or combinations of these CNMs have been developed for the detection of stress, strain, displacement, and cracks.

A hybrid smart composite rod with self-sensing capability was developed by Nanni et al. [166] to detect damage to a concrete beam. This hybrid composite consists of a conductive CNM-based core (5 wt% of CNMs, 30 nm in diameter) enclosed in an insulating GF-epoxy skin (Figure 2-7). As shown in Figure 2-8, the concrete beam exhibits good piezoresistive behaviour owing to the presence of these rods. The decay in the fractional change in resistance curves numbered from 1 to 4 represent different stages of damage, namely initial cracking, propagation, crack extension, and specimen failure, respectively.

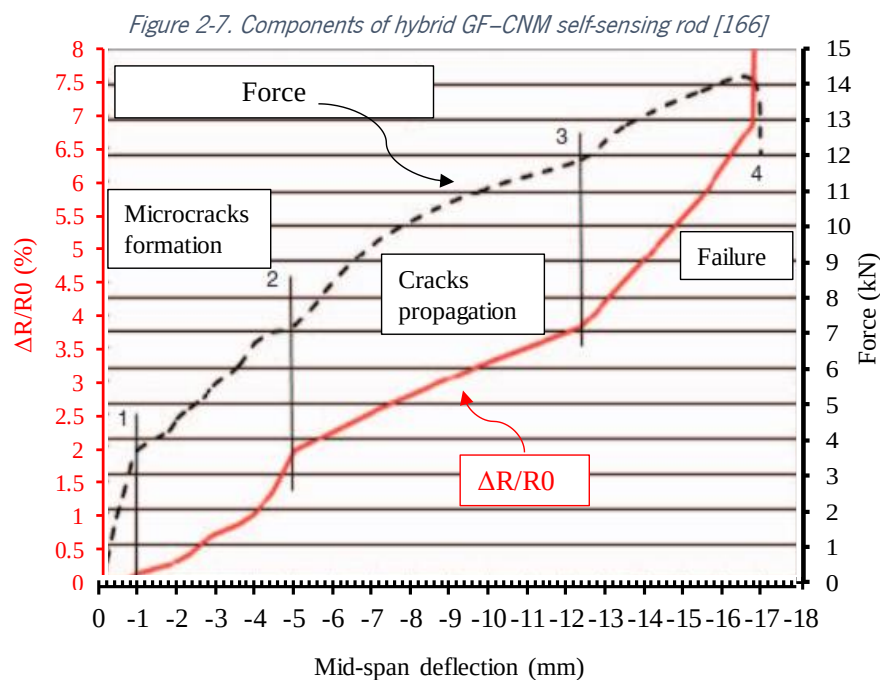
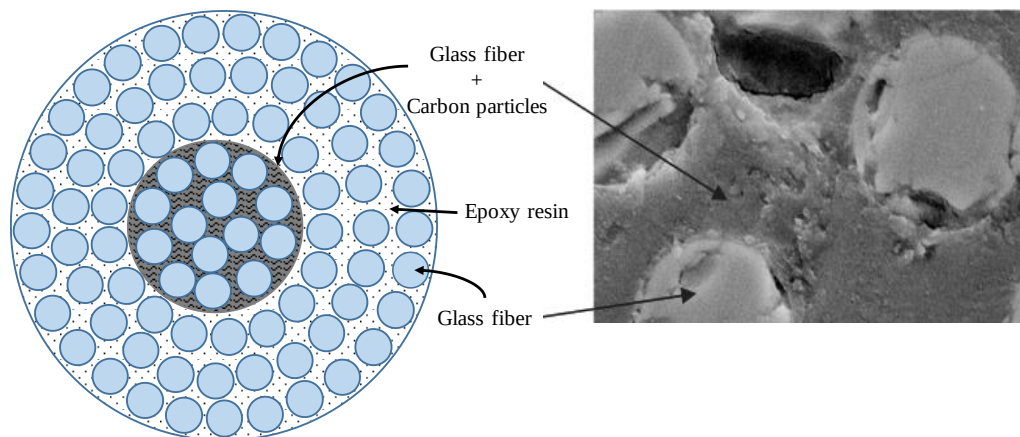


Figure 2-8. Fractional change in electrical resistance and change in load, and midspan deflection state at points 1, 2, and 3, [164].

A summary of other self-sensing composites featuring CNTs and CNFs is presented in Table 2-3.

Generally, CNFs and CNTs are the best conductive filler options for cementitious composites. The high aspect ratios and specific surface areas of these CNMs realise high piezoresistive behaviour under low strain, even at low concentrations. The nanoscale conductive paths formed by CNTs and CNFs remain stable in cement-stabilised sand with low cement content. However, the incorporation of CNMs generally can reduce the ductility of cementitious composites [176-181], the resulting brittleness of the composite leads to the formation of cracks during the initial stages of loading. In addition, the high costs of these CNMs and the challenges associated with their dispersion limit their application. While multiple studies have investigated different combinations of CNMs in terms of their self-sensing performances in cementitious composites, studies investigating the long-term piezoresistive behaviour of these particles are necessary to assess their suitability for the hostile environments of geomaterial infrastructures.

Table 2-3. Summary of studies investigating the damage monitoring performance achieved using CNTs and CNFs

Composite type	Conductor type	Conductive filler concentration (%)	Electrical factors		References
			$\Delta R/R_0$ (%)	Gauge factor	
Cement paste	CNT	0.2 of wt.	0.02	-	[182]
		0.3 of wt.	0.03	-	
		0.6 of vol.	0.04	1	
Cement paste	CNT	0.7 of vol.	0.05	50	[183]
		1.2 of vol.	0.06	2	
		0.5 of wt.	0.07	-	
Cement paste	CNF	1.0 of wt.	0.08	-	[184]
		2.0 of wt.	0.09	-	
Cement paste	CNT + CF	15 of vol. (CF)	0.10	-	[138]
		1 of vol. (CNT)	0.11	-	
Asphalt concrete	CNT	0.06 wt.	8.8% at 5.2 MPa	-	[185]
			10.3% at 8.6 MPa		
			8.4% at 5.2 MPa		
		0.1 wt.	11.4% at 8.6 MPa	-	
Mortar	CNT	2.14 vol.	6.8% at 4 MPa	-	[186]
Mortar	CNT	1	-	150	[187]
Mortar	CNT	0.3	-	254.3	[188]
Mortar	CNT	1		166.6	[189]

* Relative to the weight of the cement or total volume

2.6 Graphene-Based, Self-Sensing Cementitious Composites

Graphene or graphene nanoplatelets (GNPs) are monolayers of graphite. It is a carbon allotrope consisting of carbon atoms that are tightly bound and arranged in a hexagonal two-dimensional (2D) lattice. It is sp^2 hybridised, has an extremely thin nuclear thickness (0.345 nm), and has a honeycomb structure that features atomic carbon-carbon bonds with a length of 0.142 nm. Single-layer graphene has a tensile resistance of 130 GPa, Young's modulus of 1.0 TPa, and a loading capacity ten times higher than that of steel. Moreover, it is 1,000,000 times more conductive than copper. These remarkable features of graphene have led to it being called "super carbon" [107, 190, 191].

The incorporation of GNPs into cementitious matrices significantly improves the conductivity of the composite and increases its stress/strain sensitivity more than the incorporation of CB, NCB, CNF, and CF [192-195]. Furthermore, the incorporation of GNPs into cementitious matrices enhances the physical and mechanical properties, and durability of the composite, as well as its thermal stability, thermal

diffusivity, and heat resistance [196-200]. Lv et al. [201] showed how graphene regulates the nucleation and growth of the cement hydration products over time to achieve remarkable compressive strength (38.9%), tensile strength (78.6%), and flexural strength (60.7%) in cementitious mortar.

The inclusion of graphene as a conductive filler in a cementitious matrix not only imparts sensing ability to the composite but also enables the quantification of the degree of damage to the matrix. A correlation between fractional change in electrical resistance and degree of damage has been expressed by Le et al. [202] based on a mathematical analogy between a 2D static field and a 2D static field subjected to an antiplane shear load. The unique electrical conductivity of graphene makes it a suitable filler for electrostatic dissipative (ESD) applications. The vulnerability of a graphene-based cementitious self-sensing composite to interference is limited. By increasing the graphene concentration beyond the percolation threshold, factors, such as the water-to-cement ratio, moisture level, and hydration period, negligibly influences the composite sensing efficiency [203], which makes graphene an ideal conductive filler for geomaterials. However, the complexity of graphene production and the relatively high cost of this conductive filler limit its use. The high specific surface area of dispersed graphene facilitates the absorption of water during composite fabrication which thickens the consistency of the cementitious composite or even halts the hydration process of the cement, especially in cases with low water content [204-206]. Hence, it is advisable to combine graphene with other effective carbon conductive fillers to decrease its concentration and production costs without reducing the sensitivity of the composite or sacrificing the desirable properties of graphene [207]. The combination of 2D graphene with conductive fillers with different geometrical properties can also facilitate filler dispersion [145].

2.7 Influential Factors

The process of self-sensing composite development, including composite design (in terms of sensing performance, physical and mechanical properties, and durability), composite preparation, and electrical resistance measurements, is informed by various factors which depend on the type and application of the composite. In this section, some influential factors and their mechanism are discussed by geotechnical approaches in transportation infrastructure applications.

2.7.1. Conductive Filler Dispersion

Several factors influence conductive filler dispersion, including 1) the geometrical and morphological properties of the conductive filler (such as length, softness, roughness, and mixing content), 2) viscosity of the suspension or medium, and 3) surface features of the conductive filler [208]. Available dispersion methods are categorised as 1) physical approaches (such as ultrasonication, shear mixing, ball milling,

and raw material mixing), and 2) chemical approaches (such as covalent and non-covalent functionalisation, calendring and irradiation, and plasma). The advantages and limitations of the different methods of conductive filler dispersion are listed in Table 2-4. An ideal dispersion method exhibits minimal side effects and does not adversely affect the intrinsic properties of the conductive filler and/or composite, especially filler morphology because of its vital role in the sensing ability of the composite [209-211]. Among chemical dispersion methods, non-covalent functionalisation impairs the inherent electrical, optical, and mechanical properties of fillers and cementitious composites the least. Conductive fillers, especially CNMs, are commonly dispersed by pairing this technique with different surfactants, aromatic small molecules, polymers, the endohedral method, or bio-macromolecules [145, 212-215]. A feasible, affordable, and compatible technique for conductive filler dispersion in cementitious composites typically combines a chemical treatment, such as surfactant addition, and a physical method, such as ultrasonication, to disperse the filler in an aqueous suspension. Surfactants usually have a polar molecular structure, the hydrophobic chain end attaches to the filler and the hydrophilic chain end bonds with water molecules. Surfactants are either anionic, cationic, or non-ionic, depending on their hydrophobic chain ends and their bonding mechanism with conductive fillers. In the case of non-ionic surfactants, π - π interactions are responsible for the adsorption of the surfactant molecule on the filler surface, especially in CNMs [145]. However, in dispersion processes featuring surfactants, the temperature and concentration of the surfactant are of particular importance [145, 216]. Ultrasonication is one of the most commonly used physical dispersion methods. In ultrasonication, microbubbles are produced in the suspension by a cavitation effect that vibrate violently and release large amounts of energy. During this process, bundles of conductive fillers are exfoliated or unbundled, and the filler surface roughness increases to a certain extent. However, the applied acoustic energy should be reasonably controlled to prevent damaging the structural quality of the filler, such as the formation of edge-type defects, or reducing the aspect ratio and sp^2 domain crystallinity (L_a), which are detrimental to the electrical and mechanical performance of the filler, especially in the case of CNMs [217, 218].

Acid treatment is a chemical technique that can be used to modify the surfaces of conductive fillers. In this process, a conductive filler is soaked in acid for a relatively short time during which functional groups form on the surface of the filler to increase its wettability [219, 220]. However, this can adversely affect the conductivity and strength of the filler owing to the difficulty of regulating the degree of functionalisation [107].

Table 2-4. Advantages and limitations of different conductive-filler dispersion methods

Categories	Dispersion technique	Advantages	Limitations
Chemical dispersion	Admixture	Simple and affordable, compatible with cementitious composites in low concentration cases.	Less content cannot meet the requirement of dispersion, but more dosage can affect the filler and/or composite features.
	Surfactant		
	Conductive filler functionalisation	Relatively simple and affordable, short reaction time.	Damages filler morphology owing to difficulties in controlling degree of functionalisation.
Physical Method	Ultrasonication	Simple and affordable.	Poor dispersion, unstable over time.
Other	Optimising mixing sequence Combination of particles with different sizes.	Simple operation.	Poor dispersion, affected by the size of the particle.

2.7.2. Electrode Configuration and Resistance Measurement

The configuration and status of the electrodes are one of the influential factors on the resistance measurement accuracy in self-sensing composites. As shown in Figure 2-9, measurement methods employ four- or two-electrode configurations. In the four-electrode method, the current is typically applied to the outer electrodes, while the two inner electrodes are used to measure resistance or voltage. The materials used in the production of an electrode should exhibit several essential characteristics, including high and stable electrical conductivity. Electrodes, typically plate (such as tape, foil, flakes), grid, or rod-shaped and consisting of copper, steel, lead, brass alloy, or graphite, are either embedded into the composite or attached to its surface [221-225]. However, the electrical resistance measured using electrodes embedded into a matrix is significantly lower than the electrical resistance measured using electrodes attached to the surface of the composite owing to the greater contact of the embedded electrodes with the conductive paths in the composite [107, 226].

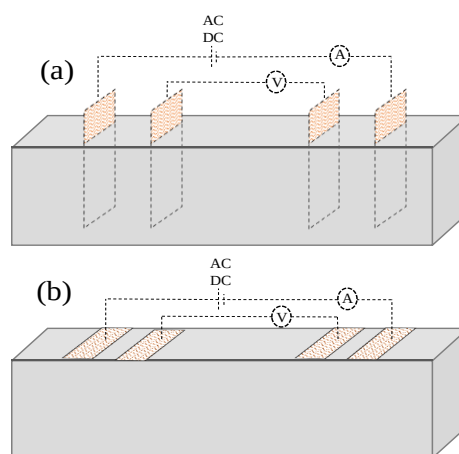


Figure 2-9. Different electrode configurations for resistance / voltage measurements in cementitious composites.

A non-contact electrical resistance measurement technique has also been developed (Figure 2-10) to avoid electrode arrangement difficulties. This measurement method is based on the transformer principle,

obviating the need for electrodes, and achieves superior accuracy [227, 228]. The type of current is also an important measurement factor that significantly affects the accuracy of sensing.

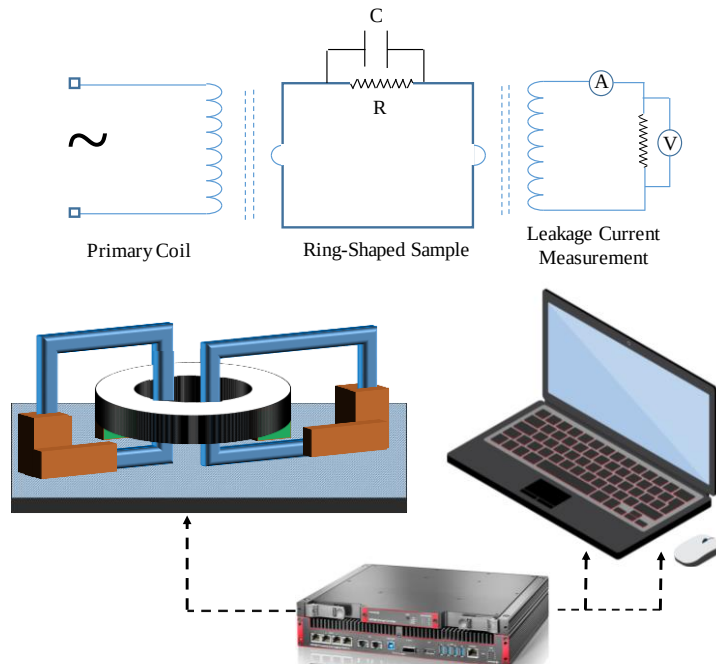


Figure 2-10. Setup of a non-contact electrical resistance measurement device and its equivalent circuit.

The application of a direct current (DC) usually increases polarisation effects and consequently increases the electrical resistance owing to the ionic conductivity of the cementitious self-sensing composite [133, 229, 230] while an AC can significantly eliminate polarisation effects during resistance measurement. The ability to adjust impedance by changing the current frequency is another advantage of using an AC during measurement [231]. Indeed, the microstructural changes of cementitious composites, such as carbonation, ion migration, steel corrosion, and cement hydration, can be analysed by regulating the AC frequency [232, 233].

2.7.3. Temperature

In a self-sensing cementitious composite, the influence of temperature variations on its piezoresistive behaviour can be described in terms of a resistance–temperature mechanism, Seebeck effect, and electro-thermal mechanism. However, resistance changes associated with the electro-thermal mechanism are negligible and, therefore, ignored [107].

In terms of the resistance–temperature mechanism, increasing or decreasing the temperature in proportion to the coefficient of expansion and contraction of the composite causes microstructure contraction (due to the temperature reduction) or its expansion (due to increasing temperature), which changes the distance between the conductive filler particles distributed in the matrix. Temperature changes can also affect the electronic transitions and electron tunnelling mechanisms of the filler. In

general, each of these phenomena affect the electrical resistance of the composite, and may be interconnected [107]. Hence, the effects of temperature on the piezoresistive behaviour of self-sensing cementitious composites can vary and should be investigated based on parameters such as temperature range, rate of change, and types of filler [234].

When a composite is exposed to high temperature, the motion and vibration of electrons at the site increase. These excited electrons tend to migrate from these high temperature regions to lower temperature regions within the matrix. This phenomenon, which is called the Seebeck effect, causes an electromotive force that changes the electrical resistance of the composite [235, 236].

2.7.4. Water-to-Cement Ratio

The water-to-cement (w/c) ratio is another important factor that affects the physical, mechanical, and piezoresistive behaviour of cementitious composites. Increasing the water-to-cement ratio greatly improves the dispersion status of conductive fillers, however, an excessive increase significantly reduces the microstructure cohesion and mechanical performance of the composite, which weakens its piezoresistive behaviour [88, 237].

The selection of a (typically low) water-to-cement ratio to fabricate geotechnical transport infrastructures, depend on the fabrication method, and should be informed by the physical, mechanical, durability, and piezoresistive response requirements.

2.7.5. Sand-to-Cement Ratio

Evidence suggests that the sand-to-cement ratio does not significantly affect the percolation threshold of cementitious composites [82]. However, at a fixed conductive-filler concentration, the electrical resistance of a composite increases with increasing sand-to-cement ratio. Although an excessive increase in the sand-to-cement ratio reduces the electrical conductivity of the composite, an optimal ratio reduces the incidence of voids in the matrix and increases the physical performance of the composite which enhances its conductivity and piezoresistive behaviour.

2.7.6. Freeze–Thaw Cycles

Repeated freezing and thawing induce the cyclical deformation of a cementitious composite resulting in the formation of cracks in the composite microstructure due to stress differences. These cracks gradually expand under service loads with increasing number of freeze–thaw cycles, irreversibly impairing the electrical conductivity of the matrix and, hence, the piezoresistive performance and sensing ability of cementitious composite [82, 222, 238].

2.7.7. Dry–Wet Cycles

In cementitious composites, the electrical conductivity in dry conditions is ascribed to the movement of electrons, whereas in wet or saturated conditions, the electrical conductivity is ascribed to the movement of ions. Furthermore, the frequent change in water content during dry–wet cycles, impairs the physical properties and, consequently, the durability of the cementitious composite, destructively impacting the conductive paths formed by its filler and reducing its conductivity and sensitivity [239, 240].

2.8 Conduction Theories and Equivalent Circuit Models in Self-Sensing Cementitious Composites

2.8.1. Conduction Theories

Generally, the formation of conductive paths in a self-sensing composite is described based on conduction theories. Currently, the literature references four main conduction theories, namely percolation theory, quantum tunnelling theory, electron field emission theory, and effective medium theory. The characteristics of the different conduction theories are listed in Table 2-5.

According to percolation theory, when the concentration of a conductive filler exceeds a certain threshold, the conductive filler particles are in physical contact or within 1 nm of each other, which form an extensive conductive network within the associated matrix [107]. This network dramatically changes the electrical resistivity of the composite, and the minimum concentration of conductive filler required to form such a network is called the percolation threshold [241]. However, when the concentration of the conductive filler is lower than the percolation threshold and the conductive paths do not form, the electrical conductivity of the matrix is described by quantum tunnelling theory. According to quantum tunnelling theory, the electrical conductivity of the composite is not enabled by the physical connection of conductive fillers, but the transfer of electrons between adjacent filler particles in the matrix under an external electric field.

In some cases, an investigation of the voltage and current variation in a self-sensing composite shows that, contrary to Ohm's law, the voltage–current relationship is not constant. In these cases, electron field emission theory is used to describe the electrical conductivity of the composite. According to this theory, a relatively high applied current produces electron field emission between conductive fillers which increases the current resulting in a non-ohmic relationship between current and voltage [242].

The effective medium theory is another theory used to describe the electrical conductivity of a composite. This theory assumes that each composite particle is in an effective medium with identical conductivity. The composite conductivity can be assessed by evaluating the conductivity of various phases [243].

Generally, the conductive behaviour of cementitious composites is the result of various phenomena described by different theories, the dominant phenomenon depends on the conductive filler concentration [244]. Ion conduction, that is, Ca^{2+} , Na^+ , K^+ , OH^- , and SO_4^{2-} , also informs the electrical conductivity of cementitious composites, particularly in porous matrices [245]. External factors affect the sensing efficiency of the composite by suppressing electron and ion conduction. However, the existing theories of conduction only consider the directional electron movement of conductive fillers, not ionic conduction. The relationships between the different conduction theories and the role of ion conduction should be investigated to formulate a more general conduction theory that considers the effects of influencing factors in describing the conductive behaviour of cementitious matrices.

Table 2-5. Characteristics of different conduction theories

Theory	Principal equations	Definition	Filler concentration	Voltage	Scale	Specifications
Percolation theory	$C = C_p [1 - V_p + V_p W \left(\frac{C_m}{C_p} \right)]$	C_m is the conductivity of the composite, V_p is the conductive filler volume fraction, and C_p is the conductive filler conductivity.	High	-	Macro	Expression and analysis of the abrupt shift in electrical resistivity associated with high conductive filler concentrations.
Effective medium theory	$\frac{(1 - \varphi) (\sigma_L^{1/t} - \sigma_m^{1/t})}{\sigma_L^{1/t} + A \sigma_m^{1/t}} + \frac{\varphi (\sigma_h^{1/t} - \sigma_m^{1/t})}{\sigma_h^{1/t} + A \sigma_m^{1/t}} = 0$ $A = (1 - \varphi_c) / \varphi_c$	φ is the conductive filler volume fraction, φ_c is the conductive filler critical volume fraction, σ_L is the low conductive phase conductivity, σ_h is the high conductive phase conductivity, σ_m is the composite conductivity, and t is the exponent. J is the density of the tunnelling current, e is the electronic charge, Δs is the barrier limit at Fermi level, $\hbar$ is Planck's constant, $\bar{\varphi}$ is the height of the average barrier, V is the voltage on both sides, $j(\varepsilon)$ is the tunnelling current when the intensity of the electric field between conductive filler particles is ε and the equivalent conductivity is j_ε , W is the width of the gap between filler particles. J is the density of the current, E is the intensity of the field, A is the frequency of tunnel, B and n are characteristic constants of the composite.	-	-	-	Wide variety of applications
Quantum tunnelling theory	$J = J_0 \{ \bar{\varphi} \exp(-A \bar{\varphi}^{1/2}) - (\bar{\varphi} + eV) \exp[-A(\bar{\varphi} + eV)^{1/2}] \}$ $j(\varepsilon) = j_0 \exp \left[- \frac{\pi X W}{2 \left(\frac{ \varepsilon }{\varepsilon_0} - 1 \right)^2} \right]$ $J_0 = e / 2 \pi \hbar (\beta \Delta s)^2$ $A = (4 \pi \beta \Delta s / \hbar) (2m)^{1/2},$ $X = \left(\frac{4 \pi m V_0}{\hbar^2} \right)^{1/2}, \quad e_0 = \left(\frac{4 V_0}{e m} \right)$	J is the density of the tunnelling current, e is the electronic charge, Δs is the barrier limit at Fermi level, $\hbar$ is Planck's constant, $\bar{\varphi}$ is the height of the average barrier, V is the voltage on both sides, $j(\varepsilon)$ is the tunnelling current when the intensity of the electric field between conductive filler particles is ε and the equivalent conductivity is j_ε , W is the width of the gap between filler particles. J is the density of the current, E is the intensity of the field, A is the frequency of tunnel, B and n are characteristic constants of the composite.	Low	Low	Micro	Describes the conductive behaviour when the conductive fillers do not physically touch.
Electron field emission theory	$J = A E^n \exp \left(- \frac{B}{E} \right)$	J is the density of the current, E is the intensity of the field, A is the frequency of tunnel, B and n are characteristic constants of the composite.	Low	High	Micro	Describes a non-ohmic relationship between current and voltage.

2.8.2. Equivalent Circuits

The piezoresistive behaviour of a self-sensing cementitious composite not only enables the monitoring of stress and strain but also the detection of microstructural changes. By combining equivalent circuit modelling and AC impedance spectroscopy (ACIS), the electrical signal changes can be analysed to reveal

microstructural changes. However, because of the complexity of ACIS results, this approach is only suitable for compounds with equivalent circuit models [246, 247].

A complex electrochemical system is created within cementitious composites when subjected to an AC. The different components of the solid, liquid, and other phases of the composite and their electrical properties can be represented by different circuit elements such as capacitors, resistors, or inductors in series or parallel. However, it is difficult to simulate the electrical system of a cementitious matrix using an equivalent circuit model because of its complex microstructure [248].

In this review, equivalent circuit models are divided into three groups according to their basis of development and geotechnical application, namely a microstructure model, conductive path model, and conduction theory model, as shown in Table 2-6.

2.9 Applications

With the advancement of self-sensing composite technology and the development of efficient smart cementitious composites, applications taking advantage of these materials have increased. The flexibility of self-sensing composites in terms of design and versatility in terms of project-specific engineering requirements have significantly widened the range of potential applications, some of which are briefly mentioned below.

2.9.1. Structural Health Monitoring

As indicated in Figure 2-11, different configurations of the self-sensing composites, including bulk, coating, sandwich, bonded, and embedded forms, have been used for structural health monitoring. Bulk refers to the construction of entire structures or key parts with self-sensing composites, which not only satisfies structural engineering criteria but also enables efficient self-sensing [249]. In coating form, one surface of a component is covered by a layer of self-sensing composite, while in the sandwich arrangement, both the top and bottom surfaces of a component are covered by self-sensing composite layers. In bonded application forms, small, self-sensing composite sensors are attached to the surface of a component.

Another common self-sensing composite application is the embedding of small self-sensing composite sensors into a component. These sensors are typically as small as or slightly larger than conventional coarse aggregates [250, 251].

The coating, sandwich, bonded, and embedded application forms can achieve wider monitoring at lower construction costs than the bulk application form, since these forms allow the targeted placement of self-

sensing composites at key positions in the structural components. With these approaches, any risk to human safety and the deterioration of embedded steel caused by the electrical conductivity of self-sensing composites can be prevented [252]. The benefit of the bulk shape, however, is that it is simple to construct, unlike the other application forms, especially the coating and sandwich types [249, 253-258].

Some of the applications of self-sensing cementitious composites in the field of structural health monitoring, as mentioned in previous studies, are summarised in Table 2-7.

2.9.2. Traffic Monitoring

Traffic data, including vehicle speed, traffic density, traffic flow rates, can be extracted, and even dynamic weighing can be performed, by incorporating self-sensing composites in transportation infrastructure, such as pavements, rail tracks, or bridges. Such applications can be independent from or secondary to structural health monitoring (Table 2-8).

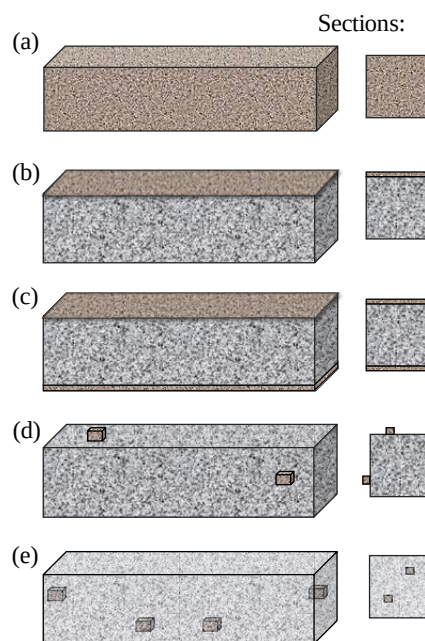


Figure 2-11. Typical application forms of self-sensing composites for structural health monitoring: a) bulk form, b) coating form, c) sandwich form, d) bonded form, and e) embedded form.

Variations in detection signals due to the polarisation of environmentally sensitive parameters are continuous and gradual, while vehicle load sensing signals are abrupt and transient. Hence, the former can be filtered out from the measured signals post-processing, and the accuracy of the monitoring will be unaffected. Accordingly, the monitoring performance of pavements or rail tracks integrated with self-sensing composites demonstrate appropriate resistance to polarisation caused by environmental factors and damage within the composite [259, 260].

2.10 Challenges

Despite many advances in the development of various self-sensing composites, few studies have explored self-sensing cementitious geocomposites which can play a crucial role in the long-term sustainability of transport infrastructures. Given the similarity between cementitious geomaterials and cementitious composites such as mortar, it is reasonable for the outcomes of studies conducted on cementitious composites to inform the development of self-sensing cementitious geocomposites.

However, there are still some challenges and unknowns regarding the feasibility of SSGs based on piezoresistive behaviour, such as:

- The sensing properties of cementitious composites arise from conductive paths which the present review shows are strongly influenced by matrix aggregate type and size. Diverse grain properties are one of the inherent characteristics of geomaterials.
- Owing to the low cement concentration used in cementitious geocomposites, a low conductive filler can be incorporated into the matrix. Hence, the type and geometrical properties of the conductive filler should be considered in such a way that a cementitious geocomposite with superior physical–mechanical properties, durability, and sensitivity is obtained.
- As discussed, the cement content of a cementitious composite determines the cohesion of the microstructure formed and, concomitantly, the stability of the conductive paths and associated piezoresistive behaviour of the composite. The efficiency of different types of conductive filler in low cement-concentration matrices needs to be investigated because of the low cement content of cementitious geocomposites.
- The water content, as one of the parameters determining mixture consistency, the physical and mechanical properties, and piezoresistive behaviour of cementitious geocomposites, requires extensive evaluation.
- In the case of carbon-based conductive fillers, a novel, effective, and affordable dispersion method is required that is compatible with the low water content used in the fabrication of cementitious geocomposites and addresses the hydrophobic nature of carbon fillers. Especially since the degree of dispersion of the conductive fillers plays a crucial role in determining the physical and mechanical properties, durability, and piezoresistive behaviour of the composite.
- Considering the effects of fabrication on the piezoresistive behaviour of cementitious composites, their application in geocomposite technology requires further investigation, taking into account the various techniques used in geotechnical engineering.

- The effects of external environmental factors such as climate cycles, carbon dioxide, temperature, chlorine ions, and humidity on the piezoresistive behaviour of cementitious composites are still unclear. Moreover, the durability and long-term monitoring ability of cementitious geocomposites are of vital importance and must be determined.
- Current conduction theories are unable to describe the conductive and piezoresistive behaviours of cementitious geocomposites and the applicability of equivalent circuit models is limited. It is, therefore, necessary to develop a universal conductive theory and an equivalent circuit model that describes the conductivity behaviour of cementitious geocomposites.
- The design flexibility of self-sensing cementitious composites, in terms of satisfying the requirements infrastructure projects, means that they cannot only be utilised in structural health monitoring of transport infrastructure (especially transition zones between structural layers), but also have the potential to detect structural damage in infrastructure such as roller-compacted concrete dams, rammed earth, and ground improvements. However, in-depth investigations on the application of self-sensing cementitious composites in the aforementioned fields are required. Future studies on the implementation of self-sensing cementitious composites should seek to develop a consistent method, guidelines, and specifications for the design and construction of self-sensing cementitious composite structures.

Table 2-6. Characteristics of different equivalent circuit models

Type of The Model	Impedance equation*	Equivalent circuit model	Definition	Specifications
Microstructure model	$Z_L = R_{S+L} + 1/(R_{INT} + j\omega C_{INT})$ $R_{S+L} = n(R_s + R_L)$ $R_1 = 2n(R_{int})$ $C_1 = C_{int}/2n$ $Z = ([R(RC)(RC)][(RC)(RC)])(RC)$		R_s is the solid-phase resistance, R_L is the liquid-phase resistance, R_{s+i} is the liquid-phase resistance plus the solid-phase resistance, C_i is the capacitance of the interface, R_i is the resistance of the interface, n is the number of units in the composite, C_m is the composite capacitance, R_m is the composite resistance, C_m' and R_m' are the capacitance and resistance increased by the conductive filler, R_m'' and C_m'' are the resistance and compensation capacitance, R_p is the conductive filler, R_c' and C_c' are the resistance and capacitance of the interface between the composite and conductive filler, R_c and C_c are the resistance and capacitance increased by polarisation effects	Used to investigate cement hydration and microstructural changes to inform structural health monitoring. Limited accuracy owing to the complexity of a cementitious matrix microstructure.
Conductive path model	$Z \approx 1/\{j\omega C_1 + \frac{1}{R_1} + \frac{1}{[R_2 + \frac{1}{j\omega C_2}]}\}$ $Z = \frac{1}{\frac{1}{R_1} + \frac{1}{R} + j\omega C_1}$		R_i is the conductive path, C_z and R_z are the partially conductive paths, and C_i is the non-conductive path-	Uses conductive paths to create an equivalent circuit model that ignores microstructural complexity. Considers microstructural conductivity changes on a macroscopic scale.
Conduction theory model	$R_{total} = f(R_L, R_c, R_t)$		R_f is the conductive filler electrical resistance, R_c is the contact resistance between the fillers, R_e is the electrical resistance affected by the tunnelling effect.	Considers both tunnelling and percolation theory. Is based on the conductive theory of CF cementitious composites.

* R is the resistance, Z is the total impedance, C is the capacitance, ω is the angular frequency, and j is imaginary.

Table 2-7. Summary of previous studies on cementitious self-sensing composites in different forms for structural health monitoring

Application forms	Conductive filler type	Component	Loading method	Detected parameters	Reference
Bulk	CSF	Concrete beam	Bending (Four point)	Load, Deflection	[261]
		Concrete ring	Parallel plate loading	Load, Diameter change	
	CSF	Concrete beam (Reinforced)	Bending (Four point)	Load, Damage	[257]
	CSF	Concrete beam	Bending (Four point)	Load	[262]
	CSF	Concrete beam	Bending (Four point)	Elastic compressive stress in the pure bending region	[263]
	PAF	Concrete pier of the bridge	Lateral	Strain, Deflection	[258]
	CSF	Concrete beam	Bending (Four point)	Fatigue, Damage	[264]
	CSF	Concrete beam	Bending (Three point)	Load, Deflection, Fracture	[265]
	CNF	Concrete column	Compression	Strain	[249]
	CSF	Concrete beam	Bending (Three point)	Compressive and tensile strain	[266]
Coating	CSF	Concrete beam	Bending (Four point)	Load, Strain	[107]
	CSF	Concrete beam	Bending (Four point)	Load, Deflection, Damage	[107]
Sandwich	CSF	Concrete beam	Bending (Four point)	Load, Deflection, Damage, Cracks	[267]
	CSF	Concrete beam	Bending (Three point)	Strain, Stress	[107, 255]
Bonded	CSF, CNF	Concrete beam (top, bottom, or side)	Bending (Four point)	Strain	[256]
		Concrete beam (embedded into the compressive zone)	Bending (Four point)	Strain, Stress	
	Hybrid CSF and CB	Concrete column (embedded in its centre)	Compression (Uniaxial)	Strain, Stress	[268]
Embedded	CSF	Concrete beam (embedded into the bottom)	Bending (Four point)	Strain, Stress, Crack	[269]
	CNT	Concrete beam (embedded into the bottom)	Bending (Three point)	Damage, Crack	[82]
	Hybrid CSF and GP	Concrete column (embedded into the centre)	Compression	Force	[270]

Table 2-8. Previous research on self-sensing composites for traffic monitoring applications.

Type of conductive filler	Self-sensing composite application style	Type of test	Goal of detection	References
CSF	Self-sensing concrete roller.	Response of roller to a rotating car tire tested in lab.	Traffic monitoring Dynamic weighing	[271]
CSF+CB	Self-sensing concrete strip component integrated into pavement.	Response of specimens tested using testing machine in lab.	Vehicle speed detection	[272]
CSF	Self-sensing concrete strip component integrated into pavement.	Response of specimens tested using testing machine in lab.	Dynamic weighing	[82]
CSF + CBP	Self-sensing concrete strip component integrated into pavement.	Response of specimens tested using testing machine in lab.	Vehicle speed Vehicle weight Traffic flow detection Vehicle type judgement	[271, 273, 274]
CNT	Self-sensing concrete strip component integrated into a pavement test section at the Minnesota Road Research Facility.	Road test performed at a road research facility with a five axle semi-trailer truck and a van.	Traffic flow monitoring	[121, 260]
NP	Self-sensing concrete arrays integrated into pavement.	Road test performed at outdoor lab with a car.	Passing vehicle detection	[275]

2.11 Concluding Remarks

Self-sensing cementitious composites are multi-phase materials composed of cement-stabilised geomaterials, conductive phases, and the interfaces between conductive fillers and the composite. The sensing capability and conductive properties of self-sensing composites are determined by factors such as conductive filler type, manufacturing technique, and material proportions. All these parameters affect the piezoresistive behaviour of cementitious geocomposites in different ways and to different degrees. Regrettably, the research on self-sensing cementitious geocomposites is limited. However, their similarity to other cementitious composites has prompted this review of the most influential parameters determining the electrical properties and piezoresistive behaviour of cementitious composites to inform the future development of self-sensing cementitious geocomposites. In order to apply SSGs in large-scale structures, the technology must be environmentally friendly, simple to implement, and economical.

According to the literature, carbon-based self-sensing composites have superior mechanical and microstructural properties, are durable, demonstrate good detection sensitivity and are appropriately compatible with cementitious matrices. Consequently, their application in the monitoring of transport infrastructures is promising. However, the combination of nano- and microscale carbon fillers is recommended in order to minimise the cost of production and prevent the brittle behaviour that is observed in composites only filled with CNMs. Combining different conductive carbon fillers, such as

CNTs and GNPs while reducing the percolation threshold without undermining the composite sensitivity, can also facilitate their dispersion. However, a feasible and compatible dispersion method is still required to properly disperse conductive fillers; non-covalent functionalisation in conjunction with an appropriate physical method may offer an affordable, compatible, and effective approach to carbon-based filler dispersion. Studies have shown that four copper electrodes embedded in a self-sensing composite under a DC and an AC enables the accurate measurement of the fractional change in electrical resistance. Since the water and cement contents of cementitious geocomposites and conventional cementitious composites differ significantly, the effects of these two parameters on the sensitivity of geocomposites incorporating different conductive fillers need to be accurately evaluated. In addition, the effects of external environmental factors on the piezoresistive behaviour of cementitious composites have to be extensively investigated considering their intended application in the long-term monitoring of transport infrastructure. Indeed, despite the promising performance of piezoresistive-based self-sensing composites for the structural health monitoring of infrastructure and inability of existing technology to realise an integrated monitoring system compatible with the mechanisms and features of geomaterials, no studies have investigated the application of these composites for the structural health monitoring of infrastructure. Hence, extensive studies that investigate the capabilities and potential of geomaterial monitoring based on smart, piezoresistive, self-sensing composites are imperative.

Chapter 3. Dispersion Method Development

«Porque eu sou do tamanho do que vejo E não do tamanho da minha altura»

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
Addressing the affecting factors	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

- Abedi, Mohammadmahdi, Raul Fanguero, and Antonio Gomes Correia. "An effective method for hybrid CNT/GNP dispersion and its effects on the mechanical, microstructural, thermal, and electrical properties of multifunctional cementitious composites." *Journal of Nanomaterials* 2020 (2020). <https://doi.org/10.1155/2020/6749150>

Abstract

This paper reports the study undertaken to achieve a compatible and affordable technique for high-quality dispersion of carbon nanotubes and graphene nanoplatelets in aqueous suspension to be used in multifunctional cementitious composites. In this research work, two noncovalent surfactants with different dispersion mechanisms (Pluronic F-127 (nonionic) and sodium dodecylbenzene sulphonate-SDBS (ionic)) have been used. The influences of various factors on the dispersion quality, such as surfactant concentration, sonication time, and temperature, have been evaluated by UV-Visible spectroscopy, optical microscopic image analysis, zeta potential, and particle size measurement. The effect of tributyl phosphate (TBP) used as an antifoam agent has been also evaluated. Besides, the optimum suspensions of each surfactant were used to produce cementitious composites, and their mechanical, microstructural, electrical, and thermal behaviors were assessed and analyzed. The best dispersed CNT+GNP aqueous suspensions using Pluronic and SDBS were obtained for concentrations of 10 and 5%, respectively, during 3 hours sonication, at 40°C, with TBP for both surfactants. Results also demonstrate that cementitious composites reinforced with CNT+GNP/Pluronic show better mechanical performance and microstructural characteristics due to the higher quality of the dispersion and the hydration rate increasing. Furthermore, composites prepared with SDBS suspension show lower electrical and thermal conductivities compared to those of Pluronic suspension due to changes in the intrinsic properties of CNTs and GNPs by the SDBS dispersion mechanism.

3.1 Introduction

Various types of multifunctional cementitious composites employing carbon fillers have attracted widespread attention due to their potential application including monitoring, thermal management, transportation, etc. [79, 116, 276, 277]. Among carbon fillers, carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) are the most widely used fillers to improve mechanical, microstructural, electrical and thermal behavior of composites due to their unique properties such as small size, low density, high stiffness, high strength, and excellent electronic and thermal conductivity [278]. Furthermore, CNTs and GNPs can act as a nucleation agent and rise the hydration rate in cementitious composites [208]. Graphene is a 2D monolayer of sp^2 -hybridized carbon atoms arranged in a hexagonal packed lattice structure and CNTs are 1D nanoscale tubes with an empty inner cavity that consists of single or multiple graphite atomic layers that are curved back on themselves [191, 278]. Besides, the high aspect ratio and large specific surface area of GNPs lead to a high contact area between composite and nanofiller and causing maximized transferred stress and phonon transference from the matrix to the nanofillers. Hence, GNP reinforced composites can be expected to exhibit better mechanical, thermal, and electrical behavior even in comparison to CNTs [279]. However, in many cases, the potential applications of GNPs and CNTs are limited due to their high aspect ratio and large specific surface which lead to large Van der Waals forces and strong π - π interactions originating easily entanglement and agglomeration [280]. This is particularly important in multifunctional cementitious composites which normally use high concentrations of the nanofillers to obtain higher electrical or thermal efficiency [190], causing agglomerate formation and reducing their mechanical and microstructural performance [281], since physical properties are of significant importance in multifunctional cementitious composite [282]. Recently several studies have been carried out to measure the CNT+GNP hybrid effects on multifunctional polymeric composites. The results showed an improvement in the mechanical, microstructural, electrical, and thermal performance in comparison to their individual usage. Hence, the synergic effects of CNT+GNP combination can decrease the required concentration of carbon nanomaterials (CNMs) by reducing the percolation threshold and subsequently alleviate concerns about the costs and formation of porosities significantly [279, 280, 283, 284]. Although no study has been found on the development of multifunctional cementitious composite by CNT+GNP combination, it is expected that similar assumptions can be assumed for this type of matrix. The combination of 1D (CNT) and 2D (GNP) nanoparticles with a high aspect ratio can increase the electron quantum tunneling effect as well as the percolation mechanism. Furthermore, CNT+GNP combinations can increase the hydration rate more than each would individually, due to the larger specific surface area which acts as a nucleation agent [201, 208]. They can also fill a

wider range of micro and nanopores which lead to denser microstructure and bridge the hydration products of cement. However, a compatible and effective technique is required for the dispersion of CNT+GNP combination in cementitious matrices, due to their agglomerate potential. Therefore, the present study proposes a compatible and affordable method for high-quality dispersion of CNT+GNP in multifunctional cementitious composites. Various chemical, biological and physical techniques such as high shear mixing, ultrasonication, ball milling, plasma, calendaring and irradiation methods, noncovalent and covalent functionalization, etc., have been extensively studied to modify and improve GNPs and CNTs dispersion in various matrices [209-211, 214, 285-290]. Although they often have an adverse effect on nanoparticles and/or composite features, preparing dispersed CNT and GNP suspensions through the non-covalent functionalization method does not alter the inherent optical, electrical, or mechanical properties of these nanoparticles. CNTs and GNPs are commonly dispersed along this route using different surfactants, aromatic small molecules, polymers, endohedral method, and bio-macromolecules [190, 209, 213, 214, 291-306]. Currently, Pluronic F-127 (PF-127) is receiving special attention among the different surfactants due to its lower toxicity and higher biocompatibility compared to other surfactants [307-311]. Pluronic F-127 (PF-127) is a non-ionic triblock copolymer consisting of a core hydrophobic chain of poly-oxypropylene (PPO) flanked by two poly-oxyethylene (PEO) hydrophilic chains. Since PF-127 has an amphiphilic structure, it has been asserted to be an efficient surfactant for the dispersion of CNTs and GNPs [216, 308]. Pluronic F-127 has great solubility in water at room temperature, allowing the preparation of CNT suspensions at a high surfactant concentration ratio. Besides, due to the long PEO chain presence, Pluronic F-127 was found to be compatible with cementitious composites and may improve its mechanical behavior and dry bulk density due to improved fluidity of mortar and dispersion of cement particles [144, 205]. Pluronic F-127 surfactant also exhibited a further ability to disperse MWCNTs in basic aqueous or extremely acidic media compared to ionic surfactants [294, 312]. MWCNTs were also effectively dispersed in ethoxy modified composite of Pluronic F-127 and a silicone surfactant (Trisiloxane). Sodium dodecylbenzene sulfonate (SDBS) is also a non-covalent surfactant that is commonly used to disperse carbon nanoparticles showing low interference with the performance of nanoparticles [144]. Effective dispersion of SWCNTs at high concentrations (up to 2 wt.%) has been obtained recently, using ionic surfactants SDBS through long-term ultrasonication treatment (24 hours) [313]. To disperse different types of CNTs and GNPs, efficiency of SDBS has been reported even more than dodecyl-/tetradecyl-/cetyl-trimethylammonium bromide (DTAB/TTAB/CTAB), gum Arabic (GA), nonylphenoxypoly(ethyleneoxy) ethanol (NP-10), cetylpyridinium chloride (CPyCl) and sodium dodecyl sulfate (SDS) [314-317]. Hence, the effect of Pluronic F-127 and SDBS on the stability of prepared suspensions by 1% of

CNT+GNP (0.5 wt% CNT and 0.5 wt% GNP), are evaluated in various concentrations, ultrasonication time, temperature and presence of tributyl phosphate (TBP, as antifoam agent) in order to propose a compatible and affordable method for high-quality dispersion of CNT+GNP in multifunctional cementitious composites. Furthermore, the effect of the optimum dispersed CNT+GNP suspensions on the microstructure and mechanical, thermal and electrical behaviors of the cementitious composites has been evaluated, in order to ensure the efficiency of the technique and the absence of adverse effects.

3.2 Materials and Methods

3.2.1. Raw materials

Carbon nanotubes and graphene nanoplatelets were purchased from CNPLUS Company (USA). Table 3-1 summarizes the characteristics of the GNPs and CNTs as provided by the manufacturer[318, 319]. Furthermore, the Raman spectroscopy (Figure 3-1) was carried out on CNTs and GNPs using a HORIBA LabRAM HR Evolution confocal Raman spectrometer through laser excitation with a wavelength of 532 nm.

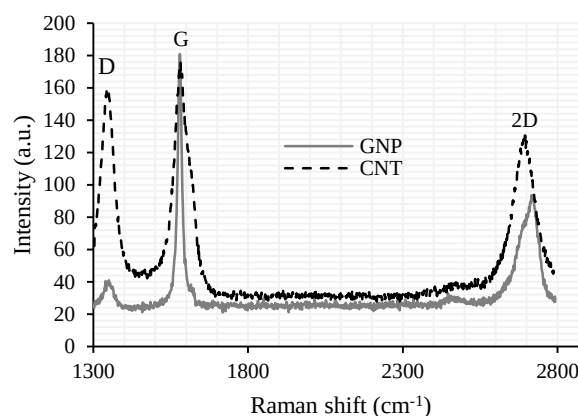


Figure 3-1. CNT and GNP Raman analysis

The Morphology of GNP and CNTs in different modes were characterized using Scanning Electron Microscope (SEM) (Figure 3-2). Pluronic F-127 and SDBS were used as surfactants in order to facilitate the CNTs and GNPs dispersion, while Tributyl phosphate (TBP) was also used as an antifoam agent with $\frac{1}{2}$ of the surfactant weight ratio. The surfactants and antifoam agent were purchased from Sigma Aldrich (Portugal) [320]. Their chemical structures are presented in Figure 3-3 [320, 321].

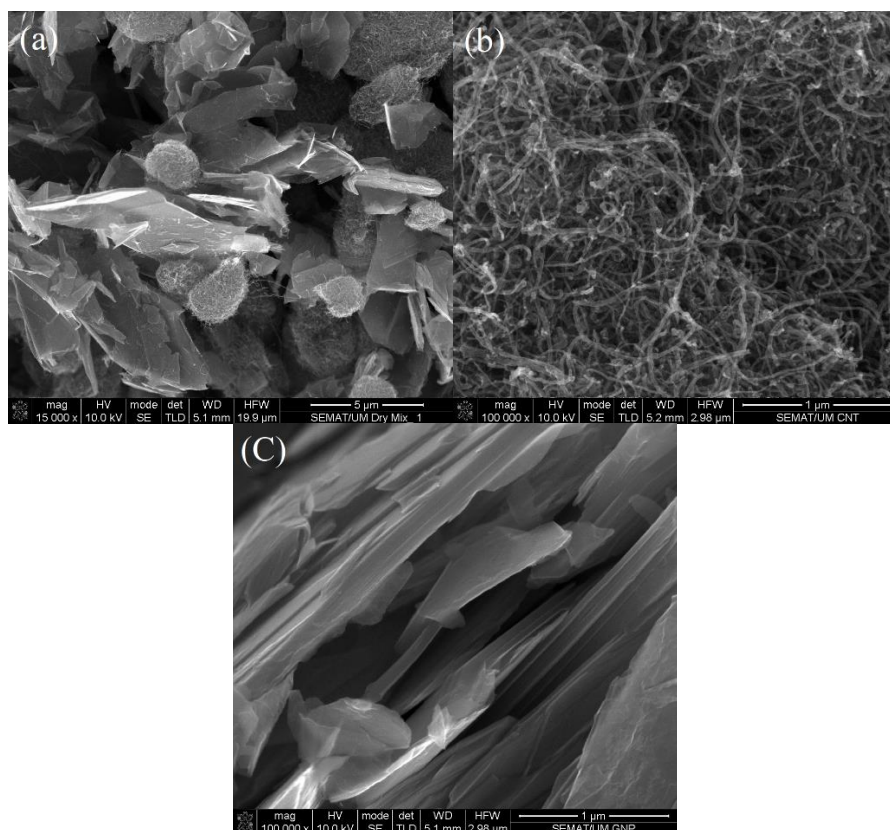


Figure 3-2: Morphology of (a) CNTs and GNPs dry mix (b) CNT (c) GNP

Table 3-1: GNPs and CNTs characteristics

GNP										
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Carbon content (%)	Tensile modulus (Gpa)	PH Value (30°C)	Tensile strength (GPa)	Layers	Dimension		Form	Part Number
120-150	0.6	>99.5	1000	7-7.65	5	<20	Thickness 4-20nm	Diameter 5-10μm	Gray Powder	TGN201
MWCNT										
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Color	Outside Diameter (nm)	Length (μm)	Ash (wt%)	Carbon content (%)	Part Number			
350	0.27	Black	<8	30-10	<1.5	>98	GCM327			

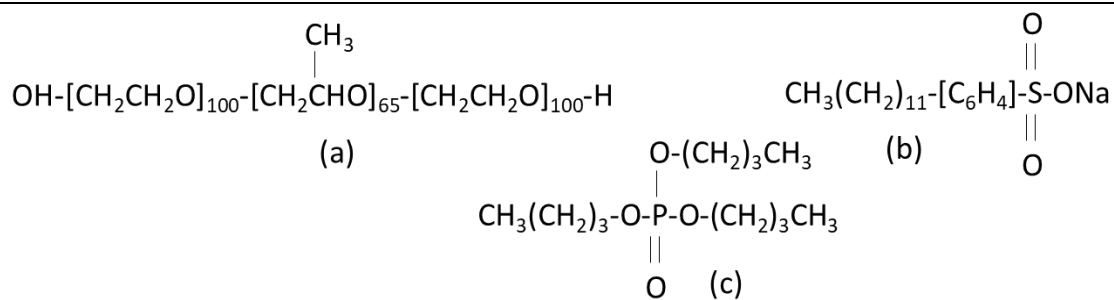


Figure 3-3: Chemical structure of : (a) Pluronic F-127 _ (b) Sodium dodecylbenzene sulfonate (SDBS)_ (c) Tributyl phosphate (TBP).

The ordinary Portland cement type I and CEN Standard sand (EN 196-1 and ISO 679: 2009) from SNL company were used to prepare cementitious composites (mortar). The particle size distribution of the sand and chemical composition of Ordinary Portland Cement (OPC) is shown in Table 3-2 and Table 3-3 [322].

Table 3-2: Particle size distribution of the sand [322]

Mesh Size (mm)	0.08	0.16	0.5	1	1.6	2
Cumulative retained(%)	99±1	87±5	67±5	33±5	7±5	0

3.2.2. CNT+GNP dispersion

1% of CNT+GNP (wt of water) was dispersed in aqueous solution by 5%, 10%, and 15% of each surfactant (wt of the carbon nanomaterials). These concentrations have been chosen based on the literature [144], and initial experiments. The surfactant was first added to water and mixed for 1 hour using a magnetic stirrer mixer with a speed of 800 rpm/min. CNT+GNP were then added to the solution and stirred for one additional hour. Samples were then placed in a sonicator bath (CREST Ultrasonicator, CP 230T) for 1, 2, and 3 h, at 45 kHz frequency and 80W power, at different temperatures.

A digital temperature regulator (NESLAB RTE-111 MP) was used to adjust the temperature during the ultrasonication process by the circulation system through a radiator and sensors. TBP was completely dissolved in water before the addition of the surfactant.

Table 3-3: Chemical composition and properties of Ordinary Portland Cement

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	TiO ₂	K ₂ O	MnO	P ₂ O ₅	SO ₃	LOI	Fineness, m ² /kg	Specific gravity
19,9	4,7	3,38	1,3	63,93	0,17	0,245	0,446	0,079	0,063	2,54	2,97	360	3,15

3.2.3. Cementitious composite fabrication

Plain and CNT+GNP reinforced specimens were prepared by mixing optimized CNT+GNP aqueous suspensions with ordinary Portland cement and standardized sand, using a laboratory mixer, following EN 196-1:1994 standard. In all samples, a cement-to-water ratio of 0.5 was used. At first, the required amount of cement (450 g) was poured in the mixer's stainless steel bowl in order to prepare the mortar mixtures. Then, the prepared CNT+GNP suspension with 125g of water was added to cement and the

required amount of sand (1350 g), and the remaining 100g of water was poured into the mixing machine's hopper. The mixer was then run for 1.5 min, with a stainless steel blade's rotational speed of 140 m/min, followed by a 30 s timeout and then run at a 285 m/min for another 2.5 min. The mixture was placed into 160 mm × 40 mm × 40 mm and 40 mm × 40 mm × 40 mm (for capillarity test) prismatic molds and placed on a jolting machine for 1 min for vibrating compaction. The molds were placed in a humid atmosphere (90%) for 24h and then the samples were de-molded and kept under the water for 28 days. Two copper meshes of 40 mm × 50 mm, were embedded in the cementitious composite as electrodes, at 5 cm from the middle of the specimen, to measure the electrical behavior.

3.2.4. Characterization of CNT+GNP Aqueous Suspensions

Different techniques were used for characterizing various parameters of the prepared CNT+GNP suspensions.

UV–visible spectroscopy

Due to additional absorption caused by 1D van Hove singularities, the dispersed carbon nanotubes and graphene nanoplatelets in aqueous suspension are active in the region of UV–Vis [292, 323, 324]. These singularities are specific points in the electronic band structure of these materials that result in sharp peaks in the density of states, leading to enhanced electronic transitions at certain wavelengths. Consequently, when UV-Vis spectroscopy is performed on such suspensions, the unique electronic structures of CNTs and GNPs cause additional absorption features, making these materials active and easily detectable in the UV-Vis spectrum.

Hence, the UV–Vis spectroscopy (Shimadzu, UV 2401 PC) was used to evaluate the dispersed CNT+GNPs concentrations in different aqueous suspensions. However, bundled carbon nanomaterials (CNMs) are extremely active in the wavelength region between 200 and 1200 nm owing to the quenching of photoluminescence, which results in a weak and wide signal [144]. A solution with the same concentration of the surfactant agent was used as the blank sample to eliminate the peaks provided by to surfactant.

Zeta potential and particle size measurement

Zeta potential and bundle size of carbon nanomaterials (CNM) were determined by the Zetasizer Malvern Nano ZS system in aqueous suspensions. Dynamic light scattering (DLS) technique has been used for measuring the bundle size of CNM in aqueous suspensions; however, this technique is more accurate for perfectly spherical particles [144, 325, 326]. The result of CNM particle size may highly be affected by

their position towards the laser source and the combined morphology due to their high aspect ratio. Therefore, the analysis of CNM particle size results was performed while considering these factors. Zeta potential (ζ) was determined by Eq. (1) and Smoluchowski approximation from electrophoretic mobility (μ).

$$\mu = \frac{\zeta \epsilon_m V}{4\pi\eta D} \quad \text{Eq. (1)}$$

Where η is the viscosity of the solution, V is the applied voltage, D is the electrode separation and, ϵ_m is the dielectric constant of the medium.

Optical microscopy and image analysis

In order to monitor the carbon nanotubes and graphene nanoplatelets agglomeration presence in the aqua suspensions and quantify the area of agglomeration, optical microscopy was used. For this purpose, a drop of the water suspension was deposited on a glass plate and covered with a coverslip. Visual inspection was performed at 10x, 20x, and 50x magnifications. For quantification analysis of the agglomeration area, 10x magnification was used. In this method, three drops were taken as samples from each aqueous suspension. Photos were accurately prepared using microscope software to completely cover the drop area. The samples' optical micrographs were investigated by ImageJ software. The total area of agglomeration was computed by the summation of the total agglomerates area measured for a sample, divided by the total analyzed image area for each drop. The approximate amount of agglomeration area was obtained from the average of three drops and expressed in percentage with standard deviation.

3.2.5. Characterization of the CNT+GNP cementitious composites

Flexural and compressive testing was carried out according to BS EN 196-1:1995 standard. Besides, apparent porosity, dry bulk density, and capillary water absorption of the samples were measured as stated by ASTM C20, BS EN 1015-10:1999, and BS EN 1015-18 standards. For evaluating the consistency of fresh cementitious composite the diameters of the paste were measured by using a flow table at two perpendicular directions according to EN 1015-3 standard. The fracture surface of specimens was characterized by Scanning Electron Microscopy (SEM-FEG, Nano SEM NOVA 200, FEI) using an accelerated voltage of 10 kV and secondary mode of the electron after coating with an Au-Pd thin film (30 nm), in a high-resolution sputter coater (Cressington 208HR), in order to investigate the microstructure. Thermogravimetric Analyzer (TGA, Perkin Elmer) in a nitrogen atmosphere (100 ml/min), at a heating rate of 10 °C/min up to 1000 °C and Energy Dispersive X-Ray Analysis (EDX) with 1.8 nm at 3 kV (Helix detector) of vacuum resolution were used. EDX tests were conducted on the cement

hydration products. The graphs were obtained by mean of at least 5 different points analysis in each specimen. The non-destructive ultrasonic test was performed for microstructural evaluation according to BS EN 12504-4 standard by Pundit Lab, using ultrasonic test device H-2984 through two probes along the longitudinal transverse axis. The electrical resistance of the specimens was measured by Agilent 34461A multimeter, after drying at 34° C, for 72 hours (To avoid possible effects caused by temperature). A portable thermal camera model FLIR 60BX, with the resolution of 320 × 240 pixels and thermal sensitivity <0.045° C with a temperature range from -20° C to + 650° C, was used to evaluate the thermal conductivity of the specimens. Photographs were taken after heating the samples at 70 °C, for 72 hours. In this route, surface calibration was done before infrared thermography by measuring emissivity and reflected temperature (ISO 18434-1:2008).

3.3 Results and Discussion

3.3.1. Aqueous Suspension Characterization

UV–Visible Spectroscopy

The UV-Vis absorption spectra diagrams for CNT+GNP suspensions prepared by Pluronic_F127 and SDBS surfactants are displayed in Figure 3-4 and Figure 3-5. The dispersion rate of CNT+GNP in liquid suspensions is directly related to the amount of energy absorbance, i.e., higher energy absorbance indicates a more homogeneous dispersion of CNT+GNP particles within the liquid. Molecules containing bonding and non-bonding electrons (n-electrons) can absorb energy in the form of UV to excite these electrons to higher anti-bonding molecular orbitals [327]. The more easily electrons are excited (i.e. lower energy gap), the longer wavelength of UV is absorbed. The maximum energy absorbance of SDBS and Pluronic suspension occurred at 275 and 260 nm respectively, which is probably due to the stronger polarity interaction between Pluronic and CNT+GNP particles, that can guarantee the long term stability of the liquid suspension.

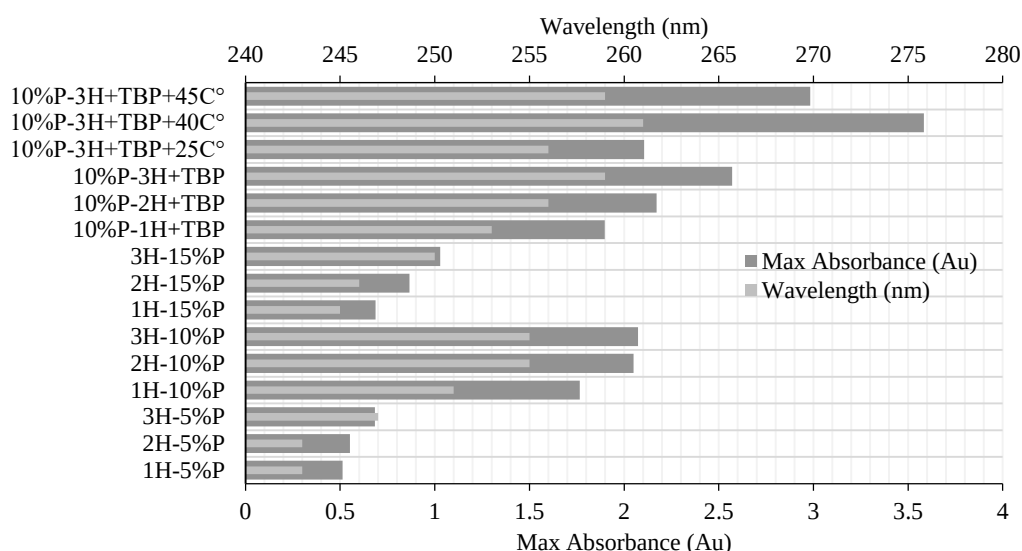


Figure 3-4: Wavelength and maximum absorbance of CNT+GNP suspensions prepared with Pluronic_F127

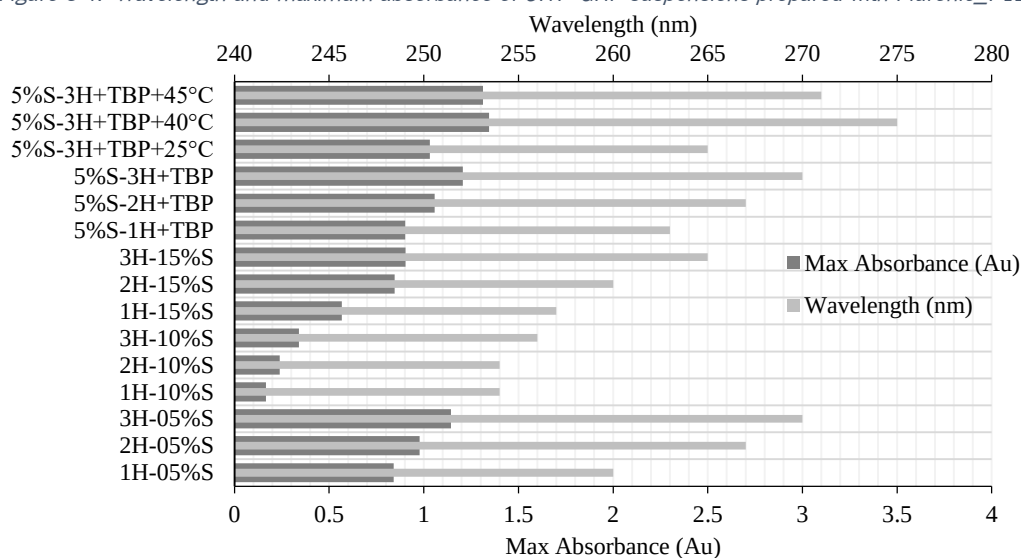


Figure 3-5: Wavelength and maximum absorbance of CNT+GNP suspensions prepared with SDBS

Shama et al. [144] also reported similar results for CNT/SDBS and CNT/Pluronic aqueous suspensions by long term storage stability evaluation. The results also indicate that the increase in the sonication time for both types of surfactants and all concentrations, improved the dispersion of the nanoparticles, but at different efficiency levels. The amount of energy absorbed (reported by spectrophotometer) in the SDBS aqueous suspension has increased by an average of 17% for each hour of sonication time increase; while for Pluronic, the average was 9%. The sonication time was not increased by more than 3 hours in order to prevent its adverse effects on CNMs structural quality such as edge-type defect, reduction of aspect ratio, and sp² domain crystallinity (La), that cause deleterious influence on their mechanical, electrical, thermal properties. The Raman spectroscopy was carried out on CNTs and GNPs which showed negligible structural damage after 3h sonication. Even though, the optimum sonication energy for CNMs dispersion can be higher than the 80W power for 3hours used in this study [217, 218]. Bath sonication dispersing

techniques exfoliates GNPs or de-bundles nanotubes into individual plates or tubes and/or thinner bundles, respectively, which consequently are stabilized through electrostatic repulsions and/or steric force of surfactants. During sonication, mechanical vibrations overcome the van der Waals interactions between the GNPs or bundles of CNTs and provide intense localized shear forces, particularly at the ends of GNP sheets or nanotube bundles. Once gaps or spaces at the bundle ends are formed, molecules of surfactant adsorbed onto the surface of the nanoparticles instantly and leading to exfoliation of them. According to the unzipping mechanism [328], surfactant molecules have to place among the small spaces into the bundle for exfoliating tubes and sheets and to prevent them from re-agglomeration. Hence, short-time ultrasonication in the presence of surfactant molecules with bulky hydrophobic groups (Pluronic) shows lower de-bundling efficiency due to hindered penetration into the inter-tube or inter-sheet regions. while, in the case of nonionic surfactants, these bulky hydrophilic groups probably due to the enhanced steric stabilization caused by longer polymeric groups, have advantages for CNT and/or GNPs dispersions. Results also demonstrate that an increase in temperature to a certain range (40°C) improves the dispersion of nanoparticles for both suspensions prepared by Pluronic and SDBS, probably by increasing the solvability of the surfactant molecules. However, excessive temperature rising (more than 45° C) causes the polarity bond breakage as well as the change in critical micelle concentration and lead to temporary re-agglomeration. Temperature fluctuations influence the critical micelle concentration (CMC) of surfactants, impacting nanoparticle dispersion stability. Higher temperatures typically lower the CMC, prompting surfactant molecules to form micelles more readily. Below the CMC, surfactants exist as monomers, less effective at preventing nanoparticle re-agglomeration due to weaker surface coverage. This temporary instability arises as reduced surfactant concentrations fail to sufficiently inhibit attractive forces between nanoparticles, like van der Waals interactions. Thus, temperature management is crucial to sustain stable nanoparticle dispersion, mitigating risks of re-agglomeration during fluctuating thermal conditions. In previous studies conducted by Yongbin et al. [216], a stable and homogeneous aqueous suspension of 0.5% graphene was obtained at low temperature (25 °C) using bath sonication with Pluronic F-127. Yan et al. [329] also found that a proper dispersion of graphene (0.56 mg · mL⁻¹) can even be achieved at 165 °C in a mixture of N, N-dimethylacetamide and water, via microwave. Results also indicated that the dispersion of CNT+GNP particles is strongly dependent on the surfactant concentration, and the best dispersion occurred with 10% of Pluronic and 5% of SDBS. It should be noted that SDBS is an ionic and Pluronic is a nonionic surfactant and their interaction mechanism with nano-carbon particles is different. In previous studies, Shama et al [144] observed that the use of 1% Pluronic for 0.1% of CNT concentration, 5% for 0.2% and 0.3% CNT and 0.8% SDBS for 0.2% CNT resulted in highly homogeneous

CNT dispersion. Yongbin et al [216] also achieved a competent aqueous suspension with 1% of Pluronic concentration for 0.5% graphene. By comparing these results with the present studies, it seems that the amount of required surfactant for dispersing CNT and GNP particles in the hybrid combination is much greater than the individual cases, in both types of ionic and non-ionic surfactants. Probably this is due to the combination of carbon nanoparticles with different geometries (1D and 2D), which may lead to an increase of the aspect ratio, as well as the specific surface area. The effects of CNMs aspect ratio on their dispersion quality were also shown by Goodman et al [330] through investigation of prepared aqueous suspensions by 1 wt% CNTs with a similar length and different diameter, dispersed by Alkali Lignin (AL), SDS and CTAB. They observed that the amount of absorbance in the UV-vis was decreased more in AL suspension when the CNT's diameters were decreased from 50 to 8 (nm). These observations reveal that the aspect ratio has a more prominent effect on the CNT suspensions when AL is used as a surfactant. This reflects the differences in how these molecules adsorb onto the surface of CNMs. In non-ionic surfactants, such as AL and Pluronic, in contrast to ionic surfactants, such as SDS, ASBS, and CTAB, π - π interactions are responsible for the surfactant molecules adsorption on CNMs surface and lower aspect ratio of CNMs exhibit lower surface curvature, which leads to stronger affinity to -COOH and -OH groups due to higher overlap between π electrons. As a result, a higher amount of non-ionic surfactant molecules is required for CNMs individualization with a high aspect ratio due to their strong tendency to agglomerate via van der Waals forces. The surfactant molecules play a crucial role by adsorbing onto the CNMs surfaces, creating a protective layer that prevents re-agglomeration and facilitates dispersion in the solvent. This increased quantity of surfactant ensures sufficient coverage of the nanomaterials' extensive surface area, thereby enhancing their stability and maintaining their unique structural properties essential for various technological applications. The interaction type and the nature of the surfactant and consequently its concentration is known to play a crucial role in the colloids phase behavior [331] as well as nanoparticle [332]. Stabilization mechanism of colloidal type particle is described in the theory Derjaguin – Landau – Verwey – Overbeek and typically relies on the surface load presence that can be induced by surface group deprotonation or through ion adsorption from the solvent to the colloidal particle surface. A diffuse layer of counter-ions has been attracted by the surface charge from the solvent and forming an electric double layer which has a diffuse nature due to Brownian motion. This can lead to an effective surface charge because of Coulomb repulsion among charged colloidal particles [281]. It can be applied to GNPs and CNTs by introducing a removable and temporary surface load through permitting the molecules of surfactant to adsorb via their hydrophobic tails on the nanoparticle surface. An ion normally dissociates itself from the head of hydrophilic groups and serves as a counter-ion. Then adsorbed

molecular ions have interaction with water [333]. The active charge signs and magnitude, through the zeta potential, is correlated with the double surface. The electrostatic potential of the layer edge is influenced by the bound tail groups [281]. For dispersing carbon nanotubes, the concentration of the surfactant has to generally exceed the critical concentration of micelles [334, 335]. Surfactants below the CMC exist as monomers and may not provide sufficient coverage to prevent nanotube agglomeration. By surpassing the CMC, surfactant molecules form micelles, which can efficiently coat the nanotubes' surfaces, reducing inter-nanotube interactions and enhancing their dispersion in the solvent. This ensures that carbon nanotubes remain individually dispersed and stable, crucial for applications in materials science, electronics, and biotechnology where uniform distribution is essential. Also, the concentration of the surfactant needs to be higher than the concentration of nanotubes. [281]. This ensures sufficient coverage of surfactant molecules on the nanotube surfaces, preventing them from sticking together due to van der Waals forces and maintaining their individualized state in the solvent. Therefore, a higher surfactant concentration relative to CNTs is crucial for achieving and maintaining stable dispersion, which is essential for various applications in materials science and nanotechnology. Regarding graphene dispersing, the latter condition is not always vindicating because some surfactants have better performance at concentrations below critical micelles and not exceeding the concentration of graphene [335]. Eventhough, the critical micelles concentration in the case of SDBS surfactant may also depend on the nanoparticles concentration [336, 337].

The quality of the dispersion and the individualization/ exfoliation degree of GNPs and CNTs dispersed by Ionic surfactant are normally measured by zeta potential. Interestingly, as opposed to carbon nanotubes, this occurs for graphene nanoplatelets within two separate ionic surfactants groups: sulfides such as SDBS, and other ionic surfactants. Since the Zeta potential reflects the potential of electrostatic at the edge of bound ion layers, one can assume that maximizing the surface charge in this layer can lead to Zeta potential increasing[338]. Hence ionic surfactant SDBS with low-molecular weight can pack tightly on the GNPs and CNTs surface and disperse them with a low concentration. In Pluronic, as a non-ionic surfactant, the lack of Coulomb repulsion for preventing GNPs and CNTs agglomeration is compensated by steric effects. Therefore, the main factor determining the dispersibility of CNTs and GNPs in aqueous suspension is the presence of branched and long polar of (PEO) chains. This leads to efficiency enhancement by increasing the molecular weight of the surfactant [339]and it is occurred due to the surfactant's affinity of π -electronic to the benzene rings of the GNPs and CNTs which can remain somewhat even after water elimination in contrast to SDBS (Figure 3-6).

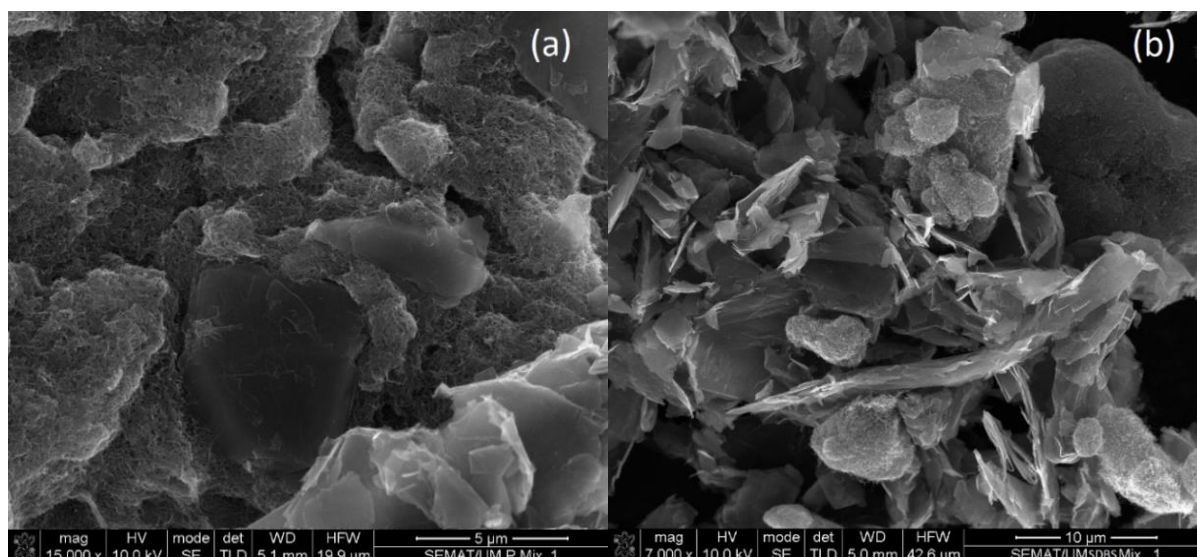


Figure 3-6: Morphology of dry CNT+GNP extracted agglomerate from aqueous suspension: (a) Prepared by Pluronic F-127_ (b) Prepared by SDBS

However, in dry mixes of CNT and GNP without the presence of surfactants, they are arranged in the form of individual spheres without interaction due to the van der Waals attraction forces between the CNTs and repulsion electrostatic forces between GNPs and CNTs (Figure 3-2). This repulsion arises from the delocalized electrons within graphene layers, preventing close interaction with CNTs. As a result, CNTs and GNPs typically remain as separate entities or form distinct clusters in the absence of surfactants. Surfactants play a crucial role in modifying these interactions by adsorbing onto the surfaces of both CNTs and GNPs, mitigating aggregation forces and potentially enabling more homogeneous mixes or composites.

Optical Micrograph Analysis

For evaluation of the agglomerates total area, CNT+GNP aqueous suspensions optical micrographs were analyzed and expressed in percentage (Table 3-4). Similarly, to the previous section, it can be observed that the dispersion quality was increased by increasing the surfactant concentration up to a certain level. Beyond this level, a further increase in concentration leads to a reduction in the dispersion quality and, consequently, to an increase on the agglomeration area.

Hence the optimum surfactant concentrations were 15% and 5% for Pluronic and SDBS respectively, which provided the lower agglomeration area. However, these concentrations are higher than their critical micelle concentrations [144]. Further increase of Pluronic and SDBS concentrations above these optimal values, not only represents a surfactants waste but also induces undesired effects. This is consistent with other studies reporting the CNTs or GNPs aqueous dispersion by various types of surfactants [292, 330,

340]. In the stable aqueous suspensions, a balance is reached between single CNMs and their bundles, which varies with the surfactant concentration.

At low concentrations, the surfactant adsorption is limited and does not efficiently counterbalance the van der Waals-induced aggregation of GNPs and CNTs. Although, when the surfactant amounts in the aqueous suspension are above a certain value, large micelles are formed around the bundles of CNMs and exert an osmotic pressure creating a depletion-induced attraction [341]. Furthermore, excessive increasing surfactant dosage at higher concentration levels than the critical micelle concentration can lead to nanoparticles re-agglomeration due to changing the micelles geometry and even changing the viscosity of the aqueous suspension system.

As mentioned before, in similar conditions for sonication, concentration, and temperature, the difference of dispersion efficiency for different surfactants is closely related to their interactions with CNMs which depends on the presence of benzene rings, the length of their alkyl chain, and the head group types. At the optimal concentrations, the individualized CNMs degree in aqueous suspension is comparable among the various dispersions, with a significant increase in the order of Pluronic > SDBS, as indicated by their respective agglomeration area.

Table 3-4: Agglomerate total area of various CNT+GNP aqueous suspensions

SDBS	Total area of agglomerates ±SD (%)	Pluronic-F127	Total area of agglomerates (%)
1%CG-1H-05%S	16.5 ± 0.95	1%CG-1H-5%P	29.6 ± 1.27
1%CG-2H-05%S	11.3 ± 0.61	1%CG-2H-5%P	22.4 ± 0.93
1%CG-3H-05%S	9.1 ± 0.48	1%CG-3H-5%P	18.3 ± 0.81
1%CG-1H-10%S	20.1 ± 1.08	1%CG-1H-10%P	12.3 ± 0.77
1%CG-2H-10%S	16.8 ± 1.1	1%CG-2H-10%P	8.1 ± 0.96
1%CG-3H-10%S	13.3 ± 0.72	1%CG-3H-10%P	3.5 ± 0.62
1%CG-1H-15%S	38.1 ± 1.36	1%CG-1H-15%P	17.4 ± 0.87
1%CG-2H-15%S	32.7 ± 1.09	1%CG-2H-15%P	13.8 ± 0.95
1%CG-3H-15%S	28.3 ± 1.22	1%CG-3H-15%P	10.6 ± 1.14
1%GC-05%S-1H+TBP	12.4 ± 0.84	1%CG-1H-10%P+TBP	10.1 ± 1.03
1%GC-05%S-2H+TBP	9 ± 0.73	1%CG-2H-10%P+TBP	6.3 ± 0.79
1%GC-05%S-3H+TBP	8.4 ± 0.66	1%CG-3H-10%P+TBP	2.9 ± 0.52
1%GC-05%S-3H+TBP+45°C	6.4 ± 0.71	1%CG-3H-10%P+TBP+45C°	2.1 ± 0.84
1%GC-05%S-3H+TBP+40°C	5.6 ± 0.5	1%CG-3H-10%P+TBP+40C°	1.2 ± 0.71
1%GC-05%S-3H+TBP+25°C	9.1 ± 0.94	1%CG-3H-10%P+TBP+25C°	5.7 ± 0.59
Aqueous Suspension without surfactant		CG: CNT+GNP(%), S: SDBS(%), P: Pluronic F-127 (%), H: Time of sonication (h), ±SD: Standard Deviation	
0,5%CG-1H	88.3 ± 2.37		
0,5%CG-2H	79.1 ± 1.61		
0,5%CG-3H	67.6 ± 1.89		

Interestingly, the presence of TBP enhanced the dispersion of nanoparticle, probably due to the TBP molecular structure that contains 3 methyl branches. Methyl group is a hydrophobic alkyl functional group derived from methane (CH₄) by removing one hydrogen atom (CH₃-), which is extremely reactive and capable of binding covalently with other carbon atoms due to its free capacity of eight radical electrons [342]. However, TBP was added to the suspension as an antifoam agent for removing bubbles which form on cementitious composite by Pluronic [144].

The optical micrographs analyzing, also shows the effect of temperature and the increasing sonication time on the nanoparticle dispersion improvement which was obtained from UV–Visible spectroscopy. Nevertheless, the aqueous suspension of 1% CNT+GNP with 10% Pluronic and 3hours sonication time,

at 40° C, with TBP, and the same suspension of 5% SDBS exhibited very low agglomerates area (only 5.6 and 1.2% respectively) indicating a proper dispersion quality (Figure 3-7). Hence, these suspension systems can be considered to be the optimized suspensions prepared using SDBS and Pluronic. However, compared to optimized Pluronic suspensions, the suspensions prepared using the optimized percentage of SDBS showed a higher agglomerate area.

Zeta Potential

As mentioned before, the stability of colloidal suspensions frequently is determined by their zeta potential which indicates the magnitude of electrostatic interaction between colloidal particles. Particles with a zeta potential higher than +15 mV or less than -15 mV are expected to be stable due to electrostatic considerations. However, colloidal suspensions with zeta potentials between +15 and -15 mV can also be stable if they are stabilized uninterruptedly [325].

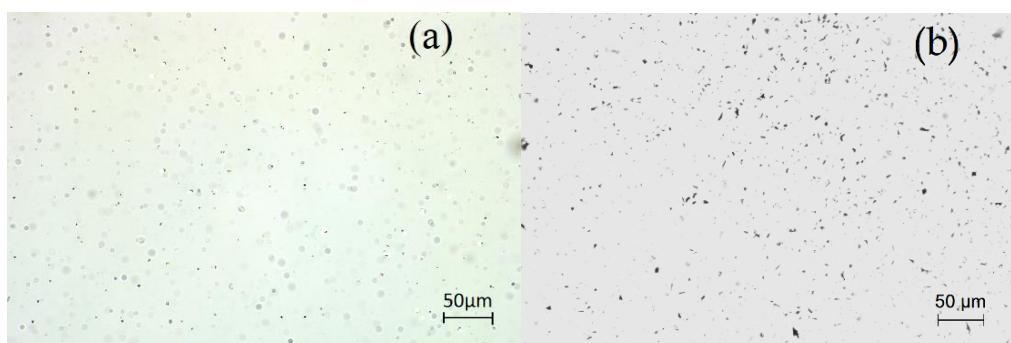


Figure 3-7: Optical micrographs of CNT/GNP suspensions, (a) Pluronic: 1%CG-3H-10%P+TBP+40°C, (b) SDBS: 1%CG-3H-5%S+TBP+40°C

The zeta potential of the CNT+GNP suspensions is listed in Table 3-5. It can be observed that the dispersion of 1% CNT+GNP in 5%, 10%, and 15% SDBS, for 3 hours sonication, showed a zeta potential of -48.6, -17.7, and -29 (mV), respectively. However, these amounts for Pluronic suspensions were around -2.9, -9.3, -5.7 (mV), respectively. The addition of 5% of TBP over SDBS, with 3 hours sonication, obtained -56.4 (mV) of zeta potential, which supports the positive effect of TBP to improve CNT and GNP dispersion. Besides, increasing temperature to 40°C leads to increasing zeta potential to -71.9 (mV) which shows more colloidal particle stability for this suspension. However, the excessive increase in temperature decreased the quality of the CNM dispersion. Similarly, increasing the temperature up to 40 °C, for 10% Pluronic prepared through 3 hours sonication with TBP, lead to the formation of more stable colloidal particles with -23.1 (mV) zeta potential. While TBP is known for its antifoaming properties in industrial applications, its direct impact on dispersion enhancement lies in its ability to adsorb onto the surfaces of nanomaterials, preventing agglomeration and promoting uniform distribution. The phosphate functional group in the TBP structure, can also push the zeta potential amount to negative values.

Table 3-5: Zeta Potential of CNT+GNP suspensions

Sample Name	Zeta Potential (mV)
3H-05%S	-48.6
3H-10%S	-17.7
3H-15%S	-29
5%S-3H+TBP	-56.4
5%S-3H+TBP+25°C	-35.5
5%S-3H+TBP+40°C	-71.9
5%S-3H+TBP+45°C	-67.3
3H-5%P	-2.9
3H-10%P	-9.3
3H-15%P	-5.7
3H-10%P+TBP	-14.6
3H-10%P+TBP+25C°	-13.1
3H-10%P+TBP+40C°	-23.1
3H-10%P+TBP+45C°	-19.9

It should be noted that the high negative amount of zeta potential for SDBS suspensions is due to the high negative surface charges of this surfactant. Consequently, due to the non-ionic chemical structure of Pluronic, CNT+GNP suspensions prepared by this surfactant presented lower negative zeta potential. Nevertheless, owing to non-electrostatic and steric interactions, Pluronic suspensions could also be stable. The poly-oxyethylene (PEO) hydrophilic groups extend into the water while hydrophobic chains of poly-oxypropylene (PPO) interact with the surface of GNPs and CNTs. The exfoliation and stabilization of separated GNPs and CNTs happen as a result of steric hindrance that was induced by the long PEO chain. Functionalized CNM zeta potential was relatively higher because of -COOH functional groups which lead to the surface negative charge on GNPs and CNTs.

Average CNT+GNP bundle size

The average size of CNM bundles and the polydispersity index (PDI) of the CNT+GNP suspensions, determined through DLS by measuring the hydrodynamic diameter, are presented in Table 3-6. PDI is used as a scale to indicate the extent of the particle size distribution range and the larger PDI, shows the wider particle size spectrum. Results indicate that the particle size of CNT+GNP suspensions dispersed

in 5%, 10%, and 15% Pluronic and SDBS, were around 512, 328 and 389 (nm) and 491, 671 and 588(nm) respectively, which shows an optimum concentration around 10% for Pluronic and 5% for SDBS, as discussed before. The small average size of CNM bundle size in Pluronic suspensions, in comparison with SDBS ones, indicate the efficiency of the -COOH functional groups on the unbundling process. Besides, lower particle size (219 nm) and lower PDI (0.315) were obtained for 10% of Pluronic, through 3 hours sonication with TBP; at 40 °C that shows low agglomeration and high quality of the dispersion. The average particle size and the amount of PDI in the SDBS dispersed suspensions, at optimum conditions, is around 393(nm) and 0.462, respectively.

Table 3-6: CNM hydrodynamic diameter in different suspensions characterized by DLS

Sample Name	Ave Bundle Size (nm)	PDI
3H-05%S	491	0.571
3H-10%S	671	0.707
3H-15%S	588	0.649
5%S-3H+TBP	448	0.514
5%S-3H+TBP+25°C	474	0.589
5%S-3H+TBP+40°C	393	0.462
5%S-3H+TBP+45°C	422	0.503
3H-5%P	512	0.576
3H-10%P	328	0.488
3H-15%P	389	0.51
3H-10%P+TBP	296	0.412
3H-10%P+TBP+25C°	311	0.438
3H-10%P+TBP+40C°	219	0.315
3H-10%P+TBP+45C°	287	0.397

3.3.2. Cementitious Composite Characterization

Mechanical Microstructural Behavior

After obtaining the best dispersed mixed design for hybrid CNT+GNP aqueous suspensions with each surfactant, mechanical and microstructural properties of the reinforced cementitious composites were evaluated to find out their effects. Mechanical and some microstructural parameters of the cementitious composites in different cases are showed in Figure 3-8 and Table 3-7. The results are obtained by the

mean of at least 3 specimens for each test performed. It can be seen that reinforcing cementitious composites by the CNT+GNP/Pluronic suspension leads to a higher bulk modulus compared to CNT+GNP/SDBS. Consequently, the time it takes for an ultrasonic wave to pass and apparent porosity of CNT+GNP/Pluronic reinforced specimens were lower than those of the CNT+GNP/SDBS ones.

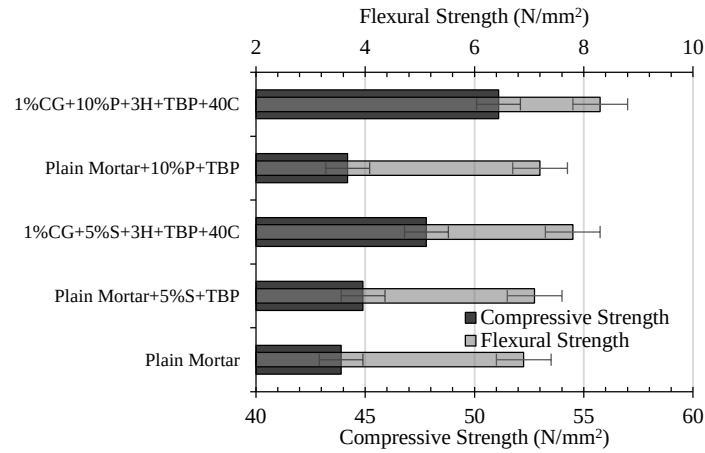


Figure 3-8: Compressive and Flexural strength of cementitious composites after 28 days of hydration period

Furthermore, the general trend of the results shows an improvement in flexural and compressive strength of CNT+GNP/Pluronic incorporated cementitious mortar. These results demonstrate more homogeneous and denser microstructure for the CNT+GNP/Pluronic reinforced cementitious composite. The positive effects of CNTs and GNPs on the improvement of the cementitious composites microstructure and their mechanical behavior, including flexural and compressive strength, have been reported in many previous studies [343-345]. The unique features of these 1D and 2D nanomaterials with high aspect ratio enhance the microstructural and the mechanical properties of the cementitious composites generally by reducing micro porosities, accelerating hydration process and bridging cement hydration products mechanism which is highly controlled by the CNMs aspect ratio and dispersion technique [208, 346, 347]. Therefore, it can be concluded that the difference between the cementitious composites mechanical and microstructure parameters in the case of CNT+GNP/Pluronic and CNT+GNP/SDBS is due to the dispersion quality and the secondary effects of the surfactant on the hydration process and/or on the nanoparticle features and/or structures. However, considering the mechanical and microstructural results for the non-reinforced composites prepared with just Pluronic and SDBS (without CNMs), the presence of these two surfactants have not adverse effects on the cementitious composite features, at least after 28days of hydration period and for these concentrations. Large agglomerations of CNTs and GNPs due to lower quality dispersion in CNT+GNP/SDBS reinforced cementitious composite lead to the formation of micro porosities. The (CNMs) agglomerates between the growing units of (C-S-H) create large gaps among them which prevent their physical connection (Figure 3-9 a). This is especially important for GNPs,

which are larger in size. As can be seen in Figure 3-10, although in this case, CNTs have shown higher reinforcement capacity in improving bridging behavior through pull-out strength due to their relatively better dispersion, the presence of large agglomerates of GNPs between hydration products has caused nanoscale discontinuities under stress, potentially facilitating the formation of microcracks. However, well-dispersed GNPs are more able to anchor the neighboring C-S-H clusters and bridge the voids between them. The large specific surface area of GNPs leads to a high contact area with hydration products and maximizes the stress transferring delaying the microcracks and macrocracks propagation. In contrast to SDBS, CNT+GNP/Pluronic reinforced cementitious composite contains lower porosities due to the better dispersion of the nanoparticles.

The results of capillary water absorption shown in Figure 3-11 also emphasize this. According to the slope of the capillarity results graphs, the rate of water absorption via capillarity in GNP+CNT/Pluronic reinforced specimens is significantly lower than that in GNP+CNT/SDBS specimens.

Table 3-7: Test results of cementitious composites according standard procedures

Name of sample	Flow values ¹ (mm)	Flexural Strength ² (N/mm ²)	Compressive Strength ² (N/mm ²)	Apparent Porosity ³ (%)	Ultrasonic wave time passing ⁴ (μs) for 150 kHz		Dry bulk density ⁵ (Kg/m ³)
					Longitudinal	Transverse	
Plain Mortar	183.2	6.9	43.9	19	36.40	9.70	2125
Plain Mortar+5%S+TBP	191.7	7.1	44.9	15.9	36.21	9.51	2130
1%CG+5%S+3H+TBP+40C	118.4	7.8	47.8	13.6	34.10	9.34	2135
Plain Mortar+10%P+TBP	198.2	7.2	44.2	13.9	35.88	9.38	2130
1%CG+10%P+3H+TBP+40C	131.5	8.3	51.1	11.4	32.97	8.93	2140

¹ BS EN 1015-3, ² BS EN 196-1:1995, ³ ASTM C20, ⁴ BS EN 12504-4, ⁵ BS EN 1015-10:1999

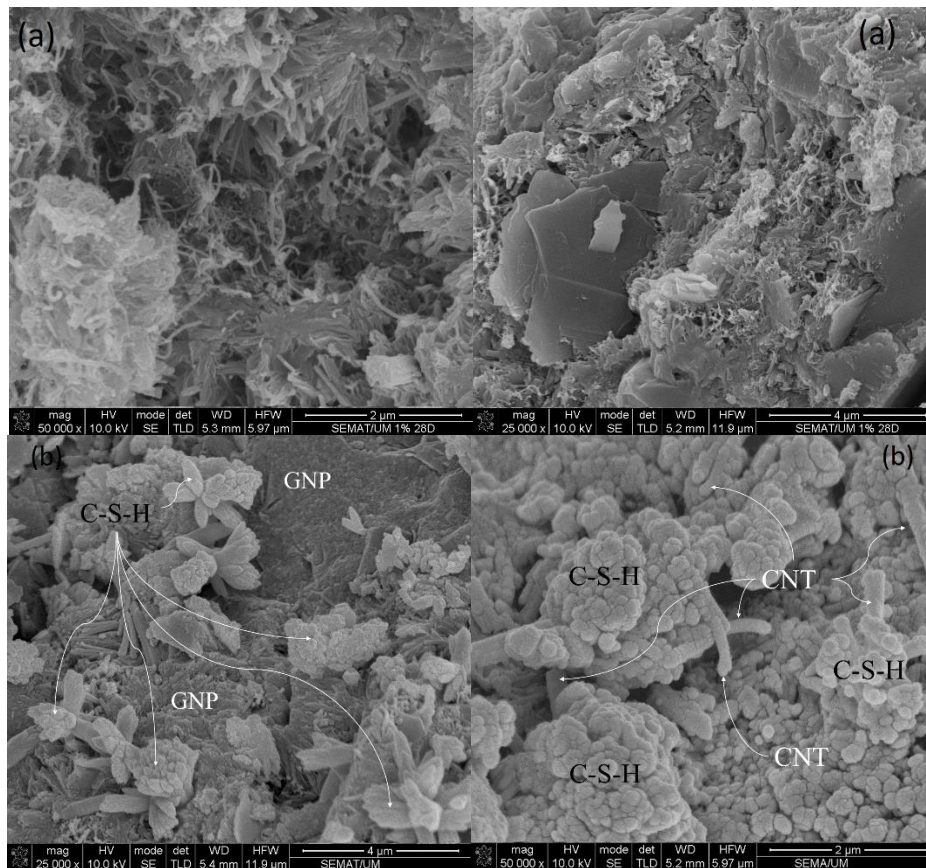


Figure 3-9: SEM morphology of reinforced cementitious composite by (a): CNT+GNP/SDBS_ (b): CNT+GNP/Pluronic

However, the rate of capillary water absorption for CNT+GNP reinforced composites prepared by both Pluronic and SDBS, is lower than that for the plain composite. It should be noted that the presence of both surfactants increased the flowability of the plain mortar. According to Table 3-7, the flow value of the specimen with 10% individual Pluronic, increased 9 and 5%, respectively, compared to the SDBS and the plain cases. The chemical structure of Pluronic F-127 contains hydrophilic PEO (polypropylene oxide) side chains similar to commonly used superplasticizers of polycarboxylate, as mentioned before. Polypropylene oxide side chains are reported to be the main chemical components of these superplasticizers responsible for the cement particles dispersion and the improvement of the flowability [348]. It is generally accepted that the cement paste fluidity is directly pertinent with the performance of hardened cement (such as pore structure and strength) [349]. However, CNT and GNP presence reduce the flowability of cementitious composite [144, 205]. Due to the large surface area of GNPs and CNTs, they can capture water molecules within the spaces between their layers; hence a significant reduction occurs in the free water content needed for lubrication, and lead to a reduction in fluidity. For this reason, the flowability of CNT+GNP reinforced samples prepared with Pluronic and SDBS, decreased by 27% and 36% compared to plain cementitious composite, respectively.

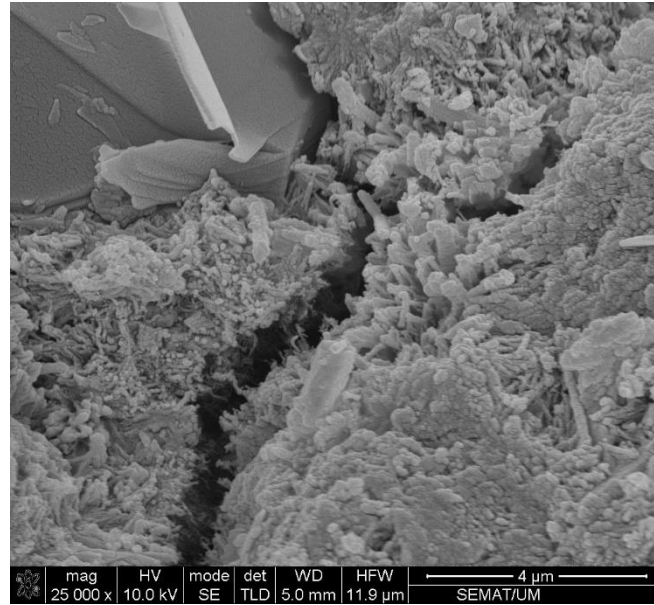


Figure 3-10: SEM morphology of CNT+GNP/SDBS sample after mechanical strength measurement

Generally, cement hydration products have a strong tendency to form on the surface of CNTs and GNPs due to the large specific surface area accompanied by a high amount of oxygen functional groups, which served as nucleation sites [201, 350]. Although C_3S and C_2S hydration produce C-S-H gel and C-H crystals, any deficiency in water content surrounding these silicates might slow down the hydration reactions [350]. This is especially true around agglomerates caused by the lower quality of dispersion (Figure 3-9a).

High magnification SEM images (Figure 3-9b) of the reinforced cementitious composite with CNT+GNP/Pluronic investigated by EDX tests indicate that CNTs and GNPs were embedded appropriately within the hydration products, in contrast to the CNT+GNP/SDBS reinforced specimen. This is due to the higher quality of the dispersion and the presence of the PEO side chains which get preferentially adsorbed on the surfaces of the C-S-H gel as well as along the CNTs length and GNPs surface [350].

In the early periods of hydration, this causes a slight discoloration of the graphene and increases the CNTs diameter due to the extremely thin layer of hydration products coating (in the form of pollen). Over time, the crystals become thicker and the carbons nanoparticles are completely embedded among them

The results of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) for different cementitious cases is shown in Figure 3-12. From the TGA curve, first decay is observed up to 105°C, which can be attributed to the removal of moisture and free water of cementitious composite samples. The second decay was observed between 105 and 400°C, due to the dehydration of chemically bonded water existing in hydrates i.e. ettringite, carbo-aluminates, and (C-S-H). The next major decay was found between 400 and 550°C, which can be attributed to dehydroxylation of CH, fourth, and the last decay ascribed to calcium carbonate coming from clinker and cementitious composite carbonation was

observed between 600 and 800°C [351, 352]. Results indicate that the amount of hydration products (measured by thermogravimetry analysis) in CNT+GNP/Pluronic reinforced specimen is more than CNT+GNP/SDBS specimen, however, hydration products amount in both cases is more than in the plain sample.

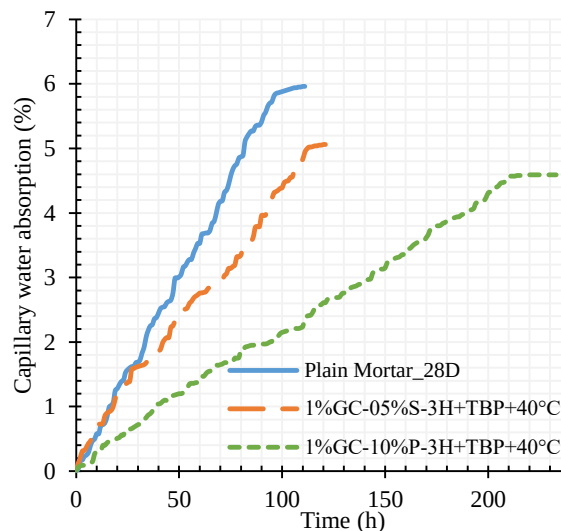


Figure 3-11: Capillary water absorption of cementitious composites

In the case of the reinforced cementitious composites with MWNTs, Konsta-Gdoutos et al. [353, 354] reported similar findings. They observed, by nano-indentation test, that multiwall carbon nanotubes could increase the formation of a larger amount of high stiffness calcium silicate hydrate gel and decrease nanoscale porosity owing to the gaps filling between gels of (C-S-H) through CNTs.

Therefore, based on the results of previous researches [347] and of the present study, one of the major effects of nanoparticles in the cementitious composite is increasing hydration rate. Energy-dispersive X-ray spectroscopy (EDX) of cementitious composites is shown in Figure 3-13. These results are obtained from the mean of 5 different points of cement hydration products for each case.

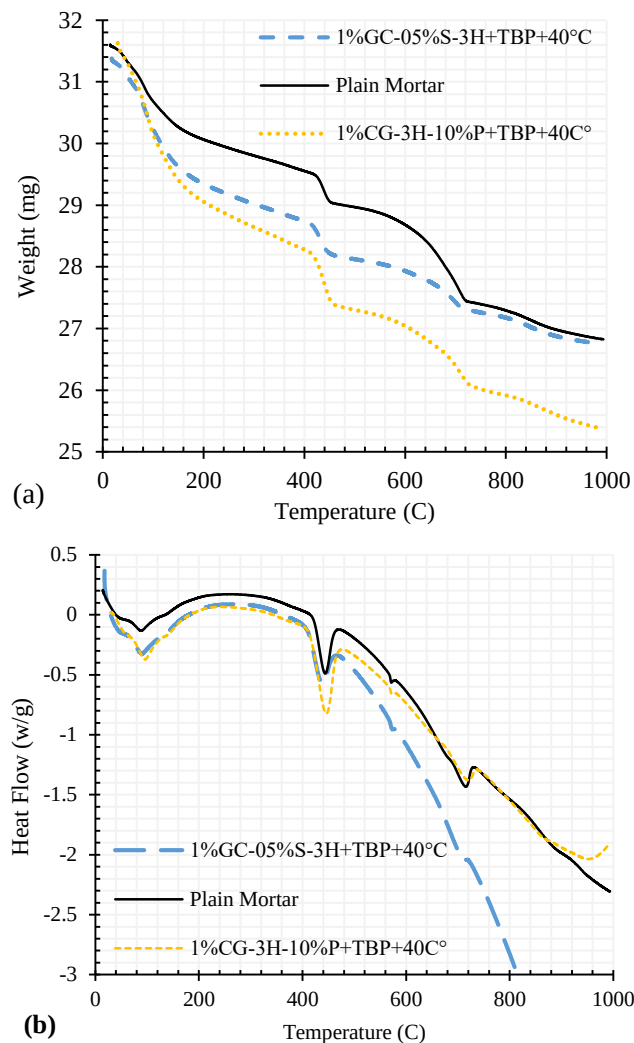


Figure 3-12: Thermal analysis of cementitious composite_ (a): TGA analysis - (b): DSC analysis

Despite higher carbon concentration in place of cement hydration products accumulation for CNT+GNP/SDBS reinforced specimen compared to CNT+GNP/Pluronic, a lower amount of hydration products is observed. The reason for this is probably the changing on nanoparticle features and/or structures due to the SDBS dispersion mechanism. This statement could be verified by evaluating electrical behavior.

Electrical Resistance

The electrical resistance of specimens after 28 days of hydration period was measured by a digital multimeter (Table 3-8). Since humidity, as an electrically conductive factor, has a relatively significant effect on the cementitious specimen electrical resistance changes [81, 82], the electrical resistance was measured after drying the specimens at 34° C for 72 hours. The electrical resistance of individual CNTs and GNPs reinforced specimens were also measured by the similar method in order to compare with hybrid CNT+GNP. The electrical resistance of the reinforced specimens with CNT+GNP/SDBS, CNT/SDBS, and GNP/SDBS was improved by 71.73, 66, and 53.2% respectively, in comparison to the

plain mortar, while these amounts were obtained by 60.94, 70.3, and 64.3% respectively, for Pluronic reinforced specimens' cases. According to the previous section discussion (3.3.2), CNT+GNP/SDBS reinforced specimen contained more and larger CNMs agglomerations compared to CNT+GNP/Pluronic due to lower dispersion quality. Although the existence of certain agglomeration quantity can act as a key factor in rising conductivity significantly and reducing percolation threshold value [281], the cementitious composite reinforced with CNT+GNP/Pluronic shows higher electrical conductivity.

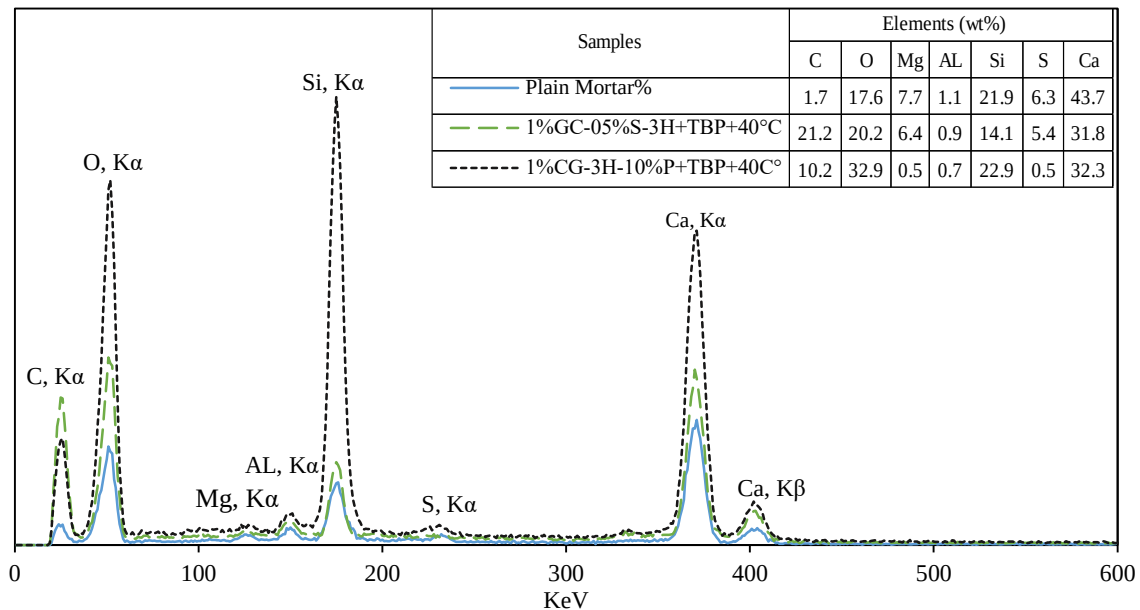


Figure 3-13: Energy-dispersive X-ray spectroscopy (EDX) of cementitious composites and their normalized elemental analysis

Table 3-8: Electrical resistance and humidity of cementitious composites specimens

Sample	Electrical Resistance (Ω)	Improvement (%)	Moisture (%)
Plain Mortar	8446.00	-	8.90
1%GC- 5%S-3H+TBP+40°C	2388.00	71.73	6.78
1%CNT- 5%S-3H+TBP+40°C	2847	66	7.7
1%GNP- 5%S-3H+TBP+40°C	3949	53.2	8.04
1%GC-10%P-3H+TBP+40°C	765.00	90.94	4.97
1%CNT- 10%P-3H+TBP+40°C	2506	70.3	6.2
1%GNP- 10%P-3H+TBP+40°C	3011	64.3	7.93

Therefore, the reason for decreasing the electrical conductivity of the CNT+GNP/SDBS reinforced composites is probably due to nanoparticle features and/or structures changes provided by the SDBS dispersion mechanism.

In previous studies, significant deterioration in the electrical properties was also reported due to disturbances of the graphene/CNTs surface π -electrons delocalization.

Furthermore, the synergic effects of hybrid CNT+GNP decreased the electrical resistance of the cementitious composites more than that observed for individual CNTs or GNPs utilization, as it expected.

Furthermore, comparing the effects of CNTs+GNPs on the electrical conductivity of cementitious composites with previous studies using individual CNTs, graphene, or other carbon-based particles (Table 3-9) clearly indicates that the hybrid combination of CNTs and GNPs is more efficient in creating a conductive path within the cementitious composite, as expected. This combination can reduce the percolation threshold while enhancing the quantum tunneling effect, facilitated by their 1D and 2D geometrical shapes.

Thermal Photography

In this study, for the first time, thermal photography was used for the evaluation of CNT+GNP dispersion in hardened specimens through the difference between the thermal diffusion coefficient and their thermal conductivity. Thermal photography of cementitious composite specimens by the same size (160 mm $\times$ 40 mm $\times$ 40 mm) in different periods of cooling time are shown in Figure 3-14, after heating to 70°C, for 72 hours. As expected, reinforced CNT+GNP cementitious composite samples have absorbed more heat due to the high thermal conductivity [355]. It was also observed that they lose heat more rapidly over time due to the high thermal transfer coefficient compared to the plain composites. As shown in Figure 3-14(a) and (b), the CNT+GNP/Pluronic reinforced specimens lose heat more rapidly in comparison to the CNT+GNP/SDBS specimen probably due to disturbances in delocalization of π -electrons on nanoparticles surface caused by SDBS dispersion mechanism, which verify previous results. Non-homogeneous heat distribution caused by considerable large agglomeration on CNMs on CNT+GNP/SDBS composites, indicates inappropriate dispersion of nanoparticles in the hardened sample, as discussed before.

3.4 Conclusion

In this work, an extensive experimental research study on hybrid CNT+GNP dispersion techniques using a high concentration of noncovalent surfactants and the aqueous suspension was conducted. For this

Chapter 3. Dispersion Method Development

purpose, a nonionic Pluronic F127 and ionic SDBS surfactants were studied for 1wt% of CNT+GNP (0.5% CNT and 0.5% GNP) dispersion, analyzing the effect of factors like surfactant concentration, sonication time, temperature and use of antifoam agent (TBP) which was used as an antifoam agent.

Table 3-9: Comparison of electrical resistance for carbon nanoparticles reinforced cementitious composites

Dispersion Method	Weight fraction (%)	Nanoparticles type	Electrical resistivity ($\Omega.m$)	References
Superplasticizer and ultrasonication	5	GNP	21.02	[190]
Dry mechanical mixing	5	GNP	78.20	[190]
Superplasticizer and high speed mixer	5	GNP	27.96	[190]
PVP and ultrasonication	0.7	CNT	24	[356]
Mixing by superplasticizer during mortar blending	10	Carbon Black	4.53	[108]
Mixing by superplasticizer during mortar blending	2	Short Carbon fiber	2.4	[124]
Pluronic F-127 and TBP, 3h sonication, 40°C	1 (half by half)	GNP+CNT	15.3	Present Study
SDBS and TBP, 3h sonication, 40°C	1 (half by half)	GNP+CNT	47.76	Present Study

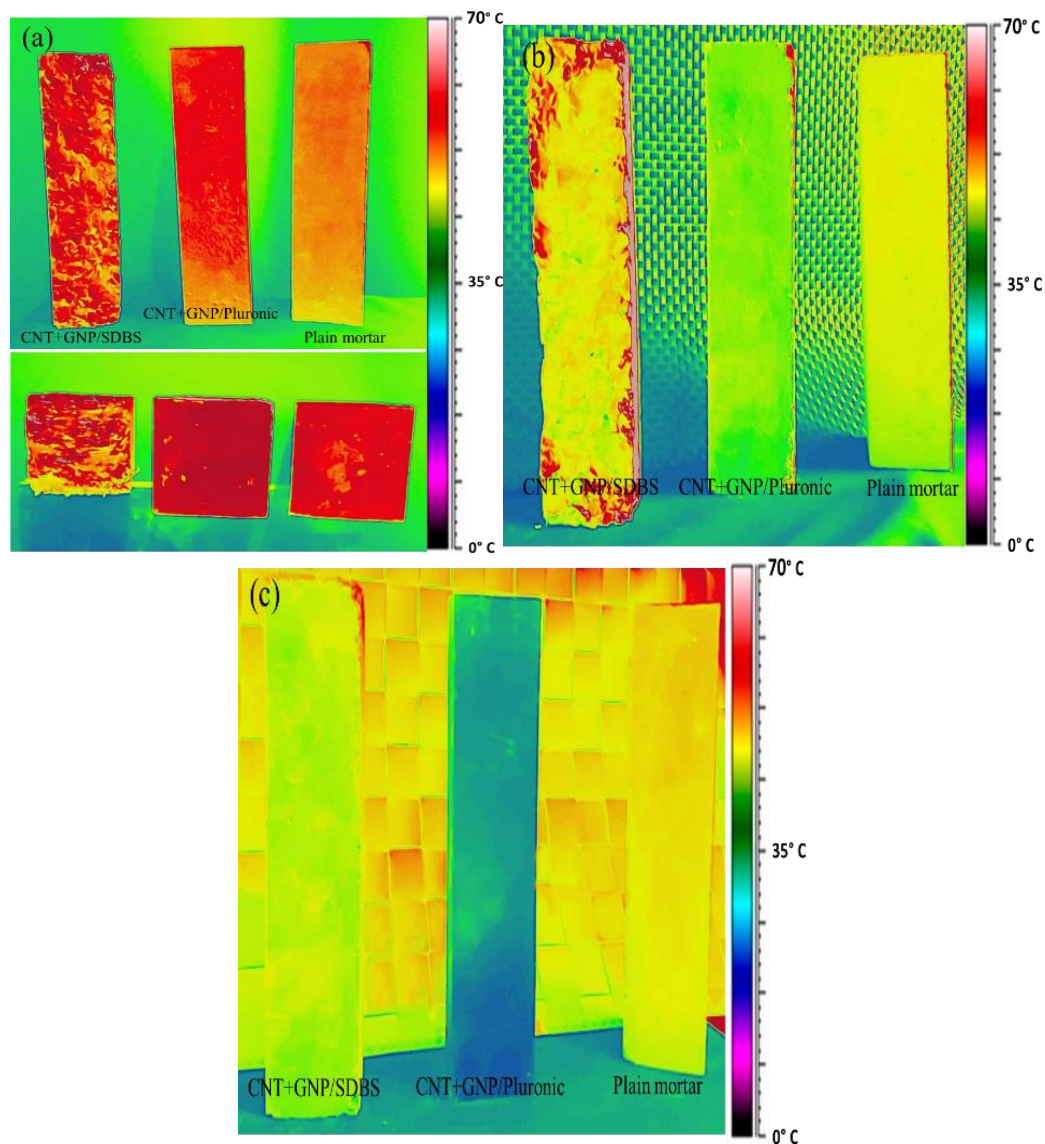


Figure 3-14: Thermography of cementitious composite at different times: (a) Immediately after heating (both cross-section and side-view), (b) After one hour, (c) After two hours.

Besides, the effects of optimized CNT+GNP suspensions prepared by SDBS and Pluronic on the mechanical, electrical, and thermal properties of cementitious composites were evaluated by various tests. UV-Spectroscopy and optical microscopy image analysis indicated that high-quality CNT+GNP dispersion with a low agglomeration area (less than 1.2%) was achieved for 10% of Pluronic (wt% of nanoparticles), with 3 hours' sonication, at 40°C, with the presence of tributyl phosphate (50 wt% of surfactant as antifoam agent). The optimum percentage of SDBS (5 wt%) leads to lower dispersion quality and a higher agglomeration area (5.6%).

Increasing temperature up to 40°C, as it mentioned before plays a positive role in the dispersion of suspensions prepared with Pluronic or SDBS, however higher temperature leads to re-agglomeration of CNT+GNP. The presence of TBP significantly reduced the agglomeration area. Increasing sonication time constantly improved dispersion in both CNT+GNP/Pluronic and CNT+GNP/SDBS aqueous suspensions

which has been also confirmed by Zeta potential and particle size measurement. SEM photographs showed that CNTs and GNPs in prepared aqueous suspensions using Pluronic presented better interaction between each other even after water elimination, compared to CNT+GNP/SDBS suspensions.

Cementitious composites reinforced with hybrid CNT+GNP have shown enhanced mechanical, microstructural, electrical, and thermal properties, for both Pluronic and SDBS dispersion cases. However, the general results trend demonstrated higher efficiency for CNT+GNP/Pluronic due to the compatible mechanism and the dispersion quality.

The results obtained for bulk modulus, water content, ultrasonic NDT test, and capillarity water absorption also indicated denser microstructure for the CNT+GNP/Pluronic reinforced composites due to the lower agglomeration caused by the high-quality dispersion. TGA, DSC, and EDX results showed that dispersed CNT+GNP/Pluronic increased the hydration rate due to the nucleation agent effects of CNTs and GNPs and oxygen functional groups of graphene which act as growing points for hydration products. SEM image analysis showed that CNMs are completely embedded in the hydration products, bridging them. However, this is not observed for CNT+GNP/SDBS due to the lower quality dispersion and also to the changing CNMs features and structures caused by the SDBS dispersion mechanism. For the same reason, CNT+GNP/SDBS reinforced specimens also presented lower electrical and thermal conductivities compared to those reinforced with CNT+GNP/Pluronic.

Chapter 4. Self-sensing Cementitious Mortar Development.

«Tudo vale a pena quando a alma não é pequena. »

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
Addressing the affecting factors	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

- Abedi, Mohammadmahdi, Raul Figueiro, Aires Camões, and Antonio Gomes Correia. "Evaluation of CNT/GNP's synergic effects on the Mechanical, Microstructural, and durability properties of a cementitious composite by the novel dispersion method." Construction and Building Materials 260 (2020): 120486. <https://doi.org/10.1016/j.conbuildmat.2020.120486>

Abstract

In this paper hybrid combination of carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) was used for developing cementitious self-sensing composite with high mechanical, microstructural and durability performances. The mixture of these two nanoparticles with different 1D and 2D geometrical shapes can reduce the percolation threshold to a certain amount which can avoid agglomeration formation and also reinforce microstructure due to percolation and electron quantum tunneling amplification. In this route different concentrations of CNT+GNP were dispersed by Pluronic F-127 and TBP with 3 hours sonication at 40 °C and incorporated into the cementitious mortar. Mechanical, microstructural, and durability of the reinforced mortar were investigated by various tests in different hydration periods (7, 28, and 90days). Additionally, the piezoresistivity behavior of specimens was also evaluated by the four-probe method under flexural and compression cyclic loading. Results demonstrated that hybrid CNT+GNP can significantly improve mechanical and microstructural properties of cementitious composite by filler function, bridging cracks, and increasing hydration rate mechanisms. CNT+GNP intruded specimens also showed higher resistance against climatic cycle tests. Generally, the trend of all results demonstrates an optimum concentration of CNT (0.25%)+GNP (0.25%). Furthermore, increasing CNT+GNP concentration leads to sharp changes in electrical resistivity of reinforced specimens under small variation of strain achieving high gauge factor in both flexural and compression loading modes.

4.1 Introduction

Cementitious composites, as one of the most extensively used materials in the structure's design and construction, are brittle and susceptible to cracking [190]. Damage and failure of cementitious composite occur mainly with time due to numerous factors such as some inherent drawbacks, porosity, heterogeneity, or aggressive environmental conditions, rebar corrosion, or overexploitation, aging of materials, overloading and lack of maintenance over their service life [357]. Among the microstructure of hardened cementitious composites, there are many nanoscale porosities and cracks. These cracks are formed in the process of fabrication or exploitation [358].

As the nanoscale cracks grow and expand over time, larger micro-scale cracks can develop, which may cause structural failures [359-361]. By timely detection of these intrinsic damages in initial phases can prevent their progression and the sudden collapse of structures and greatly minimize potential consequences of deterioration in the service life of structures [362, 363]. The process of continuously damage detection and evaluating the state of civil infrastructures which is commonly known as Structural Health Monitoring (SHM), has attracted the attention of many researchers in the past decades. SHM systems are generally organized to catching real-time data of the structure through sensors that measure condition state's parameters such as stress, displacement, strain, temperature, and etc.

Despite recent advances, most SHM techniques involve a limited number of sensors which are distributed in large areas of structures [357]. Among all SHM methods, cement-based self-sensing composites with capabilities of intrinsic damage sensing based on the piezoresistivity principle, has provided a more sustainable, real-time and practical alternative to concrete structures damage detection [82, 364, 365]. Existence of the conductive phase is essential to achieve piezoresistivity in a cementitious composite. This conductive phase creates conductive electrical networks within the none conductive cementitious matrix, based on percolation and quantum electron tunneling effects [81, 366-368]. This conducting network is disturbed when the cementitious composites are subjected to damage or deformation which results in changing the electrical resistivity [111, 138, 184].

Sensitivity and performance of the cement-based self-sensing composites are extremely dependent on the type of conductive phase, their concentration as well as their distribution [82, 281]. Although the various types of conductive fillers such as nickel powder, iron oxide nanoparticles, carbon black, carbon fiber, carbon nanofiber, graphene nanoplatelets (GNPs), and carbon nanotubes (CNTs) have been used as a conductive phase in the cementitious matrix, GNPs and CNTs have been attracted more attention for develop multifunctional composite with high gauge factor and lower percolation threshold due to their

unique electrical and mechanical properties [82, 281]. Furthermore, reinforcing cementitious composite by high dispersed CNTs and GNPs with low concentration can improve mechanical performance due to denser microstructural formation, increasing hydration rate and bridging mechanisms [208, 346, 347]. The optimum percentage of CNTs and GNPs which have been reported by researchers and lead to significant enhancement in cementitious composites mechanical performance were usually less than 0.3 % by weight of cement. However, excessive ones cause porosities formation due to agglomeration of nanoparticles. [205, 208, 347, 369]. Additionally, to achieve high-performance cement-based self-sensing composite with a high gauge factor, much more CNTs and GNPs concentration (sometimes up to 30 times higher) is required [113, 190, 370, 371].

Besides, the presence of a certain amount of nanoparticle agglomeration was reported as a crucial issue in significant increasing electrical conductivity as well as reducing the percolation threshold [372-375]. Therefore, high sensitive and applicable cement-based self-sensing composites mostly have weakness microstructural behavior with low mechanical and durability performances.

Consequently, piezoresistivity behavior of composite can have sharp changes and scatters in electrical resistance over the time or at the end of each loading cycle due to the destruction of conductive networks. In this study, in order to overcome some of these drawbacks, a hybrid combination of CNT+GNP has been used as a conductive part in order to develop a novel high sensitive affordable cementitious composite with a low percolation threshold. The mixtures of these two nanoparticles with different 1D and 2D geometrical shapes can cause amplification of percolation and electron quantum tunneling which decreases the percolation threshold to a certain amount that can avoid agglomeration formation and reduce the production cost.

Besides the synergic effects of CNTs and GNPs can improve mechanical, microstructural, and durability performances by filler function, bridging and/or deviation of cracks, and increasing hydration rate. As a first step of this achievement, an effective compatible and affordable technique for dispersion of hybrid CNT+GNP high concentration with Pluronic F-127 was proposed by Abedi et al. [145]. Using this technique different concentrations of CNT+GNP were dispersed and incorporated in a cementitious mortar.

These composites were further characterized in terms of mechanical, microstructural, and durability by various test methods. Besides the piezoresistivity behavior of the composite specimens was evaluated under compression and flexural cyclic loading tests.

4.2 Raw Materials and Characterization

Table 4-1 summarizes the characteristics of the GNPs and CNTs as provided by the manufacturer [145]. The Morphology of GNP+CNTs (dry mix) was characterized using Scanning Electron Microscope and depicted in Figure 4-1.



Figure 4-1: Morphology of CNTs and GNPs dry mix.

For CNTs and GNPs dispersion in aqua suspension, a noncovalent surfactant (Pluronic F-127) was used in this study. Pluronic F-127 is a non-ionic triblock copolymer surfactant composed of two hydrophilic chains of polyoxyethylene (PEO) which placed in two sides of a central polyoxypropylene (PPO) hydrophobic chain. These side chains of hydrophilic PEO are similar to superplasticizers of polycarboxylate which are typically used in cementitious composites [144]. For this reason, Pluronic F-127 was found to be compatible with cementitious composites and could possibly improve its mechanical behavior and dry bulk density due to improved fluidity of mortar.

According to previous research, to prevent formation of any porosities caused by surfactant function, tributyl phosphate (TBP) with $\frac{1}{2}$ of surfactant weight ratio was used as antifoam [205]. Their chemical structures are presented in Figure 4-2, [145].

The ordinary Portland cement type I (CEM I 42.5R) and CEN Standard sand (EN 196-1 and ISO 679: 2009) were used to prepare mortar mixtures. The chemical composition and grain size curve of cement and sand are presented in Table 4-2, Table 4-3, and Figure 4-3, [145].

Table 4-1: GNPs and CNTs characteristics .

GNP										
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Carbon content (%)	Tensile modulus (Gpa)	PH Value (30 °C)	Tensile strength (GPa)	Layers	Dimension		Form	Part Number
120-150	0.6	> 99.5	1000	7-7.65	5	< 20	Thickness	Diameter	Gray Powder	TGN201
							4-20nm	5-10µm		
MWCNT										
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Color	Outside Diameter (nm)	Length (µm)	Ash (wt%)	Carbon content (%)		Part Number		
350	0.27	Black	< 8	30-10	< 1.5	> 98		GCM327		

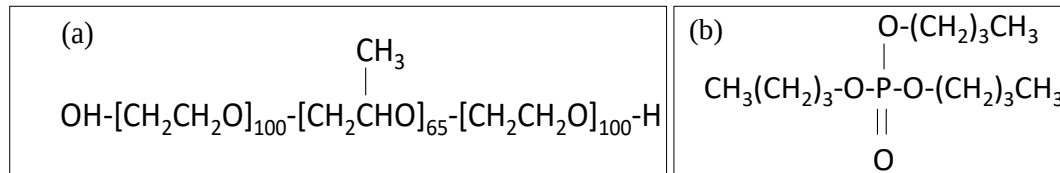


Figure 4-2: Chemical structure of: (a) Pluronic F-127_ (b) Tributyl phosphate 97% [145].

Table 4-2: Particle size distribution of the sand [145].

Mesh Size (mm)	0.08	0.16	0.5	1	1.6	2
Cumulative retained(%)	99±1	87±5	67±5	33±5	7±5	0

Table 4-3: Chemical composition and properties of Ordinary Portland Cement.

SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	CaO (%)	Na ₂ O (%)	TiO ₂ (%)	K ₂ O (%)	MnO (%)	P ₂ O ₅ (%)	SO ₃ (%)	LOI (%)	Initial settin g time (min) ^a	Soundnes (mm) ^a	Blaine's surface (cm ² /g) ^a
19	4.7	3.38	1.3	63.9	0.17	0.24	0.44	0.07	0.06	2.5	2.9 7	194	1.1	4220

^a(Loss on ignition)EN 196-2, ^b EN 196-3.

4.2.1. CNT+GNP Dispersion Method

Nowadays, a successful feasible and compatible technique for hybrid CNT+GNP dispersion in aqueous suspension to be used in multifunctional cementitious composites has been developed by 10% Pluronic F-127 with the addition of TBP through 3hours sonication (80w, 45kHz) at 40 °C [145]. In this route, first TBP (50 wt% of Pluronic) was completely dissolved in 225ml water by 12 hours magnetic stirrer 800 rpm/min. Thereafter, 10% Pluronic (wt of CNMs) added to the water and the suspension was mixed for 1 more hour by magnetic stirrer mixer. Further on, CNT+GNP was added and stirred continuously for 1hour. Afterward, suspensions were placed in a sonicator bath. A digital temperature regulator was used

to adjust the temperature during the ultrasonication process by the circulation system through a radiator and sensors.

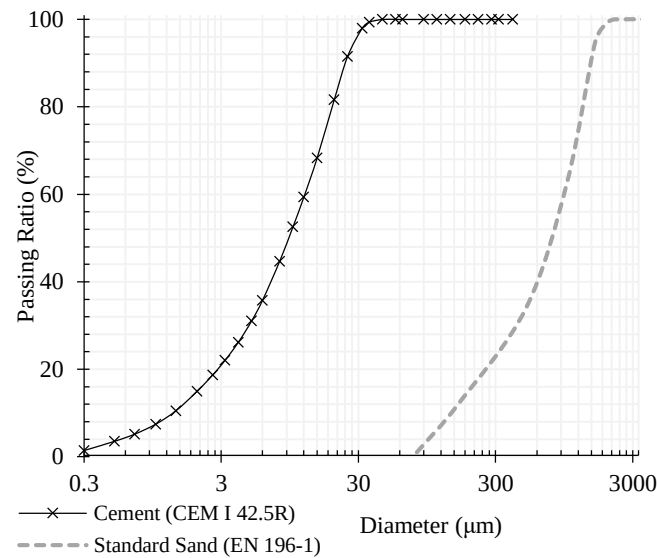


Figure 4-3: Grain size curves of cement and sand.

Under these mild mixing conditions, negligible structural damage is expected for the CNTs and GNPs. The Raman spectroscopy (Figure 4-4) was carried out on CNTs and GNPs using laser excitation with wavelength 532 nm to avoid adverse effects on CNMs structural quality such as edge-type defect, reduction of aspect ratio and sp² domain crystallinity (La), that cause deleterious influence on their mechanical and electrical properties [217, 218].

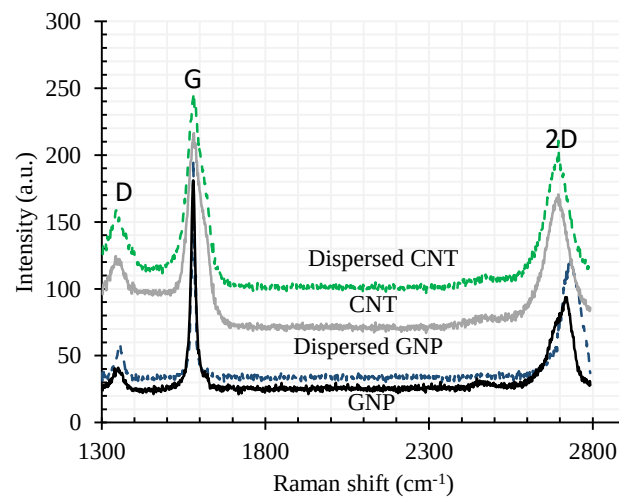


Figure 4-4: CNT and GNP Raman analysis results.

4.2.2. Cementitious Composite Fabrication

Plain and CNT+GNP reinforced specimens were prepared through the mixing of prepared CNT+GNP aqua suspensions with ordinary Portland cement and standardized sand using a laboratory mixer following EN 196-1:1994 standard.

First, the required amount of cement (450 g) was poured into the mixer's stainless steel bowl in order to prepare the mortar mixes. Thereupon, the prepared CNT+GNP aqueous suspension with 225ml water was added to cement (after re-measurement to ensure that water does not evaporate before and after the dispersion process) and the required amount of sand (1350 g) were poured into the mixing machine's hopper.

The mixer was then run for 1.5 min, with stainless steel blade's rotational speed at 140 m/min, followed by a 30 s timeout and then run at a 285 m/min higher speed for another 2.5 min. Next, the mixture was shed into 160 mm × 40 mm × 40 mm prismatic molds and placed on a jolting machine for 1 min to vibrating compaction.

For 24 hours the molds were placed in a humid atmosphere (99%) and then the samples were de-molded and kept underwater for 28 days of the hydration process. Four copper mesh in 40 mm × 50 mm dimensions, were embedded as electrodes in 40 and 60 mm distance from the middle of the specimens which were used for piezoresistivity behavior evaluation (Figure 4-5).

The samples were identified by the variation of CNMs concentration in such a way that specimen GC(0.1%), GC(0.3%), GC(0.5%), GC(0.7%), and GC(1.0%) are contained 0.1%, 0.3%, 0.5%, 0.7% and 1.0% of CNT+GNP respectively (1:1 by weight of cement). For all specimens the content of cement and W/C are constant and equal to 33.3% by weight, and 0.5, respectively.

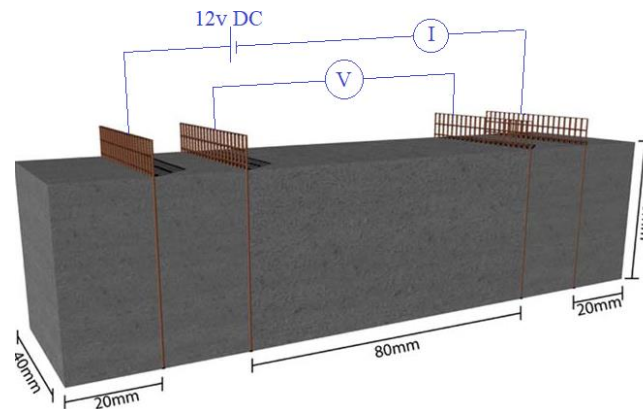


Figure 4-5: Representation of the geometrical parameters and resistivity measurement principle.

4.2.3. Mechanical and Microstructural Characterization

Flexural and compressive strength tests were carried out according to BS EN 196-1:1995 standard. In addition, apparent porosity and dry bulk density of the samples were measured as stated by ASTM C20 and BS EN 1015-10:1999 standards.

The results are obtained by the mean of at least 3 specimens for flexural and 6 for compressive test according the test procedure. Compressive and flexural moduli at rupture moment (E_{Cr} and E_{Fr}) were also calculated by Eq. 1 and 2 respectively:

$$E_{Cr} = \frac{F_c L_0}{A \Delta L} \quad (\text{Eq. 1})$$

$$E_{Fr} = \frac{F_f L^3}{4 \delta w h^3} \quad (\text{Eq. 2})$$

Where F_c is the compressive load at rupture, L_0 initial length of the specimen in the direction of loading, A is the section area of specimen perpendicular to the loading, ΔL is the L_0 changes at rupture, F_f is the flexural load at rupture, L is the length of the specimen, δ is the displacement of the specimen at rupture at the middle and under the loading axis, w and h are also width and height of the section.

The fracture surface of specimens was characterized by Scanning Electron Microscopy using an acceleration voltage of 10 kV and secondary mode of electron after coating with an Au-Pd thin film (30 nm) in a high-resolution sputter coater (Cressington 208HR) in order to investigate the microstructure. Furthermore, the ultrasonic test was performed for microstructural evaluation according to BS EN 12504-4 standard by ultrasonic test device through two probes along the longitudinal transverse axis. Besides, specimens weight loss percentage was measured as a criterion of cementitious composite durability against freeze-thaw cycles.

In this route, saturated specimens with similar dimensions were tested in the temperature range of -20 °C to 30 °C after 28 days of curing. The relative humidity of the chamber was 90% in 30 °C and the duration of each cycle was considered 12 hours (Figure 4-6). Furthermore, relative dynamic modulus of elasticity were calculated by Eq. 2 [376]:

$$RDM = \left(\frac{t_{s,0}}{t_{s,n}} \right)^2 \quad (\text{Eq. 2})$$

where $t_{s,n}$ is the transmit time after n cycles and $t_{s,0}$ is the initial transmit time.

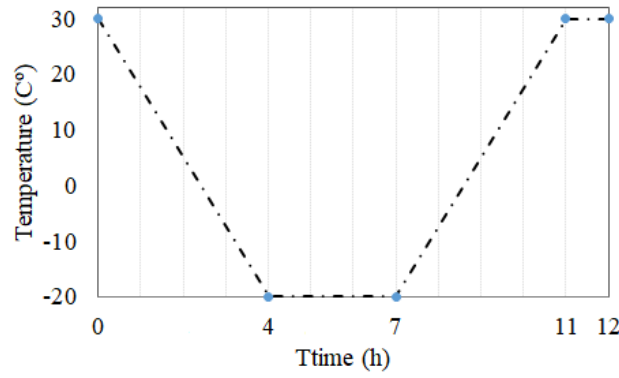


Figure 4-6: Freeze-Thaw temperature cycle

4.2.4. Piezoresistivity Measurement

Specimens with embedded electrodes used for piezoresistivity tests were dried at 70° C for 72 hours after 90 days of curing to avoid moisture effect on electrical conductivity. In this study four-probe method (Figure 4-4) by applying DC current was used to evaluate cementitious composite piezoresistivity behavior under cyclic compression (10KN by rate of 50N.s⁻¹) and flexural (500N by rate of 2.5 N.s⁻¹) loading. Electrical resistance was measured by two digital multimeter and one programmable power supply. One multimeter was connected to the power supply and to the outer electrode for measuring the intensity of current and the other one was connected to the inner electrode for measuring of voltage difference.

The electrical resistivity $\rho(t)$ of each specimen was obtained from the average of five time resistance measurements and was calculated by combining first and second ohm's law equations (Eq. 3 and Eq. 4 respectively) as presented in Eq. 5:

$$R = \frac{V}{I} \quad (\text{Eq. 3})$$

$$R = \rho \frac{L}{A} \quad (\text{Eq. 4})$$

$$\rho(t) = \frac{V(t)}{I(t)} \times \frac{A}{L} \quad (\text{Eq. 5})$$

where R is electrical resistance, I(t) is the current between outer electrodes, V(t) voltage difference between inner electrodes is the applied voltage, L is the spacing between the inner electrodes, A is the contact surface between electrode and composite. For the following assessment of cementitious composite piezoresistivity, the fractional change in resistivity (FCR) was calculated by Eq. 6:

$$FCR = \frac{\rho(t) - \rho_0}{\rho_0} \quad (\text{Eq. 6})$$

where ρ_0 is the initial electrical resistivity which is measured before loading and $\rho(t)$ is the resistivity at time t during the test. To evaluate the sensitivity of CNT+GNP reinforced specimens, the gauge factor (GF) is defined as the relative change in electrical resistivity over the strain (Eq. 7):

$$GF = \frac{FCR}{\varepsilon} \quad (\text{Eq. 7})$$

where ε is the applied strain along the axis of force and bottom of the specimen in compressive and flexural loading respectively.

4.3 Results and Discussion

4.3.1. Mechanical Characterization of Cementitious Composite

Mechanical properties of the reinforced cementitious composite were evaluated by the flexural and compression tests. The results of these tests for cementitious composites reinforced by different CNT+GNP concentrations are shown in Figure 4-7 after 7, 28, and 90 days of the hydration period, along with their corresponding coefficient of variation, which did not exceed 4%.

According to the results, the presence of CNTs and GNPs in cementitious composites enhances mechanical properties. Reinforcing cementitious composite by 0.1, 0.3, 0.5, 0.7 and 1% concentration of CNT+GNP lead to increasing flexural strength by 12, 31, 37, 23 and 25% respectively, when compared to the plain mortar after 7 days of hydration period and 14, 23, 41, 16 and 13% after 28 days.

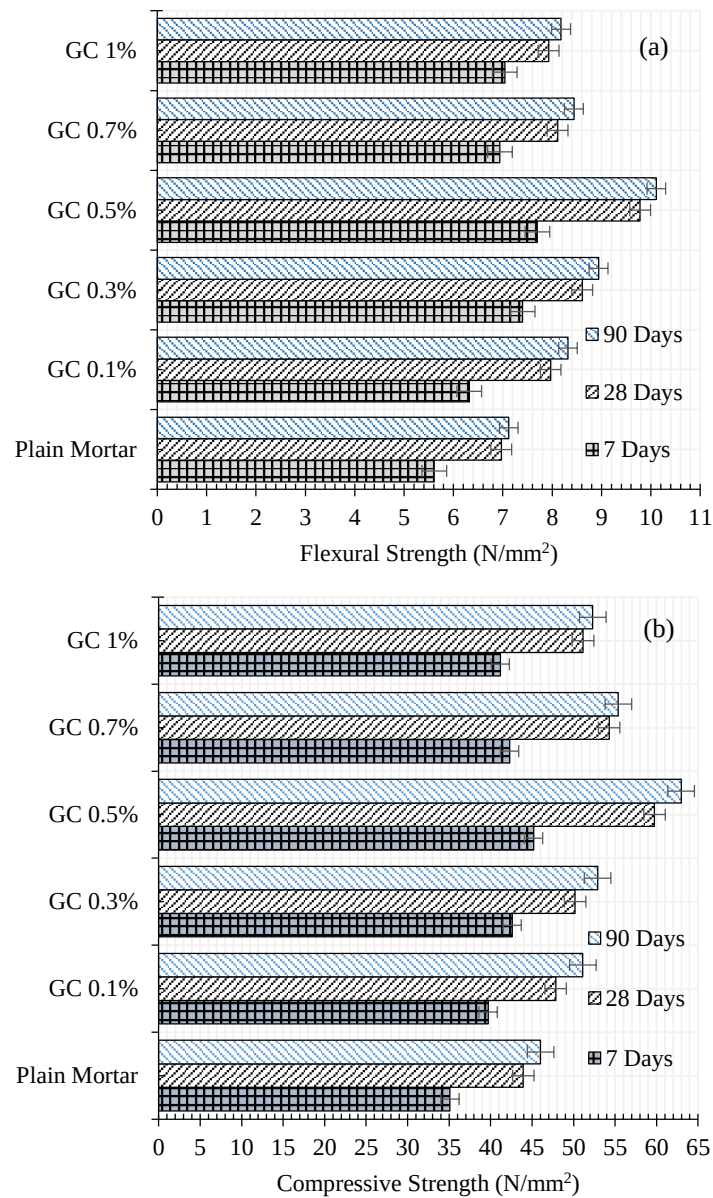


Figure 4-7: a) Flexural, and b) compressive strength of CNT+GNP incorporating cementitious composites at different curing ages

The same analysis for compressive strength shows an increase of 13, 21, 28, 20, and 17% after 7 days and 8, 14, 36, 23, and 16% after 28 days of hydration period. The normalized results (Bernoulli normalization) of flexural and compressive strength for different concentrations of CNMs and hydration periods are presented in Figure 4-8. As can be seen, the overall trend shows that the optimal dosage, around 0.5% concentration of CNT+GNP, achieves the best mechanical properties in the reinforced composite.

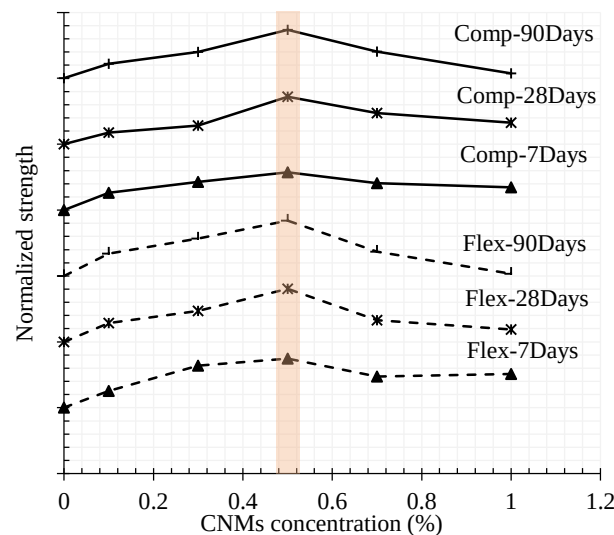


Figure 4-8: Normalized compressive (Comp) and flexural (Flex) strength of plain and CNMs reinforced mortar for different curing times

Flexural and compressive (rupture) modulus of plain mortar and CNM/mortar composites are provided in Figure 4-9.

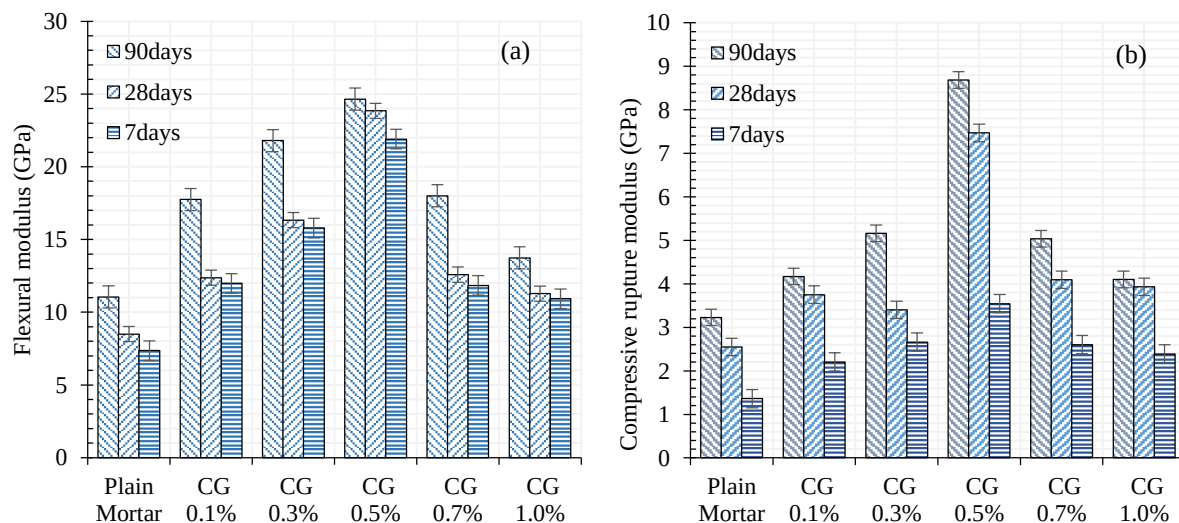


Figure 4-9: Plain and CNMs reinforced rupture modulus in different hydration periods: a) Flexural, b) Compressive

The results indicated significantly higher flexural modulus of CNMs reinforced composites as compared to plain mortar. It is also clear that increasing CNMs concentration up to around 0.5% presented more improvement in flexural and compressive modulus as compared to plain mortar. The improvement in flexural and compressive modulus was quite high reaching up to 123% and %168 respectively in the case of 0.5% reinforced samples after 90days of curing. Specimens failure modes under flexural and compressive loadings have been shown in Figure 4-10.



Figure 4-10: Images of specimens after tests: a) specimen GC(0.5%) after flexural test, b) specimen GC(0.5%) after compression test.

Indeed, incorporating CNT+GNP into cementitious composites enhances mechanical performance by leveraging the bridging mechanism and reducing porosities. Firstly, their high aspect ratios and nanoscale dimensions allow them to effectively reinforce the cement matrix. When dispersed uniformly, these nanomaterials form a network that enhances load transfer and distributes stress more efficiently throughout the composite. This reinforcement leads to a stiffer and stronger material overall.

Additionally, CNTs and GNPs possess superior mechanical properties themselves, such as high tensile strength and stiffness, which contribute directly to increasing the composite's modulus. Furthermore, their ability to reduce microcracks and improve the interface bonding with cementitious materials also plays a significant role.

By mitigating crack formation and propagation, CNTs and GNPs prevent premature failure and maintain the structural integrity of the composite, thereby enhancing its modulus and overall mechanical performance. However, an excessive increasing of CNMs concentration can cause agglomeration and porosities formation which consequently can decrease flexural and compressive strengths [377].

Despite this decrease, it is noticed that the flexural and compressive strengths values of specimen GC (1.0%) are still greater than the value of plain mortar. These results demonstrate that the hybrid combination of these carbon nanoparticles CNT+GNP is more efficient to mechanical properties enhancement than reinforced cementitious composites that used individual CNMs (Table 4-4).

This can be explained by the capacity of load carrying between CNMs and the adjacent cementitious matrix. Besides, GNPs can facilitate the CNTs dispersion which leads to an increase in overall strength [378]. Additionally, in nano intruded cementitious composite, crack propagation is stopped by various types of inclusions: pores, grains, fibers, aggregates, and particles [379].

The synergy between these nanomaterials leverages CNTs' high aspect ratio for effective load transfer and GNPs' large surface area to improve dispersion and compatibility within the cement matrix. This combination enhances mechanical properties such as strength and modulus more efficiently than either material individually, while also promoting better electrical and thermal conductivity.

CNTs and GNPs also block the cracks by deviating and/or arresting their propagating tips, like an obstacle (Figure 4-11).

Table 4-4: Comparison of mechanical properties with previous studies for CNMs reinforced cementitious mortar after 28 days of hydration.

Dispersion Method	Nanoparticles type and weight fraction(%*)	Results and improvement (%)	Standard	References
Ultrasonication	CNT (1%)	(6%) Compressive	N.A	[380]
Nitric and sulfuric acid by carboxylation plus Sonication	CNT (0.5%)	(19%) Compressive _ (25%) Flexural	N.A	[381]
Modified acrylic polymer and acetone plus sonication and superplasticizers	CNT (0.5%)	(11%) Compressive _ (33%) Flexural	UNI-EN 196-1	[382]
Modified polycarboxylate admixtures and naphthalene-sulfonate plasticizer	CNT (0.3%)	(12%) Compressive _ (34%) Splitting Tensile _ (14%) Modulus	NBR 7215, 8522,7222	[383]
Ultrasonication	CNT (0.5%)	(15%) Compressive _ (36%) Splitting Tensile	ASTM C39 C496	[384]
Polycarboxylate superplasticizer	GO (1.5%)	(48%) Tensile	N.A	[385]
Pluronic F-127 and Tributyl phosphate by optimized temperature and ultrasonication	GNP+CNT (0.5% half by half)	(36%) Compressive _ (41%) Flexural	BS EN 196-1:1995	Present Study

* by weight of cement

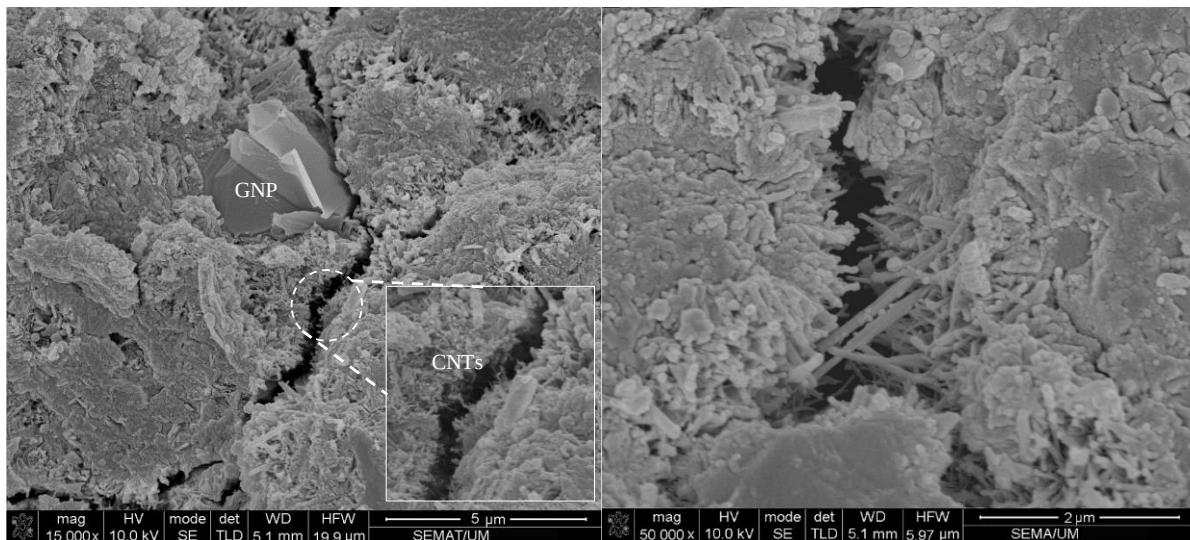


Figure 4-11: SEM morphology of CNT+GNP specimen after mechanical strength measurement

4.3.2. Microstructural Investigation

The results of apparent porosities, dry bulk density, and ultrasonic wave passing time for different nano intruded cementitious mortar are shown in Figure 4-12, Figure 4-13 and Table 4-5.

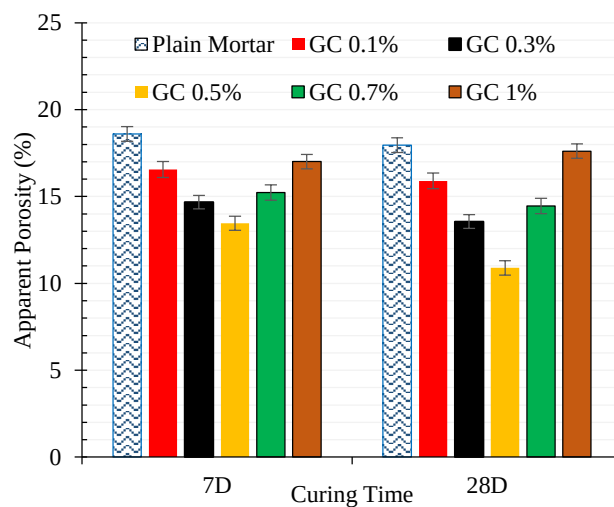


Figure 4-12: Apparent porosity of nano intruded cement mortars

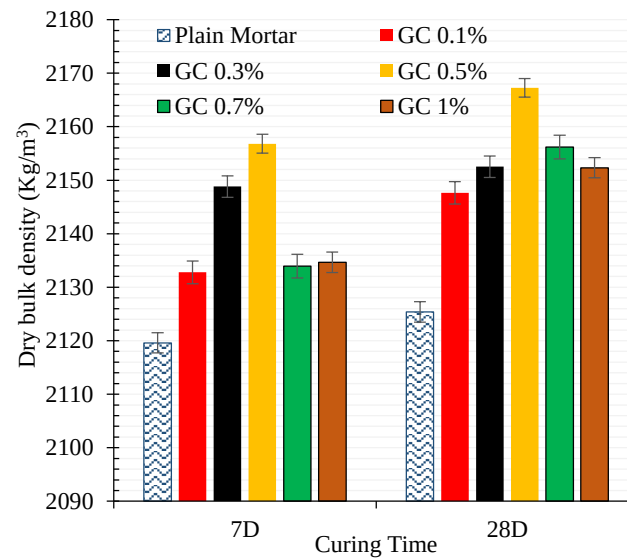


Figure 4-13: Dry bulk density of nano intruded cement mortars

These results show that incorporating CNT+GNP into the cementitious composite generally leads to reducing apparent porosities (denser microstructure) and consequently increase the density and decreases the ultrasonic wave passing time.

The addition of 0.1, 0.3, 0.5, 0.7, and 1% CNT+GNP to the cementitious composite has decreased the apparent porosities by 2.1, 4.0, 5.2, 3.4, 1.6% after 7 days and 2.0, 4.4, 7.1, 3.6 and 0.4% after 28 days of hydration period, respectively. It is also noticed an increase of dry bulk density about 2% by increasing CNT+GNP concentration to around 0.5%.

However, by further increasing nanoparticles percentage, it will increase micro porosities resulting from GNP+CNT agglomerates formation in cementitious composite inducing in a decrease of the dry bulk density and mechanical properties of the composite.

A similar trend was also observed for ultrasonic wave passing time (Table 4-5). Indeed, increasing CNT+GNP to around 0.5% leads to reducing passing time, while an increasing CNT+GNP more than 0.5% results in an increase of passing time.

Furthermore, an increase of the curing age, e.g of the increase of hydration products, results in a decrease of apparent porosity and an increase of dry bulk density with a decrease of ultrasonic waves passing time as a consequence of a denser microstructure.

Table 4-5: Ultrasonic wave passing times of nano intruded cement mortars

Sample ID	Age	Ultrasonic wave time passing (μ s) for 150 kHz	
		Longitudinal	Transverse
Plain Mortar	7D	36.46	10.69
	28D	36.40	9.60
GC 0.1%	7D	35.90	10.17
	28D	34.70	9.38
GC 0.3%	7D	35.20	9.61
	28D	34.10	8.93
GC 0.5%	7D	33.24	9.21
	28D	33.18	8.72
GC 0.7%	7D	33.70	9.43
	28D	32.90	8.64
GC 1%	7D	34.10	9.93
	28D	33.97	9.49

4.3.3. Durability of CNT+GNP reinforced cementitious mortar

Figure 4-14 shows the weight loss percentage of cementitious composites as a function of freeze-thaw cycles numbers after 180 cycles.

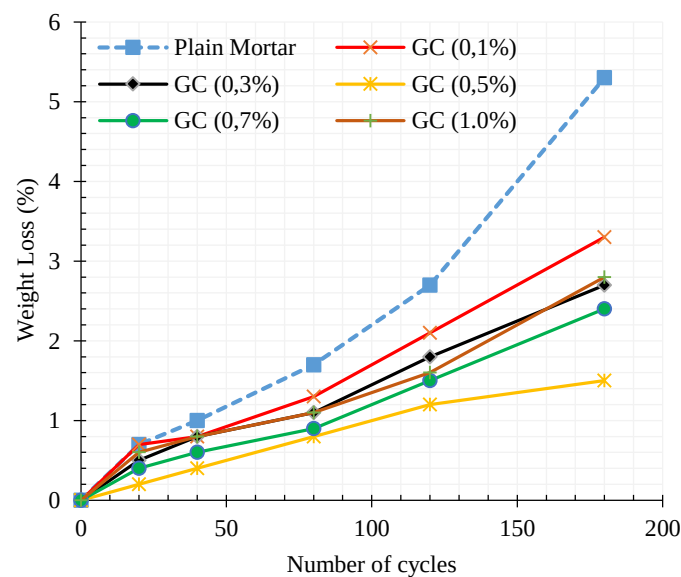


Figure 4-14: The weight loss rate of cementitious composites in different freeze-thaw cycles.

These results reveal the same trend of the previous test results with an optimum percent, around 0.5% of CNT+GNP concentration for the maximum resistance against climatic cycles. Besides, the increase of hydration rate of reinforced specimens due to nucleation effects of CNMs [206], may lead to a decrease of the destruction of cementitious composite with the increase of the number of cycles. This is clearly observed by the decrease ratio of weight loss with the number of cycles in comparison with the results of plain specimen.

The same tendency of the optimum around 0.5% of CNT+GNP concentration was also observed for the relative dynamic elasticity modulus after 180 freeze-thaw cycles as depicted from Figure 4-15. Incorporating 0.1, 0.3, 0.5, 0.7, and 1.0% CNT+GNP into the cementitious mortar increases the dynamic modulus by 5, 19, 23, 17, and 4%, respectively.

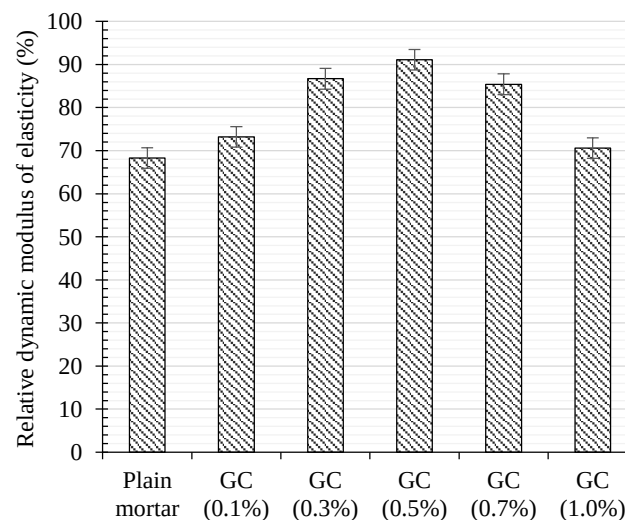


Figure 4-15: Relative dynamic elasticity modulus of CNT+GNP intruded cement mortar after 180 freeze-thaw cycles

4.3.4. Piezoresistive Behavior

Electrical resistance results

The results of electrical resistance of plain and CNT+GNP reinforced specimens in different hydration period are presented in Figure 4-16.

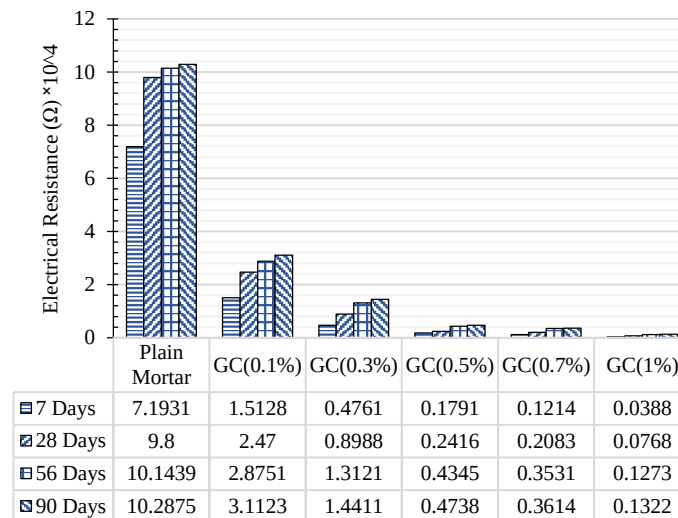


Figure 4-16: The electrical resistance of reinforced cementitious composite by different CNT+GNP concentration and curing age.

As can be noticed, reinforcing cementitious composite by CNT+GNP leads to a significant decrease in electrical resistance. Incorporating 0.1, 0.3, 0.5, 0.7, and 1% CNT+GNP (1:1) into the cement mortar reduced electrical resistance 78, 93, 97, 98, and 99%, respectively after 7 days of hydration period compared to the plain specimen. These results after 90 days of curing slightly decrease for 69, 85, 95, 96, and 98%, respectively. It also shows that the percolation threshold for reinforced cementitious composite by CNT+GNP is probably between the concentration of 0.5% and 0.7% due to no noticeable alterations in electrical resistance with the rise in CNM concentration. Additionally, it can be observed that in all reinforced and plain cementitious composites, the electrical resistance increased by increasing curing age and hydration period. This is because of the formation of finer pores due to cement hydration which leads to a decrease in pore water and cut off the conductive pathway of water [386]. Moreover, cement hydration process also can make a gap in CNT+GNP conductive paths by changing in cementitious composite microstructure and surrounding the CNMs as visible in Figure 4-17.

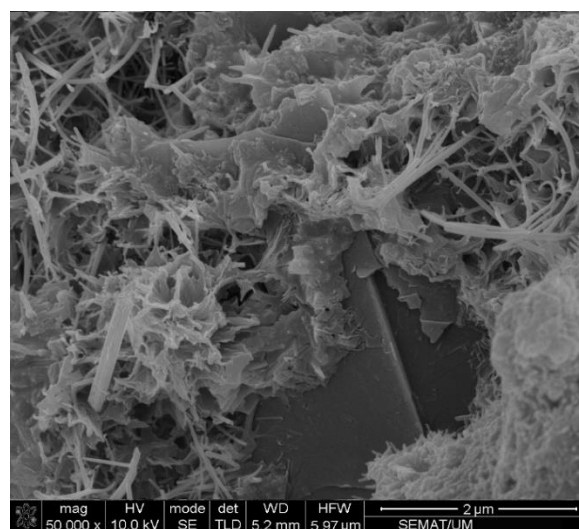


Figure 4-17: Buried GNPs and CNTs among of the hydration products (characterised by EDX).

However, the increase of electrical resistance with curing time for the specimens reinforced with more than 0.5% concentration is lesser in comparison with other specimens, especially after 7 days of curing. The reason for this can be explained by the fact that the concentrations of CNT+GNP greater than the percolation threshold leads to the well-established continuous CNT+GNP conductive pathways which are not affected significantly by dried pores [386, 387]. Table 4-6 summarizes results of other studies and shows the high efficiency of hybrid CNT+GNP used in this research. Indeed, the synergic effects of CNT+GNP create more electrical conductive paths by lower concentration and consequently reduces electrical resistivity significantly.

Table 4-6: Comparison of electrical resistance for CNM reinforced cementitious composites after 28 days of curing

Matrix	Nanoparticles type	Weight fraction wt (%)	Electrical resistivity ($\Omega.m$)	References
Mortar	GNP	5	21.02	[190]
Paste	CNF	2	11	[184]
Mortar	GNP	5	78.2	[190]
Mortar	GNP	5	27.96	[190]
Mortar	CNT	0.7	24	[356]
Mortar	Graphene	1	4000	[388]
Mortar	Carbon Black	10	4.53	[108]
Paste	GNP	4.8	20	[358]
Mortar	Short Carbon fiber	2	2.4	[124]
Mortar	GNP+CNT	1 (half by half)	15.3	Present Study

Figure 4-18 illustrate clearly that the combination of CNTs and GNPs with 1D and 2D geometrical shapes and large surface area can increase the possibility of the formation of long-range connectivity in a random system. GNPs provide additional pathways for electron transport across the cement matrix, effectively bridging gaps between CNT networks. This increased connectivity facilitates the formation of a more continuous conductive network throughout the composite material, improving its overall electrical and mechanical properties. Thus, the combination of CNTs and GNPs synergistically enhances the structural integrity and functional performance of cement composites, making them suitable for advanced applications in construction and infrastructure.

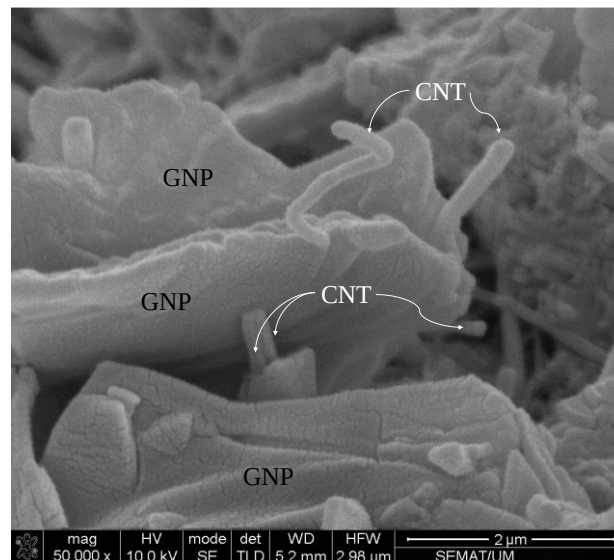


Figure 4-18: SEM image of incorporated CNT+GNP into the cement mortar

The high specific surface area of GNPs can also increase the free surface for electron transmission by the quantum tunneling mechanism schematically illustrated in Figure 4-19.

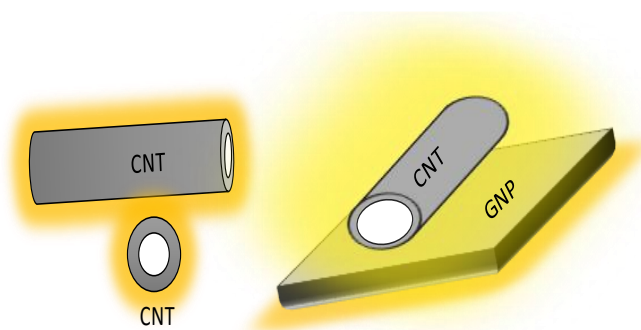


Figure 4-19: Schematic of tunneling effect for CNT/CNT and CNT/GNP cases.

Cyclic compression test results

Figure 4-20 shows the results of the fractional change in resistivity together with cyclic compression response for reinforced cementitious composite by different CNT+GNP concentration in function of time.

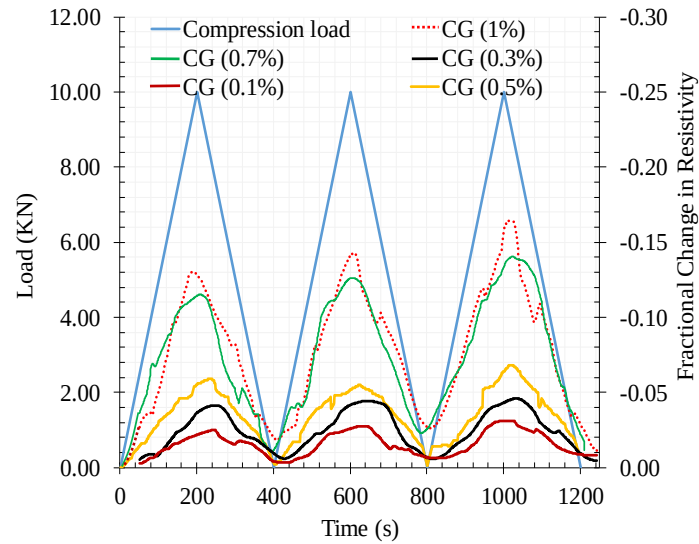


Figure 4-20: The fractional change in resistivity together with cyclic compression response for reinforced cementitious composite by different CNT+GNP concentration

As can be seen, increasing the compression load leads to a decrease in the electrical resistivity by making conductive paths closer to each other and in opposite, removing the load causes an increase in electrical resistivity.

The maximum fractional change in electrical resistivity of cementitious composite was also increased by increasing CNT+GNP concentration. In general, the fractional change in resistivity (FCR) value was negative under compression loading due to the decrease in electrical resistance during loading compared to the primary electrical resistance of specimen which was measured before loading (Eq .5). However, in all CNT+GNP concentrations, except 0.5%, after fully load elimination, the amount of specimen electrical resistance does not return to its original value. This can be a consequence of some internal defects (i.e. holes, native cracks, etc.) in the microstructure which are gradually reduced in the successive cycles of compression loading. In order to get more information, these results are also presented in terms of the axial strain as illustrated in Figure 4-21. It can be observed an increase of the strain level with the increase of number of loading cycles, except for 0.5% of CNT+GNP concentration.

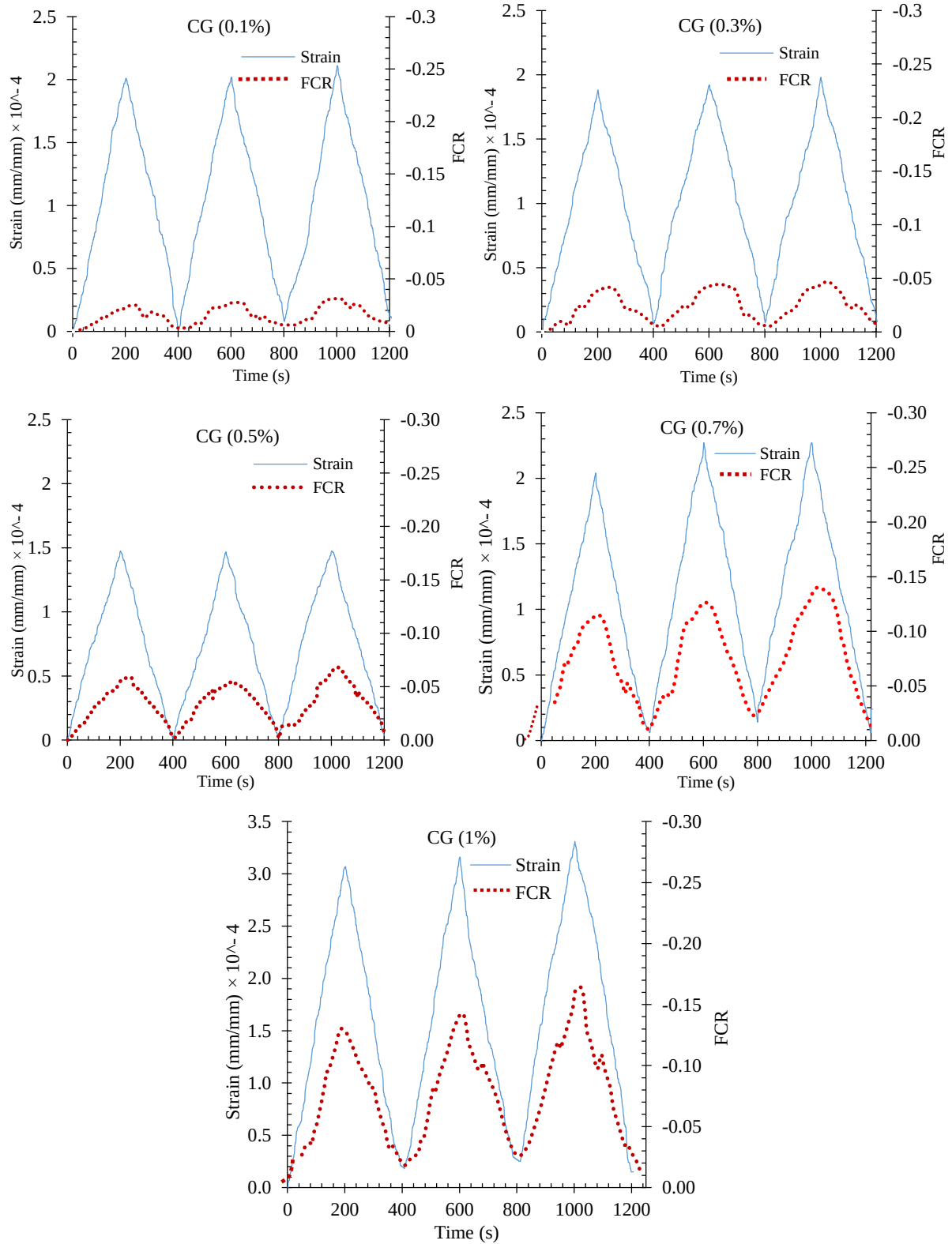


Figure 4-21: The fractional change in resistivity together with axial strain under cyclic compression loading

The ratio of the compressive modulus at 10 KN loading (E_{c10}) to the compressive modulus at rupture (E_{cr}) also has been shown in Figure 4-22.

As can be seen, the difference of modulus between rapture moment and at the top of each loading cycle (10 KN) for specimen CG0.5% is more compared to the rest. Hence, the residual strain of specimen CG 0.5% was lower at the end of each loading cycle. As noted in section 4.3.1, the sample CG 0.5% exhibited greater improvements in physical performance.

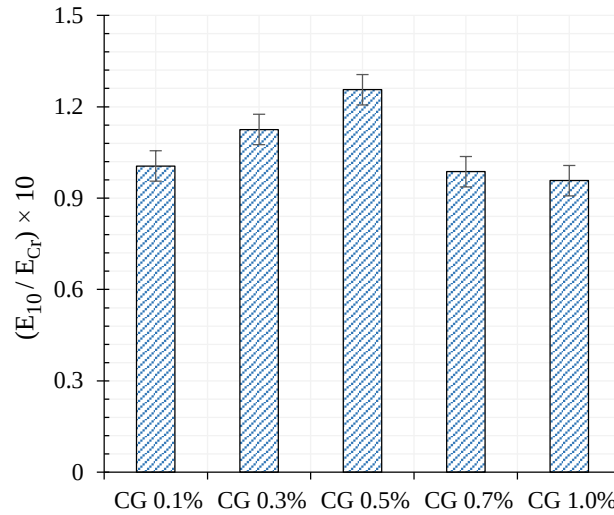


Figure 4-22: The ratio of rupture compressive modulus (E_{cr}) to compressive modulus at 10KN loading (E_{c10})

The relationship between the Fractional Change in Resistivity (FCR) with strain is shown in Figure 4-23 for the different CNT+GNP concentrations. The results of adjustment of these relationships by power function regressions are also presented.

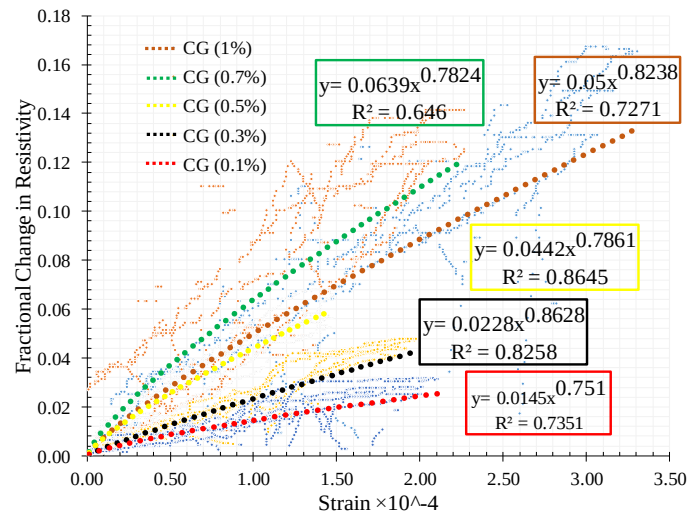


Figure 4-23: Variation of strain with the fractional change in resistivity for CNT+GNP reinforced specimens.

It can be observed that increasing CNMs concentration causes an increase in the fractional change in resistivity with an optimum sensitivity of the composite to strains around 0,7% of CNT+GNP concentration. Indeed, for an excessive increase of CNT+GNP concentration reduces the sensitivity of the composite under compression loading due to specimen saturation of the conductive paths.

Cyclic flexural test results

Figure 4-24 and Figure 4-25 are the correspondent results for the flexural tests adopting the same analysis as for the compression tests. From Figure 4-24 it can be observed that contrarily to the compression tests the electrical resistivity increases with the flexural loading. This can be explained by the mixed mechanisms of compression and tensile in the beam section in conjunction with the susceptibility of the micro cracking network created in the bottom of the section under tension. Therefore, conductive paths are disrupted by these cracks, resulting in an overall increase in electrical resistivity with the increase in flexural loading.

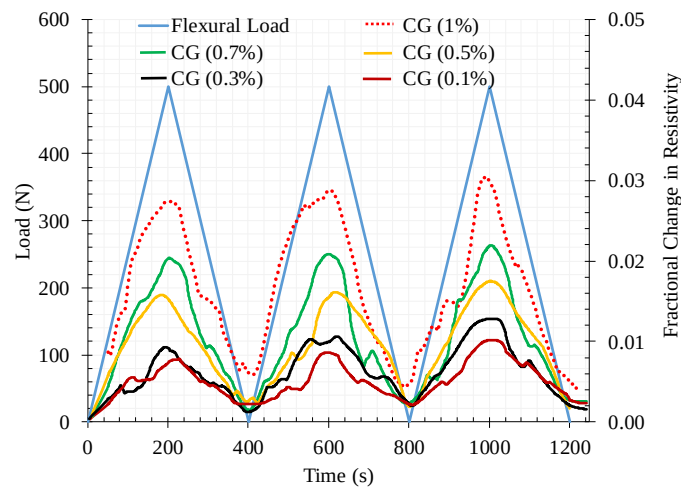


Figure 4-24: The fractional change in resistivity together with the cyclic flexural response for reinforced cementitious composite by different CNT+GNP concentration

As for the compression tests, the FCR increases with the increase of CNT+GNP concentration and after unloading it is also observed that the electrical resistance does not return to its initial value, being this offset more significant with the increase of number of cycles (Figure 4-25). In contrast to compression loading mode, the FCR values were generally positive under flexural loading due to the increase in electrical resistance during loading compared to the primary electrical resistance of specimen which was measured before loading (Eq .6).

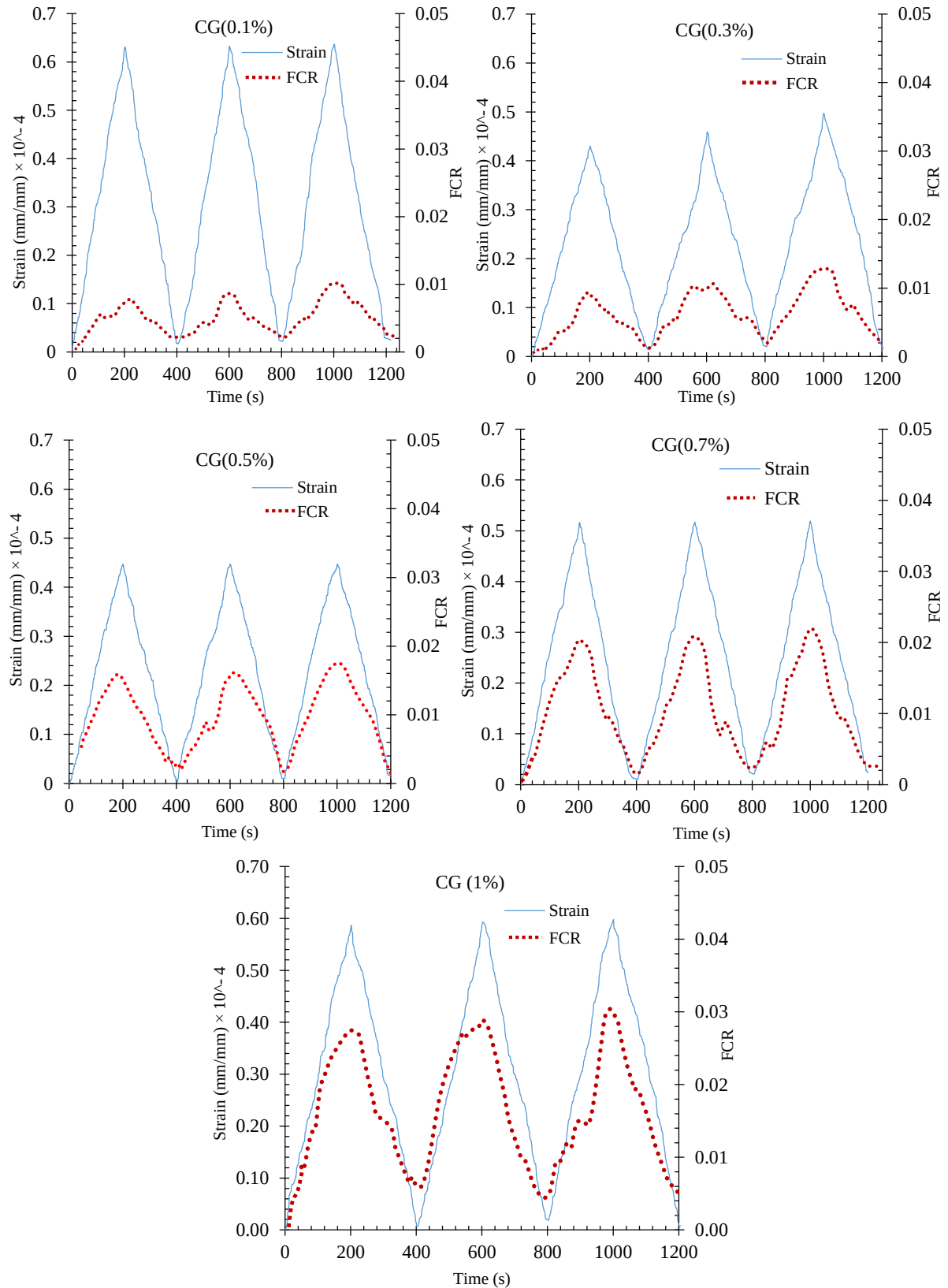


Figure 4-25: The fractional change in resistivity together with axial strain under cyclic flexura loading

The ratio of the flexural modulus at 500 N loading (E_{F500}) to the flexural modulus at rupture (E_{Fr}) are shown in Figure 4-26. As can be observed, specimen CG 0.5% and CG 1.0% showed higher and lower stiffness respectively compared to the other specimens at the top of each loading cycle.

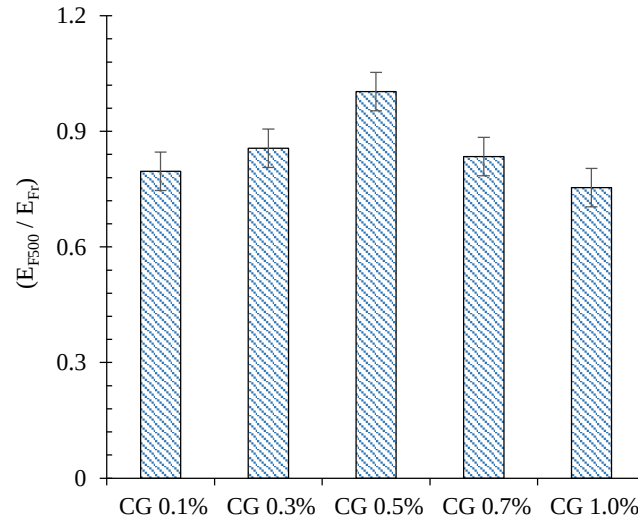


Figure 4-26: The ratio of rapture flexural modulus (E_r) to flexural modulus at 500N loading (E_{F500})

Concerning the relationship between the FCR with strain under flexural loading for the different CNT+GNP concentrations it is observed a big scatter (Figure 4-27) when compared with the compression tests (Figure 4-23). It is noticed that increasing CNMs concentration caused an increase in the slope of the FCR curve which shows more sensitivity.

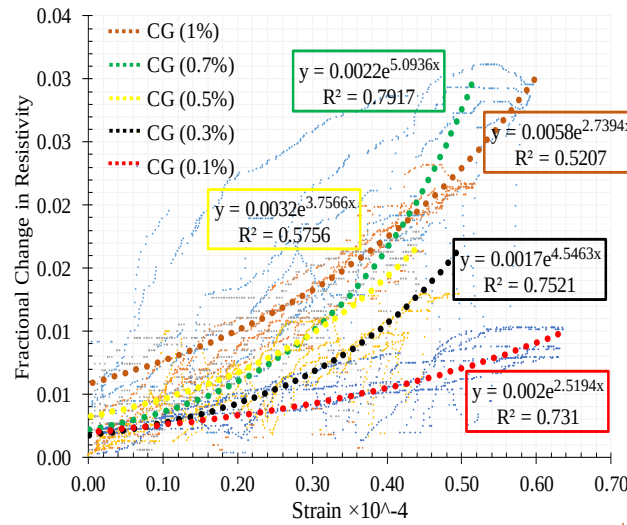


Figure 4-27: Variation of strain with the fractional change in resistivity for CNT+GNP reinforced specimens under flexural cyclic load

Results also indicated a greater curvature in the FCR curves of CNMs reinforced mortar with high concentrations which can be due to lower stiffness or micro cracks formation. Higher concentrations of CNMs led to the formation of agglomerates and decreased physical performance.

Gauge Factors

To better illustrate the strain sensing capabilities of CNT+GNP reinforced cementitious composite, the gauge factor for both types of flexural and compression cyclic loading is presented in Figure 4-28.

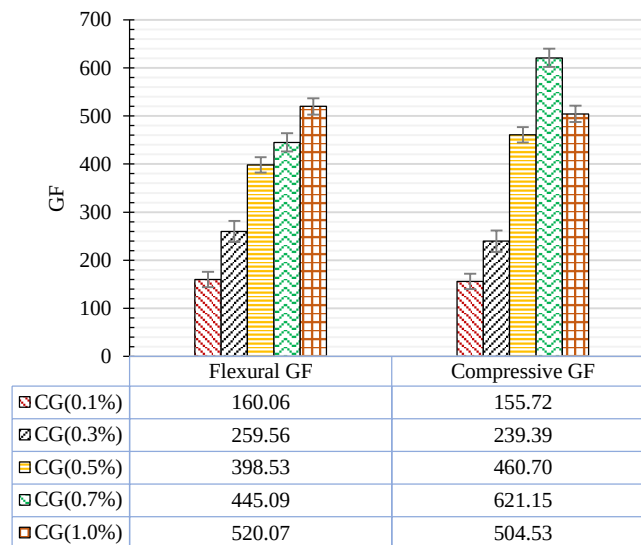


Figure 4-28: Variation of gauge factors in flexural and compression loading

Table 4-7 shows that higher gauge factors can be achieved, giving enhanced sensitivity, for CNT+GNP reinforced cementitious composite for 0.5%, when compared with previous studies that used individual CNTs or GNPs. These results are very encouraging showing that the hybrid combination of these nanoparticles (CNT+GNP) is very efficient to achieve a sensitive self-sensing cementitious composite with good performance in terms of durability and mechanical performances.

Table 4-7: Comparison of gauge factors for cementitious composite under compressive loading.

Matrix	Filler	Filler Ratio (Vol%)	GF	References
Cement Paste	Nanographite Plates	5	156	[371]
Mortar	GNP	2.4	30	[370]
Mortar	GNP	3.6	100	[370]
Mortar	GNP	2.1	90.8	[190]
Mortar	GNP	1.5	110	[190]
Mortar	CNT	0.15	240	[389]
Mortar	CNT+GNP	0.12	460	This study

4.4 Conclusion

In this study, mechanical, microstructural, and durability of hybrid CNT+GNP reinforced cementitious mortar were evaluated in different hydration periods (7, 28, and 90d). Besides the sensitivity of specimen to the strain and stress were investigated under cyclic flexural and compression loading by measuring fractional changes in the electrical resistivity. The dispersion of nanoparticles (0.1, 0.3, 0.5, 0.7, and 1% CNT+GNP with equal proportions) was achieved by using Pluronic F-127 and TBP with 3 hours' sonication at 40°C and following outcomes were obtained:

Chapter 4. Self-sensing Cementitious Mortar Development.

- An optimum concentration of CNT+GNP around 0.5% (1:1) shows the best performance in terms of durability (resistance against freeze-thaw cycles), microstructure and mechanical behaviour.
- Incorporating 0.5% CNT+GNP into the cementitious mortar led to increasing flexural strength by 37, 41, and 43% after 7, 28, and 90 days of curing respectively. these amounts for compressive strength were 28, 36, and 46% respectively.
- The improvement in flexural and compressive moduli was quite high reaching up to 123% and %168 respectively in the case of 0.5% reinforced samples after 90days of curing.
- Scan electron microscopy, dry bulk density, apparent porosities, and ultrasonic wave passing time also showed the denser microstructure for reinforced mortar by 0.5% CNT+GNP.
- Evaluation of relative dynamic modulus and weight loss of specimens after 180 freeze and thaw cycles also showed the best performance in terms of durability for the reinforced specimen by 0.5% CNT+GNP.
- Incorporating 0.5% CNT+GNP into the cementitious mortar led to the sharp change in electrical resistivity under cyclic loading which caused flexural and compression gauge factors by 398 and 460 respectively. This optimum percentage reveals to have significant higher gauge factors when compared with the available results using individual nanoparticles.

The results of this study provide proof of the concept that incorporating a low concentration of hybrid combination of carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) in a cement mortar, can provide self-sensing capabilities of the reinforced cementitious composite enhancing microstructure, durability and mechanical performances.

Chapter 5. Self-sensing Cementitious Geocomposite Development.

«The best way to predict the future is to create it.»

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

- Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "Ultra-sensitive affordable cementitious composite with high mechanical and microstructural performances by hybrid CNT/GNP." Materials 13, no. 16 (2020): 3484. <https://doi.org/10.3390/ma13163484>

Abstract

In this study, a self-sensing cementitious stabilized sand (CSS) was developed by the incorporation of hybrid carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) based on the piezoresistivity principle. For this purpose, different concentrations of CNTs and GNPs (1:1) were dispersed into the CSS, and specimens were fabricated using the standard compaction method with optimum moisture. The mechanical and micro-structural, durability, and piezoresistivity performances of CSS were investigated by various tests after 28 days of hydration. The results showed that incorporation of 0.1%, 0.17%, and 0.24% CNT/GNP into the stabilized sand by 10% of cement caused an increase in UCS of about 65%, 31%, and 14%, respectively, compared to plain CSS. Excessive increase in the CNM concentration beyond 0.24% to 0.34% reduced the UCS by around 13%. The addition of 0.1% CNMs as the optimum concentration increased the maximum dry density of the CSS as well as optimum moisture reduction. Reinforcing CSS with the optimum concentration of CNT/GNP improved the hydration rate and durability of the specimens against the severe climatic cycles including freeze–thaw and wetting–drying. Addition of 0.1%, 0.17%, 0.24%, and 0.34% CNMs into the CSS resulted in gauge factors of about 123, 139, 151, and 173, respectively. However, the Raman and X-Ray analysis showed the negative impacts of harsh climatic cycles on the electrical properties of the CNT/GNP and sensitivity of nano intruded CSS.

5.1 Introduction

Among all monitoring methods, self-sensing composites with intrinsic stress, strain, and damage sensing capabilities based on piezoresistivity provide a more integrated, real-time, and practical solution for infrastructure damage detection, considering geomaterial properties and nature. Although several studies have evaluated self-sensing in various composites, such as concrete, cement paste, polymer, and asphalt [81, 82, 207, 390, 391], little research exists that assesses the impact of this method in geomaterials. Self-sensing composites arise from the dispersion of the conductive phase in the non-conductive composite [392-394]. These conductive components form a conducting electrical network within the composites. When the composites are subjected to strain, stress, or any other external factors, this conducting network is disturbed, leading to a change in the electrical resistivity [387, 395-397]. However, sensitivity and performance of self-sensing composite are affected by some factors such as electrodes status, current, temperature, humidity, and loading, among which the type of conductive phase, their concentration, and distribution have particular importance [398-401]. Although various types of fibrous and nanomaterials have been used as the conductive phase in the cementitious matrix, carbon nanomaterials (CNMs) have attracted more attention due to their unique electrical, physical, and mechanical properties [187, 281, 402-404]. Furthermore, reinforcing cementitious composites by highly dispersed CNMs with low concentration can improve their mechanical, microstructural, and durability performance [107, 396, 405-408]. Recently, studies have been conducted on the synergic effects of carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) on the mechanical, microstructural, durability, and piezoresistivity performances of cementitious mortar using compatible, affordable, and effective dispersion techniques [145, 207, 409]. These researches reported that hybrid CNTs/GNPs can improve percolation and electron tunneling effects, which increase the conductive path and consequently reduce the percolation threshold significantly. Hence, the synergic effects of CNTs/GNPs also decrease the required concentration of conductive filler and eliminate concerns about rising production costs and porosity formation. Accordingly, it is possible that hybrid CNTs/GNPs combinations can also achieve similar performance in cementitious stabilized sand (CSS). Taking this into account, this study aims to take advantages from the synergic effects of these CNMs to advance existing knowledge developing a novel piezoresistive self-sensing cementitious geocomposite. In this route, different concentrations of hybrid CNT/GNP (1:1) were incorporated into the mixture of the sand and 10% cement (by weight of the sand). The optimum moisture of different nano-intruded mixture was measured and cylindrical specimens were fabricated using the standard compaction method. The mechanical performance of specimens was evaluated by unconfined compressive strength (UCS) tests. Microstructural properties of CNM-reinforced

CSS were also investigated by various tests. The durability of the specimens was investigated against hybrid climatic cycles, including a combination of both freeze–thaw and drying–wetting cycles, which represents harsh climatic conditions. In addition, the piezoresistivity behaviors of nano-intruded specimens were evaluated using the four probes method under cyclic compression loading. To investigate the sustainability and long-term performance of this novel self-sensing CSS, the effects of water content and severe climatic cycles on its piezoresistivity properties were also evaluated.

The outcomes of this study provide an extensive contribution to the new era of smart cementitious geocomposites, with primary applications in transport and energy infrastructure, such as roller-compacted concrete dams, rammed earth, ground improvement, and, in particular, pavement structural layers and critical zones, including transition zones.

5.2 2. Materials and Methods

5.2.1. Raw Materials

A multi-layer type of GNP and multi-wall CNT (MWCNT) with a purity of approximately 99.5% and 98% respectively, were utilized as conductive fillers in this study. The characteristics of the GNPs and MWCNTs are summarized in Table 5-1 [145, 409]. The morphology of hybrid GNPs/CNTs in the dry mix state was also investigated by scanning electron microscopy (SEM), as depicted in Figure 5-1.

Table 5-1. Characteristics of graphene nanoplatelets (GNPs) and carbon nanotubes (CNTs).

GNP									
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Carbon content (%)	Tensile modulus (Gpa)	PH Value (30 °C)	Tensile strength (GPa)	Layers	Dimension		Form
120-150	0.6	> 99.5	1000	7-7.65	5	10 < n < 60	Thickness 4-20nm	Diameter 5-10µm	Gray Powder
MWCNT									
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Color	Outside Diameter (nm)	Length (µm)	Ash (wt%)	Carbon content (%)	Part Number		
350	0.27	Black	< 8	30-10	< 1.5	> 98	GCM327		

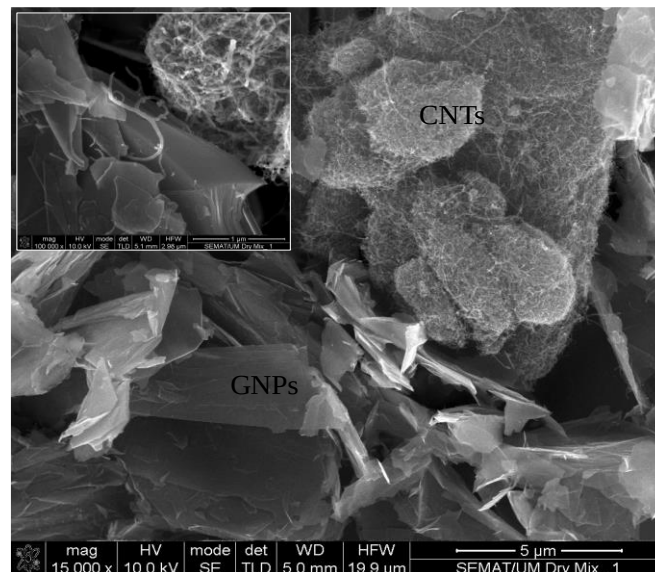


Figure 5-1. CNT and GNP morphology (dry mix).

In order to hybrid CNT/GNP dispersion, Pluronic F-127 (PF-127) was used as a noncovalent surfactant. This surfactant is a nonionic triblock copolymer surfactant and has specific bipolar molecule structure [145]. In addition, tributyl phosphate 97% (TBP) with $\frac{1}{2}$ of the surfactant weight ratio was used as an antifoaming agent to prevent porosity formation caused by surfactant function following previous research [205]. The TBP and PF-127 molecular structures are illustrated in Figure 5-2 [145].

The sand used for this study was CEN Standard sand, with siliceous and clean particles which classified as well-graded sand according to the Unified Soil Classification System (USCS). The physical properties of the sand are presented in Table 5-2. The ordinary Portland cement type I (CEM I 42.5R) was also used as a binder to prepare the CSS.

Table 5-2. Sand particle size distribution.

Mesh Size (mm)	0.08	0.16	0.5	1	1.6	2
Cumulative retained (%)	99±1	87±5	67±5	33±5	7±5	0
Specific gravity G_s	2.67	C_u^a		7.5	C_c^b	1.8

^a Uniformity coefficient, ^bCurvature coefficient

5.2.2. CNT and GNP Dispersion Method

Nowadays, a compatible and effective method for hybrid CNT/GNP dispersion in aqueous suspension to be used in multifunctional cementitious composites has been developed. In this technique, high dosage of CNT/GNP (1%, by the water volume, (1:1)) were dispersed by 10% PF-127 (by weight of CNMs) with the addition of 50% TBP (by weight of surfactant) via 3 hours of sonication (80 W output power and 45 kHz frequency) at 40 °C [145]. A similar method was used to CNT/GNP dispersion [145].

Under these specific mixing conditions, negligible structural damage was expected for the carbon nanomaterials. Raman spectroscopy (Figure 5-2) was also carried out on CNMs using laser excitation with a wavelength of 532 nm to ensure the absence of adverse effects on the structural qualities of CNTs and GNPs, such as edge-type defects, a reduction in aspect ratio, and sp^2 domain crystallinity (L_a), which have a deleterious influence on their mechanical and electrical properties [217, 218].

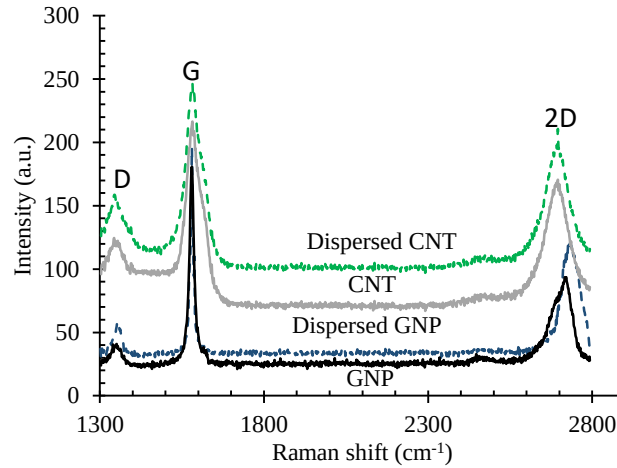


Figure 5-2. Raman analysis results of CNTs and GNPs.

5.2.3. Specimen Fabrication

In stabilized sands, the cement content usually varies around 10% owing to the target strength of the sand–cement [147, 148]. In this study, 10% cement concentration (by weight of the sand) was also utilized in order to CSS fabrication. First, the cement and sand were added to a steel bowl and blended with a stainless steel blade at a rotational speed of 140 rpm for 1.5 min. Then, CNM suspensions composed of 0.1%, 0.17%, 0.24%, and 0.34% CNT/GNP (by weight of the sand and equal portions, 1:1), prepared by ω_{opt} (for each CNM concentration) was sprayed into the mixture and blended at a 285 rpm higher speed for another 2.5 min. Thereafter, the mixture was poured into a plastic bag to prevent moisture loss. The CSS cylindrical specimens were fabricated by the compaction method according to the standard compaction ASTM D698. Split molds with dimensions of 101.6×116.4 mm were filled with the wet mixture in three equal-height layers. A calculated amount of the well-mixed wet mixture was measured (to an accuracy of 0.01 g), poured into the split mold, and then compacted carefully by a metal tamper to the desired height (controlled by a caliper to an accuracy of 0.02 mm). After the molds were filled with the compacted mixture, they were sealed at both ends and extracted after a 24 h period for hardening; then, the specimens were cured underwater for 28 days. For the specimens that were used for piezoresistivity tests, four square meshes of copper with dimensions of 50×50 mm were embedded as electrodes at distances of 38.8 and 19.4 mm from the middle of the specimens (Figure 5-3).

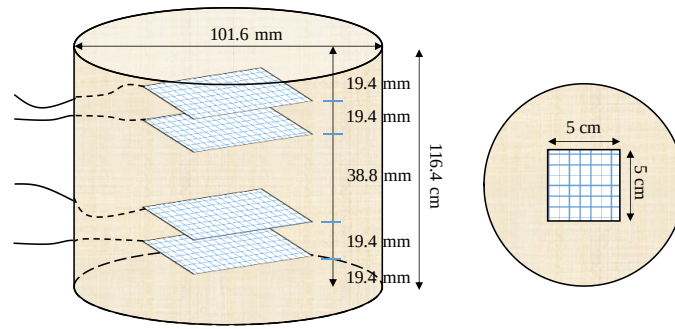


Figure 5-3. Representation of the specimen geometrical and electrode layout.

The amount of the mixture moisture after compaction was also measured to ensure the lack of water evaporation. Sample identification was conducted based on the variation of CNM concentrations in such a way that specimen GC (0.1%), GC (0.17%), GC (0.24%), and GC (0.34%) were composed of 0.1%, 0.17%, 0.24%, and 0.34% of CNT/GNP (by weight of dry sand), respectively. In order to evaluate the water content effects on piezoresistivity behavior of the nano-intruded specimens, two specimens were made with different water content and the same CNM concentration as specimen GC (0.34%). These specimens, labelled GC (0.34+2%w) and GC (0.34-2%w), contained 2% more and less water than the optimum content, respectively.

5.2.4. Mechanical and Microstructural Characterization

The unconfined compressive strength test was used following the ASTM/D2166M standard for the evaluation of mechanical properties of cylindrical specimens. The results were calculated by the mean of at least 3 specimens. Because the modulus of elasticity for cementitious geomaterials is typically expressed by the modulus at 50% of the peak stress [181, 410-412], the Tangent $E_{(50\%)}$ of reinforced CSS by different CNMs concentrations was calculated at 50% of the maximum compression stress.

The effects of different CNM concentrations on maximum dry density and ω_{opt} were investigated according to the ASTM D697-78 standard. Thermal analysis and X-ray diffraction analysis (XRD) was conducted for the evaluation of cement hydration process [409]. The specimens' fracture surfaces were also investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectrometer EDX [145, 409]. Furthermore, an ultrasonic nondestructive test was performed for the microstructural investigation following the BS EN 12504-4 standard through two probes along the longitudinal axis. To evaluate the effects of the adversarial environmental conditions on physical and electrical performances of the nano-intruded specimens, a hybrid climatic cycle, including both freeze-thaw and drying-wetting cycles, as illustrated in Figure 5-4, was used. This cycle represents the harsh climatic conditions in deserts. The percentage of the specimens' weight loss and the ultrasonic wave passing time after 12 cycles were utilized as a criterion for determining the resistance of specimens to this climatic cycle. The

quantitative approach via Raman spectroscopy was utilized to investigate the defect status of CNMs evolved under the effect of climatic cycles. Raman analysis was performed on dry CNTs and GNPs using an excitation wavelength of a 514.5 nm (2.41 eV) Ar ion laser at room temperature.

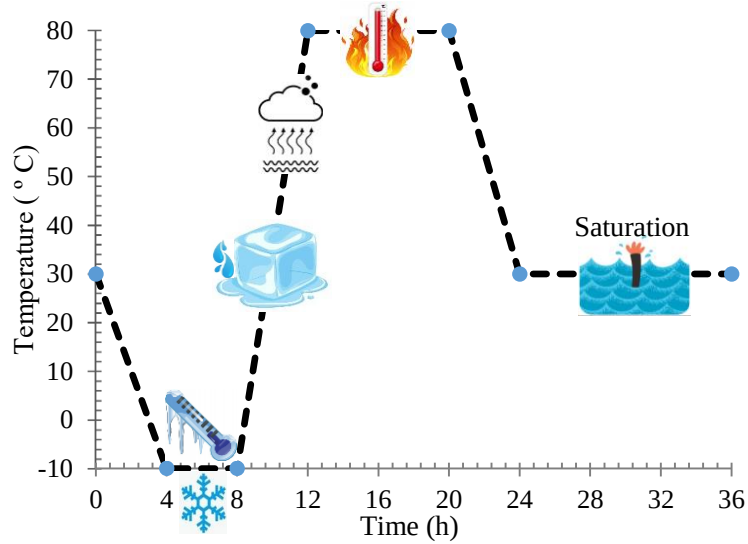


Figure 5-4. A schema of the climatic cycle protocol.

5.2.5. Piezoresistivity Measurement

To investigate the piezoresistivity performance of CNM-reinforced CSSs, the specimens with embedded electrodes were first dried at 70 °C for 72 hours after 28 days of hydration to ensure the absence of moisture effects on electrical conductivity values. As can be seen in Figure 5-3, a four-probe technique was used in this study to measure the fractional changes in electrical resistivity (FCR) under cyclic axial compression loading. In this circuit, a reference resistor of 100 Ω was connected to the outer probes in series powered by a direct current (DC) source with a constant value of 20 V. Power was supplied for 40 minutes, and it took between 25 and 30 minutes to stabilize the supply in CNM-reinforced CSS. Two digital multimeter were used to measure the voltage variation of the two inner probes and resistor. Three cycles of 4 kN axial compression loading at a rate of 40 N/s were used in order to evaluate CSS piezoresistivity behavior. The electrical resistivity $\rho(t)$ of each sample was obtained from the average of three voltage measurements and calculated by combining first and second Ohm's law equations (Eq. 1 and Eq. 2, respectively) as presented in Eq. 3 [396]:

$$R(t) = \frac{V_1(t)}{I(t)} = \frac{V_1(t)}{V_2(t)/100} \quad (\text{Eq. 1})$$

$$R(t) = \rho(t) \frac{L}{A} \quad (\text{Eq. 2})$$

$$\rho(t) = \frac{V_1(t)}{V_2(t)/100} \times \frac{A}{L} \quad (\text{Eq. 3})$$

where $V_1(t)$ and $V_2(t)$ are the inner probes and resistor voltage, respectively; $R(t)$ is the resistance between the two inner probes; $I(t)$ is the current between the outer electrodes; A is the contact surface between the electrode and CSS; and L is the spacing between the inner electrodes. For the following assessment of CSS piezoresistivity, the FCR was calculated by Eq. 4:

$$FCR = \frac{\rho(t) - \rho_0}{\rho_0} \quad (\text{Eq. 4})$$

where ρ_0 is the initial electrical resistivity measured before loading and $\rho(t)$ is the resistivity at time t during the test. To evaluate the sensitivity of CNT/GNP reinforced CSS, the gauge factor (GF) is also defined as the relative change in electrical resistivity over the strain (Eq. 5):

$$GF = \frac{FCR}{\varepsilon} \quad (\text{Eq. 5})$$

where ε is the applied strain along the force axis.

5.3 Results

5.3.1. Compaction Tests Results

The compaction curves of plain and reinforced CSS by different CNM concentrations are presented in Figure 5-5. As can be observed, the addition of 10% cement increased the maximum density of pure sand and reduced the optimum moisture.

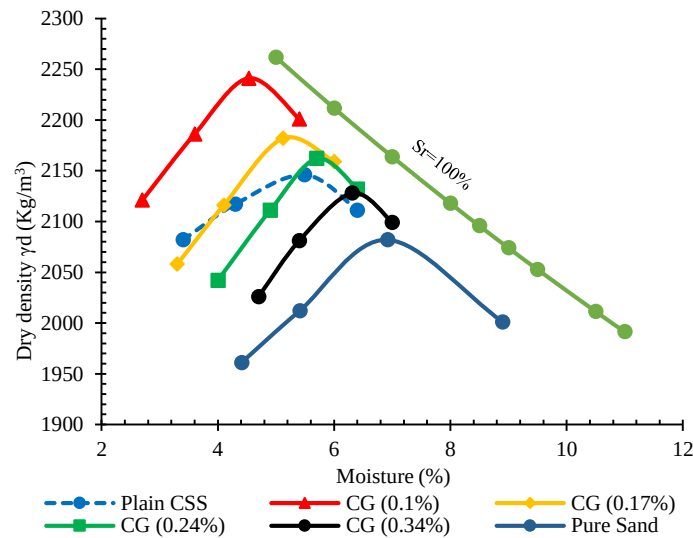


Figure 5-5. Compaction curves of plain and reinforced cementitious stabilized sand (CSS) by different carbon nanomaterial (CNM) concentrations.

Reinforcing CSS by different concentrations of CNM also caused similar trends. The incorporation of 0.1% hybrid CNT/GNP increased the maximum dry density to around 2241 kg/m³. However, specimens composed of 0.17% and 0.24% CNM also showed higher maximum dry density, they were accompanied

by a declining trend, such that an excessive increase in the CNM concentration beyond 0.24% led to a decrease in the maximum dry density compared to plain CSS. The results of the saturation degree (Sr) are also indicated in Figure 5-6.

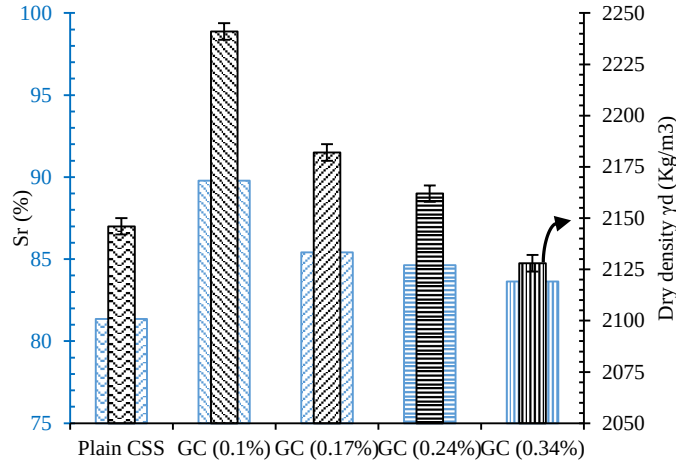


Figure 5-6. Maximum dry density and degree of saturation for plain and reinforced CSS with different CNMs concentration.

A similar trend to that of the compaction results was also observed for the saturation degrees. The augmentation of the density affected by CNM reinforcement decreased the void ratio, which consequently led to an increase in saturation degrees. The maximum saturation degree of around 89% was achieved via the incorporation of 0.1% CNM into the plain CSS, and an excessive increase in CNM concentration beyond 0.24% caused a decrease in saturation degree.

An interesting relationship between “ $\gamma_d/\gamma_{d(max)}$ ” and “ $Sr-(Sr)_{opt}$ ” was appeared, as illustrated in Figure 5-7. A similar trend has been reported in recent studies [413], indicating these laboratory results can be applied directly in the field. This is a major issue to support the use of these novel materials in practice.

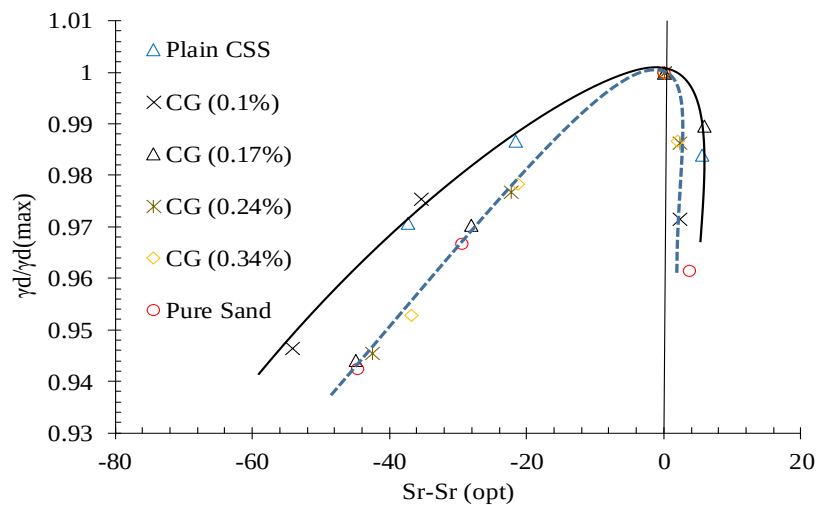


Figure 5-7. “ $\gamma_d/\gamma_{d(max)}$ ” vs. “ $Sr-Sr_{(opt)}$ ” relationship.

Although increases of maximum dry density caused by the addition of CNMs were less significant, optimum moisture reductions were considerable. Reinforcing CSS by 0.1% and 0.17% CNMs reduced the optimum moisture by about 21% and 14%, respectively, whereas excessive increase in the CNM percentage increased the optimum moisture by around 8% and 17% compared to plain CSS. However, the filler function of hybrid tubular CNTs and plate-shaped GNPs was not ineffective; this mechanism does not play a crucial role in the augmentation of the maximum dry density due to the low concentration of these nanomaterials. In fact, their role as a moisture regulator is more effective in increasing the slipperiness of cement and sand grains, and consequently CSS density improvement. CNTs and GNPs absorb and decrease the free water among the cement and sand grains, and also prevent the inhomogeneous distribution of water [144, 205]. In addition, these nano-scaled particles with a water-impregnated surface and high aspect ratio encourage the slipping and latching of cement and sand grains together (Figure 5-8).

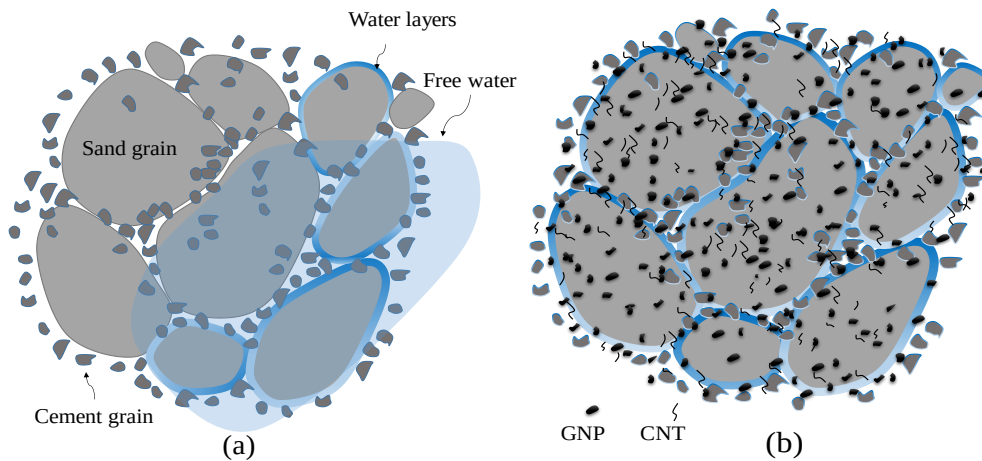


Figure 5-8. A schema of compacted: a) Plain CSS, reinforced, b) CSS by CNMs.

It should be noted that, in the case of high CNM concentrations, the required moisture increases abruptly, which can cause the gaps between the particles to reduce the maximum dry density. This amount of moisture is possibly converted to free water at high compaction energies. By starting the cement hydration process, the mechanical performance of CSS gradually increases due to the microstructural improvement. The results of ultrasonic non-destructive tests after 28 days of hydration period, as presented in Figure 5-9, also indicate this issue.

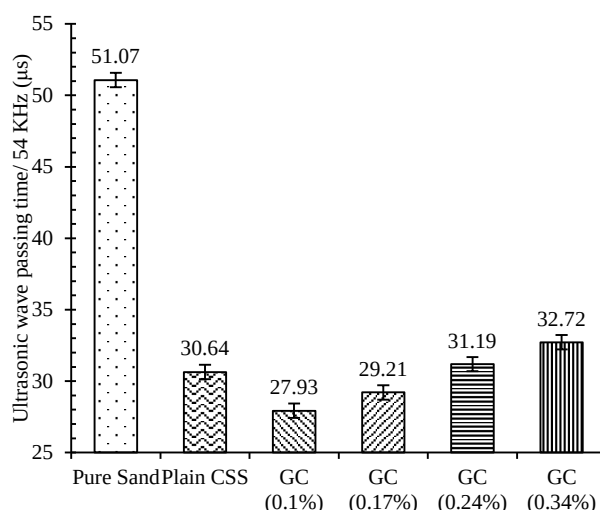


Figure 5-9. An ultrasonic wave passing time for plain and reinforced CSS with different CNM concentrations.

As can be observed, the ultrasonic wave passing time of compacted pure sand was reduced by about 40% by the addition of 10% cement after 28 days of curing. In the case of CNM-reinforced CSS, the passing times of the ultrasonic wave were also reduced by incorporating certain amounts of CNMs into the plain CSS due to a more cohesive microstructure which were in line with the above compaction tests outcomes and UCS results discussed in Section 3.3. The lower amount of ultrasonic wave passing time was achieved for CSS reinforced by 0.1% CNM, and excessive increase in CNM concentration beyond 0.24% increased the passing time compared to that of plain CSS.

Generally, reduction in optimum water content and increasing maximum dry density of the cementitious stabilized soil as a result of different CNMs addition, frequently has been reported. Alsharef et al.[414], reported a 13.6% decrease in optimum water content and a 1.5% increase in dry density by the dispersion of 0.075% MWCNTs into the clayey sand. For the soil sample reinforced by carbon nanofibers (CNFs), the maximum decrease in optimum water content was 14% at 0.075% CNFs content. Furthermore, dry density increased from 1.86 to 1.88 (g/cm³).

5.3.2. Microstructural Investigations

In general, reinforcing the cementitious composite by specific amounts of CNMs enhanced the physical performance due to three main mechanisms, such as filler function, bridging and/or divination of nano cracks, and increasing the cement hydration rate.

Although, incorporation of an optimum concentration of hybrid CNTs and GNPs with different 1D and 2D geometrical shapes can fill wider ranges of porosities and among the hydration products that led to their reinforcement, a high concentration of CNMs caused the formation of agglomeration and consequently increased the porosities (Figure 5-10).

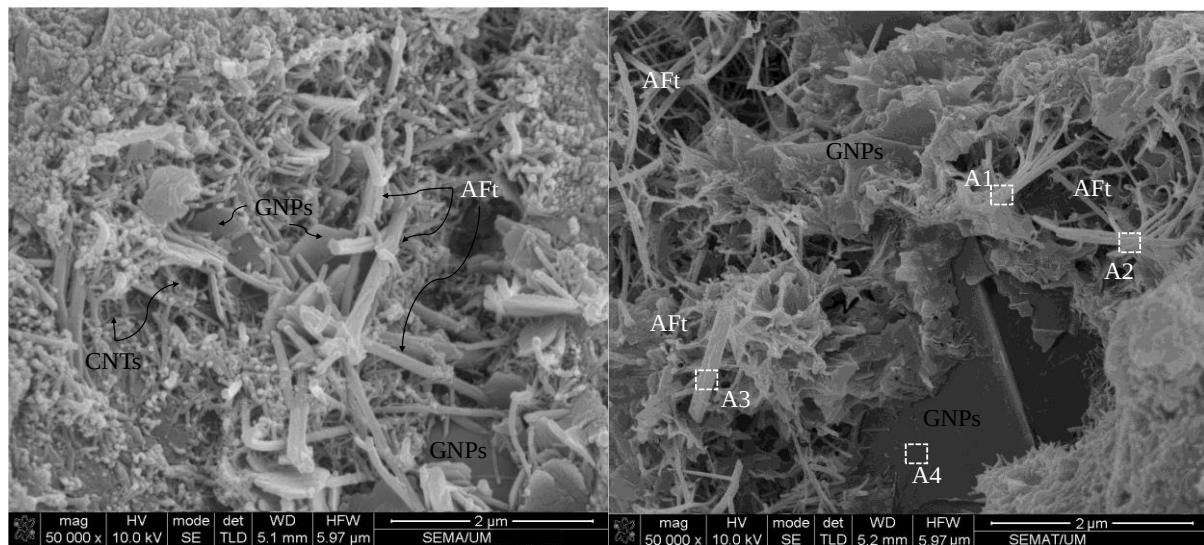


Figure 5-10. Scanning electron microscopy (SEM) images of the reinforced CSS by high CNM concentration CG (0.34%) (The areas with white markings are the areas selected for EDX analysis).

In addition, the EDX results, which are listed in Table 5-3, indicate that the needle-shaped crystals around the agglomerates have similar chemical composition to that of ettringite. The CNMs agglomerates are proper place in terms of ettringite formations due to the high quantities of accumulated water. The ettringite crystals have a needle-shaped, brittle, and porous structure, which can act as a starting point for micro-cracks, and consequently reduce the physical performance. In contrast, incorporation of an optimum concentration of hybrid CNTs and GNPs can prevent nano- and micro-scale cracks propagation via their bridging mechanism. Furthermore, these CNMs increase the required energy of crack expansion by the deviation of its path or direction (Figure 5-11).

Table 5-3. Cement hydration crystal chemical composites.

Position	Elements (%)									
	C	O	Ca	Al	Si	S	Mg	K	Fe	P
Figure 5-10 (A1)	8.72	36.53	35.81	3.67	10.47	1.29	0.52	0.19	0.29	0.61
Figure 5-10 (A2)	11.29	36.59	33.23	4.57	12.01	1.11	0.27	0.37	0.15	0.41
Figure 5-10 (A3)	9.3	38.44	31.73	6.18	11.71	1.95	0.43	0.11	0.27	0.24
Figure 5-10 (A4)	81.31	4.27	7.91	-	5.72	-	-	-	-	0.79

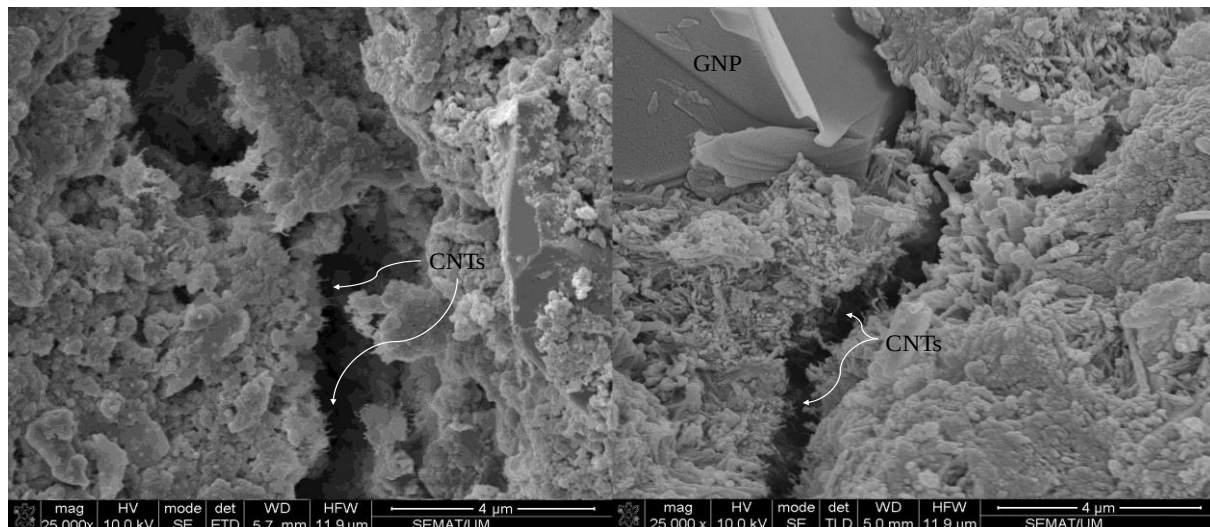


Figure 5-11. SEM morphology of CNTs and GNPs crack bridging and deviation mechanism in CNM-reinforced CSS.

5.3.3. Cement Hydration Evaluation

Tricalcium silicate (Ca_3SiO_5 , abbreviated C_3S), dicalcium silicate (Ca_2SiO_4 , C_2S), tetra calcium aluminoferrite ($\text{Ca}_4\text{Al}_n\text{Fe}_{2n}\text{O}_7$, C_4AF), tricalcium aluminate ($\text{Ca}_3\text{Al}_2\text{O}_6$, C_3A), and small amounts of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and clinker sulfate (Na_2SO_4 , K_2SO_4) are the main components of the chemical composition of cement grains in the anhydrous state [409]. C_3A , C_3S , C_4AF , and C_2S , react with water following specific chemical reactions during the hydration process to produce monosulfonate (AFm), hydrate of calcium silicate (C-S-H) gel, ettringite (AFt), and calcium hydroxide (CH) [201, 415]. During these reactions, the formation of CH, C-S-H, AFt, and AFm with various geometries and crystal shapes, as the most influential hydration products, plays a key role in determining the final properties of the cement and, eventually, by the evolution of the hydration process, they form a dense microstructure [349]. One of the major techniques used for the investigation of cement hydration products is thermogravimetric analysis (TGA). The results of TGA tests are illustrated in Figure 5-12 (a). Besides the results of differential scanning calorimetry (DSC) have been presented in Figure 5-12 (b) in order to better observe the temperature range associated with each weight decay. A range of weight losses for the powder of cementitious composite samples was observed in the TGA curves between 105 and 1000 °C, as found in previous studies [416, 417].

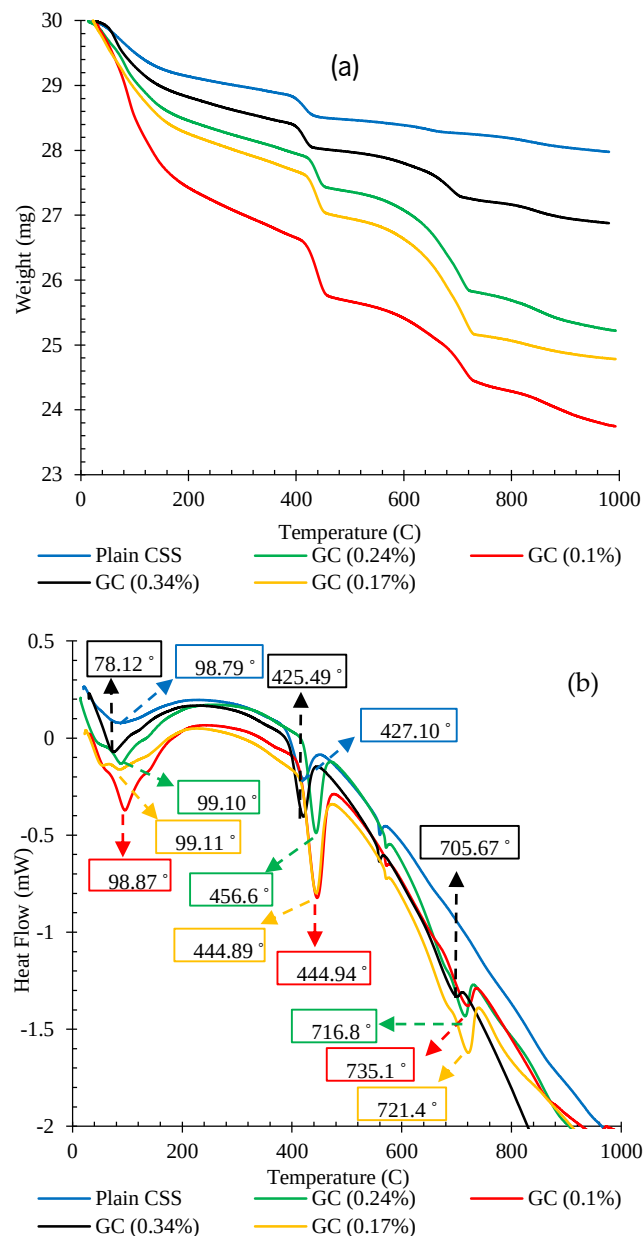


Figure 5-12. Thermal analysis of plain and reinforced CSS by different concentrations of CNMs: a) TGA thermograms, b) DSC thermograms.

From the TGA thermogram, the first decrease was up to 105 °C, which can be attributed to the elimination of sample-free water and moisture of CSS. Due to the dehydration of chemically bound water existing in hydration materials such as ettringite, C-A-S-H, carbo-aluminates, and C-S-H, the second decline in the TGA curves occurred between 105 and 400 °C. The next big decline was detected between 400 and 550 °C, which can be linked to the CH dehydroxylation. The fourth and final decay was due to calcium carbonate, and clinker carbonation was found between 600 and 800 °C [418, 419].

Results showed that the incorporation of an optimum concentration of CNMs into the CSS increased the hydration products and specifically raised the amount of the CH and C-S-H gel [420, 421]. The maximum quantity of cement hydration products was achieved by the addition of 0.1% hybrid CNT/GNP, and excessive increase in the CNM concentration caused the reduction in hydration. The positive effects of CNMs on the cement hydration process have been reported in previous studies [422, 423]. CNMs can increase and accelerate the formation of numerous cement hydration products owing to their nucleation agent effects and their surficial functional groups, thereby resulting in strong bonding performance between CNMs and the cementitious matrix [409]. Comparing the results of this study with previous studies which used similar CNMs shows that in samples made by compaction method, the higher amount of hydration products was obtained in a lower dosage of this nanomaterial. This can be directly related to the reduction of water content in this fabrication method [409].

It should be noted that the excessive increase in CNM concentration absorbs the water needed for the cement hydration process, which reduces the hydration products and, in some cases, stops the hydration process of the cement. As mentioned in this study PF-127 was used in order to the dispersion of the CNMs. The molecular structure of the PF-127 composed of a central hydrophobic chain of polyoxypropylene (PPO) and two hydrophilic chains of polyoxyethylene (PEO) placed on two sides. The PPO chains are absorbed on the surface of the CNMs during specific reactions and PEO chains also trap water molecules on the surface and between layers of CNMs and their agglomerates [145].

The XRD analysis result of hardened plain CSS and CSS reinforced by different CNM concentrations, presented in Figure 5-13, also showed a similar trend. As can be observed, the amount of the hydration product was increased by the incorporation of a certain concentration of CNM (0.1%) while excessive increase in CNMs caused a reduction in CH and C-S-H, in addition to increasing unhydrated cement components, such as C2S and C3S.

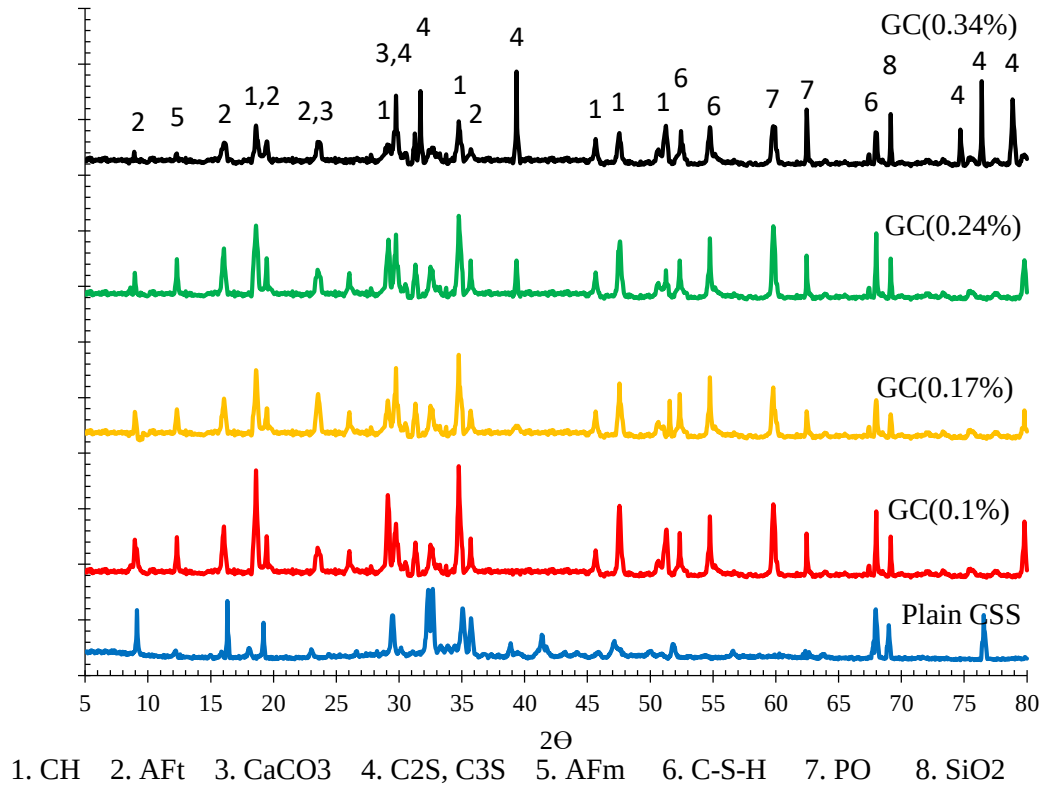


Figure 5-13. X-ray diffraction analysis (XRD) patterns of hardened plain and reinforced CSS by different CNM concentrations.

5.3.4. Mechanical Characterizations

The results of axial unconfined compressive strengths in addition to the axial strain at failure (maximum stress) and the tangent modulus at 50% of the peak stress ($E_{(50\%)}$) for plain and reinforced CSS by different CNM concentrations are illustrated in Figure 5-14 (a) and (b).

As can be seen, incorporation of 0.1%, 0.17%, and 0.24% CNMs into the CSS caused an increase in UCS of about 65%, 31%, and 14%, respectively, compared to the plain CSS. While excessive increase in the CNM concentration beyond 0.24% to 0.34% led to reduction in UCS by around 13%.

Furthermore, results showed that reinforcing CSS by specific CNM concentrations caused to reducing the ductility of specimens, such that the addition of 0.1%, 0.17%, and 0.24% CNM increased the $E_{(50\%)}$ of plain CSS by about 48%, 32%, and 16%, respectively. The specimen GC (0.34%), which was composed of a high CNM concentration, did not show a noticeable change in the amount of rupture modulus.

Concerning the failure strain, a similar trend was observed. However, incorporation of 0.1%, 0.17%, and 0.24% of CNM into the CSS reduced the maximum strain by around 25%, 19%, and 9%. Excessive increase in the CNM concentration up to 0.34% caused an increase in maximum strain of about 6%. Indeed, reinforcing cementitious composites by CNMs reduced their ductility and increases their stiffness.

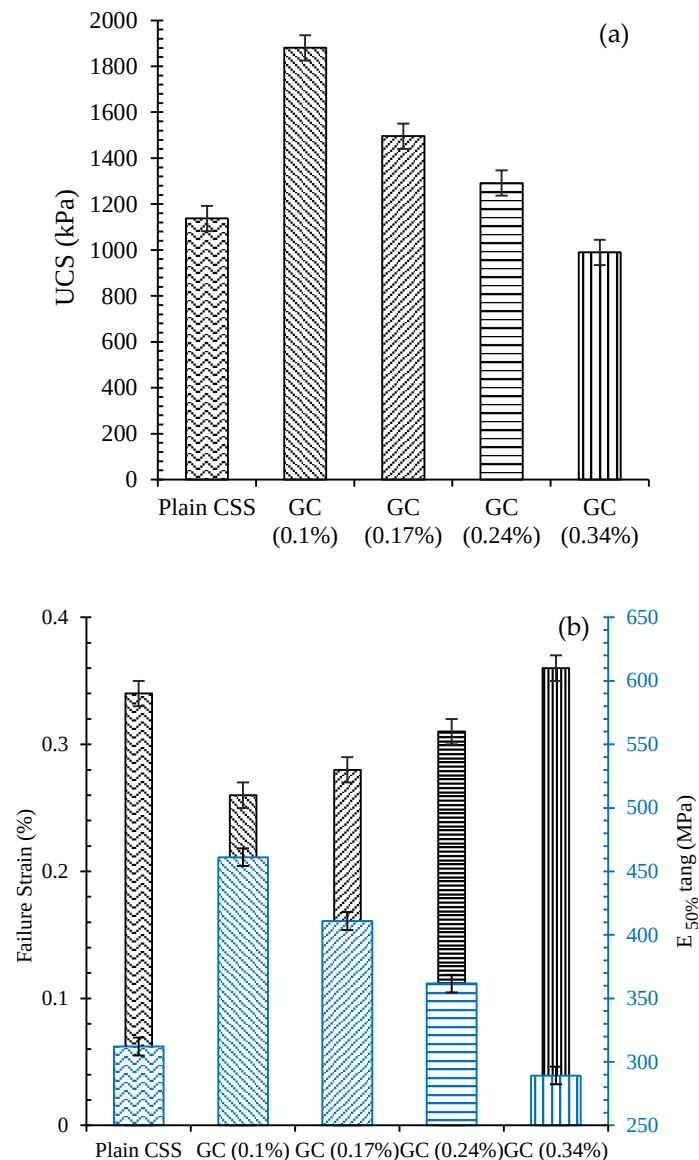


Figure 5-14. a) Unconfined compressive strengths of different CSSs, b) the amount of strain at the rupture point and $E_{(50\%)}$ after 28 days of hydration.

Correia et al.[181] also reported an increase in UCS and $E_{(50\%)}$ by around 65% and 145% respectively for clayey sand which compacted by 11% and 1% cement and MWCNT respectively.

5.3.5. Durability Investigation

The percentage of the specimens' weight loss and their ultrasonic wave passing time after 12 climatic cycles, as the criteria of the specimens' resistance and durability against the climatic cycle, is illustrated in Figure 5-15. As can be observed, the plain specimen showed a weight reduction of around 4.2%, whereas this amount for specimens composed of 0.1%, 0.17%, and 0.24% CNM was 2.4%, 3.2%, and 3.5%, respectively. These results show the positive effects of CNMs in terms of durability augmentation of cemented sand against harsh climatic cycles. However, increasing the concentration of the CNMs beyond 0.1% caused a reduction trend for the specimens' resistance to the climatic cycle; in such a way that the

incorporation of 0.34% CNM caused a greater weight loss than that of the plain specimen, equal to 4.51%. The dense and highly cohesive microstructure of the CSS caused by the intrusion of specific CNM concentrations increased the resistance of the sample to decay, contractions, and expansions caused by the climatic cycle. The results of the ultrasonic non-destructive tests also show a similar trend. The ultrasonic wave passing time along the longitudinal axis of the plain specimen was around 35.5 μs after 12 cycles, whereas the passing times for the specimens containing 0.1%, 0.17%, 0.24%, and 0.34% CNM were obtained 30.1, 32.2, 34, and 37.1 μs , respectively.

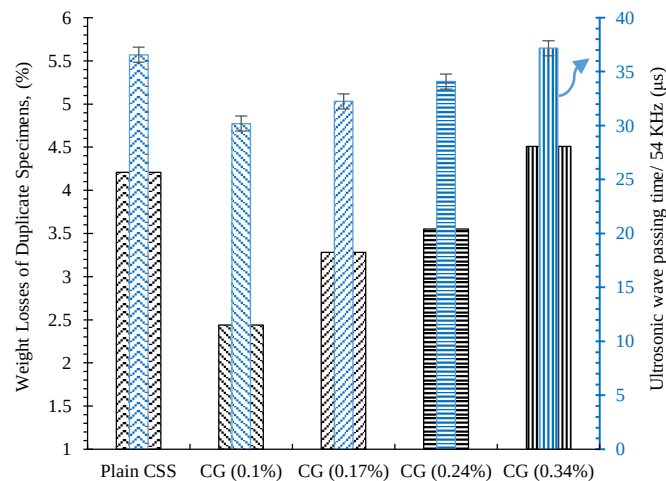


Figure 5-15. Weight loss and ultrasonic wave passing time after 12 climatic cycles.

5.3.6. Piezoresistivity Behavior Investigation

Electrical conductivity

The electrical resistivity of plain and reinforced CSS by different CNM concentrations is shown in Figure 5-16. As expected, the electrical conductivity of the CNM-reinforced specimen increased significantly due to the conductive path formation by percolation and electron tunneling mechanisms.

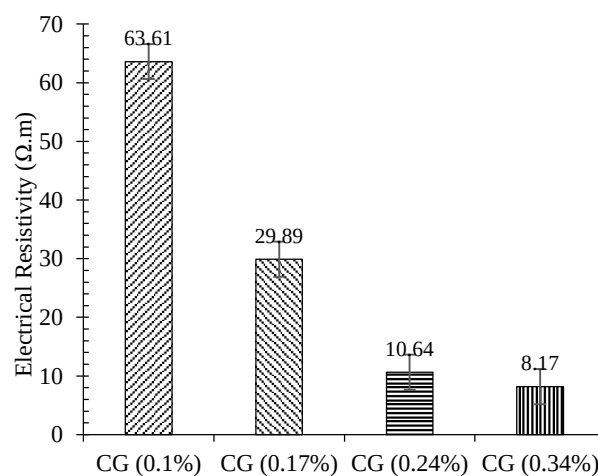


Figure 5-16. Electrical resistivity of hardened plain and reinforced CSS by different CNM concentrations.

Increasing the carbon concentration continuously increased the conductivity of the composite in such a way that the electrical resistivity of specimens composed of 0.1%, 0.17%, 0.24%, and 0.34% CNM decreased by around 46%, 74%, 92%, and 95%, respectively, compared to the plain CSS. According to these results, the percolation threshold for this type of reinforced CSS could be around 0.24% due to the insignificant effect of CNM on reducing electrical resistivity by its increment beyond 0.24%.

By comparing the results with other studies that used similar types of sand, cement, dispersion method, and CNM in order to investigate piezoresistivity and electrical conductivity of cementitious mortar ($W/C = 0.5$) [145, 207], it can be observed that decreasing the cement concentration in CNM-reinforced cementitious composite led to the reduction in electrical resistivity. Comparing these results also showed that the use of compaction fabrication method can reduce the electrical resistivity by up to 80%.

Effects of water content on electrical conductivity

The electrical resistivity of the specimens with different water content and the same CNM concentration are shown in Figure 5-17. These specimens were composed of 0.34% CNM and fabricated with optimum, 2% more than optimum, and 2% less than optimum water content.

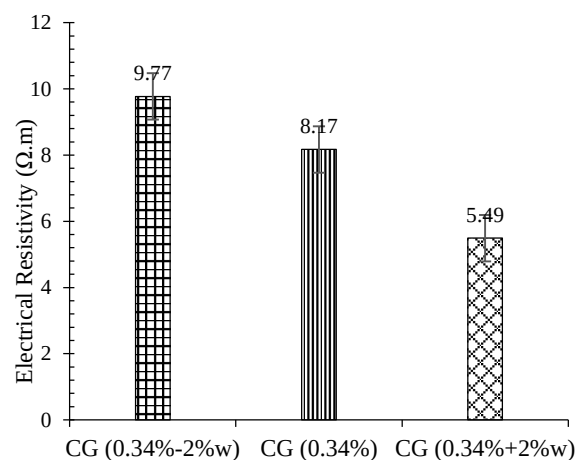


Figure 5-17. The electrical resistivity of the specimen CG (0.34%) at optimum, 2% more than optimum, and 2% less than optimum water content.

As shown in Figure 5-17, the electrical resistivity of the specimens containing 2% of water more than the optimum concentration was reduced around 33% compared to the specimens fabricated at the optimum water content, while for the specimen composed of 2% water less than the ω_{opt} the electrical resistivity was increased about 20%. Generally increasing the water content assists CNM dispersion, which causes an increase in electrical conductivity [313, 332, 424]. In addition, increasing the water content increased the formation of ettringite (AFt) crystals, which are filled with water molecules and charged ions. These

needle-shaped crystals also increase the electron tunneling effect and percolation mechanism, which reduce the electrical resistance.

Effects of climatic cycles on electrical conductivity

The electrical resistivity of the reinforced specimens with different concentrations of CNM after climatic cycles is shown in Figure 5-18. By comparison with the results of the electrical resistivity of the specimens before climatic cycles (Section 3.5.1.), it is clear that the electrical conductivity of the samples drastically decreased following the climatic cycles.

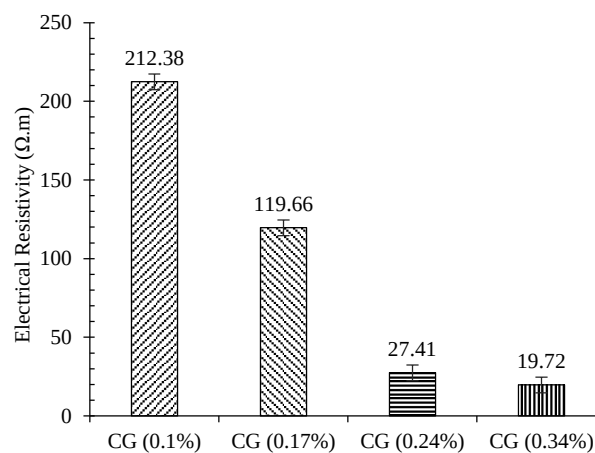


Figure 5-18. Electrical resistivity of the reinforced specimens with different concentrations of the CNM after climatic cycles.

As can be seen, the electrical resistivity of the specimens composed of 0.1%, 0.17%, 0.24%, and 0.34% CNM increased by around 5, 3, 1.5, and 1.4 times, respectively, compared to the normal specimens. Destructive effects of wetting–drying combined with freeze–thaw cycles on the conductive system, including CNMs and free charged ions, are the major reason for the decrease in the electrical conductivity of the composite.

The morphology of CNMs after climatic cycles at different magnification is illustrated in Figure 5-19. The SEM images clearly indicate the surficial structure of GNPs suffered from relatively severe degradation and damage. The surface texture of GNPs and their layers disintegrated due to the destructive effects of hybrid wetting–drying and freeze–thaw cycles, which caused a reduction in the electrical conductivity of the composite. In addition, EDX analysis was performed for CNMs, and their chemical compositions are listed in Table 5-4

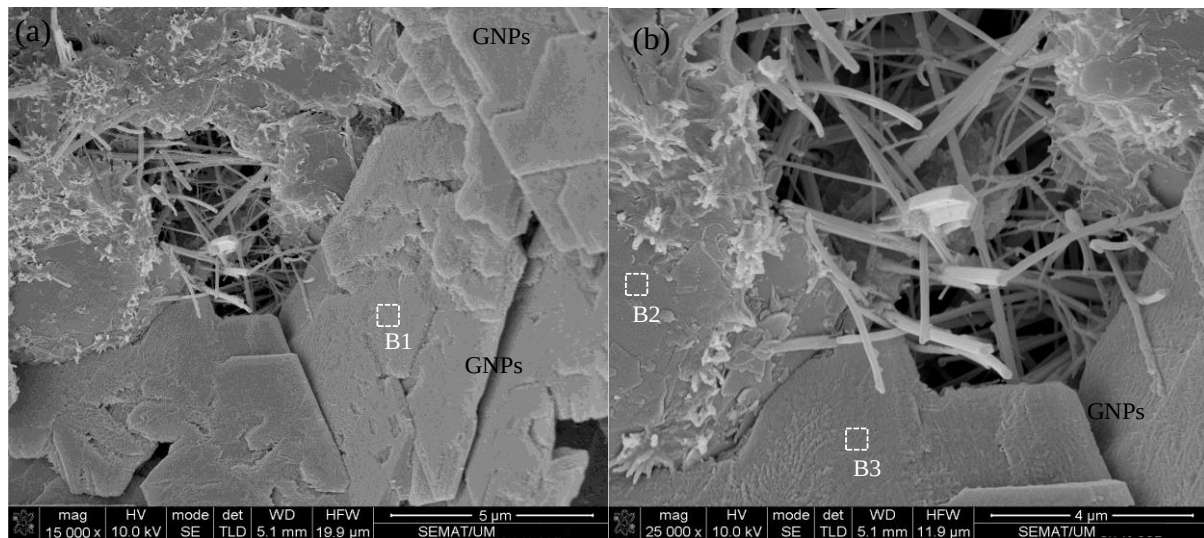


Figure 5-19. CNM morphologies after climatic cycles (the areas with white markings are the areas selected for EDX analysis).

Table 5-4. CNMs' chemical composition.

Position	Elements (%)									
	C	O	Ca	Al	Si	S	Mg	K	Fe	P
Figure 5-19 (B1)	29.46	37.61	15.7	4.81	8.15	2.64	0.71	0.14	-	0.83
Figure 5-19 (B2)	34.51	35.18	14.64	3.19	7.12	3.91	0.29	0.18	0.27	0.71
Figure 5-19 (B3)	32.77	39.41	11.92	3.89	7.51	3.24	0.42	0.12	0.15	0.58

The results show that the quantity of carbon element in GNP chemical compounds was greatly reduced by climatic cycles compared to the normal specimens (Table 5-3, Position A4). However, increasing the oxygen concentration is evidence of GNPs' excessive oxidation, which also caused GNPs to be surrounded by other elements and ions, such as Fe, Mg, Ca, Si, S, Al, and K that can eliminate the free capacity of the π -electron in GNPs and CNTs. It should be noted that the π -electron plays as a crucial rule in the electrical conductivity of the CNMs [425, 426]. Raman spectroscopy was also carried out on CNTs and GNPs in order to more accurately investigate the CNMs after climatic cycles; and its analysis results are shown in Figure 5-20.

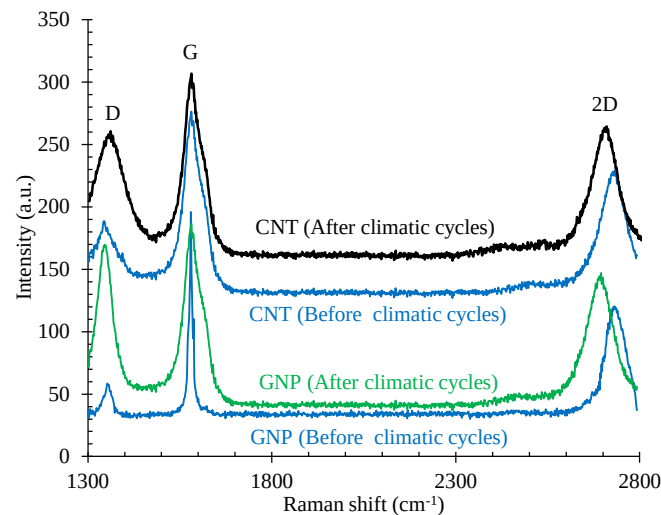


Figure 5-20. Raman spectra of the CNMs after climatic cycles.

In the Raman spectra of CNTs and GNPs, the D band refers to the presence of defect concentration and disturbance in C-C bonds, which are also referred to as non- sp^2 (sp^3) structural defects. Indeed, in the perfect hexagonal sp^2 -based carbon crystal structure, the D band is not as high but, owing to the disturbance of the aromatic system, it is active for defective and small layers [218, 427]. The G band in GNPs and CNTs defines a graphitic crystallinity state that can be ascribed to the C-C bonds' tangential vibration (sp^2 hybridization) and upshift to high wave numbers under the chaotic state [428]. Eventually, the 2D band is an overtone of the defect-related D band and purity grade of the sample, which is specified with its corresponding intensity and broadening [429, 430]. Hence the variations in the characteristics of G, D, and 2D in Raman spectra represent the evolution of structural specification [218]. As can be seen, the intensity of the D and 2D band in both CNMs and GNPs after hybrid climatic cycles was significantly increased, which testifies to the presence of significant (sp^3) structural defects and the reduction of the sp^2 domain (La). This can greatly reduce the inherent electrical conductivity of CNTs and GNPs [218].

Piezoresistivity investigations

The fractional change in resistivity against the axial strain under the cyclic compression loading for reinforced CSS by different CNM concentrations is indicated in Figure 5-21.

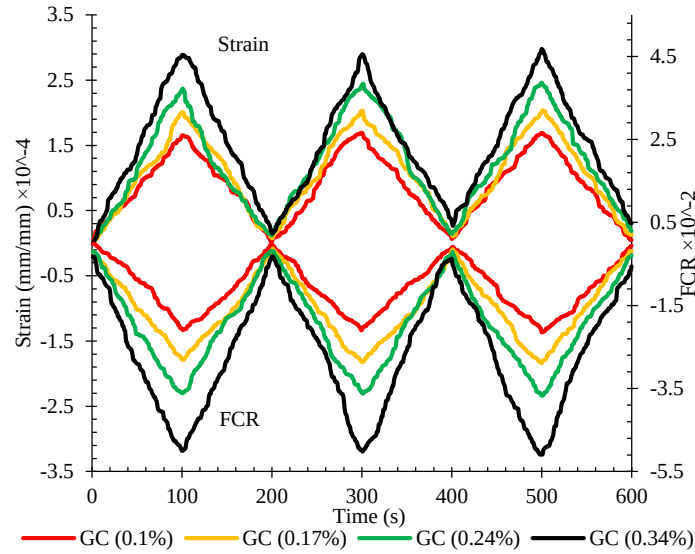


Figure 5-21. The fractional change in resistivity and axial strain under cyclic compression loading for reinforced CSS by different CNM concentrations.

As can be seen, the negative values were achieved for FCR. In compression loading, the CNM particles and conductive paths become closer to each other, which consequently decreases the second resistance and caused negative values for FCR following Eq 4. The FCR amounts did not reach zero at the end of each loading cycle and a negligible amount of FCR remained after unloading, which is consistent with the strain remaining at the end of each loading cycle and was increased by increasing load cycles. These remaining amounts were lower than those of previous studies [207] due to the compaction fabrication methods. Variation of the strains with the fractional changes in resistivity for reinforced CSS by different CNM concentrations is also illustrated in Figure 5-22. A power function with a proper approximation was utilized to express the relationship between strain and FCR. Generally, the slopes of the diagrams consist of two parts.

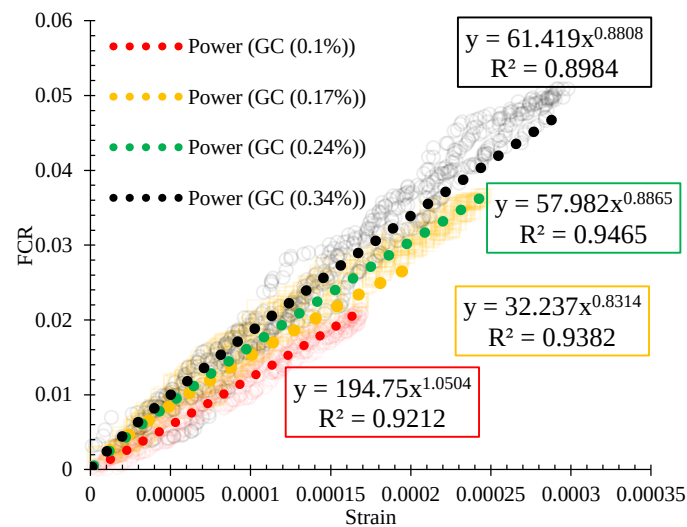


Figure 5-22. Variation of strain with the FCR for reinforced CSS by different CNMs concentrations.

Although the slopes of the first part were increased by the increase in the CNM concentration, the specimens composed of lower CNM dosage showed higher slopes in the second part. By increasing the CNMs concentration beyond the percolation threshold, the sensitivity of the composites against the high amount of stress and/or strain was decreased due to the saturation of the composite structure from the conductive paths. By comparing these results with previous studies, it can be seen that the scatters of the data have been decreased [207].

Effects of water content on piezoresistivity performance

The fractional changes in electrical resistivity vs the axial strain for specimens CG (0.34%) fabricated with $\pm 2\%$ of water relative to the optimum water content, under the cyclic compression loading have been shown in Figure 5-23.

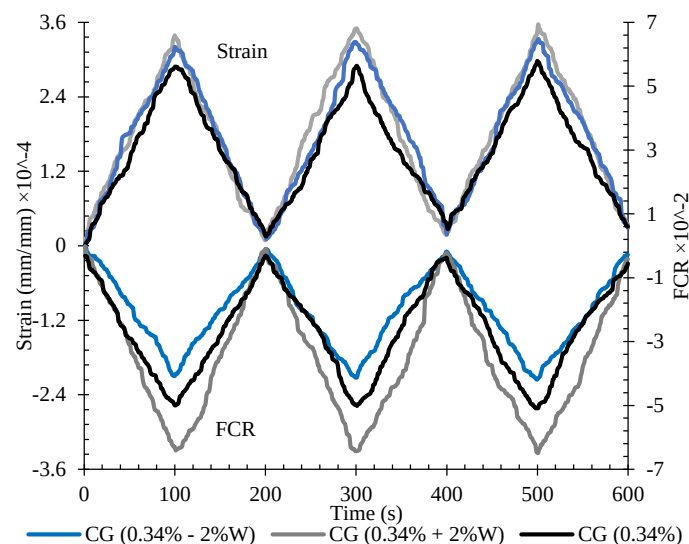


Figure 5-23. The fractional change in resistivity together with axial strain under cyclic compression loading for specimen CG (0.34%) at optimum, 2% more than optimum, and 2% less than optimum water content.

The results showed that fractional changes in electrical resistivity were increased by increasing the water content while decreasing water content caused a reduction in fractional changes in electrical resistivity. As mentioned, increasing the water content in addition to enhancement of the dispersion of nanoparticles and improving conductive behavior by ionic conduction, also increase the amounts of strain due to higher amounts of porosities among the microstructure of the specimen (Figure 24). Hence, the conductive paths are more affected and eventually leads to more fractional changes in the electrical resistivity.

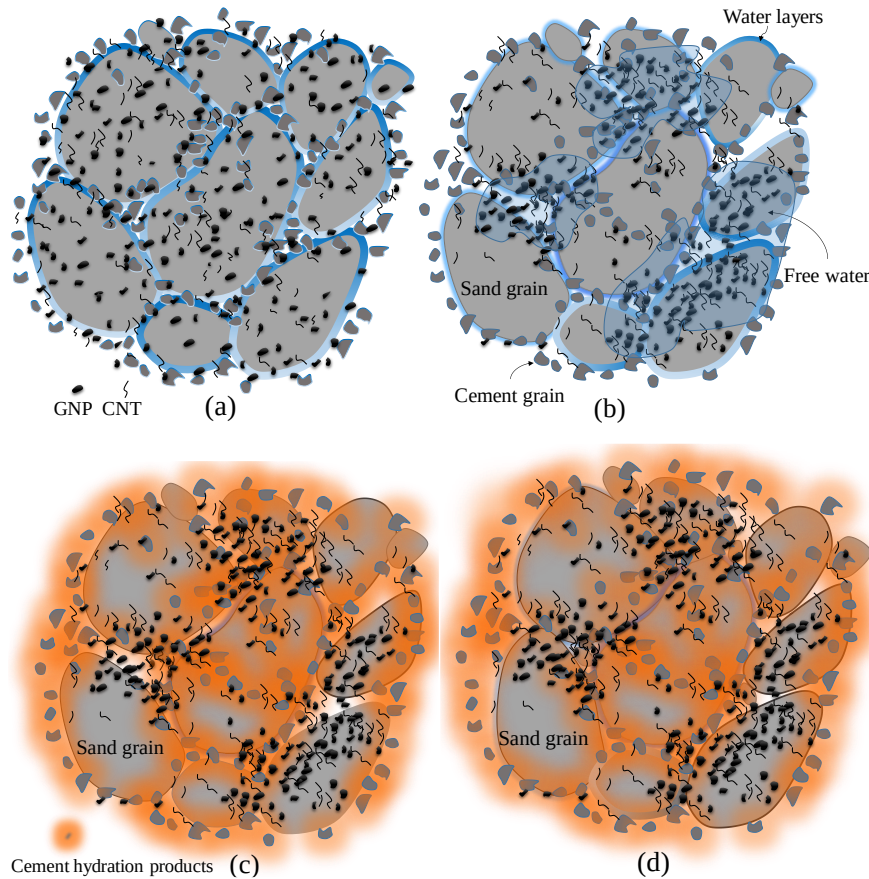


Figure 5-24. A schema of the compacted specimen composed of the CNMs: a) fresh sample at optimum water content, b) fresh sample at the higher or lower water content c) hardened specimen at the higher or lower water content d) hardened specimen at the higher or lower water content, subjected to the compression loading.

Although in specimens composed of lower water content, the grains are not well locked together and the amount of strain is also larger due to the high amount of porosity, the fractional changes in electrical resistance are lower due to the poor ionic conduction and dispersion of CNMs. As indicated in Figure 5-24, increasing the water concentration until the optimum, increases density of the samples and after it, a decrease in density is observed. the presence of the extra water content between the grain causes gaps between them and finally lead to porosities formation. By increasing effective stress or after drying, the free water will be eliminated and ultimately cause the strain to increase. In the case of lower water content, the lack of water required for lubrication prevents grains from locking properly in each other, which finally leads to the formation of porosity and increased strain when the specimen is subjected to the load. However, in low water content, the formation of the porous and needle-shaped crystals of ettringite is lower which can lead to lower strain compared to the higher water content cases.

Variation of strain against the fractional changes in electrical resistivity for specimen contained 0.34% of CNMs at optimum, 2% more than optimum, and 2% less than optimum water content, have been illustrated in Figure 5-25.

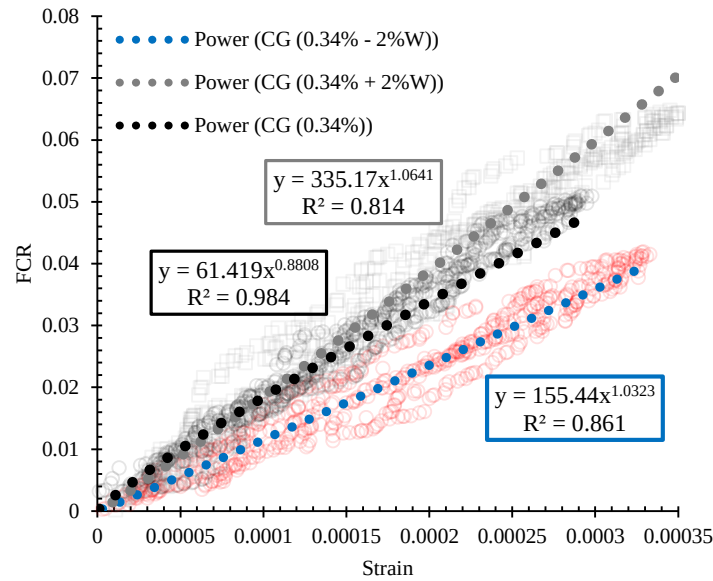


Figure 5-25. Variation of strain with the FCR for specimen CG (0.34%) at optimum, 2% more than optimum, and 2% less than optimum water content.

As can be seen, the slope of the diagram for the specimen composed of higher water content is steeper than specimens fabricated at optimum and lower water content which is evidence of its higher sensitivity. Additionally, the data scatter of the specimen CG (0.34%) is lower compared to the other specimens. Besides, the specimens contained higher water content showed higher data scatter which may be due to its porous microstructure.

Effects of climatic cycles on piezoresistivity performance

The fractional change in resistivity vs strain for nano intruded specimens after climatic cycles have been shown in Figure 5-26. As can be seen, the specimen CG (0.1%) didn't show the piezoresistivity behavior after climatic cycles. In addition, the fractional changes in electrical resistivity of other specimens were decreased significantly. However, the amounts of their strain at the peak of loading and end of the loading were significant growth.

Despite such circumstances, increasing the CNMs concentration also caused increasing of the fractional changes in electrical resistivity as well as peak and residual strains.

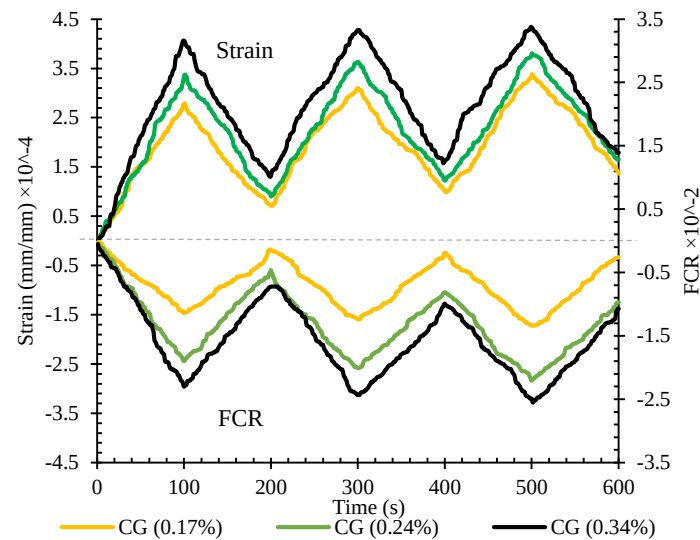


Figure 5-26. The fractional change in resistivity together with axial strain under cyclic compression loading for reinforced CSS by different CNMs concentrations after climatic cycles.

Generally, the destructive effects of the climatic cycles can be categorized into two main groups of adverse effects on composites' microstructure, and inherent properties of CNMs. The effects of successive expansion due to water freezing along with the decay of hydration products due to frequent wetting-drying cycles, reduce the cohesiveness of composite microstructure and cause its deterioration and nano cracks formation.

This feeble matrix with defective microstructure does not have the needful strength to maintain conductive paths and by increasing the load a sudden interruption occurred in conductive paths. Meanwhile, as mentioned in section 3.5.3, the electrical conductivity of the CNMs have been extremely reduced due to the severe weather cycle. The relation between fractional changes in electrical resistivity of specimens and this axial strain after climatic cycles, under compression loading, has been illustrated in Figure 5-27.

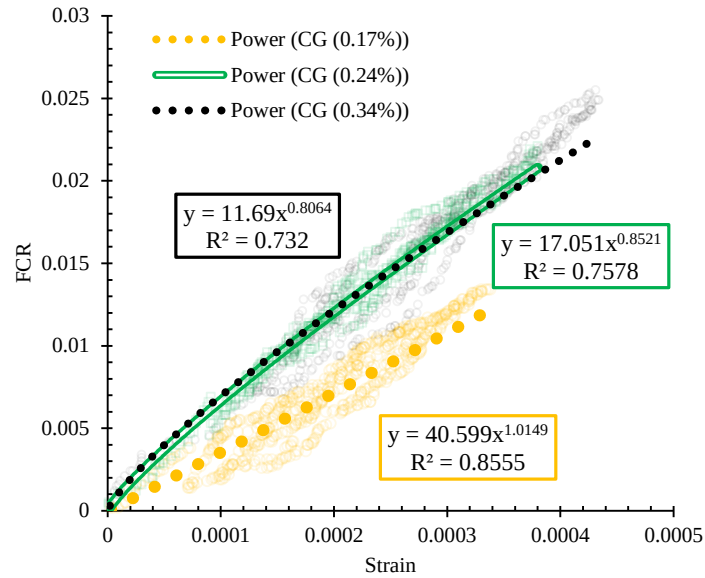


Figure 5-27. Variation of strain with the fractional changes in electrical resistivity (FCR) for nano-intruded specimens after climatic cycles.

As shown, increasing CNM concentration resulted in a steeper slope. However, the slopes of the specimens composed of 0.24% and 0.34% CNM were almost equal. Furthermore, the scattering of the data was increased compared to the normal nano-intruded specimens (Figure 5-22).

Gauge factor

Variation of the gauge factors for reinforced CSS with different CNM concentrations and water content, in normal conditions and also after climatic cycles, is shown in Figure 5-28. Results indicate that, in normal conditions, increasing the CNM concentration raised the gauge factors, which shows the sensitivity improvement of the specimens against the strain. Generally, increasing the CNM concentration from 0.1% to 0.17%, 0.24%, and 0.34% led to the enhancement of the gauge factor by around 13, 22, and 40%, respectively.

Comparing these results with previous studies which used hybrid CNT/GNP for reinforcing mortar [207], showed that the use of the compaction fabrication method can somewhat reduce the sensitivity and gauge factor.

As shown in Figure 5-28(b), increasing water content increased the gauge factor; increasing water content by 2% more than optimum content increased the gauge factor of specimen CG (0.34%) around 10%, whereas reducing the water by 2% compared to the optimal value reduced the gauge factor by 34%.

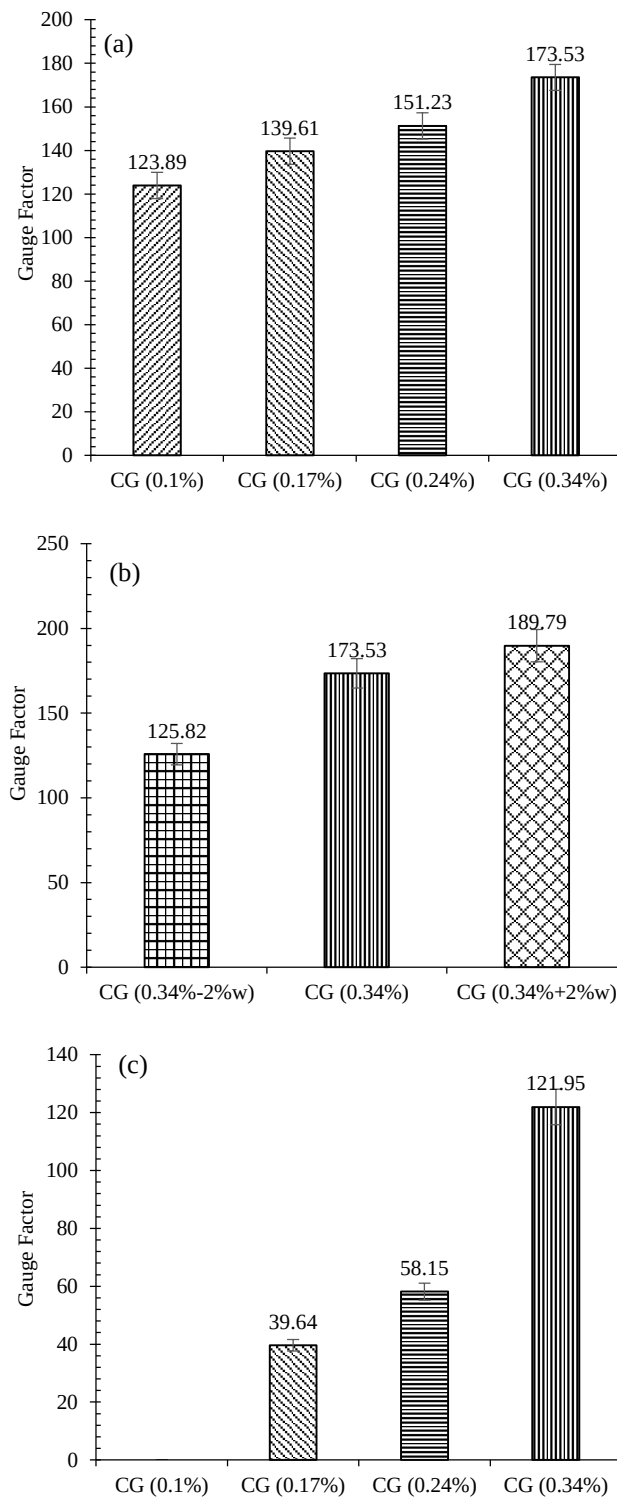


Figure 5-28. Variation of CNM reinforced specimens' gauge factors under compression loading: a) in normal condition, b) with different water content, c) after climatic cycles.

Investigation of the specimens' gauge factors after climatic cycles (Figure 5-28(c)) clearly shows that the gauge factors decreased compared to those of the normal case due to the destructive effects of the climatic cycle. Indeed, the gauge factors of the specimens composed of 0.17%, 0.24%, and 0.34% CNM reduced by 72%, 61%, and 30%, respectively, compared to the normal specimens.

5.4 4. Conclusion

This study was a systematic effort to develop a novel self-sensing cementitious geocomposite (cementitious stabilized-sand, (CSS)) based on piezoresistivity behavior with high physical and mechanical performance.

The series of tests carried out on specimens with different concentrations of hybrid CNTs and GNPs (1:1) dispersed into cementitious stabilized sand (10% of cement of dry weight of the sand), compacted at the optimum water content, revealed that 0.1% of carbon nanomaterials was the optimum concentration to achieve the best mechanical behavior (strength and stiffness), the maximum electrical resistance and enhanced durability of the geocomposite against severe climatic cycles, including freeze-thaw and wetting–drying. These results were explained by the microstructure of the geocomposite (SEM, EDX, XRD, and TGA tests) and showed the combination of 1D and 2D carbon nanomaterials (CNMs), in addition to filling the nano and micro porosities, can bridge and/or deviate the cracks, which can prevent their expansion. However, reinforcing CSS by CNMs generally increased the modulus and consequently reduced the ductility of the composite. Although reinforcing the CSS by 0.1% hybrid CNT/GNP increased the rate of the cement hydration due to their nucleation effects, excessive increase in the CNM concentration reduced hydration products due to the required water absorption of the hydration process.

The outcomes of this study clearly showed the significant benefits of the compaction method, which is representative of transport and energy infrastructure works, in terms of mechanical and piezoresistivity performance enhancement, compared with mixing procedures typically used for cementitious composites. In summary, we believe that this novel approach contributes to the new era of smart composite materials in intelligent structures.

Chapter 6. Effects of The Fibers on Physical, Mechanical, and Piezoresistivity Performances of Cementitious Geocomposite.

«The journey of a thousand miles begins with a single step. »

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

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Abstract

This study is a systematic attempt to develop a self-sensing fiber-reinforced cemented sand with high physical, mechanical, durability, and piezoresistivity performances. In this route, different concentrations of Dyneema, glass, and polypropylene (PP) fibers were incorporated into cemented sand (CS) containing 0.17% hybrid carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs). The specimens were fabricated using the standard Proctor compaction method and tested at the optimum water content. The mechanical, microstructural, and durability performances of the specimens were evaluated through various types of tests. Further, the piezoresistivity of the specimens was evaluated under compression cyclic loads using the four probes method. The incorporation of 1.0% glass and Dyneema fiber as the optimum percent increased the unconfined compression strength (UCS) (29% and 82%, respectively) and the maximum dry density of the CS; however, reinforcing of the specimens with PP fiber at a concentration in the range of 0.5% to 1.5% generally reduced the UCS of the specimens. The pullout test results exhibited a considerable interfacial performance for the Dyneema fiber. The CS reinforced with 1.0% Dyneema and glass fiber demonstrated a lower weight loss after 12 wetting and drying cycles compared to other specimens. The maximum gauge factors were also achieved for Dyneema fiber-reinforced CS. The outcomes of this study, balanced with sustainable issues, contribute to the development of the new era of smart structures, with applications to roller-compacted-concrete dams, rammed earth, and particularly in structural layers in transportation infrastructure.

6.1 Introduction

Currently, various structural health monitoring techniques, i.e., electric strain gauges, PZT-based piezoelectric sensors, fiber Bragg grating sensors, etc., have been introduced to detect the actual stress or strain state of infrastructures for applications in different fields, such as monitoring damage, transportation, and traffic flow [121, 431, 432]. Contrary to many advances, most of these available techniques involve a limited number of sensors distributed in a large area of the infrastructures. Furthermore, the low durability, heterogeneity, production complexity, and incompatibility of their detection mechanism with geomaterial mechanical behaviors have made many of these techniques incompetent [187, 359, 433].

Among all structural health monitoring methods, cement-based piezoresistive self-sensing composites have attracted more attention from researchers to overcome these limitations and achieve a real-time integrated system for infrastructure monitoring [84, 138, 184, 389, 434-438]. The main idea of a self-sensing cementitious geocomposite is to monitor the stress/strain states in cementitious geomaterials based on a fractional change in the electrical resistance. This means that the cementitious geomaterials must be conductive [207].

For this purpose, different kinds of conductive fillers may be dispersed into the non-conductive cementitious composite [183, 439, 440]. The continuous conductive pathways generated by conductive fillers based on percolation and electron tunneling effects, have a considerable contribution to the transmission of electric current among the cementitious composite structure [441-443]. These conducting paths are disturbed when the composites are subjected to stress, strain, damage, or deformation, which results in changing the electrical resistivity [444-446]. The type of conductive phase is one of the effective factors in the sensitivity and performance of the cement-based self-sensing composites [81, 82, 207].

Although, the various types of conductive fillers, such as steel fibers, nickel powder, iron oxide nanoparticles, carbon black, and carbon fiber, have been used as a conductive phase in the cementitious matrix [189, 281, 447, 448], graphene nanoplatelets (GNPs), and carbon nanotubes (CNTs) have attracted more attention for developing piezoresistive self-sensing cementitious composite due to their unique electrical and mechanical properties [82, 402, 449].

Besides, recent studies have proven the potential of CNT/GNP synergic effects to create ultrasensitive affordable self-sensing cementitious composites [145, 207, 449]. The hybrid combination of these 1D

and 2D carbon nanomaterials (CNMs) with high aspect ratios, can improve the percolation and electron tunneling mechanism, which leads to the reduction of the percolation threshold and consequently eliminates the concerns regarding the production costs as well as the porosity and agglomeration formation. The nanoscale conductive paths formed by these CNMs are also more sensitive against the small strain and stress compared to the microscale conductive paths created by micro fiber fillers [207, 450].

The incorporation of these CNMs into the cementitious composite also enhances the mechanical, microstructural, and durability performances by the bridging and/or deviation of cracks, increasing the cement hydration rate and their filler function mechanism [346, 451-453]. However, the low ductility of the cementitious composites which is also intensified by CNTs in some cases [180] still remains as one of the cement base's self-sensing weaknesses.

The brittle behavior of nano-intruded cementitious composite exacerbates their weakness against cracking. This is particularly important in cementitious stabilized sand (CS), which is one of the most widely used geomaterials in infrastructure construction. What's more, it should be noted that the more ductile composite behavior leads to a more appropriate piezoresistive response against the stress/strain and, consequently, the data scatter will be reduced. Based on the literature, fiber reinforcement is one of the common and widely used methods to improve the ductility behavior of CSs [176, 454, 455].

Fibers can radically moderate brittle behavior and enhance mechanical performance [456]. Glass and polypropylene (PP) fibers are two conventional available fibers utilized for CS reinforcement [457, 458]. Due to the low stiffness of PP fibers, they adapt well with the CS in terms of ductility. CS reinforced with the glass fiber (GF) with non-crystallizable components has attracted substantial attention in different applications predominantly owing to being fairly light, cost-effective, and enjoying rather fine relative mechanical properties, including flexural and tensile moduli as well as flexibility during processing and biodegradation. In addition, they have the least harm to the processing equipment and ample renewable resources [459-465].

In the last decade, a novel fiber with a high strength-to-weight ratio was developed, called Dyneema. Dyneema is an ultra-high-molecular-weight polyethylene (UHMWPE) that is stronger than steel and aramid fibers up to 15 and 1.4 times, respectively [466]. High strength and modulus, high durability and resistance against aggressive chemical agents, and low toxicity have made Dyneema attractive [466]. Hence, in this study, a CNT/GNP intruded CS was reinforced by these fibers to develop a self-sensing cementitious geocomposite with high ductility. Indeed, one of the main purposes of fiber inclusion into

the CS is to decrease the stiffness with an increase in the maximum strain and to consequently prevent micro crack propagation as well as to produce long-term performance enhancement.

The outcomes of this study provide an extensive contribution to the new era of smart structures, in this case with applications to roller-compacted-concrete dams, rammed earth, ground improvement, and particularly in structural layers in transportation infrastructures, especially in critical zones, such as transition zones. This self-sensing composite material will have the capacity to detected material damages, anticipate maintenance needs, and avoid failures, which, in some structures, may cause fatalities.

In this route, and following the previous findings applied to a mortar [145, 207, 409], different concentrations of glass, PP, and Dyneema fibers were incorporated into the cylindrical specimens after surficial acid treatment, and the maximum dry density and optimum water content were evaluated. This approach in the manufacture of the specimens is different from previous studies, aiming for applications for the type of structures mentioned earlier. The mechanical properties of the specimens were investigated by unconfined compressive strength (UCS) tests after 28 days of hydration as well as by the tangent modulus at 50% of the maximum strength. These properties are important in the characterization and design of several geotechnical structures [412, 467-469]. Additionally, the microstructural performance of reinforced CS was investigated using various tests. The interfacial properties of the fibers and matrix were evaluated by mechanical and chemical analysis. The durability of the specimens against wetting and drying cycles was also evaluated (long term performance). Furthermore, the piezoresistivity behaviors of reinforced CS were investigated under cyclic compression loading by the four probes method to evaluate the self-sensing capacity of the novel materials.

6.2 Materials and Methods

6.2.1. Raw Materials

To fabricate CS, CEN Standard sand, with siliceous and clean particles was used. This sand is classified as well-graded sand following the Unified Soil Classification System (USC). The particle size distribution (PSD) curve and physical properties of the sand are presented in Figure 6-1 and Table 6-1. The grading, measured by sieving, complies with the EN 196-1 and of ISO 679: 2009 standard requirements. In this study, ordinary Portland cement type I (CEM I 42.5R) was also utilized as a binder in order to stabilize the sand. The grain size curve of cement is presented in Figure 6-1.

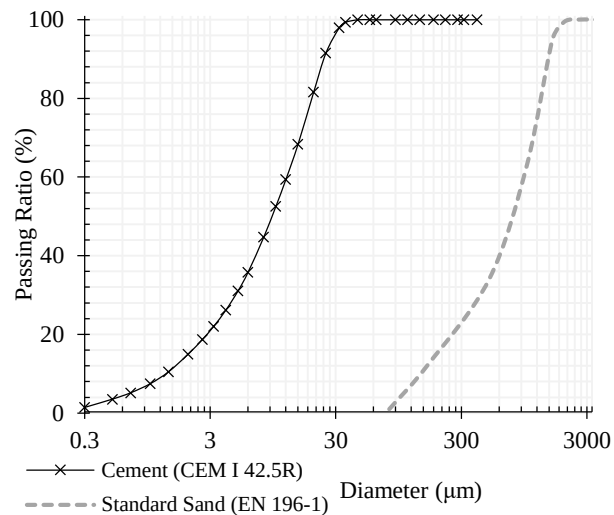


Figure 6-1. The particle size distribution (PSD) curve of the sand and cement [207, 409].

Table 6-1. Physical properties of the sand [207].

Mesh Size (mm)	0.08	0.16	0.5	1	1.6	2
Cumulative retained (%)	99±1	87±5	67±5	33±5	7±5	0
Specific gravity G_s	2.67		Cu	7.5	Cc	1.8

A multi-wall CNT (MWCNT) and multi-layer type GNP were utilized as conductive fillers in this study. The specifications of the MWCNTs and GNPs are summarized in Table 6-2 [145]. The morphology of hybrid GNPs/CNTs in a dry mix state was also investigated using a scanning electron microscope (SEM), and this is depicted in Figure 6-2. The X-ray diffraction (XRD) patterns of CNTs and GNPs are shown in Figure 6-3.

Table 6-2. Multi-wall carbon nanotube (MWCNT) and graphene nanoplatelets (GNP) specifications [145].

GNP										
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Carbon content (%)	Tensile modulus (Gpa)	PH Value (30 °C)	Tensile strength (GPa)	Layers	Dimension		Form	Part Number
120-150	0.6	> 99.5	1000	7-7.65	5	10 < n < 60	Thickness 4-20nm	Diameter 5-10μm	Gray Powder	TGN201
MWCNT										
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Color	Outside Diameter (nm)	Length (μm)	Ash (wt%)	Carbon content (%)	Part Number			
350	0.27	Black	< 8	30-10	< 1.5	> 98	GCM327			

In order to hybrid the CNT/GNP dispersion, Pluronic F-127 was used as a noncovalent surfactant. This surfactant is a nonionic triblock copolymer surfactant composed of a central hydrophobic chain of polyoxypropylene (PPO) and two hydrophilic chains of polyoxyethylene (PEO) placed on two sides. The molecular structure of Pluronic F-127 is similar to polycarboxylate, which is typically used as a cementitious composites superplasticizer [144].

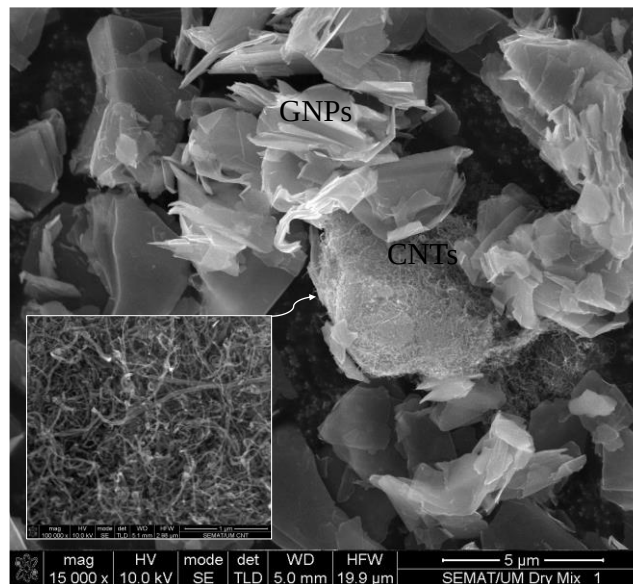


Figure 6-2. GNP and CNT morphology (dry mix).

Hence, this surfactant was found to be compatible with cementitious composites and could possibly enhance its dry density owing to the improved consistency of CS. In addition, tributyl phosphate (TBP, 97%) with $\frac{1}{2}$ of the surfactant weight ratio was used as an antifoam to prevent porosity formation caused by surfactant function following the previous research [205]. The TBP and PF-127 molecular structures are depicted in Figure 6-4, [145]. Since the average optimum length of fiber used for cementitious composite reinforcement is around 10~14mm [470-472], in this study Alkali-resistant glass fibers (AR-GF), split film type of polypropylene (PP), and Dyneema fibers with an average length of 12 mm were used for CS reinforcement. The physical properties of the fibers are listed in Table 6-3.

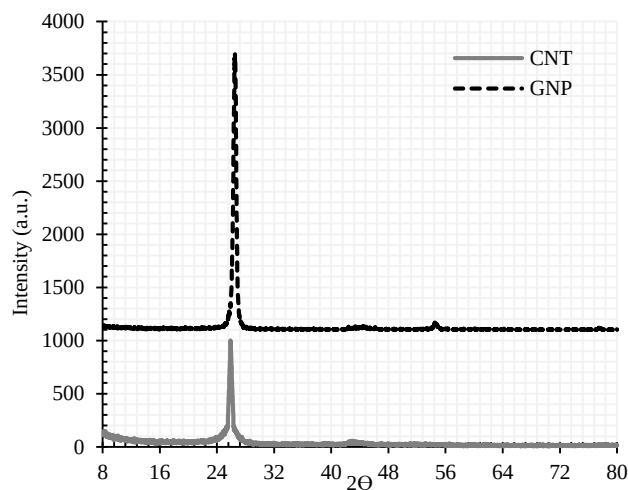
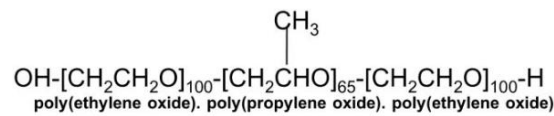
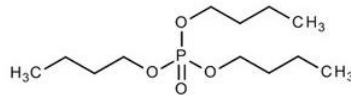


Figure 6-3. X-ray diffraction (XRD) patterns of CNT and GNP.



(a)



(b)

Figure 6-4. Chemical structures of: (a) Pluronic F-127_ (b) Tributyl phosphate 97%, [145].

In this study, sulfuric acid 97% and hydrofluoric acid 40% were used for surface treatment of the fibers.

Table 6-3. Physical properties of glass, polypropylene, and Dyneema fibers.

Alkali-resistant (AR)-glass fiber (GF)						
Tensile strength (GPa)	linear mass density of fibers (g/Km)	Density (kg/m3)	Elastic modulus (GPa)	Elongation at break (%)	Length (mm)	Diameter (μm)
3.2	9600	2700	73.1	4.4	12	31
Ultra-high-molecular-weight polyethylene (UHMWPE) (Dyneema)						
Tensile strength (GPa)	linear mass density of fibers (dtex)	Elastic modulus (GPa)		Elongation at break (%)	Length (mm)	Diameter (μm)
3	440	10.3		5.6	12	14.5
Polypropylene (Split film)						
Tensile strength (MPa)	Density (kg/m3)	Elastic modulus (GPa)		Elongation at break (%)	Length (mm)	Section Dimension (μm)
54	910	4.31		15	12	36 × 640

6.2.2. Dispersion, fabrication and characterization methods

Dispersion Method of CNT/GNP

The presence of strong surface van der Waals forces between carbon nanomaterials due to their high specific surface area leads to their high tendency to agglomeration. Hence, the effective transfer of CNT/GNP supreme properties to the composite is strongly dependent on their dispersion status. Besides, the dispersion method should be attentively chosen to prevent adverse effects on the cement hydration process and/or inherent properties of nanomaterials [146, 473-475]. Following the above discussion, a feasible and compatible dispersion method that was developed recently was used in this study [145]. In this route, TBP was first dissolved into the required optimum water content of fiber-reinforced CS over 12 hours with a magnetic stirrer at 800 rpm/min. It should be noted that the required optimum water content

(ω_{opt}) concentration for each case of fiber-reinforced CS was measured by the compaction standard test, and, for this purpose, dry CNT/GNP was dispersed among the dry materials using the mechanical mixer and with gradual water concentration increases. After completely dissolving the TBP, 10% of Pluronic F-127 (by weight of CNMs) was added to the suspension and mixed for 1 more hour using the magnetic stirrer mixer at the same speed.

Based on the literature, the optimum hybrid CNT/GNP concentration that was obtained for the development of a self-sensing cementitious composite was around 0.17% (1:1, by weight of the sand) by considering the physical behavior [207, 409]. Hence, in this study, the same concentration was used. The hybrid CNT/GNP was added to the suspension and stirred continuously for 1 more hour, and the suspensions were then placed in a sonicator bath with 80 W output power and, 45 kHz frequency for 3 hours. In addition, a digital temperature regulator composed of the circulation system, sensors, and a radiator was used to adjust the temperature during the ultrasonication at 40 °C. Under these specific mixing conditions, negligible structural damage was expected for the carbon nanomaterials.

Raman spectroscopy (Figure 6-5) was also performed on CNMs using laser excitation with a wavelength of 532 nm in order to ensure about the absence of adverse effects on the nanomaterials structural quality, such as edge-type defects, reduction of the aspect ratio, and sp² domain crystallinity (La), which cause a deleterious influence on the mechanical and electrical performances [145, 217, 218].

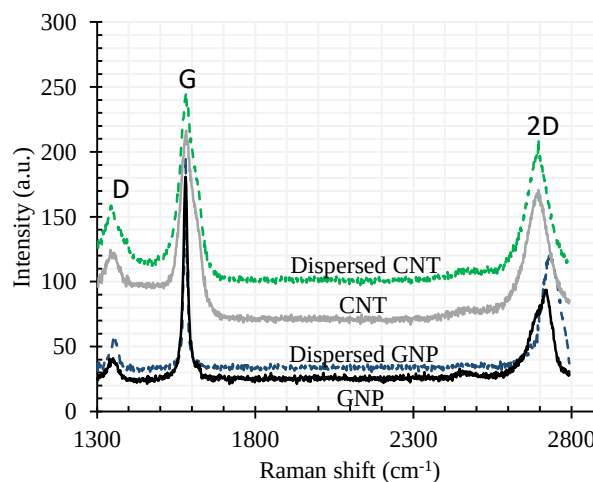


Figure 6-5. Raman spectroscopy of CNTs and GNPs after dispersion.

Surface Treatment of the Fibers

The surfaces of the fibers were treated using the chemical method to increase the interaction and adhesion between the cementitious matrix and fibers [476-480]. In this route, The PP and Dyneema fibers were submerged into a bath of sulfuric acid and sodium dichromate solution for 24 h at 50 °C, and the

glass fibers were dipped into hydrofluoric acid 40% for 18 h at 40 °C. Thereafter, the fibers were washed with water and dried at room temperature. This treatment might cause asperity on the fiber surface, and likely causes a chemical reaction between the fibers and the cement matrix.

CS Specimens Fabrication

In the case of sand, the required cement concentration for soil improvement usually varies by around 10% owing to the target strength of the enhanced soil [147, 148]. In this study, 10% of the cement (by weight of the dry sand) was utilized for CS fabrication, and all of the fiber-reinforced specimens were fabricated at the optimum water content (ω_{opt}) of the Standard Proctor test.

In this route, the mixing sequence adopted was as follows: the cement and sand were poured into the Hobart mixer and then blended with the stainless steel blade's rotational speed at 140 m/min for 1.5 min. Then, a nanomaterial suspension prepared by ω_{opt} (for each fiber concentration) was sprayed into the mixture and blended at a 285 m/min higher speed for another 2.5 min. Thereafter, surficial treated fibers were added to the mixture and stirred for a further 1.5 minutes by the same rotational speed. The concentration of the fibers used in this study were 0.5%, 1.0%, and 1.5% by weight of the dry sand. The mixture was then poured into a plastic bag to prevent moisture loss.

The fiber-reinforced CS cylindrical specimens were fabricated by the compaction method according to the standard compaction following ASTM D698. Split molds with dimensions of 101.6 × 116.4 mm were filled with the wet mixture in three equal-height layers. A calculated amount of the well-mixed wet mixture was measured (to an accuracy of 0.01 g), shed into the split molds, and then compacted carefully by a metal tamper to the desired height (controlled by a caliper to an accuracy of 0.02 mm).

After compaction, the surfaces of the specimens were carefully leveled and sealed. The specimens were extracted after 24h and cured under the water for 28 days. As shown in Figure 6-6, four copper mesh (as electrodes) were also embedded into specimens used for piezoresistivity experiments.

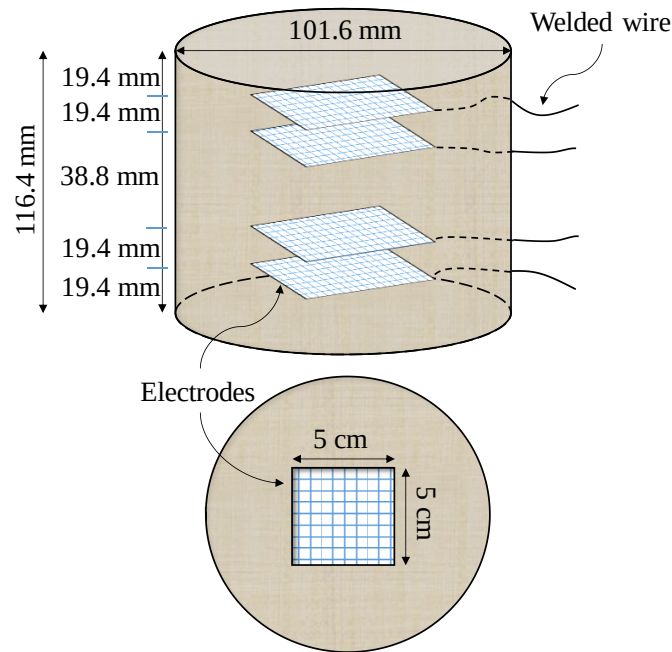


Figure 6-6. Representation of the specimen geometry and electrode layout.

The mixture moisture also was measured after compaction to ensure minimal water evaporation. The specimen's identification was conducted based on the variation of fiber types and concentration as listed in Table 6-4.

Table 6-4. Mix proportions of the reinforced cemented sand (CS). Carbon nanomaterial (CNM), polypropylene (PP).

Sample ID	CNM (%)	Glass Fiber (%)	PP Fiber (%)	Dyneema Fiber (%)	Cement (%)
Plain CG	0.17	-	-	-	10
CGG (0.5%)	0.17	0.5	-	-	10
CGG (1.0%)	0.17	1	-	-	10
CGG (1.5%)	0.17	1.5	-	-	10
CGP (0.5%)	0.17	-	0.5	-	10
CGP (1.0%)	0.17	-	1	-	10
CGP (1.5%)	0.17	-	1.5	-	10
CGD (0.5%)	0.17	-	-	0.5	10
CGD (1.0%)	0.17	-	-	1	10
CGD (1.5%)	0.17	-	-	1.5	10

*All of the concentration are by weight of the dry sand.

**By equal proportion (1:1).

In other to investigate the interfacial properties of the fibers and cement matrix, single-fiber pullout tests were used. Due to the small size and fragility of the fibers, a special method was used following the

literature [481, 482]. For the preparation of the specimen, special plastic molds were used which showed in Figure 6-7 (a)).

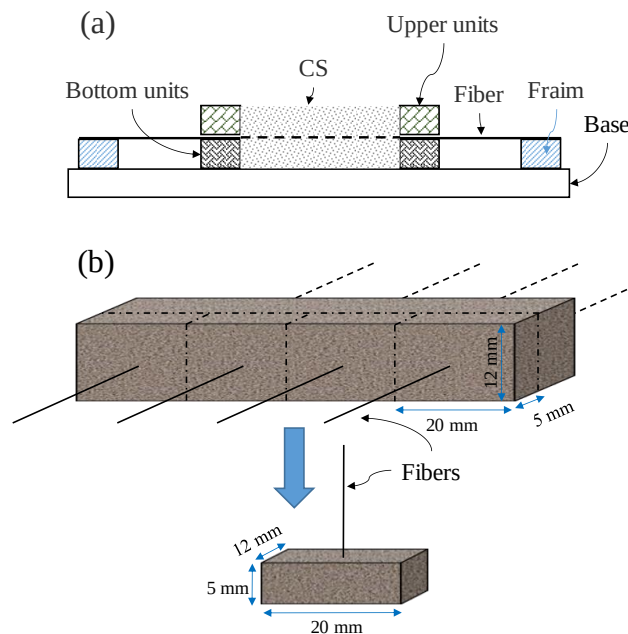


Figure 6-7. The process of pull-out specimen fabrication: a) cross-section of the mold, b) dimensions of the specimen.

The thickness of the initial cured samples is too wide and hence, the single fiber would break during the test. Following Li [483], the theoretical length of fiber was calculated around 6 mm for the successful pullout of the fiber without rupture, which indicates that the specimen's thickness, i.e., the fiber embedded length in the matrix, would not exceed 6mm. In fact, to successfully obtain the entire force-displacement curve during pulling out, the real CF embedded length should be further limited. Hence, after 28 days of curing, specimens were cut to dimensions of 20 mm × 12 mm × 5.0 mm as shown in Figure 6-7 (b).

Mechanical, Microstructural, and Durability Evaluations

For the mechanical evaluation of fiber-reinforced CS, we used the unconfined compressive strength tests following the ASTM/D2166M standard. The results were calculated by the mean of at least three specimens. As the modulus of elasticity for cementitious geomaterials is typically expressed by the modulus at 50% of the peak stress [410-412], the Tangent $E_{(50\%)}$ of fiber-reinforced composites by different fiber types and concentrations was calculated at 50% of the maximum compression stress.

In order to evaluate different fiber concentrations on the maximum dry density and ω_{opt} of fiber-reinforced CS, standard compaction tests were carried out following the ASTM D697-78 standard. Besides, the specimen fracture surfaces were investigated using scanning electron microscopy (SEM) through the secondary electrons mode and an acceleration voltage of 10 kV after coating with an Au-Pd

thin film (30 nm) in a high-resolution sputter coater (Cressington 208HR). The energy-dispersive X-ray spectroscopy (EDX) analysis was performed at 3 kV using an energy-dispersive X-ray spectrometer coupled with the SEM.

Fourier transform infrared spectroscopy (FTIR) and energy-dispersive X-ray spectroscopy (EDX) were performed to analyze the surface structure of the treated fibers. For this purpose, the fibers were purified by washing and precipitation with deionized water and then dried for 10 h at 50 °C in a vacuum dry container. An ultrasonic nondestructive test was conducted for microstructural investigation following the BS EN 12504-4 standard through two probes along the longitudinal axis of the specimens. The weight loss of the fiber-reinforced CS was measured in terms of a composite durability investigation after 12 cycles of wetting and drying according to ASTM D 559 – 96.

The single fiber pullout tests were conducted at a rate of 0.02 mm/s, by a universal testing machine. The test results are an average of at least five specimens and three bonding parameters including bond strength (τ_{max}), frictional bond strength (τ_{fr}), and chemical debonding energy (G_d) were calculated by Eq. 1 to 3 as follow [484, 485]:

$$\tau_{max} = \frac{P_{max}}{\pi d_f l_e} \quad (\text{Eq. 1})$$

$$\tau_{fr} = \frac{P_{fr}}{\pi d_f l_e} \quad (\text{Eq. 2})$$

$$G_d = \frac{2(P_{max} - P_{fr})^2}{\pi^2 d_f^3 E_f} \quad (\text{Eq. 3})$$

where (P_{fr}) is frictional pullout load, (P_{max}) is maximum pullout load, l_e is fibers embedded length, d_f is fibers diameter, and E_f is fibers elasticity modulus. In the case of PP fibers, the cross section was assumed to be circular with a diameter of 7 μm . This assumption has no quantitative effect on the circumference of the fiber.

Piezoresistivity Behavior Investigations

For the piezoresistivity investigation of the fiber-reinforced CS, the cylindrical specimens with embedded electrodes were dried first at 70 °C for 72 hours after 28 days of hydration to eliminate the effects of moisture on electrical conductivity. In this study, the four probes technique (Figure 6-6) was used for the evaluation of fractional changes in the electrical resistivity (FCR) of reinforced CS subjected to cyclic axial compression loading. In this route, a 100 Ω reference resistor was connected to the outer probes in a series powered by a direct current (DC) source (20 V).

The power supply was conducted for 45 minutes to stabilize the supply in the fiber-reinforced specimens. The variation of voltage between the two inner probes and resistor were measured by a digital multimeter. As illustrated in Figure 6-8, three cycles of 5 kN axial compression loading with a rate of 50 N/Sec were used for the CS piezoresistivity behavior evaluation.

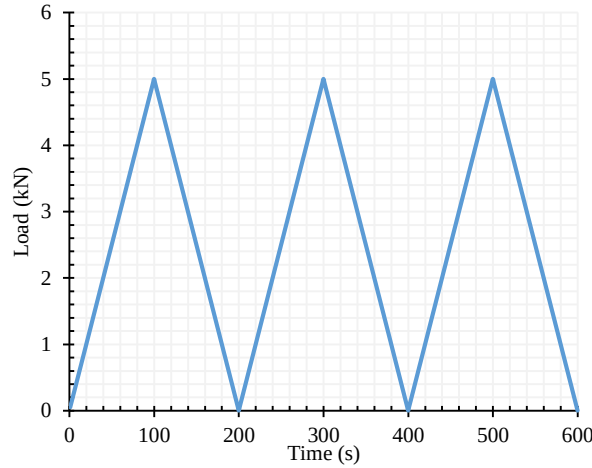


Figure 6-8. Cyclic compression loading pattern.

The voltage variations of the fiber-reinforced CS were obtained from an average of three measurements. The resistivity $\rho(t)$ of the specimens was calculated by combining the first and second Ohm's law equations (Eq. 4 and Eq. 5 respectively) as presented in Eq. 6 [396]:

$$R(t) = \frac{V_1(t)}{I(t)} = \frac{V_1(t)}{V_2(t)/100} \quad (\text{Eq. 4})$$

$$R(t) = \rho(t) \frac{L}{A} \quad (\text{Eq. 5})$$

$$\rho(t) = \frac{V_1(t)}{V_2(t)/100} \times \frac{A}{L} \quad (\text{Eq. 6})$$

where $V_1(t)$ and $V_2(t)$ are the inner probes and resistor voltage, respectively; $I(t)$ is the current between the outer electrodes; $R(t)$ is the resistance between the two inner probes; A is the contact surface between the electrode and specimen; and L is the spacing between the inner electrodes. The FCR was calculated using Eq. 7 for the following assessment of CS piezoresistivity:

$$FCR = \frac{\rho(t) - \rho_0}{\rho_0} \quad (\text{Eq. 7})$$

where ρ_0 is the initial electrical resistivity, which is measured before loading, and $\rho(t)$ is the resistivity at time (t) during the test. In order to evaluate the sensitivity of fiber-reinforced CS against the strain, the gauge factor (GF) is also defined as the relative change in electrical resistivity over the strain (Eq. 8):

$$GF = \frac{FCR}{\varepsilon} \quad (\text{Eq. 8})$$

where ε is the applied strain along the force axis.

6.3 Results and analysis

6.3.1. Fiber Surficial Characterizations

The FTIR spectra of the pure and treated fibers are illustrated in Figure 6-9. The results confirmed the presence of different functional groups on the surface of the fibers caused by the sulfuric acid treatment of the PP and Dyneema fibers and hydrofluoric acid treatment of the glass fibers.

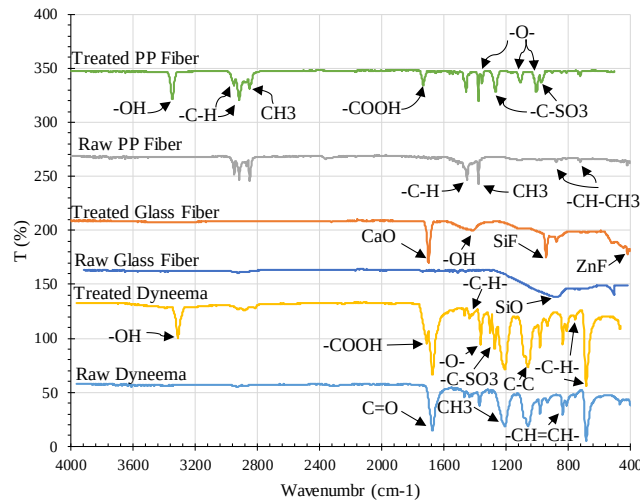


Figure 6-9. Fourier transform infrared spectroscopy (FTIR) spectra of raw and treated fibers.

As can be observed, the treated PP and Dyneema fibers had the oxygen groups of -O-, -OH, -C-SO₃, and -COO- supported by new peaks at 1360 cm⁻¹, 1102 cm⁻¹, 1025 cm⁻¹ (-O-), 3350 cm⁻¹ (-OH), 1360 cm⁻¹, 1264 cm⁻¹, 985 cm⁻¹ (-C-SO₃), and 1730 cm⁻¹ (-COOH). The FTIR analysis of the pure PP and Dyneema fibers showed the characteristic peaks at 1375 cm⁻¹, from the symmetric bending vibration of CH₃; at 1450 cm⁻¹, from the symmetric stretching vibration of -C-H in CH₃; at 1166 cm⁻¹, 972 cm⁻¹, 840 cm⁻¹, and 808 cm⁻¹, from the vibration rocking of -CH₂ and CH₃, and the stretching of CH-CH₃ and CH-CH₂. Hydrofluoric acid treated glass fiber also demonstrated some functional groups, including -OH, CaO, SiF, and ZnF supported by 1419 cm⁻¹ (-OH), 1700 cm⁻¹ (CaO), 1031 cm⁻¹ (SiF), and 417 cm⁻¹ (ZnF). The morphology of the fiber surfaces was also investigated with SEM, which is shown in Figure 6-10 to Figure 6-12.

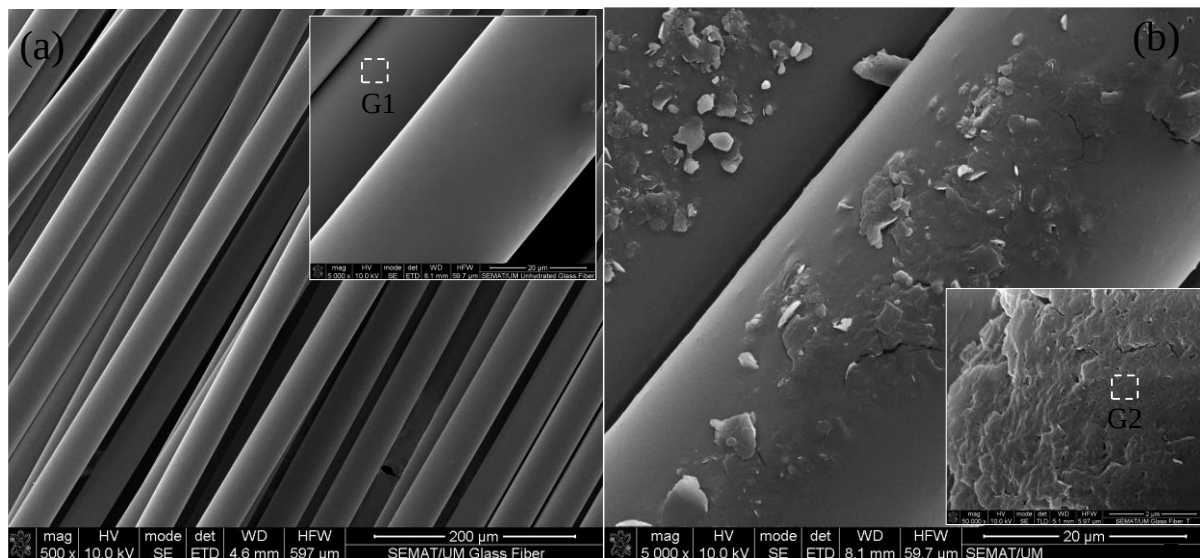


Figure 6-10. Surface morphology of glass fibers: a) pure glass fiber, b) hydrofluoric acid treated glass fiber. (The marked areas in the scanning electron microscopy (SEM) images are the areas selected for energy-dispersive X-ray spectroscopy (EDX) analysis.)

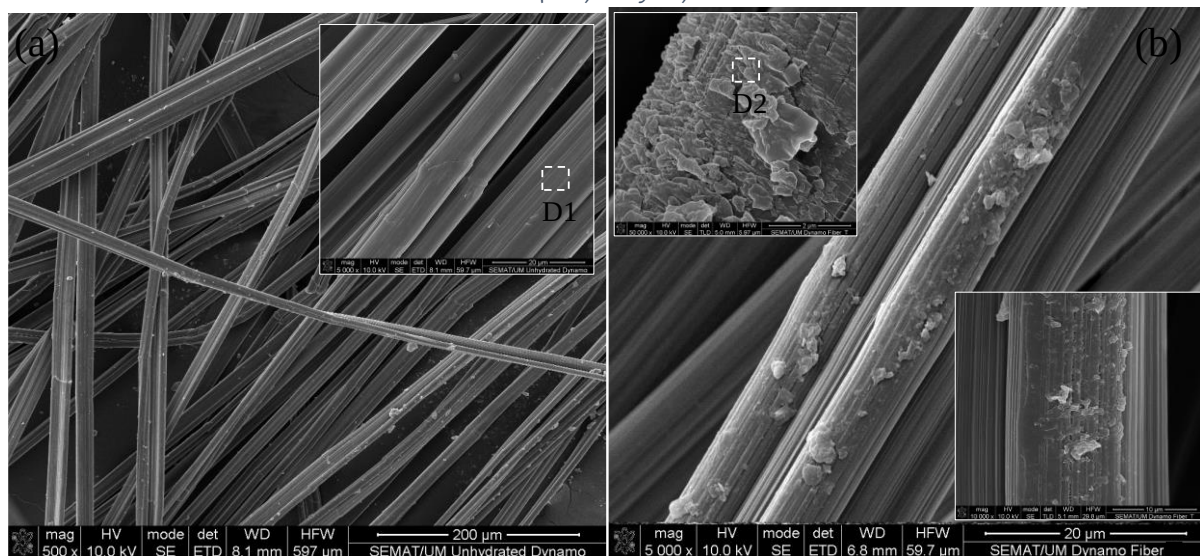


Figure 6-11. Surficial morphology of Dyneema fiber: a) pure Dyneema fiber, b) sulfuric acid treated Dyneema fiber. (The marked areas in the SEM images are the areas selected for EDX analysis.)

As can be seen, the chemical reaction between fibers and acids during the treatment process, led to some irregularity and roughness on the surface of the fibers. These surface defects can cause a slight reduction in the tensile strength of the fibers, increasing the interaction and bonding between CS and fibers that can improve the anchor mechanism of the fibers and, consequently, increase the mechanical performance of the composite [486].

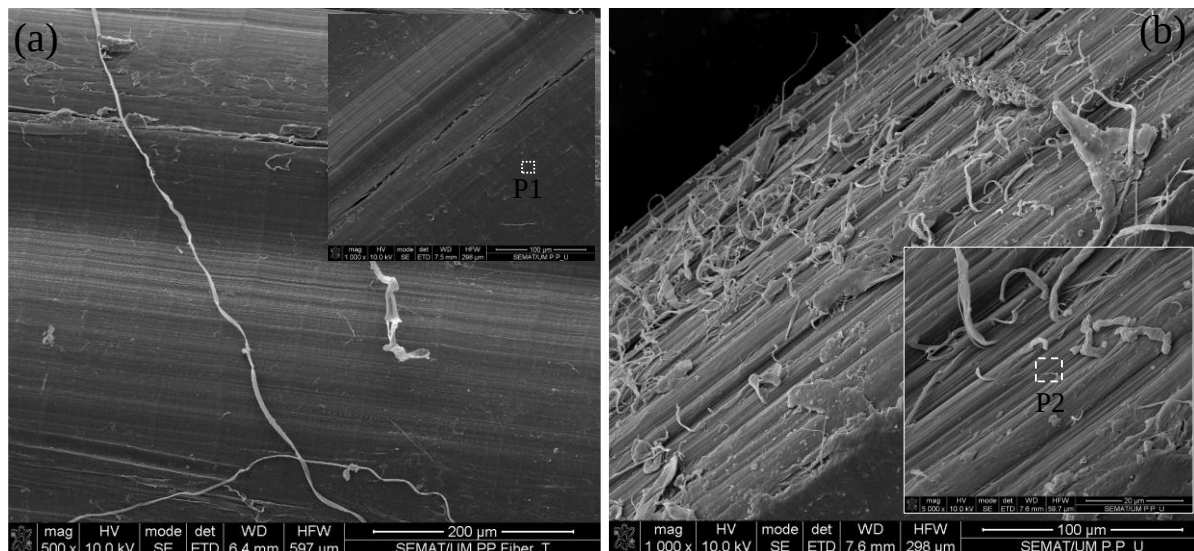


Figure 6-12. Surficial morphology of PP fiber: a) pure PP fiber, b) sulfuric acid treated PP fiber. (The marked areas in the SEM images are the areas selected for EDX analysis.)

The chemical analysis results of the fiber surface are also listed in Table 6-5. The results showed that the chemical composition of the pure PP and Dyneema fibers almost included carbon, nitrogen, and oxygen elements. However, the EDX analysis results illustrated the presence of sulfur (S) and an increase in the amounts of oxygen after treatment by sulfuric acid. In the treated glass fibers, the existence of the fluorine element and increase of oxygen was clear due to the chemical reaction of glass with hydrofluoric acid during the treatment process.

Table 6-5. Chemical analysis of pure and treated fibers.

Position	Elements (%)										
	C	O	N	Ca	Al	Si	S	Mg	F	Zn	Zr
Figure 6-10 (G1)	20.3	13.72	-	18.1	5.62	24.1	-	1.17	-	4.1	12.9
Figure 6-10 (G2)	15.8	29.6		12.7	2.14	19.8	-	1.1	7.4	3.3	8.2
Figure 6-11 (D1)	78.09	5.6	16.3	-	-	-	-	-	-	-	-
Figure 6-11 (D2)	61.7	16.2	9.1				12.9				
Figure 6-12 (P1)	78.75	3.95	17.3	-	-	-	-	-	-	-	-
Figure 6-12 (P2)	64.8	14.7	11.9				8.6				

6.3.2. Compaction and Microstructural Investigations

The compaction curves of plain CS containing 0.17% CNMs and fiber-reinforced CS with different types and concentrations of the fibers are presented in Figure 6-13.

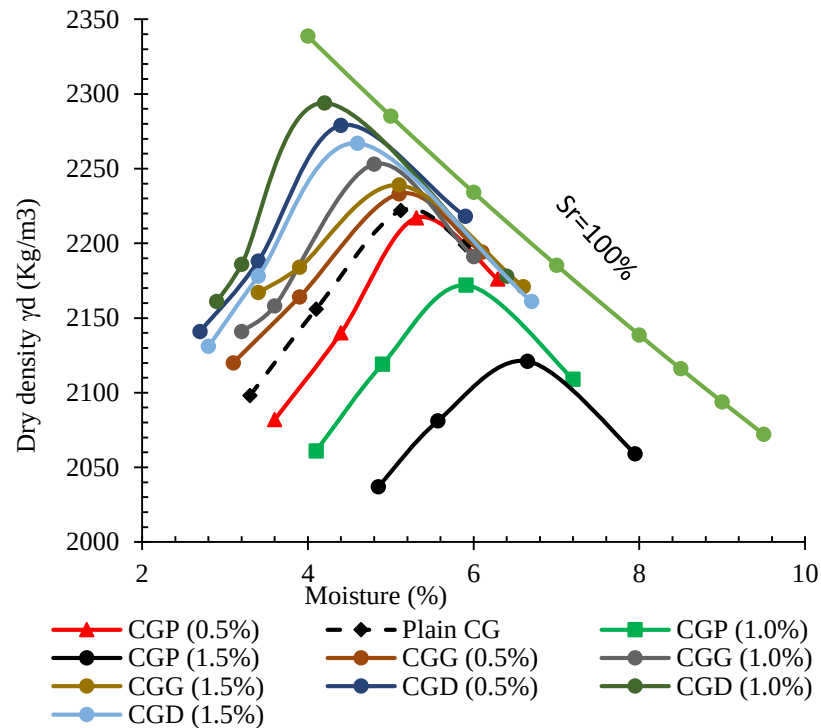


Figure 6-13. Compaction curves of reinforced CS with different types and concentrations of the fibers.

The results showed that the incorporation of a certain amount of fiber concentration into the CS caused an increase of the maximum dry density. Indeed, an excessive increase in the fiber content reduced the dry density. The filler function of the fibers is heavily dependent on their geometry shapes and aspect ratio and the ductility of the fibers as subjected to the compaction energy.

A schematic representation of the fibers filler mechanism is illustrated in Figure 6-14. As can be observed in Figure 6-14 (b), the rectangular cross-section of the PP fibers makes it difficult to embed them into the free spaces between this type of sand grains. Further, by increasing the PP fiber concentration, the gaps between the sand and cement particles were increased, which led to an increase in the optimum water content in the form of free water.

In the case of the glass and Dyneema fibers (Figure 6-14 c and d), however, the circular cross-section of the fiber with smaller dimensions improved the maximum dry density of the composite, and excessive increasing of fiber concentration caused an increment in porosity content and consequently reduced the maximum dry density of the CS.

In order to better evaluate the relation between fiber types, and concentration with the maximum dry density and optimum water content are illustrated in Figure 6-15. As can be observed, the optimum concentration of glass and Dyneema fibers were obtained around 1.0%.

However, the maximum dry density of Dyneema reinforced CS at the optimum concentration was more than with the glass fibers due to the higher aspect ratio of Dyneema fibers. Besides, as presented in Figure 6-14, the incorporation of the PP fibers into the CS composite by a concentration in in range of 0.5% to 1.5%, generally reduced the maximum dry density and increased the optimum water content.

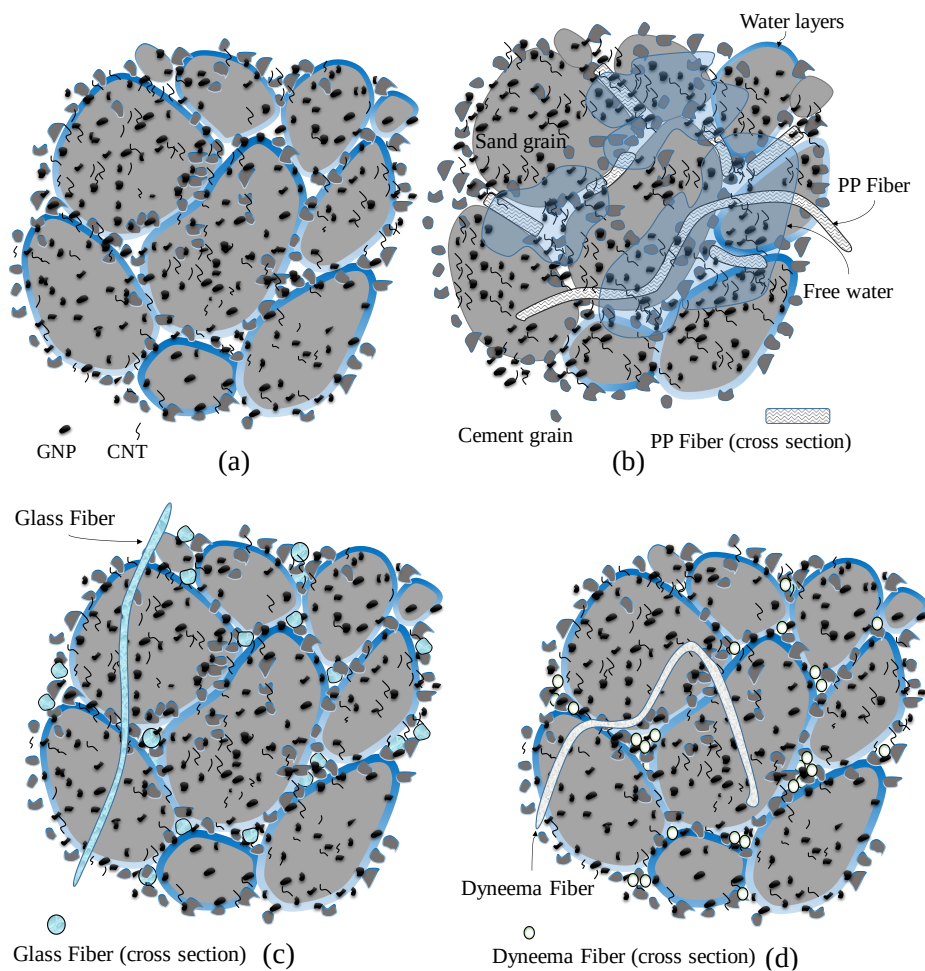


Figure 6-14. A schema of different fiber filler functions in CS: a) without fiber, b) reinforced with PP fibers, c) reinforced with glass fibers, d) reinforced with Dyneema fibers.

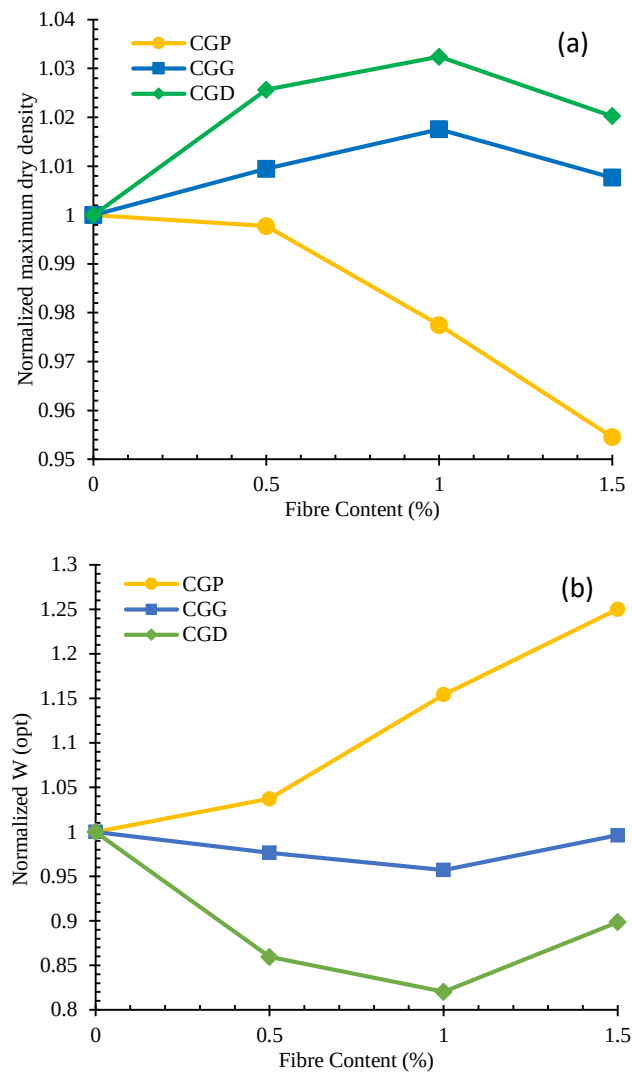


Figure 6-15. The relation between fiber types and concentration by the: a) maximum dry density, b) optimum water content. The results of the saturation degree, which are shown in Figure 6-16, also show the efficiency of Dyneema and glass fiber-reinforced CS in terms of the compressibility. The results showed that the saturation degree of the composite was generally reduced by the addition of the PP fibers, due to an increase in amounts of the void ratio.

In the case of glass and Dyneema fiber-reinforced composites, increasing the fiber concentration to 1.0% increased the saturation degree by reducing the void ratio, while increasing the fiber concentration beyond 1.0% caused a decreasing trend in terms of the saturation status. These very interesting findings can be alternatively represented by $\gamma_d/\gamma_{d(max)}$ vs. " $S_r-(S_r)_{opt}$ " as illustrated in Figure 6-17. This unique relation agrees with the recent studies [413] that demonstrated that these laboratory results can be applied directly in the field. This is a major issue to support the use of these novel materials in practice.

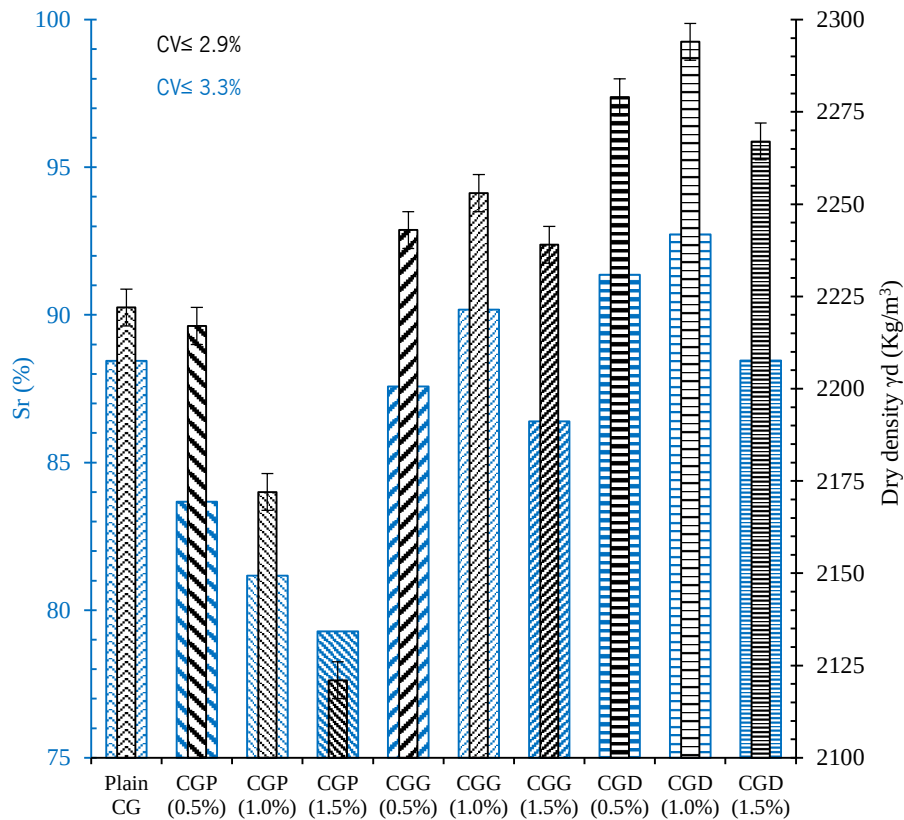


Figure 6-16. The saturation degree of reinforced CS with different fiber types and concentrations.

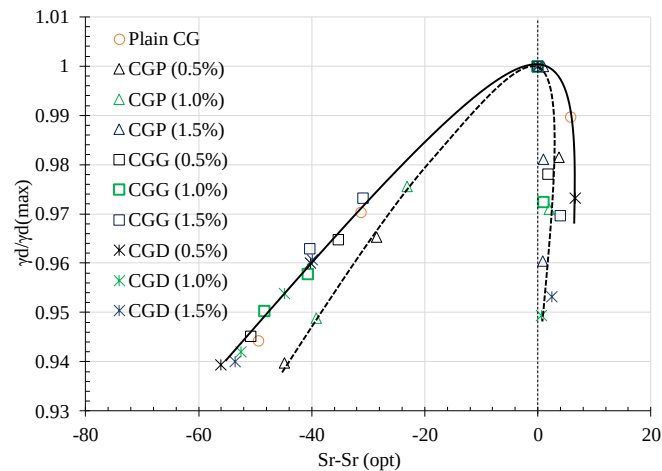


Figure 6-17. $\gamma_d/\gamma_d(\max)$ vs. " $S_r - S_r(\text{opt})$ " relations.

The results of the ultrasonic nondestructive test for reinforced CS by different fiber types and concentration after 28 days of the hydration period are shown in Figure 6-18. The time of ultrasonic wave passing through the composite depended on the microstructure and density of the specimens. As can be observed the ultrasonic passing time was decreased for specimen CGP 0.5%, which contained 0.5% PP fiber. This indicates that the density of the specimen was increased after 28 days of cement hydration due to the growth of hydration products into the porosities and around the fibers that led to the embedding of them.

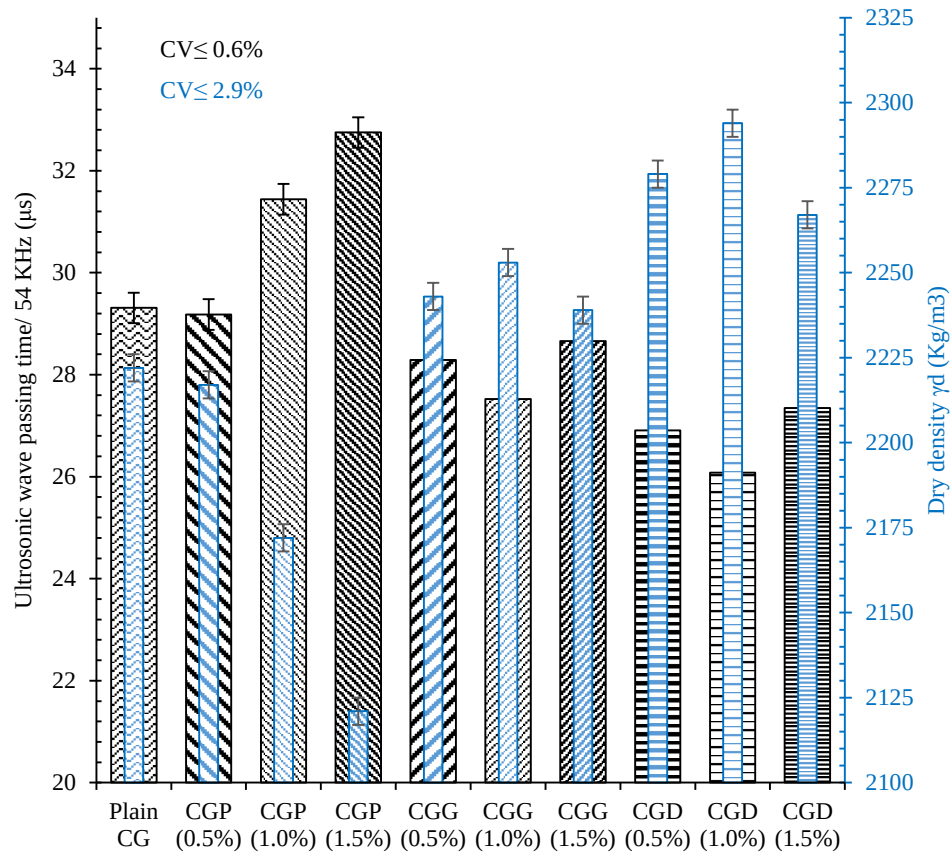


Figure 6-18. Ultrasonic wave passing times vs. the maximum dry density for reinforced CS by different fiber types and concentrations after 28 days of hydration.

It should be noted that the excessive increase in the PP fiber concentration increased the ultrasonic wave passing time that was evidence of a density reduction. A similar trend was observed for glass and Dyneema fibers, except that the optimum percentage of these types of fibers were obtained around 1.0%. The above outcomes are in line with the results of the mechanical investigations. Although the micro crack bridging and/or deviation mechanism of the fibers (Figure 6-19) is the key factor in CS mechanical improvement, the filler function of the fiber was also not ineffective.

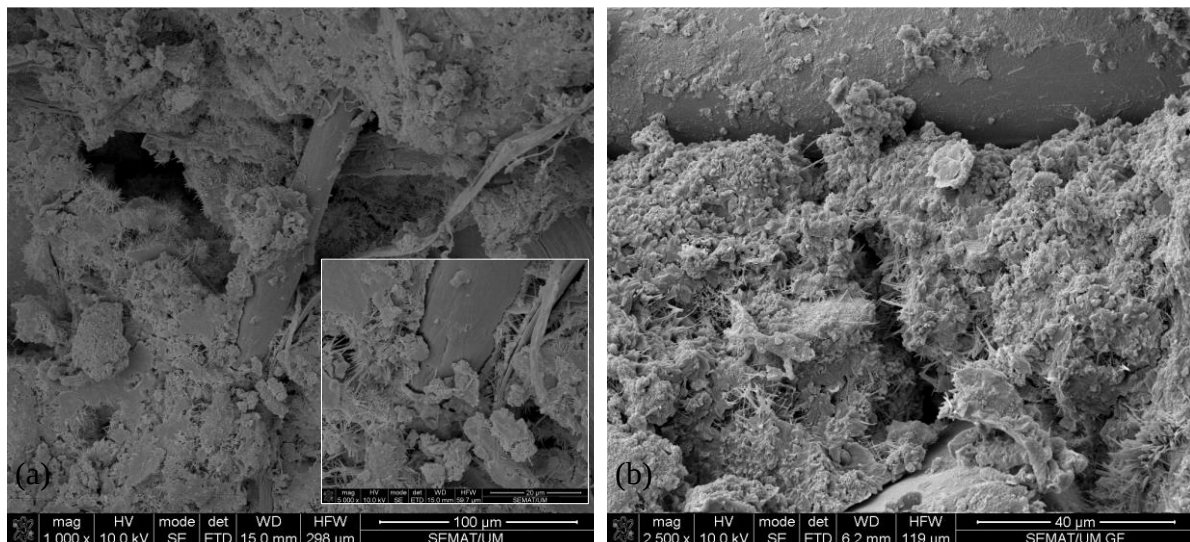


Figure 6-19. The mechanism of crack bridging and deviation: a) Dyneema fiber, b) glass fiber.

By incorporating a certain amount of the fiber into the CS, this fills the pores and spaces between the sand particles. The fibers buried among the hydration products during the cement hydration process can reinforce and bridge those products. When the plain CS is subjected to the load, usually in the early stages of loading, microscale cracks form inside the composite microstructure due to the brittle behavior of cementitious composites. While the optimum fiber-reinforced CS shows more resistance to cracking in the initial loading phase. When increasing the loads, nano-scale cracks grow into larger cracks at the micro-scale.

In the case of the proper bonding of fibers and the matrix, as long as the tensile force along the fiber length does not exceed the interaction force between the fibers or its pullout strength, the specimen withstands the increasing load, and its ductility will also be increased.

The proper interaction between Dyneema and glass fiber surfaces with the cement matrix, which can be seen in Figure 6-19, led to a halt in the crack expansion process by bridging and also deviation of the path. In next step, by the excessive increase of the load, two mechanisms are likely to occur, fiber extruding and fiber rupture (Figure 6-20).

In the case of low interaction between the fiber surface and/or expansion of cracks in the region around the fibers and low shear strength of the matrix near them, the fibers are extruded from the cementitious composite.

As can be observed in Figure 6-20, the low interaction between the PP fiber and cement matrix led to the fiber extruding, while the proper interaction between glass and Dyneema fibers led to glass fiber rupture and elongation in the Dyneema fibers due to withstanding the tension.

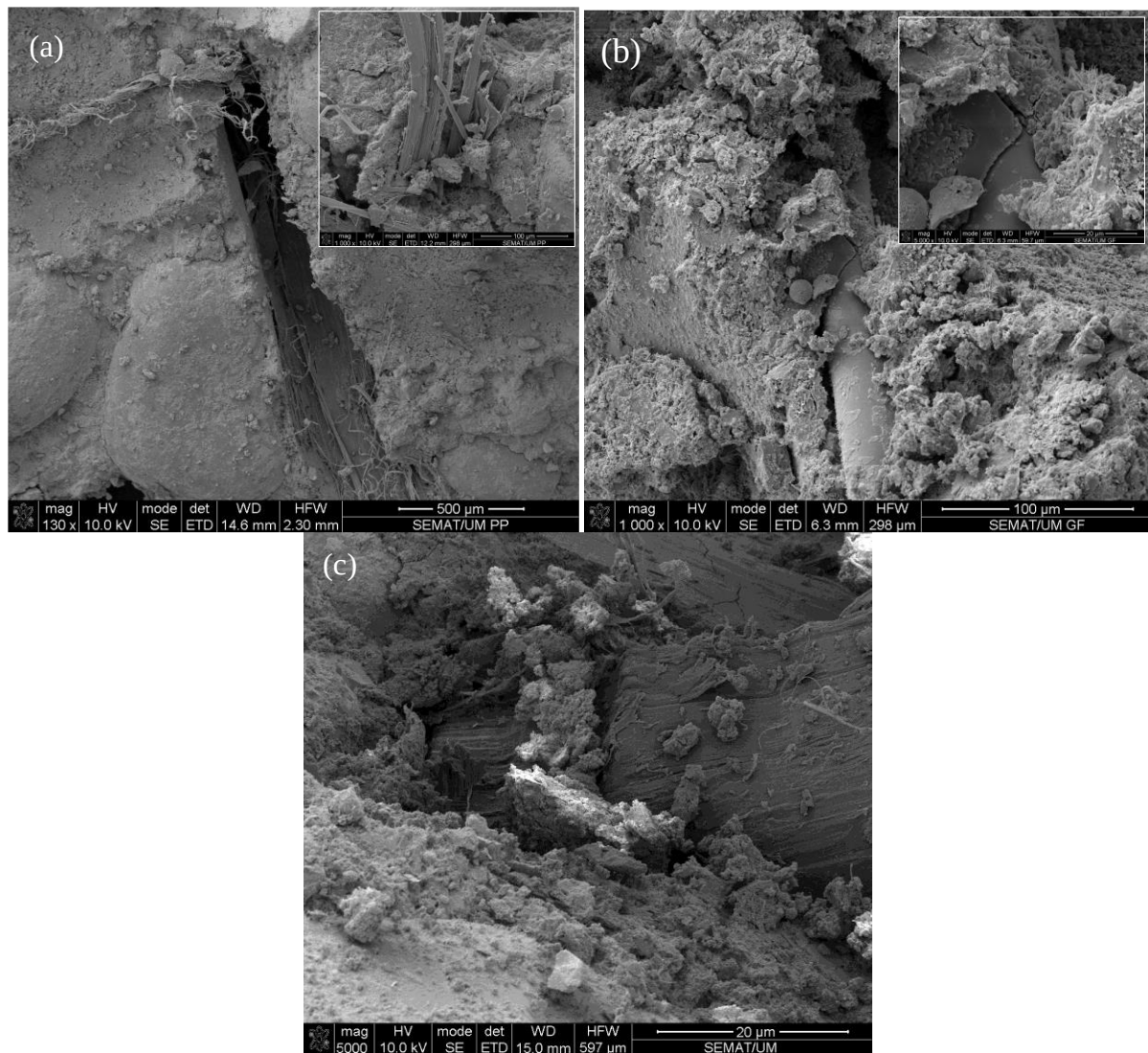


Figure 6-20. Morphology of the CS reinforced by fibers: a) PP fiber (fiber extruding), b) glass fiber (fiber rupture), c) Dyneema fiber (fiber elongation).

The dimensional and geometrical shape of the fiber cross-section, roughness, and status of the fiber surface in terms of rippling and porous texture are some of the crucial factors to enhance the fiber adhesion with a composite. Besides the existence of effective chemical functional groups can increase the fiber surface bonding with the cementitious matrix. The morphology of the Dyneema, glass, and PP fiber extracted from cementitious composite are illustrated in Figure 6-21 and Figure 6-22. As can be seen, the oxygen functional groups caused by acid treatment, which are discussed in section 3.1, made an approximately strong bond with the cement hydration products. On the surface of glass fiber, the oxygen and fluorine functional groups, made the surface ideal for growing hydration products. In the case of PP fibers, despite the presence of oxygen functional groups caused by acid treatment, no proper bonding was observed between the surface and the cement matrix and only the surface roughness statute provided the required interaction with the composite.

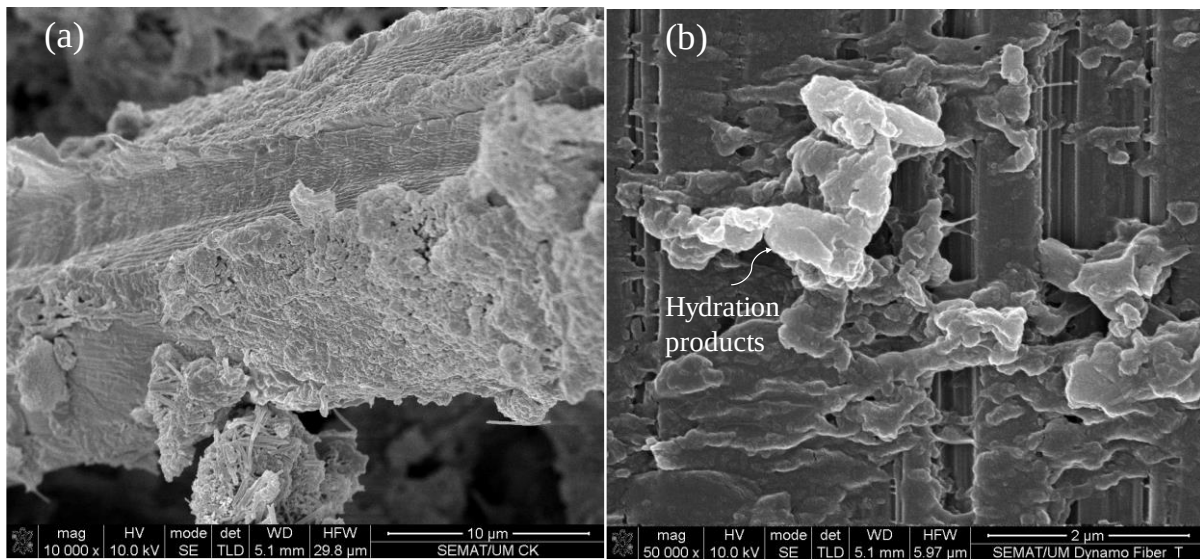


Figure 6-21. Dyneema fiber surface morphology, a) low magnification, b) high magnification.

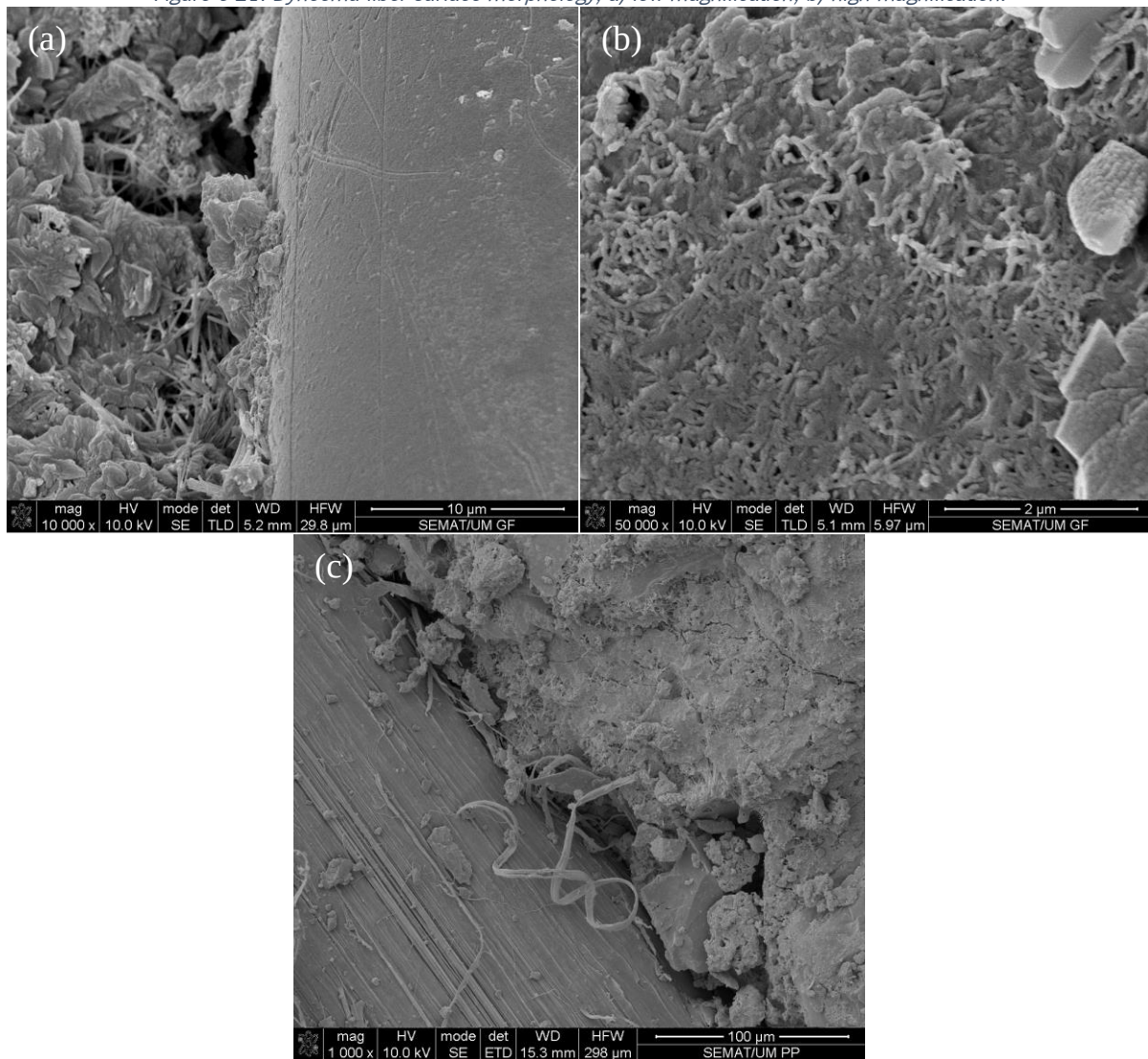


Figure 6-22. Glass and PP fiber surface morphology, a) low magnification of glass fiber, b) high magnification of glass fiber, c) PP fiber.

The XRD patterns of treated and extracted fibers from the CS, as illustrated in Figure 6-23, clarified new aspects of this issue. In Dyneema fibers, the chemical reaction between cement particles and oxygen functional groups led to the formation of calcium hydroxide (CH) on the surface of the fibers while in PP fibers, ettringite (AFt) crystals were formed on the surface. The dense structure of calcium hydroxide was more resistant vs. the brittle needle-shaped crystals of ettringite, which can be the reason for the proper bonding and interaction of Dyneema fibers with the cement matrix.

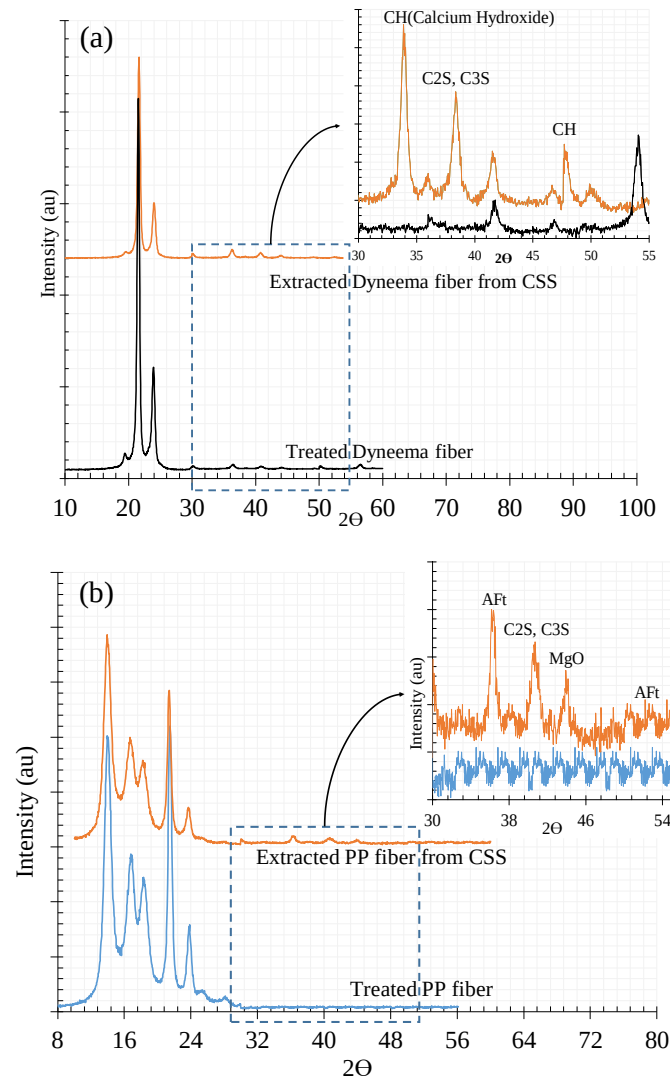


Figure 6-23. The XRD patterns of treated and extracted fiber from the CS: a) Dyneema fiber, b) PP fiber.

In order to, more accurate investigations on the interfacial performance of the fibers-matrix, the single fiber pullout tests were performed and interfacial properties such as bond strength (τ_{max}), frictional bond strength (τ_i), and chemical debonding energy (G_d) were measured. The results of the typical successful pullout tests are presented in Figure 6-24. Three main regions were observed at the pullout load slip curve in Figure 6-24 (a). Initially, a stable debonding process of the fibers happens along with the interface of fiber and matrix at the first region. In this zone, the embedded end of the fibers doesn't move. Meanwhile,

the debonded length of the fiber increases until reaches the embedded length, and the fiber's pullout resistance grows up to P_{max} .

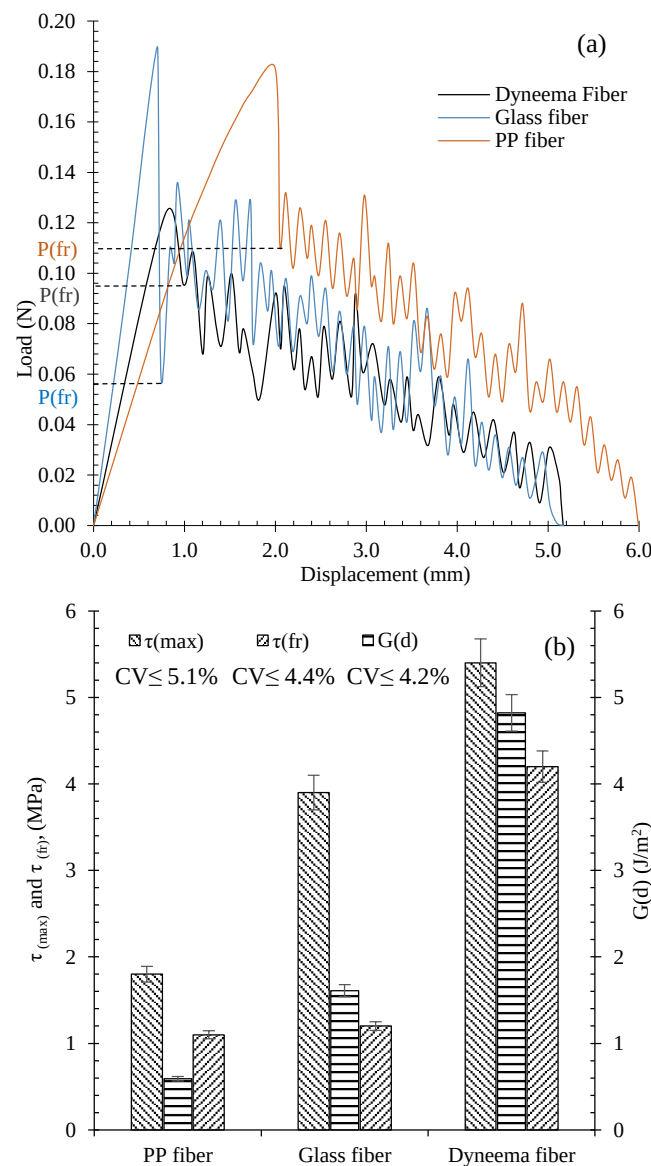


Figure 6-24. The single-fiber pullout test results of different fibers: a) Pull-out load/ displacement curves, b) Interfacial properties.

The measured displacements at this phase attributed only to the debonded fiber elastic stretching and the free length of the fibers. In the second region, the load suddenly declines from P_{max} to P_{fr} which corresponds to the breaking of the chemical bond between the fiber and the matrix. Finally, at the third zone, at the slippage region, the load of the fibers is resisted by frictional forces [484, 487, 488]. Three bonding factors were calculated based on this behavior and results are shown in Figure 6-24 (b). As can be apperceived the chemical debonding energy (G_d) and frictional bond strength (τ_{fr}) of the PP fiber are about 0.59 J/m² and 1.1 MPa respectively, and the bond strength (τ_{max}) for this fiber was obtained around

1.8 MPa. This result shows that frictional bond strength acts a crucial role in the PP bond strength among the cementitious matrix.

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6.3.3. Mechanical Properties

The unconfined compressive strength (UCS) of reinforced CS with different types and concentration of fibers after 28 days of hydration is shown in Figure 6-25. As can be observed the incorporation of certain concentrations of the fibers into the CS caused an increase in the UCS of the composite where this concentration was heavily dependent on the properties of the fibers. Indeed, reinforcing CS containing 0.17% of CNMs by 0.5% PP fibers, increased the UCS by around 21% compared to the plain specimen while excessive increasing of the PP fibers concentration to 1.0%, and 1.5%, led to the reduction of the CS of around 11% and 31%, respectively.

In the case of glass fibers, the incorporation of 0.5% and 1.0% glass fiber into the composite increased the UCS by about 8% and 29%, respectively, and increasing the fiber concentration beyond 1.0% to 1.5% caused a UCS reduction of around 6%. The trend of the results in Dyneema fibers showed a different narrative, reinforcing the CS by all concentration of Dyneema fibers in the range of 0.5% to 1.5% led to the improvement of the UCS in such a way that incorporation of 0.5%, 1.0%, and 1.5% caused the UCS increment by around 32%, 82%, and 40% compared to the plain CS.

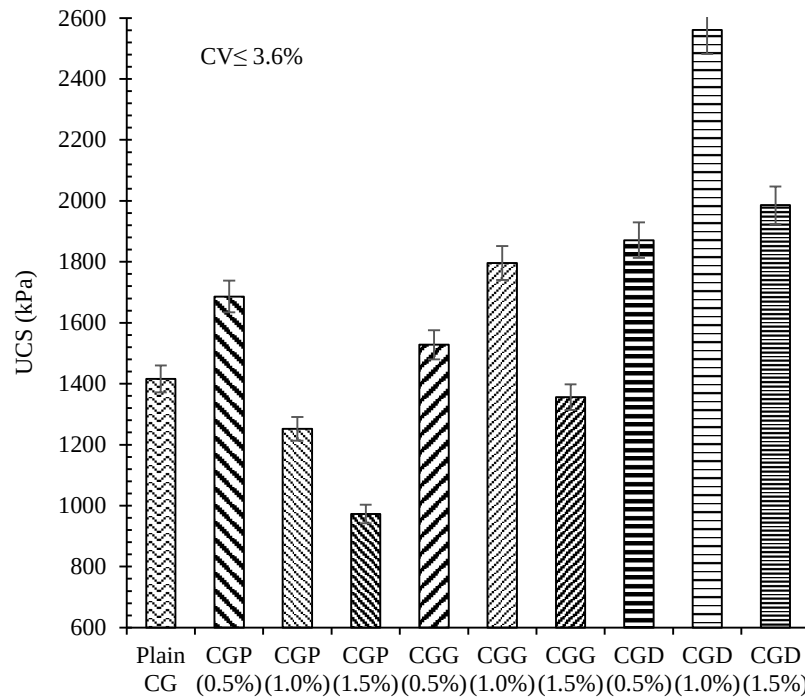


Figure 6-25. Unconfined compressive strength (UCS) of reinforced CS with different types and concentrations of the fibers after 28 days of hydration.

Hence, based on the above results, the optimum concentration of the PP was obtained around 0.5% in terms of the UCS enhancement, while this amount for glass and Dyneema fibers was approximately 1.0%. The reason for this issue is dependent on the surface condition, aspect ratio, and ductility of the fibers. Indeed, a certain ratio of the ductility and strength of fiber is required to avoid the occurrence of the shear rupture on the surface and/or around the fiber particularly in the case of high interaction between the fiber and composite. The stress–strain curves of reinforced CS with different types and concentration of the fibers after 28 days of hydration, are shown in Figure 6-26.

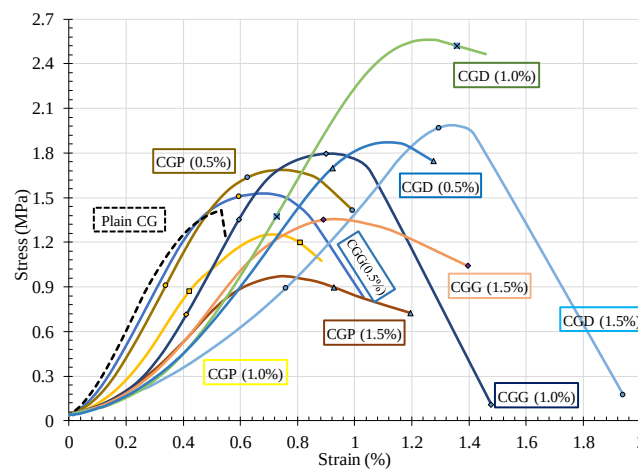


Figure 6-26. The stress–strain curves of reinforced CS with different types and concentration of the fibers after 28 days of hydration.

The results showed that incorporation of fibers into the CS increased the ductility of the composite. A similar trend was reported in other studies performed on the effects of PP and glass fibers on the mechanical behavior of cementitious stabilized sand [176, 489-492]. As illustrated in Figure 6-26, specimens composed of Dyneema fibers showed more ductile behavior and also more dissipated energy after failure. The axial strain at failure (maximum stress) and the tangent modulus at 50% of the peak stress ($E_{(50\%)}$) of reinforced CS with different types and concentrations of the fibers, which is shown in Figure 6-27, also proved this issue. Indeed, as can be seen, the incorporation of 0.5%, 1.0%, and 1.5% PP fibers into the CS containing 0.17% of CNMs, increased the failure strain by around 30%, 38%, and 44%, while this amounts for glass fiber reinforcement, were obtained 26%, 67%, and 75% respectively. The failure strain increment of CS affected by Dyneema fiber reinforcement were 106%, 142%, and 153%, respectively.

The addition of fiber also decreased the $E_{(50\%)}$ of the CS such a way that the incorporation 0.5%, 1.0%, and 1.5% PP fiber into the composite reduced the $E_{(50\%)}$ by around 12, 27, and 50% in comparison to the plain CS. Reinforcing of the CS by similar concentration of glass fibers also caused a reduction in the $E_{(50\%)}$ by 10, 17, and 40%, respectively, while these amounts for Dyneema fibers were around 35, 25, and 46%.

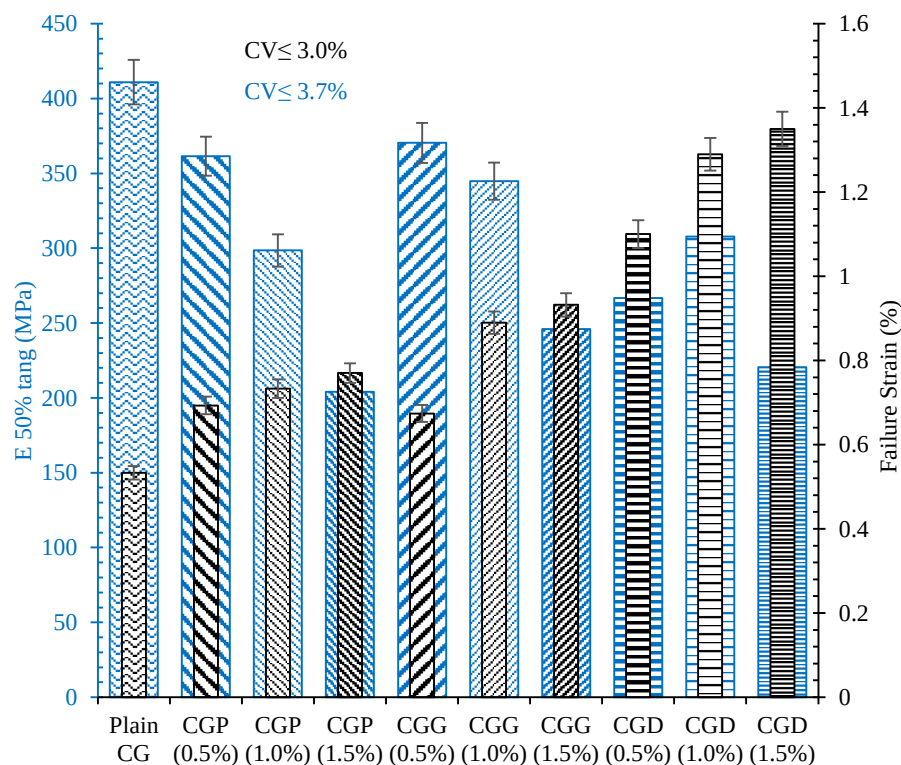


Figure 6-27. The amounts of the strain at rupture point and $E_{(50\%)}$ for reinforced CS with different types and concentrations of the fibers after 28 days of hydration.

6.3.4. Durability evaluations

In order to evaluate the durability of fiber-reinforced CS, we calculated the weight loss of the composite during 12 cycles of wetting and drying, and this is presented in Figure 6-28.

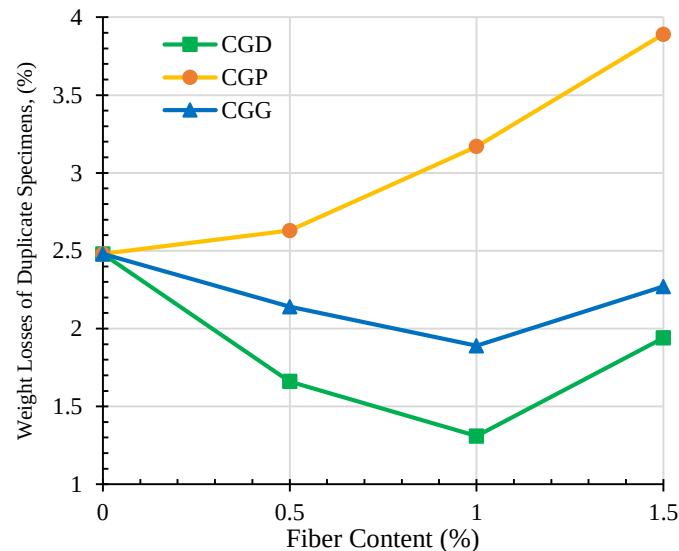


Figure 6-28. Weight loss of reinforced CS with different types and concentrations of the fibers against wetting and drying cycles.

The results showed that the incorporation of PP fibers in the range of 0.5% to 1.5% into the CS composite led to the reduction of composite resistance against wetting and drying cycles in such a way that specimens composed of 0.5%, 1.0%, and 1.5% PP lost weight of around 2.63%, 3.17%, and 3.9 %, respectively.

Reinforcing plain CS containing 0.17% CNMs with glass and Dyneema fibers increased the composite durability; however, excessive increases of the fiber concentration beyond 1.0% caused an increase in the weight loss against wetting and drying cycles that showed a downward trend in terms of durability.

The incorporation 0.5%, 1.0%, and 1.5% glass and Dyneema fiber into the plain CS demonstrated 2.1%, 1.8%, and 2.2% as well as 1.6%, 1.3%, and 1.9% weight loss, respectively, while this amount for plain CS was obtained at around 2.5%.

6.3.5. Piezoresistivity Investigations

Electrical resistivity

The results of the electrical resistivity measurements for reinforced CS with different types and concentrations of the fibers after 72 h of oven-drying are shown in Figure 6-29. It is clear that incorporation of microfiber into the plain CS, which contained 0.17% hybrid CNT/GNP, reduced the electrical conductivity of the composite.

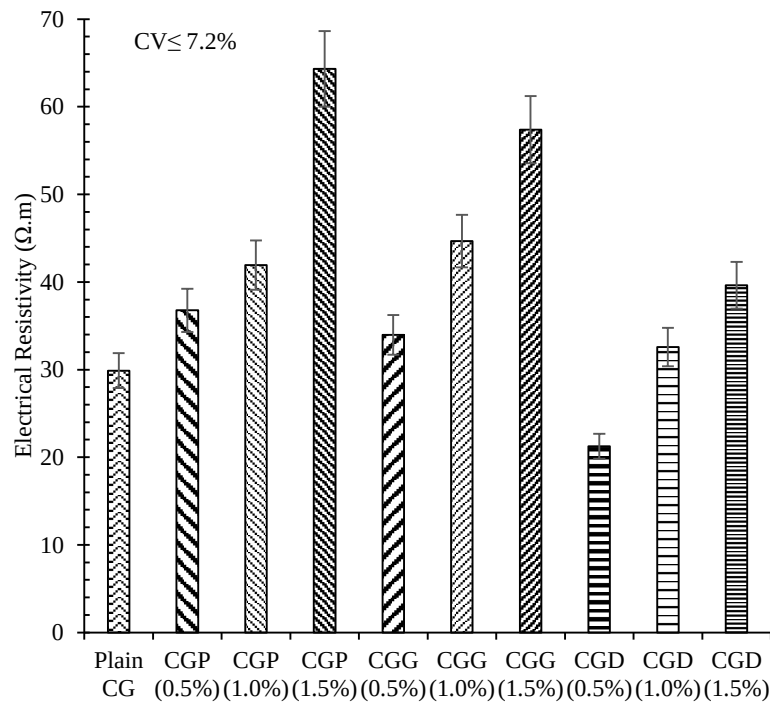


Figure 6-29. Electrical resistivity of reinforced CS with different types and concentrations of fibers after 72 h of oven-drying. As can be observed reinforcing the CS by 0.5%, 1.0%, and 1.5% PP and glass fibers caused a reduction in the electrical conductivity by around 23%, 40%, and 115% as well as 13%, 49%, and 92%, respectively. This indicates that the reinforcing of the composite by microfibers is likely to increase the percolation threshold. Besides the rectangular shape of the PP cross-section with relatively large dimensions increased the electrical resistivity of the composite more than with glass fibers. The non-conductive microfiber with a dense microstructure also acted as a large obstacle and reduced the electron tunneling effects.

In the case of Dyneema fiber, a different trend was observed. Although, the addition of the low fiber concentration to the CS increased the conductivity of the composite, increasing the fiber concentration reduced this. The specimen composed of (0.5) % of Dyneema fiber showed a resistivity reduction of around 29%, while the electrical resistivity of the specimens containing 1.0% and 1.5% Dyneema demonstrated 10 and 32% more electrical resistivity than plain CS, respectively. The smaller cross-section and porous structure of the Dyneema fiber which can absorb a negligible amount of water is likely the reason for this issue.

Piezoresistivity response under cyclic compression test

The variation of fractional changes in the electrical resistivity and axial strain under cyclic compression loading for different fiber-reinforced composites is illustrated in Figure 6-30. Generally, under compression cyclic loading, the negative values were achieved for FCR due to the decrease in electrical resistance

during loading compared to the primary electrical resistance (Eq. 7). In compression loading, the conductive path becomes closer together, which causes an increase in the electrical conductivity and, consequently, reduces the electrical resistivity. The results showed that the changes in electrical resistivity increased by increasing the strain affected by the load increasing, and, in contrast, the FCR was decreased by load reduction.

At the end of each loading cycle, a lower residual strain and FCR remained in the fiber-reinforced specimen compared to the plain CS, which contained 0.17% CNM. This is likely due to the ductility improvement of the fiber-reinforced composite. The maximum amount of strain and the FCR in addition to the residual strain and FCR at the end of the third cycle of the loading for fiber-reinforced and non-reinforced specimens are shown in Figure 6-31. The results showed that the maximum amounts of strain and FCR for fiber-reinforced specimens at the pick of the load were higher than for the plain composite. However, the residual strain and FCR at the end of unloading were much lower, which indicates the positive effects of fiber in terms of ductility behavior enhancement and, consequently, the more stable and reliable piezoresistivity behaviors.

What's more, the incorporation of the fiber into the CS can prevent crack propagation due to a reduction of the stiffness, which can improve the long-term performance. By comparing the results of this study with previous research [207], we also found that the residual strain and FCR in the fiber-reinforced specimen were significantly lower than the composite, which was only composed of CNT and GNPs.

The results also showed an increase in the FCR and strain at the end of loading that becomes increase by increasing the loading cycle number. These amounts were more obvious in the PP and glass fiber-reinforced specimens, particularly with high fiber concentrations. As can be observed in Figure 6-30 (a) and (b), in PP and glass fiber-reinforced composites, the amounts of the FCR increased with the increasing fiber concentration due to the increasing strain and ductility of the specimens.

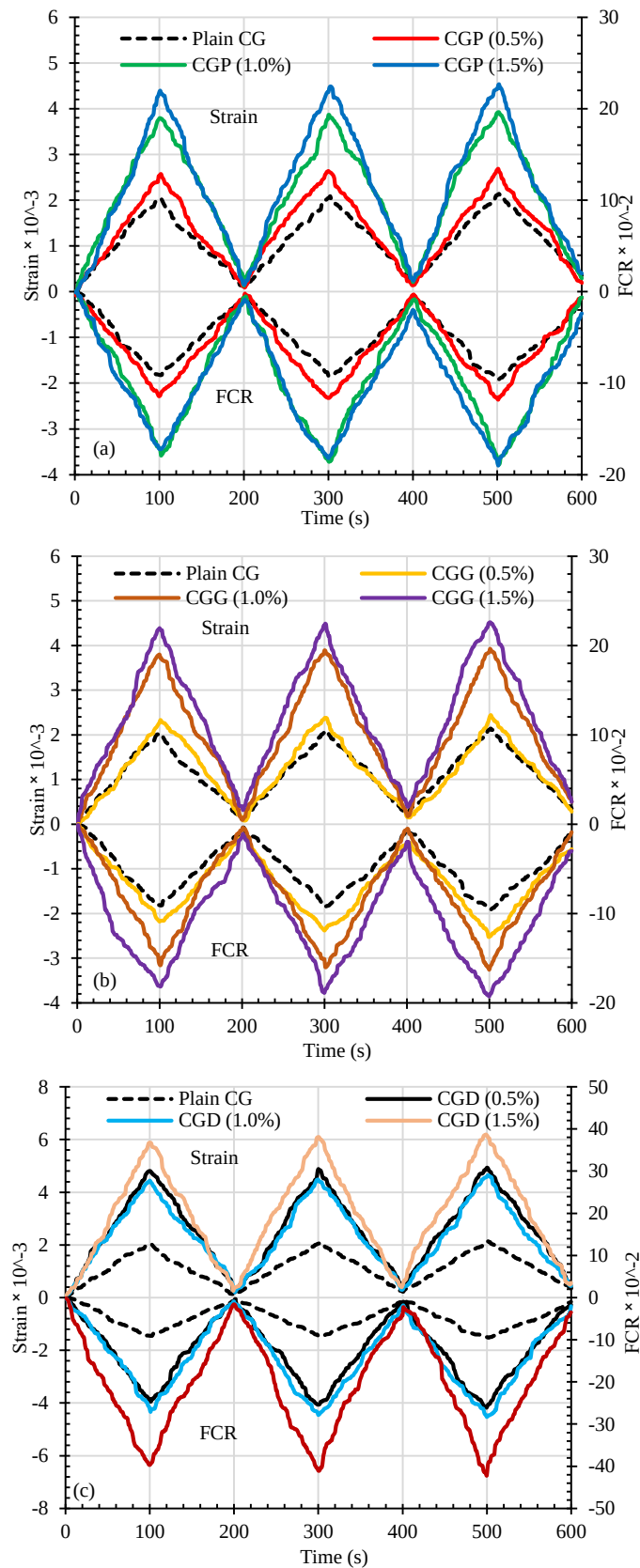


Figure 6-30. Variation of the fractional changes in the electrical resistivity (FCR) vs. the strain for reinforced CS with different types and concentrations of the fibers: a) PP fiber, b) glass fiber, c) Dyneema fiber.

In the case of Dyneema fiber-reinforced CS (Figure 6-30 (c)), increasing the fiber concentration from 0.5% to 1.0% reduced the FCR, due to the strain reduction affected by the higher stiffness of this specimen,

while by increasing the fiber concentration to (1.5) %, the strain and consequently FCR was increased again.

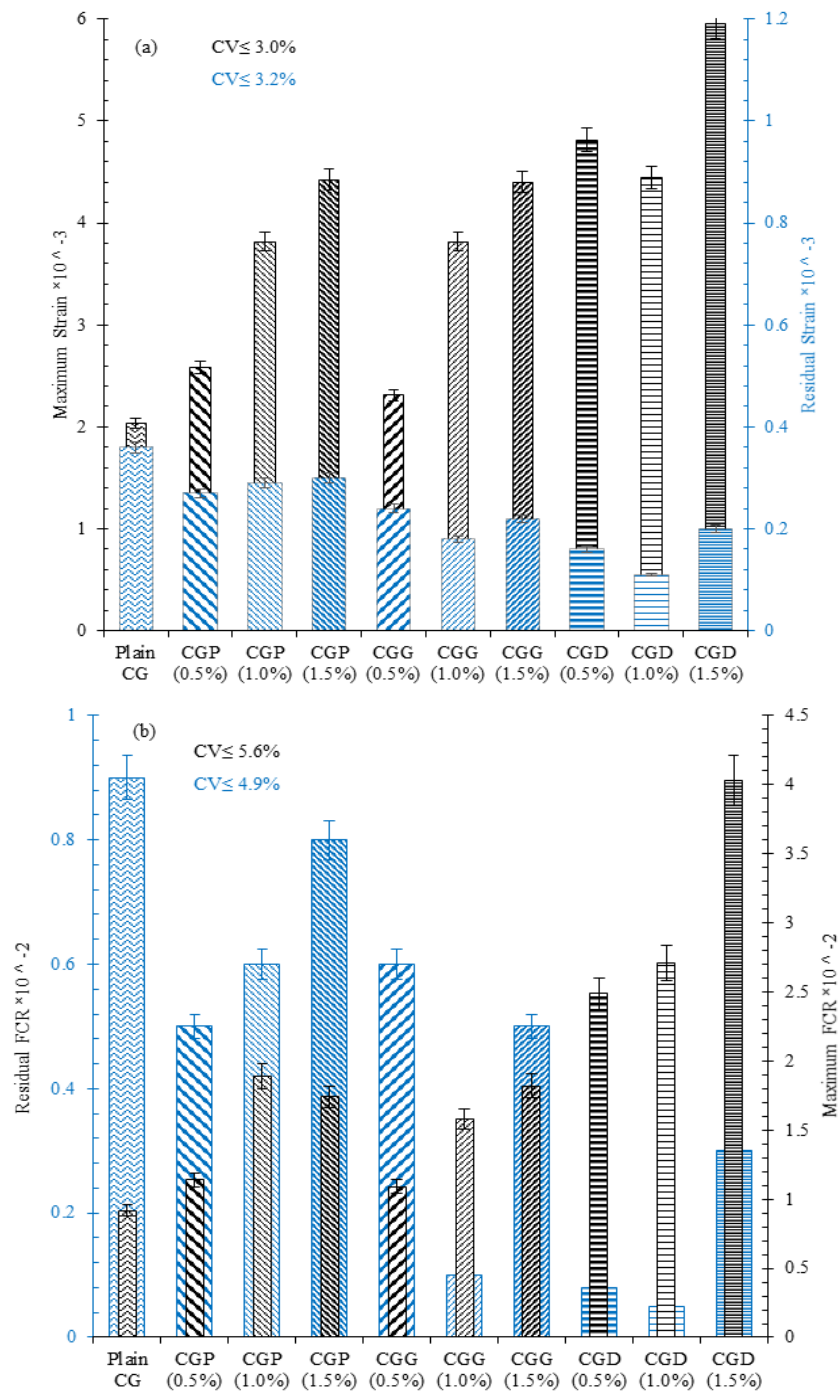


Figure 6-31. The strain and FCR for fiber-reinforced and non-reinforced specimens: a) maximum and residual strain, b) maximum and residual FCR.

In order to better evaluate the relation between fractional changes in the electrical resistivity and strain under cyclic compressive loading, the variation of FCR together with the axial strain for different fiber-reinforced specimens is presented in Figure 6-32. Generally, the power functions were found to better express the relation between the FCR and strain.

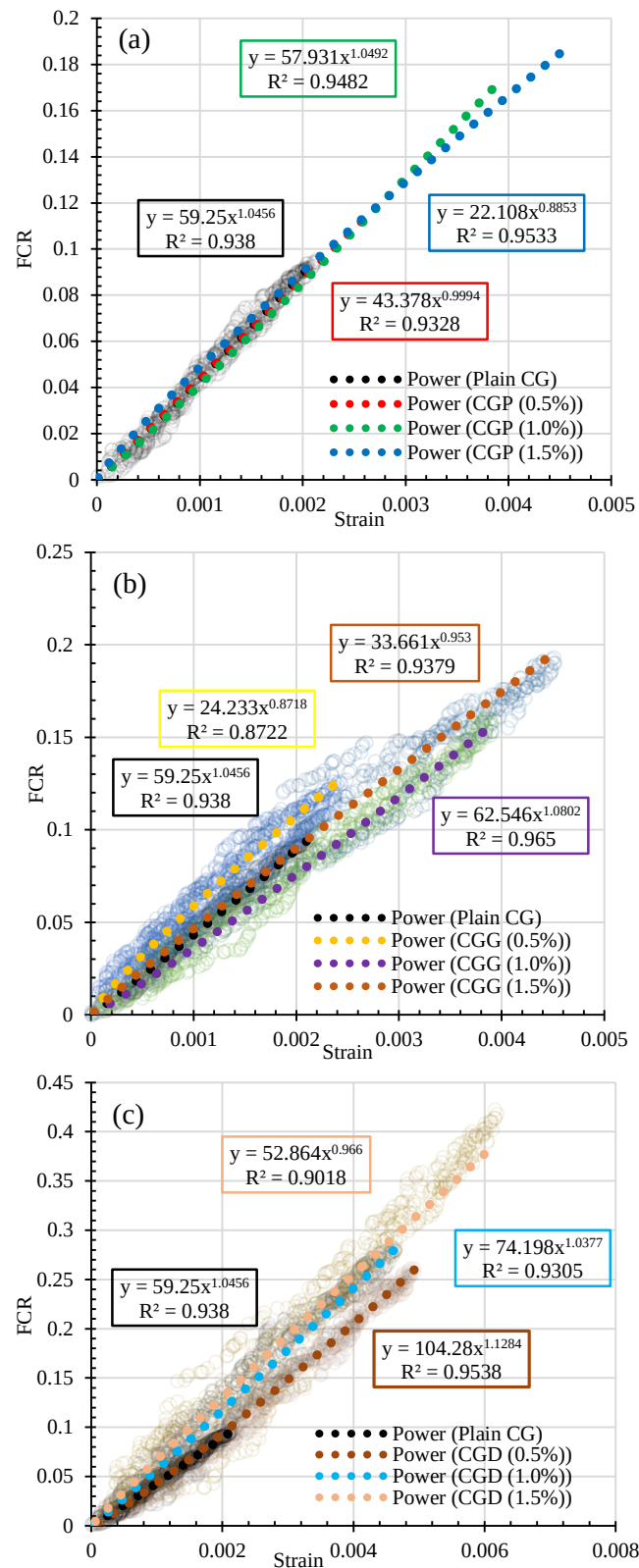


Figure 6-32. The relation between the FCR and strain for reinforced CS with different types and concentrations of fibers: a) PP fiber, b) glass fiber, c) Dyneema fiber.

In PP fiber-reinforced CS (Figure 6-32, (a)) however, the slopes of the curves were increased sharply at the initial phase of the loading, the slope was decreased by excessive load increasing, which indicates an

excessive reduction of the distance between the conductive paths due to high strain, which prevents significant changes.

This ambivalent trend was more explicit in specimen CGP (1.5) %, which contained a higher amount of PP fiber and showed more ductile behavior. It should be noted that the scatters of the data were also decreased by increasing the PP fiber concentration. In the case of glass fiber-reinforced CS (Figure 6-32, (b)), this ambivalent trend was observed in the slopes of the curves, especially in specimen CGG (1.5) %. In the glass fiber-reinforced composites, the scatter of the data was also reduced by increasing the fiber concentration. In the Dyneema reinforced specimens (Figure 6-32, (c)), a reverse trend was observed, and the scattering of the data was increased by increasing the fiber concentration due to the mechanical interactions between the composite components and fibers under the load. In Dyneema reinforced specimens, however, the slopes of the curves were gradually raised by the increasing fiber concentration, the ambivalent trend was still visible in the curve slopes.

Gauge Factors

The results of calculated gauge factors for reinforced CS with different types and concentration of the fibers are demonstrated in Figure 6-33.

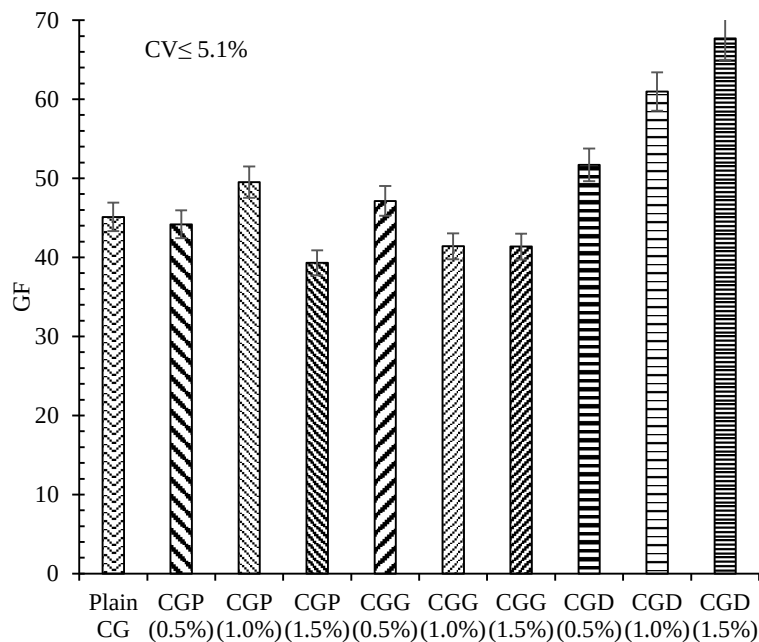


Figure 6-33. Gauge factor variations for reinforced CS with different types and concentrations of the fibers: a) PP fiber, b) glass fiber, c) Dyneema fiber.

The amounts of the strain and FCR are two effective parameters in terms of determining the sensitivity or the gauge factor of the specimen (Eq. 8). By considering this issue and the above discussions, as can be observed in Figure 6-33, the maximum gauge factors were achieved for Dyneema fiber-reinforced

specimens in such a way that specimens composed of 0.5%, 1.0%, and 1.5% of Dyneema fibers showed gauge factors equivalent to 52, 61, and 68, respectively, while the gauge factor of the plain CS containing 0.17% CNMs was about 45.

In the case of PP and glass fiber-reinforced composite, the incorporation of 0.5%, 1.0%, and 1.5% fibers into the CS, led to a gain in the gauge factors equal to 44, 49, 39, and 47, 42, 41 respectively.

6.4 Sustainable Issues

Sustainability focuses on addressing the desires of the present without undermining the ability of future generations to fulfill their requirements. The concept of sustainability is composed of three pillars: environmental friendliness, economics, and social values also known informally as the planet, profits, and people [493].

Generally, the AR-glass fiber has lower environmental impacts compared with polymer-based fibers, such as PP and Dyneema [494, 495]. Given that the approximate price of AR-glass fiber and propylene is the same and is about one-third of the Dyneema fibers, this makes the AR-glass fibers appear more ideal, despite the high impact of Dyneema compared to glass fibers. Indeed, in a project where high sensitivity, ductility, and strength of the composite is not required, the remarkable effects of Dyneema fiber in terms of 82% UCS improvement, 25% elastic modulus reduction, and a gauge factor equal to 61, can be balanced with a low cost solution satisfied by glass fiber with 29% UCS improvement, 17% modulus reduction, and a gauge factor equal to 42.

6.5 Conclusions

In this study, the effects of the fibers on the physical, mechanical, durability, microstructure, and piezoresistivity behavior of reinforced cemented sand incorporating hybrid carbon nanotubes (CNT) and graphene nanoplatelets (GNP) were evaluated to achieve a novel ductile self-sensing fiber-reinforced cementitious composite. For this purpose, 0.5%, 1.0%, and 1.5% of a novel fiber called Dyneema and two conventional fibers, glass and polypropylene, were treated by chemical methods and incorporated into the cemented sand (CS) containing 0.17% hybrid CNT/GNP (1:1). From the experimental results obtained and deeply analyzed after 28 days of hydration in at least 144 of the compacted composites, our conclusions are summarized as follows:

- Scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and Fourier transform infrared (FTIR) analysis of the fibers showed that the surface treatments of PP and Dyneema fibers with sulfuric acid, in addition to surface roughing

and rippling, caused the formation of different oxygen functional groups on the fiber surface including: (-OH), (-COO), (-O-), and (-C-SO₃). Treatment of the glass fiber with hydrofluoric acid also led to the formation of (-OH), (-SiF), and (-ZnF) functional groups on its surface.

- The single fiber pullout test also showed great interfacial performance for Dyneema fiber so that chemical debonding energy, frictional bond strength, and bond strength of this fiber were obtained 3, 3.5, and 1.3 times more than Glass and 8.1, 3.8, and 3 times more than PP fiber respectively.
- The incorporation of PP fiber with concentrations in the range of 0.5% to 1.5%, into the plain CS, which contained 0.17% carbon nanomaterials (CNMs), reduced the UCS. Although increasing the glass and Dyneema fiber up to 1.0% increased the UCS of the CS (29% and 82% respectively). An excessive increasing of the fiber concentration led to a UCS reduction.
- Evaluation of the tangent modulus at 50% of the peak stress ($E_{(50\%)}$) for the fiber-reinforced specimens demonstrated that Dyneema, glass, and PP fibers were respectively more efficient in terms of ductility improvement of the composite.
- Reinforcing the plain CS with 0.5%, 1.5%, and 1.5% of PP fiber led to increasing the optimum water content as well as a reduction in the maximum dry density. While the incorporation 1.0% of the glass and Dyneema fiber increased the maximum dry density and caused a reduction in the optimum water content. It should be noted that increasing the fiber concentration beyond 1.0% reduced the maximum dry density and increased the optimum water content. A quasi unique relation between ρ_{dmax} vs. "Sr-Sr (opt)" showed the potential for real field applications.
- The non-destructive ultrasonic test also showed a denser microstructure after 28 days of hydration for reinforced CS by 1.0% of Dyneema and glass fibers compared to the other specimens.
- The Dyneema and glass fibers showed better bonding and interactions with the cementitious matrix.
- Specimens reinforced by 1.0% of Dyneema and glass fibers demonstrated a lower weight loss after 12 wetting and drying cycles (durability) compared to the other specimens.
- The maximum gauge factors were achieved for Dyneema fiber-reinforced specimens in such a way that specimens composed of 0.5%, 1.0%, and 1.5% Dyneema fibers showed gauge factors

equivalent to 52, 61, and 68 respectively, while the gauge factor of the plain CS containing 0.17% CNMs obtained about 45. In the case of PP and glass fiber-reinforced composites, the incorporation of 0.5%, 1.0%, and 1.5% fibers into the CS, led to a gain in the gauge factors equal to 44, 49, and 39, as well as 47, 42, and 41, respectively.

- From a sustainable perspective, the reinforcement with glass fiber could be a compromise to achieve a low cost and environmentally friendly solution for the development of a novel self-sensing fiber-reinforced cementitious composite.

In summary, we believe that this novel approach contributes to the new era of smart composite materials in intelligent structures. However, the high stiffness and low ductility of the CNMs reinforced composite makes them prone to cracking which necessitates further research in order to achieve solutions to increase their ductility such as fiber reinforcements.

Chapter 7. Effects of Size and Electrodes Layout on Sensing Potential of Cementitious Geocomposite.

«The devil is in the details. »

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

- Abedi, Mohammadmahdi, Raul Fanguero, and António Gomes Correia. "Effects of electrodes layout and filler scale on percolation threshold and piezoresistivity performances of a cementitious-based geocomposite." *Nanomaterials* 12, no. 10 (2022): 1734. <https://doi.org/10.3390/nano12101734>

Abstract

An extensive experimental study was conducted to investigate the co-effects of surface area and distance between electrodes as well as filler scales on the percolation threshold of piezo-resistive cement stabilised sand. In this route, the electrical resistivity of numerous specimens of different sizes and composed of different content of carbon-based conductive fillers, including carbon nanotubes (CNTs), graphene nanoplatelets (GNPs), and carbon fibres (CFs) with different aspect ratios, were measured. In addition, the numerical relations between the electrical percolation threshold and matrix dimensions were expressed for different conductive fillers. Furthermore, the electrical percolation threshold of two large-scale specimens with different shapes (a $10 \times 10 \times 85$ cm³ beam, and a 15 cm size cube) were predicted through numerical relations, and their piezoresistivity performances were investigated under compression cyclic loading (cube) and flexural cyclic loading (beam). The mechanical properties of the specimens were also evaluated. The results showed that the changes in the length, width, and thickness of the matrix surrounded between electrodes had a significant effect on the electrical percolation threshold. However, the effects of length changes on the percolation threshold were greater than the width and thickness changes. Generally, increasing the aspect ratio of the conductive fillers caused a reduction in the electrical percolation threshold of the cementitious geocomposite. The appropriate piezoresistivity response of the large-scale specimens composed of filler content equal to their percolation threshold (obtained by the numerical relation presented in this study) showed the adequacy of the results in terms of threshold dosage prediction and self-sensing geocomposite design. The results of these studied addressed a crucial factor for design of self-sensing composites and pave the way for the development of field applicable smart cementitious geocomposite.

7.1 Introduction

Intrinsic self-sensing cementitious-based geocomposites (SCGs) are ideal engineering materials for infrastructure monitoring applications. These intelligent materials can help to develop smart infrastructure integrated with health monitoring and sensing abilities, thus improving the safety, serviceability, durability, and reliability of the infrastructure [82, 496]. SCGs provided a new approach for maintaining sustainable development in roller-compacted concrete dams, rammed earth, ground improvement, and particularly in structural layers in transportation infrastructures, especially in critical zones, such as transition zones [56, 497]. A piezoresistive cementitious-based geocomposite is a compounded material, which is composed of a conductive phase distributed in a non-conductive matrix. The conductive phase is often made with one or a mixture of carbon nanomaterials (CNMs) or metallic alloys with different geometrical shapes [46, 107, 498, 499], whereas the non-conductive cementitious phase, or the matrix of the sensor, is commonly compacted cemented sand, mortar, and concrete [500-502]. Indeed, the conductive phase is vital for providing the piezoresistive composites with the ability to sense deflection, strain, stress, cracks, temperature, and humidity [503]. The conductive phase dopes the non-conductive phase through two primary mechanisms, namely, percolation and quantum tunnelling effect [139, 504]. The percolation phenomenon can be explained by the formation of randomly conductive paths that contribute to increased electrical conductivity [81]. However, studies have indicated that the percolation mechanism cannot justify and interpret the piezoresistivity of sensors containing discontinuous conductive fillers. Hence, to explain the electron movement in the material, quantum tunnelling effect theory is also often required for the exploration of electrical conductivity in the quantum realm [505]. Based on this theory, there is also a nonzero probability that electrons with lower energy can overcome a potential energy barrier, and thus, even with high electrical resistivity and a low external voltage, a minority of electrons can still create a current and move to a place with low potential energy [506-508]. The distance beyond which the electrons are not able to pass through a non-conductive matrix or at which tunnelling conductivity becomes invalid is called the “tunnelling distance.” Considering both the percolation mechanism and quantum tunnelling effect, the critical volume fraction of a conductive phase at which a composite can undergo a transition from a non-conductor to a conductor is called the “percolation threshold” [81, 509]. However, percolation does not imply a fully physical connection, owing to the tunnelling conductivity. Indeed, percolation threshold content and type of the filler are the most important factors to achieve economical self-sensing cementitious composites with proper mechanical properties and potential of use in the fields [3, 46, 56]. Literature assessment shows that many studies have been conducted on the factors affecting the percolation threshold including the type of conductive fillers, their aspect ratio and geometrical shapes,

dispersion, type of non-conductive matrix, size and shape of aggregate, etc., [125, 362, 510-512]. However, to transfer laboratory findings to field applications, it is crucial to study the effects of different measurement systems and electrodes configurations on the percolation threshold. In field applications, many laboratory measurement methods may lose their effectiveness due to limitations and scale changes. In addition, in field applications, particularly in bulk forms, by changing the distance, shape and cross-section of the electrodes, the percolation threshold might be changed due to factors such as polarization and contact resistance between electrodes and composite [82]. Chacko et al. [140] and Banthia et al. [84], assessed the effect of electrodes distance on the electrical resistivity of cementitious composites. They showed that the measured electrical resistivity increased with rising electrode space. Indeed, they reported that the electrical resistivity would become constant when the space of electrodes reached a threshold. Based on the hypothesis proposed by Banthia et al. [81, 84], the diminished capacitance effect with rising electrode spaces may be the reason for this phenomena. Furthermore, a larger space of electrodes may also reduce interference from the surface resistance while measuring volume resistance [81]. Although, many studies assessed the effects of electrodes configuration on the electrical resistance of cementitious composites, the coeffects of the electrode surface area and electrode distance on the percolation threshold have received less attention. The importance of this issue becomes more apparent when a special electrode layout is required in field applications, due to special constraints such as size or physical shape. Indeed, insufficient addressing of this point has led to a few successful experiences in self-sensing composites employment in the field particularly in bulk form. The co-effects of electrodes distance and surface area, which have great effects on the percolation threshold content, can be reflected in the boundary conditions or volume of the surrounded matrix between the electrodes (Figure 7-1). Accordingly, in this study, the volume of the matrix refers to the volume surrounded between the two electrodes which means considering the combined effects of electrode surface area and electrodes space. This approach has a crucial contribution to the design for the field application of self-sensing composites.

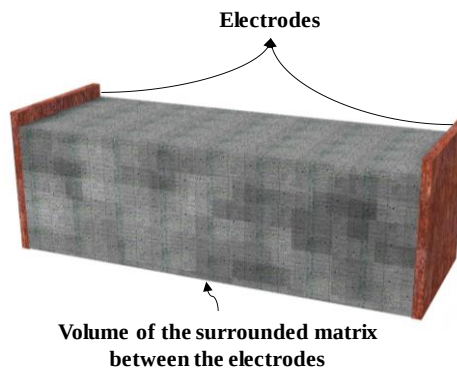


Figure 7-1. The volume of the surrounded matrix between the electrodes.

Hence, in order to address this issue, different content of carbon-based conductive fillers was dispersed into the specimens fabricated using compacted cementitious stabilised sand of different sizes and shapes. The copper electrodes were installed on both ends of the specimens. In this investigation, carbon microfibers (CFs) and carbon nanotubes (CNTs) with different lengths and a hybrid combination of CNT and graphene nanoplatelets (GNPs) were used as conductive fillers. In each case, the electrical resistance of the specimens was measured after 28 d of hydration in order to achieve the percolation threshold of the specimens. The numerical relations between the percolation threshold, volumetric electrical resistance, and matrix volume were also expressed for different conductive fillers. Finally, two large-scale specimens were prepared using the obtained numerical relations to ensure their adequacy. The mechanical performance and piezoresistivity behaviour of these specimens were also investigated under compression and flexural loading cycles. Indeed, the present study elucidate the path for passing from small scale laboratory investigations towards large scale self-sensing geocomposites.

7.2 Materials and Methods

7.2.1. Materials

In order to evaluate the effects of the aspect ratio of the fillers on the electrical percolation threshold, three types of multiwall carbon nanotubes (MWCNTs) and CFs with different lengths were used. The GNPs used in this study were of a multilayer variety with a diameter of approximately 5-10 μm . Carbon nanomaterials and CFs were supplied by Sigma Aldrich and Toho Tenax company respectively. Additional specifications of these carbon-based fillers are presented in Table 7-1 [46, 145, 409].

Table 7-1. Characterization of Multi-wall carbon nanotube (MWCNT), graphene nanoplatelets (GNP), and short carbon fibre (CF)

Characterization	Carbon content (%)	Length (μm)	Outside Diameter (nm)	Density (g/cm^3)	Layers	Form
CNT (5-10)	> 98	5-10	7-15	2.21	-	Black powder
CNT (10-30)	> 98	10-30	< 8	2.27	-	Black powder
CNT (40-50)	> 95	40-50	8-15	2.34	-	Black powder
GNP	> 99.5	5-10	4-20	2.25	$10 < n < 60$	Gray powder
CF (5)		5000				
CF (10)	> 93	10000	7000	1.87	-	Black microfiber
CF (20)		20000				

A compatible noncovalent surfactant with a central hydrophobic chain of polyoxypropylene (PPO) and two hydrophilic side chains of polyoxyethylene (PEO), Pluronic F-127 (Sigma Aldrich), was used to disperse the CNMs. In addition, tributyl phosphate (TBP, 97%, Sigma Aldrich) was used as an antifoaming agent and dispersion booster, according to a previous study [145]. In this study, siliceous and clean particles of CEN-standard sand were used. The classification of this sand following the unified soil classification system (USC) was a well-graded class. The physical properties of the sand are listed in Table 7-2. The grading was measured by sieving according to ISO 679: 2009 and EN 196-1 standard requirements. In this study, ordinary Portland cement type I (CEM I 42.5R) from Secil company was also used as a binder for SCG fabrication [409].

Table 7-2. Sand particle size distribution.

Mesh Size (mm)	0.08	0.16	0.5	1	1.6	2
Cumulative retained (%)	99 $\pm$ 1	87 $\pm$ 5	67 $\pm$ 5	33 $\pm$ 5	7 $\pm$ 1	0
Specific gravity G_s	2.67		C_u^a	7.5	C_c^b	1.8

^a Uniformity coefficient, ^b Curvature coefficient

7.2.2. Samples Preparation

The benefits of the synergistic effects of CNT/GNPs when combining them to develop economical self-sensing composites with high sensitivity and low percolation thresholds have been mentioned in several studies [46, 112, 513-518]. Based on this different content of CNT/GNP combination (at a 1:1 ratio) were used to investigate the role of the matrix shape and size effects. The specimens were fabricated by gradually increasing the dimensions in different directions, as shown in Figure 7-2.

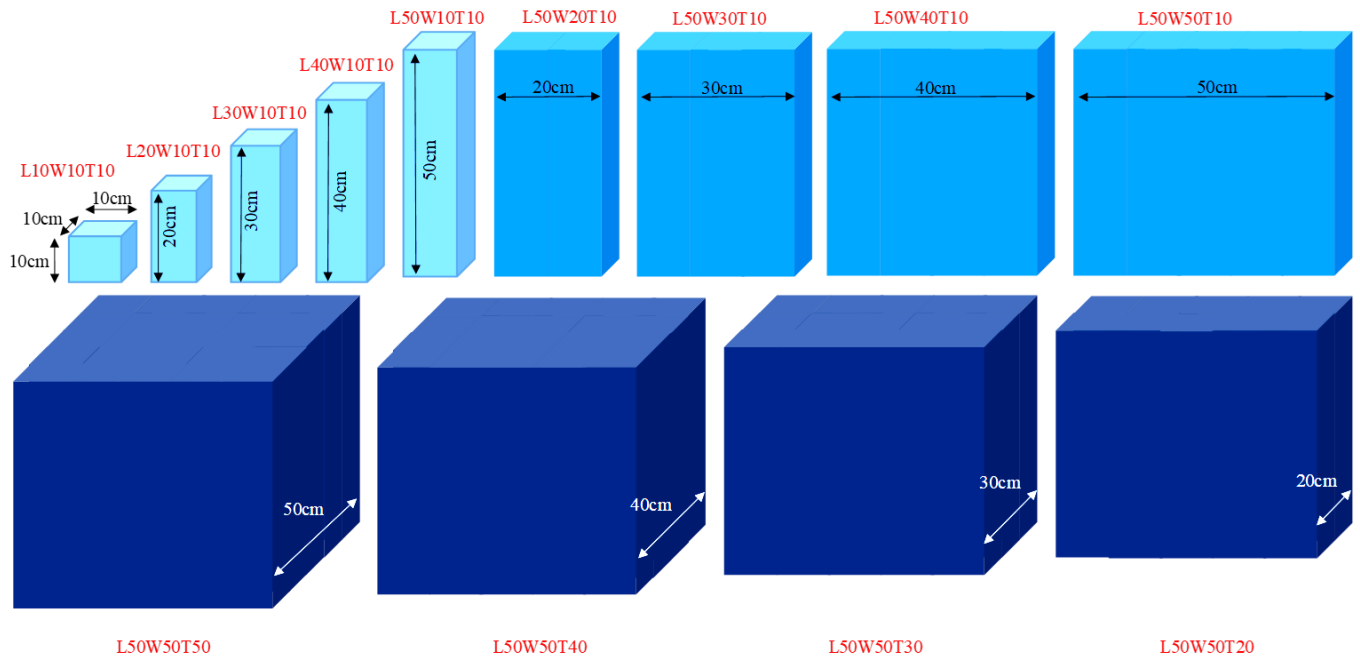


Figure 7-2. Schematic diagram of the specimens' dimensions (reinforced by hybrid CNT/GNP, 1:1), L: length, W: width, T: thickness

In order to evaluate the effects of the aspect ratio of the conductive fillers on the electrical percolation threshold in different matrix volumes (surrounded between electrodes), three different lengths of CF and CNT at different content were separately incorporated into cubic specimens with similar aspect ratios. The dimensions of the specimens gradually increased, as shown in Figure 7-3. The reason for choosing cubic specimens was to ignore the effect of changes in the aspect ratio of the matrix.

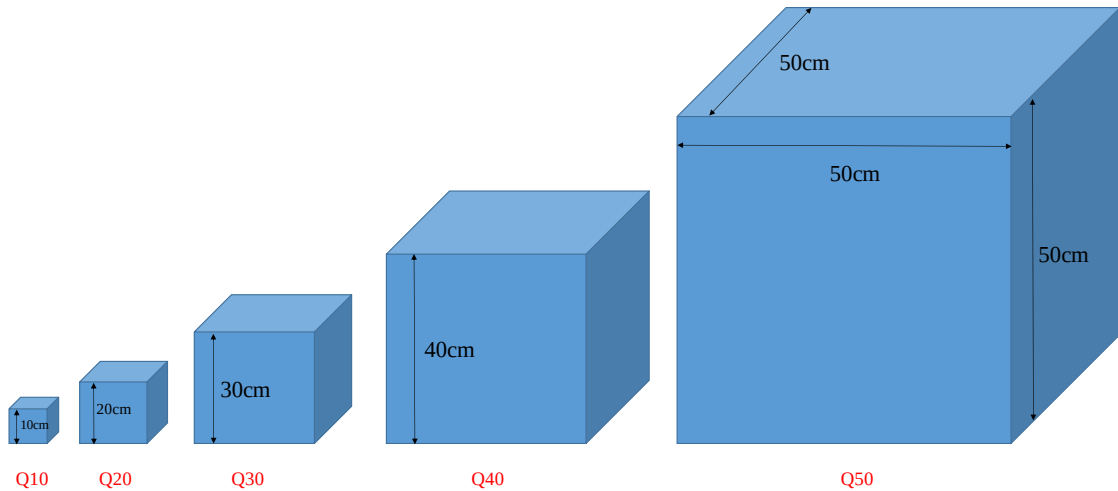


Figure 7-3. Schematic diagram of the specimens' dimensions (reinforced by different length of CF and CNT, separately).

Since, in sand stabilization, the cement content usually varies by approximately 10% owing to the target strength of the sand–cement [147, 148], in this study, 10% cement (by the weight of the dry sand) was also utilized for SCG preparation. Based on the literature [3, 56, 57], the optimum water content for the CNM-reinforced stabilised sand with similar cement content and sand grading was around 7%. Hence,

the amount of water content for all the specimens was considered to be 7% (by the weight of the dry sand) and they were fabricated by the compaction methods. In this route, the CNMs were first dispersed into water through a compatible and effective dispersion method [145, 409]. Then, cement and sand were added to a steel bowl (Grade 304) and blended with a stainless-steel (Grade 304) blade at a rotational speed of 140 rpm for 3 min. Then, the CNM suspensions were sprayed into the mixture and blended at a speed of 285 rpm for another 3 min. In the case of CF, the fibre was first de-bundled through the air in a specialized flask at high pressure and blended at a speed of 285 rpm with sand and cement for 5 min. Then, water was sprayed into the mixture and blended at the same speed for another 3 min. Thereafter, the mixture was stored into a plastic bag to retain a constant moisture content. The split moulds were filled with the wet mixture in three equal-height layers. For each layer, the well-mixed wet mixture was poured into the split mould and then compacted carefully using a metallic electrical hammer to the desired height (controlled by Vernier calliper of accuracy 0.02 mm). The compaction of each layer continued as long as the weight of the specimen was not changed (to an accuracy of 0.5 g). In all specimens, two copper meshes (mesh size $\approx 2.5 \times 2.5$ mm) with dimensions equal to the cross-section of the specimen were placed at both ends as electrodes (along the length, top, and bottom in Figure 7-2 and Figure 7-3) to completely cover the matrix. For the piezoresistivity behaviour of the geocomposite only one specimen was used in each case.

7.2.3. Electrical Resistivity Measurement

As mentioned, in this study the volume of the matrix refers to the volume of the cross-section between the two electrodes (Figure 7-1). For this reason, in each case, the size of the electrode was considered equal to the cross-sectional size of the specimens and placed at the boundary of them to completely surround the specimen.

The biphasic DC electrical measurement approach was used in this study to avoid the polarization effects [519]. The biphasic DC method is an effective technique to determine fractional changes in the electrical resistance of cementitious composites [519, 520]. To assess the electrical resistance of the specimens, a periodic measure/discharge cycle in the form of a 6 Hz square wave ranging from -5 V to +5 V with a duty cycle of 50% was used. The periodic signal was supplied by National Instruments equipped with the Arduino Mega 2560 R3 microcontroller board (Pin 10 and 5).

In this route, the specimens were oven-dried at 70 °C for 72 h to avoid the effects of moisture content on the electrical conductivity values and then was connected in series to a 1000 Ω reference resistor as shown in Figure 7-4.

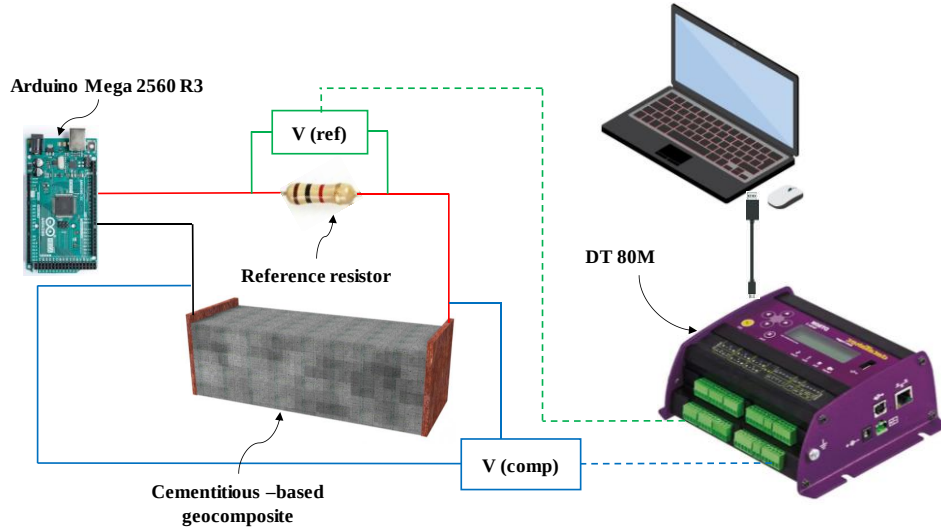


Figure 7-4. Experimental test configuration for electrical resistance measurement.

The potential differences between $V_{(ref)}$ and $V_{(comp)}$ were measured using a data taker DT 80M (accuracy: 10^{-5} V) at time instants of 80% of the positive constant part of the biphasic signal [519]. Eq. (1) was used to calculate the electric current (I) flowing in this circuit, where $V_{(ref)}$ is the voltage drop across the reference resistor and $R_{(ref)}$ is the electrical resistance of this resistor (1000Ω). Due to the complete polarization of the composite, the electrical current (I) achieved a plateau, and the electrical resistance $R_{(comp)}$ was calculated using Eq. (2) [519, 520].

$$I = \frac{V_{(ref)}}{R_{(ref)}} \quad (\text{Eq. 1})$$

$$R_{(comp)} = \frac{V_{(comp)}}{I} \quad (\text{Eq. 2})$$

The volumetric electrical resistance (ρ) for the system composite and electrodes can be estimated using the electrical resistance $R_{(comp)}$, as shown in Eq. (3). It's vital to remember that this value represents more than just the specimen genuine electrical resistivity. The electrical resistance of the electrodes and the contact resistance between composite and electrodes are two other smaller electrical resistances that are related to this characteristic.

$$\rho = \frac{R_{(comp)}}{L} \times A \quad (\text{Eq. 3})$$

where A is the contact surface area between the electrode and the specimen (cross-section), and L is the spacing between the electrodes (length of the specimen). The results were plotted as the volumetric electrical resistance vs conductive filler content diagrams [81, 132, 133, 190]. The percolation threshold content was also calculated from the derivative ($dy/dx=0$) of the relationship between the conductive filler content and the volumetric electrical resistance of the specimens.

7.2.4. Piezoresistivity Investigations

The electrical percolation threshold for a large-scale beam with dimensions of $10 \times 10 \times 85 \text{ cm}^3$ and a cubic 15 cm length specimen were calculated using the relations obtained by hybrid CNT/GNP. To investigate piezoresistivity, a four-probe method and direct current (DC) source was used (Figure 7-5) [56]. The four-probe method is highly recommended for piezoresistivity investigations in previous studies as a more accurate technique for eliminating the influential resistance from current lines, joints, interfaces and polarization effects [81, 225, 521, 522]. The electrical properties and piezoresistivity behaviour of these specimens were evaluated under flexural (three points) and compression cyclic loading, as shown in Figure 7-6. A similar electrical circuit was used in both flexural and compressive loading modes which its details are the same as section 2.3. As shown in Figure 7-5, the strain in compressive and flexural loading was measured by LVDT (RDP ACT 500A, accuracy: 10^{-6} m) and strain gauge (TML/PL 60, gauge resistance: $120 \pm 3 \Omega$, accuracy: 10^{-6} m) respectively.

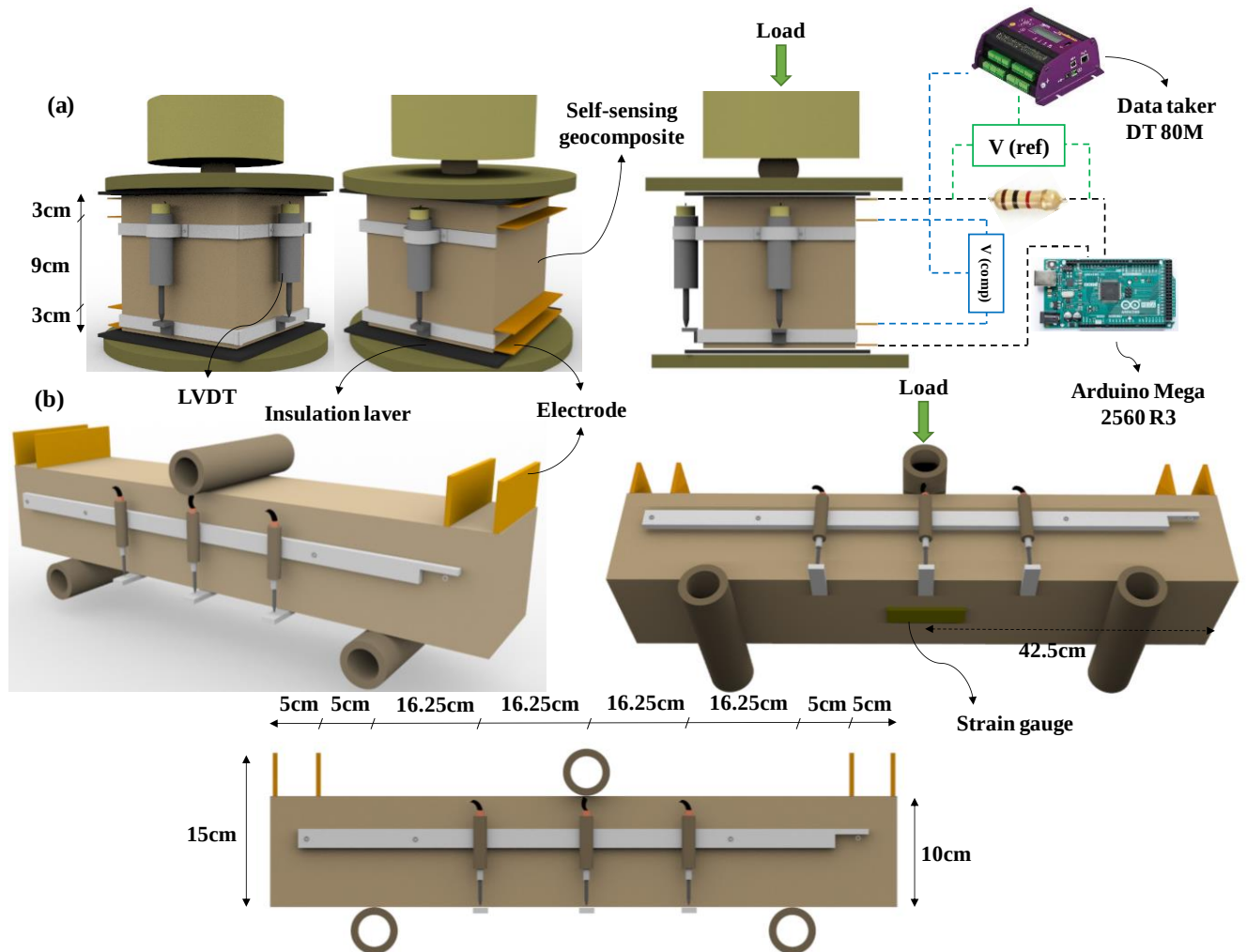


Figure 7-5. Test setup and electrode configuration: a) compression, b) flexural.

Eq. (4) was used to obtain the fractional changes in electrical resistivity (FCR) for the following assessment of composite piezoresistivity:

$$FCR = \frac{\rho(t) - \rho_0}{\rho_0} \quad \text{Eq. (4)}$$

where $\rho(t)$ is the resistivity during the test at time t and ρ_0 is the initial electrical resistivity (before loading). The gauge factor (GF) is also defined as the relative change in electrical resistivity over the strain (Eq. (5)) in order to evaluate the sensitivity of composites:

$$GF = \frac{FCR}{\varepsilon} \quad \text{Eq. (5)}$$

where ε is the axial and flexural strain for the compressive (mean of three LVDT) and flexural (strain gauge) loading modes respectively.

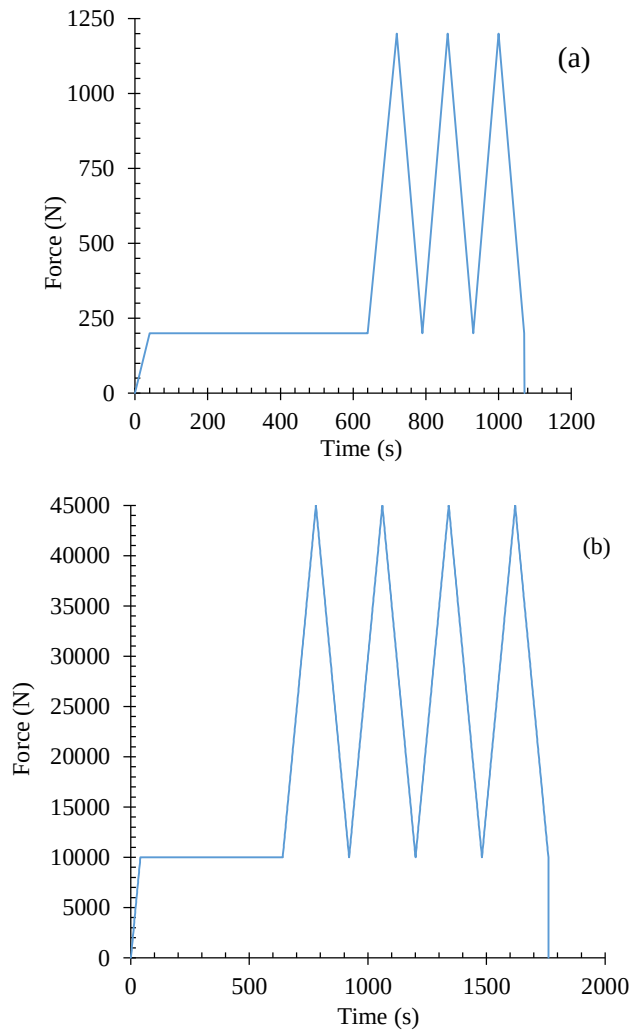


Figure 7-6. Cyclic loading patterns used for piezoresistivity behaviour investigations: a) flexural mode, b) compression mode.

7.2.5. Mechanical properties investigation

The three-point flexural strength of the beam was also measured following EN 1015-11 [46]. Three cubic specimens ($100 \times 100 \times 100 \text{ mm}^3$) were also cut from the beam after the test, and their axial compression strength was evaluated along with the large cubic ($150 \times 150 \times 150 \text{ mm}^3$) specimen. Compressive and flexural elastic moduli were also calculated [46]. The results are obtained by the mean of 3 specimens.

7.3 Results

7.3.1. Effects of Length Changes

The volumetric electrical resistances of specimens with different lengths (surrounded between electrodes) are illustrated in Figure 7-7. The specimens were composed of different content of hybrid CNT/GNP.

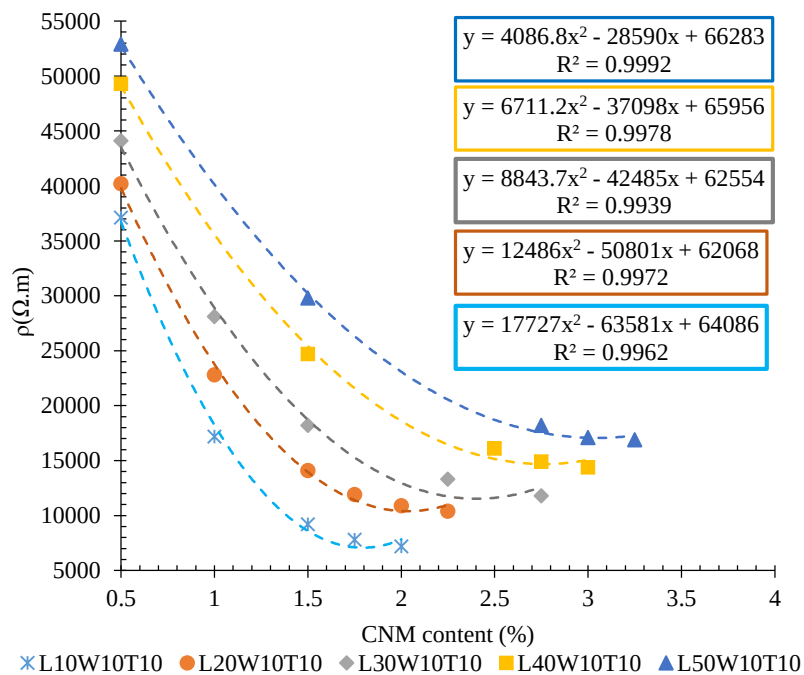


Figure 7-7. Volumetric electrical resistance of CNT/GNP-reinforced specimens with different lengths (surrounded between electrodes).

As can be observed, increasing the length of the matrix (surrounded between electrodes) significantly increased the electrical resistivity of the cementitious composite. Indeed, increasing the length of the specimen, which was composed of 0.5% CNT/GNP, from 10 cm to 20, 30, 40, and 50 cm, caused an increase in volumetric electrical resistance of approximately 10, 19, 33, and 43%, respectively. While these amounts for the specimens containing 1.5% hybrid CNT/GNP were around 53, 97, 168, and 257%, respectively. In fact, the increase in the percolation threshold with the increase in the matrix length might have been one of the reasons for the significant increase in the resistivity of the specimens containing

1.5% CNMs compared to that of the samples containing 0.5% CNMs. To better interpret this issue, the results of electrical resistivity for the specimens with different lengths (surrounded between electrodes), containing 0.5% and 1.5% CNT/GNP, are presented in Figure 7-8. In the specimens with lower lengths, due to the lower percolation threshold (and its proximity to 1.5%), the degree of resistivity reduction caused by increasing the CNM content from 0.5% to 1.5%, was higher than in longer specimens.

In fact, increasing the length of the specimen surrounded by two electrodes means increasing the distance between the electrodes. Accordingly rising in the electrical resistance of composites affected by increasing the distance between the electrodes have been reported in several studies [84, 140]. Generally, the physical and geometrical parameters of the composite have significant effects on the electrical resistance of the cementitious composite. Obviously, by increasing the length of the specimen surrounded by two electrodes and consequently increasing the volume of the covered matrix, the electrical resistance will be an increase. Although the results of the present study show an increase in percolation threshold with increasing electrical resistance, the lack of a clear numerical relationship between the percolation threshold and electrical resistance poses challenges against interpreting the percolation threshold changes and electrical resistance based on physical parameters. However, by a simple approach, it can be conceivable that increasing the matrix volume rise the need for conductive fillers to achieve a lower and approximately constant electrical resistance. This issue required further investigation.

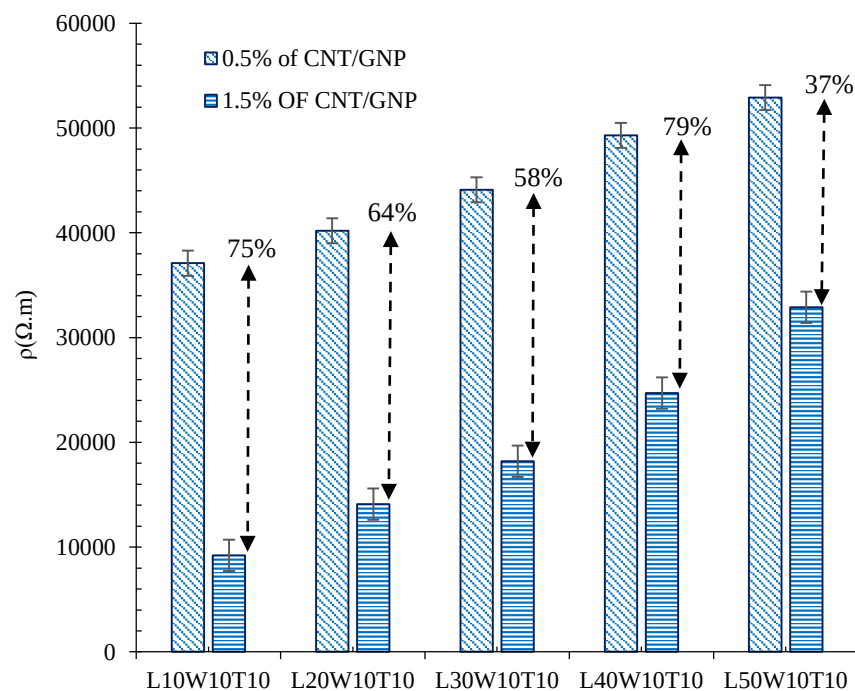


Figure 7-8. Volumetric electrical resistance of the specimens with different lengths and reinforced by 0.5 and 1.5% CNT/GNP.

As shown in Figure 7-7, the slope of the curves generally decreased with increasing length of the specimens (surrounded between electrodes), which might demonstrate the lower performance of these CNMs in terms of increasing the electrical conductivity of the large specimens. Hence, this could be evidence that there is a relationship between the dimensions of the conductive filler and the matrix, which could then be manipulated to achieve high conductivity. However, this issue needs to be further assessed. The electrical percolation threshold of the specimens was also obtained from the derivative of the relationship between the percentage of CNMs and their electrical resistivity, as depicted in Figure 7-9. As can be observed, the percolation threshold of the composite increased with increasing length and, consequently, the volume of the matrix (surrounded between electrodes). A well-defined relationship between the percolation threshold and matrix volume was also obtained with a low approximation using a linear function.

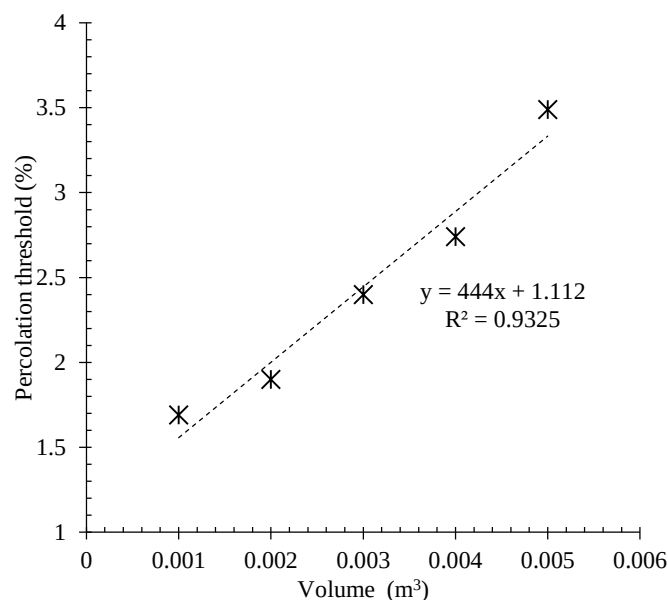


Figure 7-9. Relation between electrical percolation threshold and matrix volume of CNT/GNP reinforced specimens with different lengths.

7.3.2. Effects of Cross-Section Changes

The electrical resistivity of the CNT/GNP-reinforced specimens with different section widths and thicknesses are shown in Figure 7-10. It should be noted that widths and length of matrix cross section (thicknesses) reflect the surface area of the electrodes. With respect to changes in the cross-sectional area, increasing the width of the specimen containing 0.5% hybrid CNT/GNP from 10 cm to 20, 30, 40, and 50 cm increased the volumetric electrical resistance by approximately 10%, 16%, 23%, and 31%, respectively. The results also showed that by increasing the CNM content to 1.5%, the effect of changes in the width of the cross-section on the increase in electrical resistivity also increased, in such a way that increasing the specimen width to 20, 30, 40, and 50 cm led to an increase in the resistivity by

approximately 19, 32, 49, and 62%, respectively. The results of the electrical resistance for specimens with different thicknesses also showed similar trends. As can be seen in Figure 7-11 (b), the volumetric electrical resistance of the specimens containing 0.5% CNMs with thicknesses of 20, 30, 40, and 50 cm increases by approximately 12%, 19%, 27%, and 34%, respectively, compared to the specimen with 10 cm thickness. The calculated percolation thresholds for the specimens with different cross-sectional widths and thicknesses are shown in Figure 7-11 (a) and (b). The relationship between the electrical percolation threshold and variation in the cross-sectional area of the matrix was also expressed by the logarithmic and linear functions. As can be observed, increasing the width or thickness of the matrix cross-section increased the percolation threshold of the composite. However, the slope of the curve of the thickness variation was lower than the width changes, which shows that the width changes had a greater effect on the electrical percolation threshold.

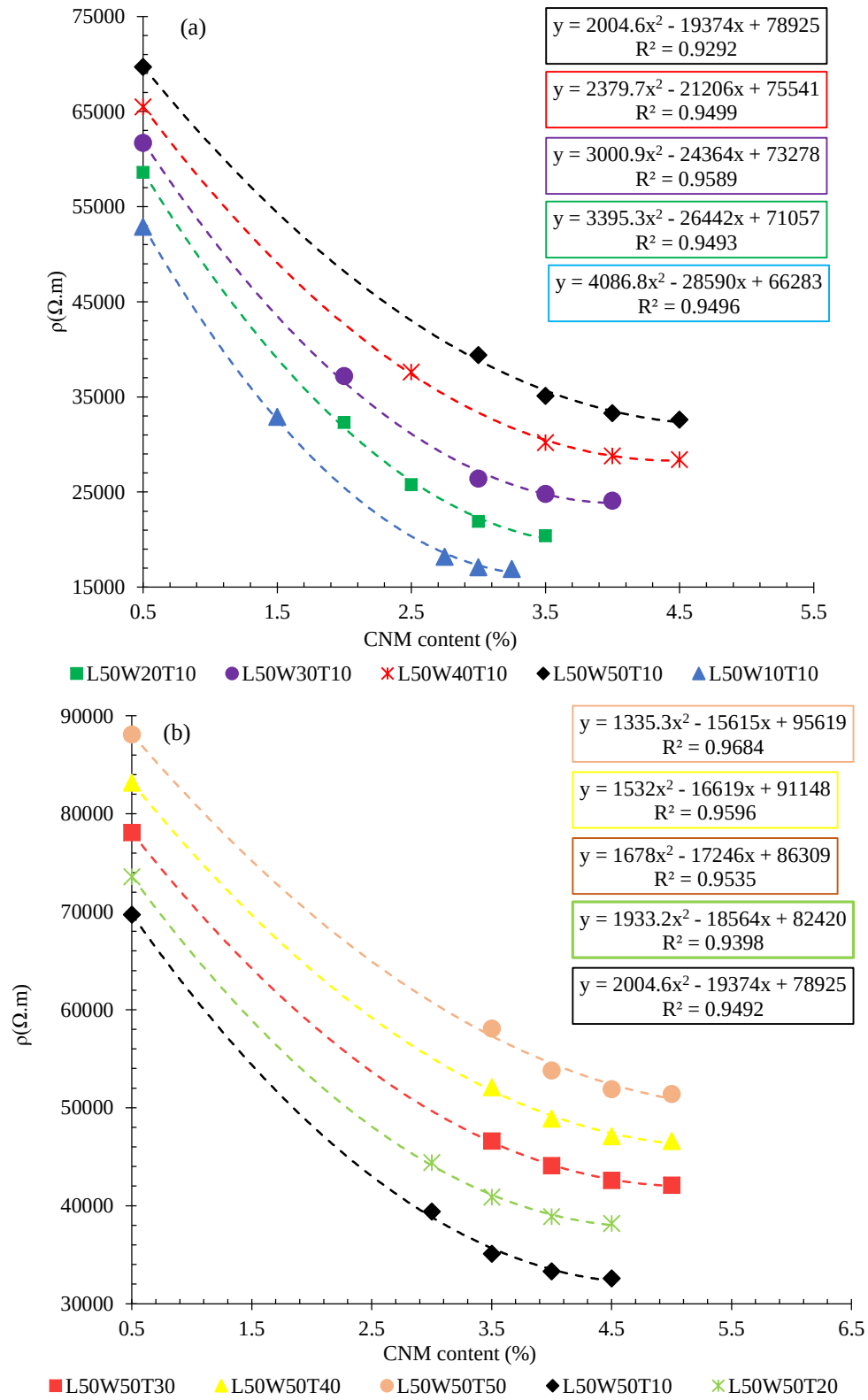


Figure 7-10. Volumetric electrical resistance of CNT/GNP reinforced specimens with: a) different width, b) different thickness.

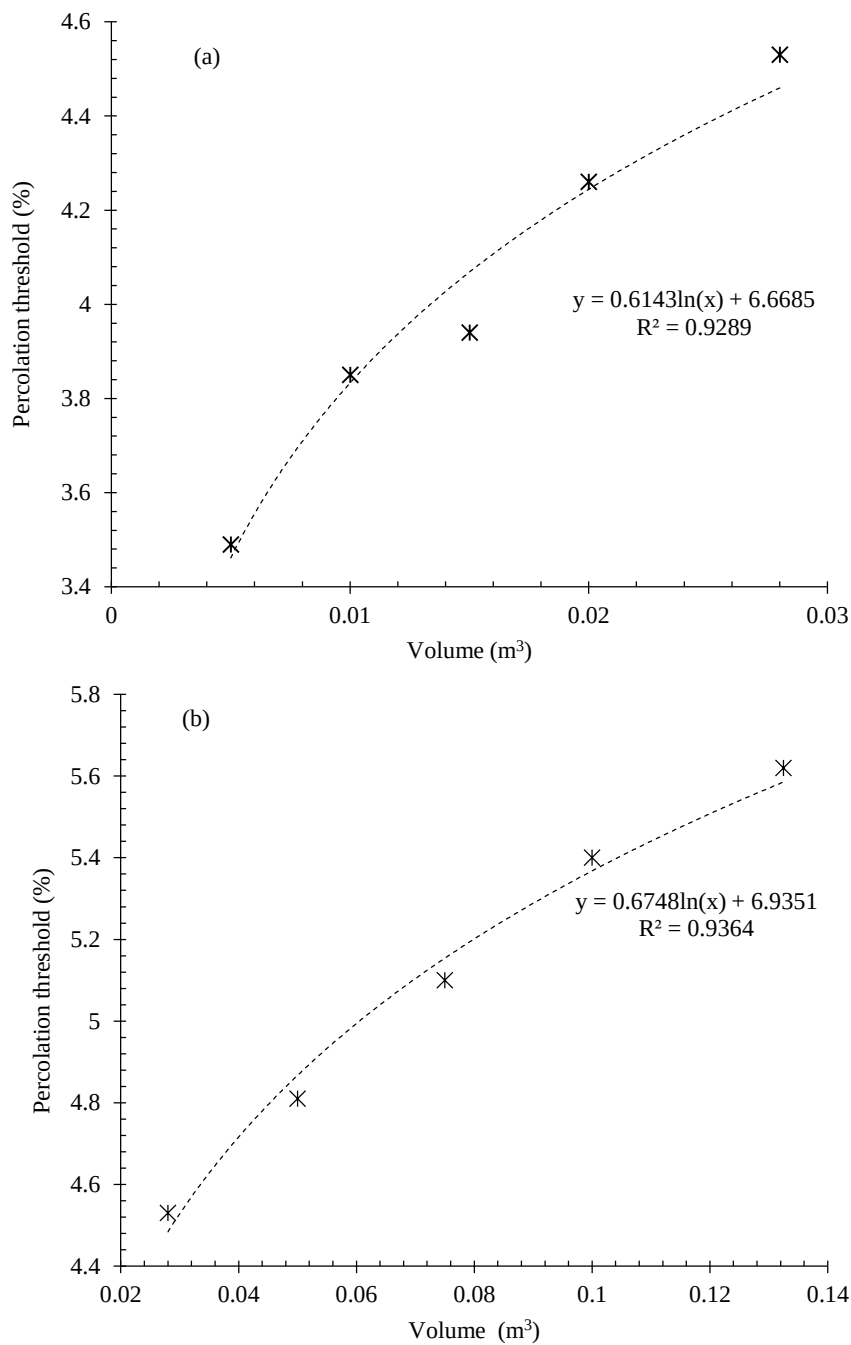


Figure 7-11. Relation between electrical percolation threshold and matrix volume for CNT/GNP reinforced specimens with: a) different width, b) different thickness.

In order to better evaluate the contribution of each dimension change in the variation of electrical resistivity, and consequently the percolation threshold, the electrical resistivity of the $10 \times 10 \times 10 \text{ cm}^3$, $50 \times 10 \times 10 \text{ cm}^3$, $50 \times 50 \times 10 \text{ cm}^3$, and $50 \times 50 \times 50 \text{ cm}^3$ specimens were calculated, as shown in Figure 7-12.

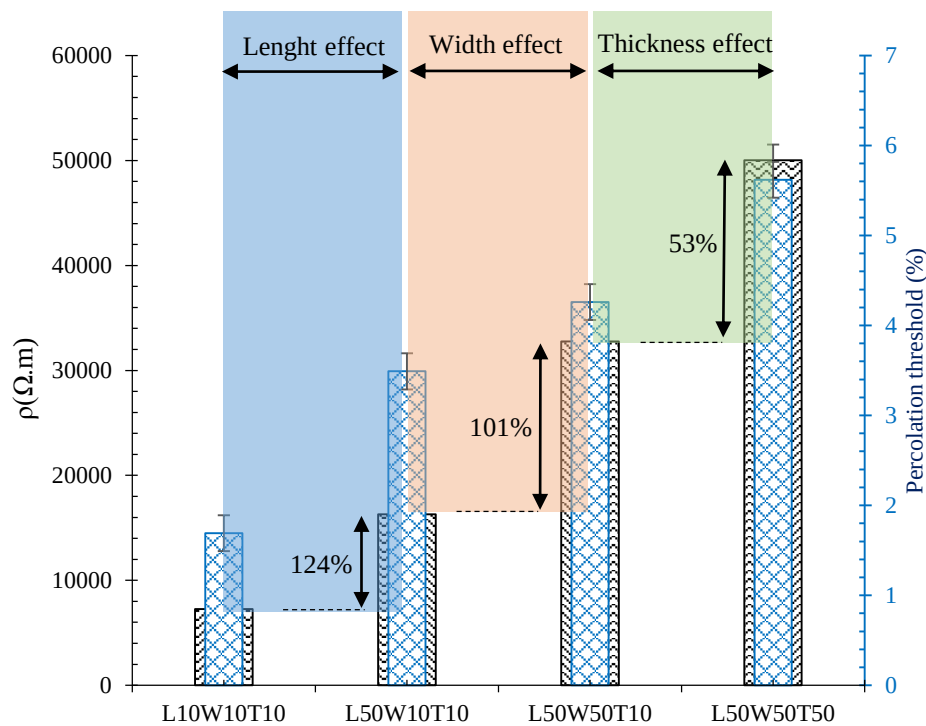


Figure 7-12. Electrical resistivity of specimens reinforced with hybrid CNT/GNP at the percolation threshold content.

Clearly, the effect of length changes was greater than the width and thickness changes in such a way that increasing the length, width, and thickness of the specimens from 10 cm to 50 cm, caused an increase in electrical resistivity of approximately 124, 101, and 53%, respectively.

7.3.3. Effects of Volume Changes

Figure 7-13 shows the variation in the electrical percolation threshold of the specimens reinforced with CNT/GNP versus the matrix volume (surrounded between electrodes) changes by considering specimens with dimensions of $20 \times 20 \times 20 \text{ cm}^3$, $30 \times 30 \times 30 \text{ cm}^3$, and $40 \times 40 \times 40 \text{ cm}^3$.

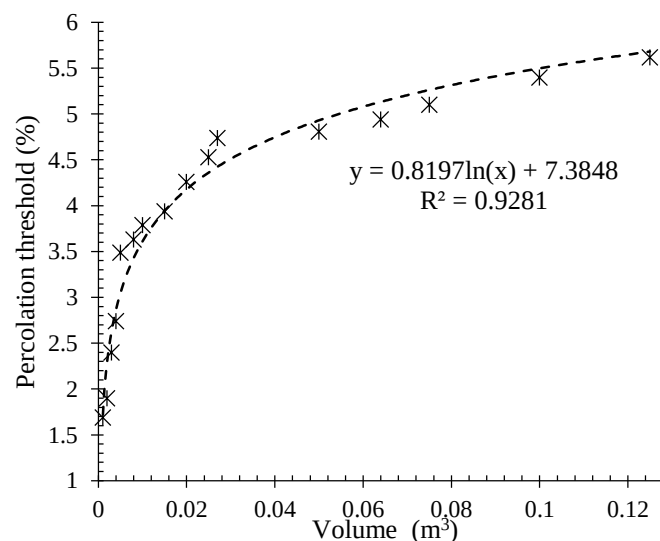


Figure 7-13. Relation between electrical percolation threshold and matrix volume for CNT/GNP reinforced specimens with different volumes.

Based on Figure 7-13, there is a well-defined trend between the matrix volume (surrounded between electrodes) and its electrical percolation threshold, which is expressed by a logarithmic function. With this proposal, design engineers can easily predict the electrical percolation threshold, which is one of the key factors in smart composite design and development [523, 524].

7.3.4. Effects of Fillers' Aspect Ratio

As mentioned, in order to evaluate the effect of the filler aspect ratio on the percolation threshold of the matrix, different content of the three different types of CFs and CNTs, which varied in terms of their length, were used as conductive fillers. Square cubic specimens with different volumes were also used to remove the effects of the matrix aspect ratio. The variation of percolation threshold relative to the aspect ratio of the fillers for the specimens are illustrated in Figure 7-14.

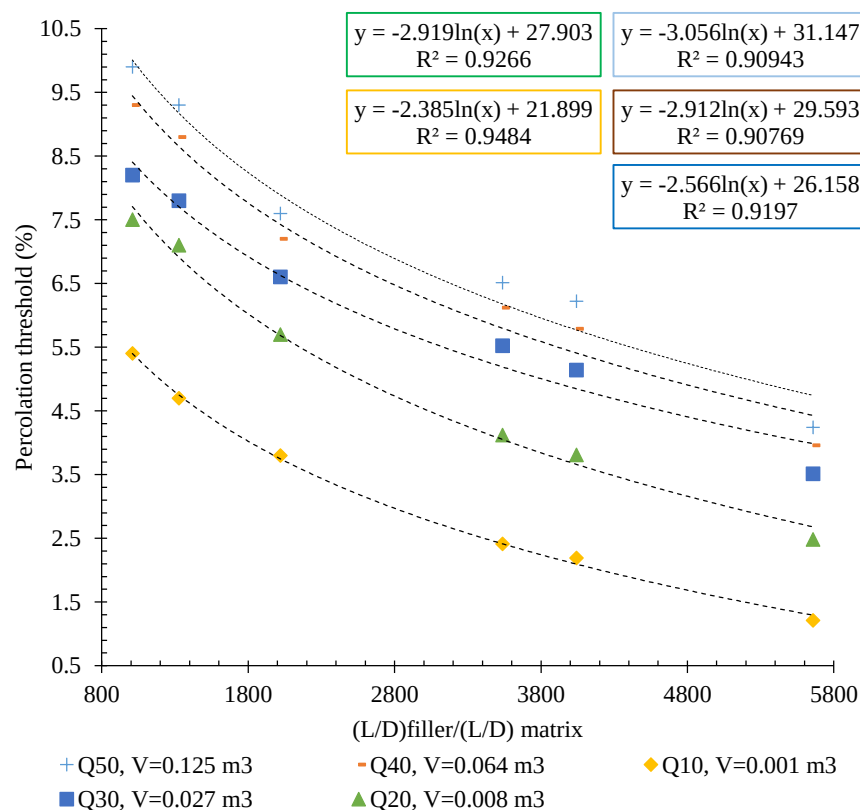


Figure 7-14. The relative aspect ratio of the carbon-based fillers vs percolation threshold for the specimens with different volumes.

As can be observed, increasing the aspect ratio of the conductive fillers relative to the matrix caused a reduction in the electrical percolation threshold percentages. A same trend was reported in the literature [129, 132, 525]. They reported that the percolation threshold value increases when the aspect ratio decreased. Chiarello and Zinno [525] expressed that the threshold value is defined as the conductive filler volume where the clusters of the conductive filler start to be in contact with one another, forming the conductive network through the insulating matrix.

In fact, to form the conductive network a greater content of short than long filler is necessary, because the latter are more likely to be connected themselves to form the percolation path.

Another point of interest is that by evaluating the slope of the graphs for different matrix volumes, it can be seen that increasing the aspect ratio of the filler relative to the matrix had a greater effect on reducing the percolation threshold in larger specimens. In other words, at larger volumes, fillers with higher aspect ratios exhibited higher performance.

The results of the percolation threshold for the specimens reinforced with CFs and CNTs of different lengths are also shown in Figure 7-15.

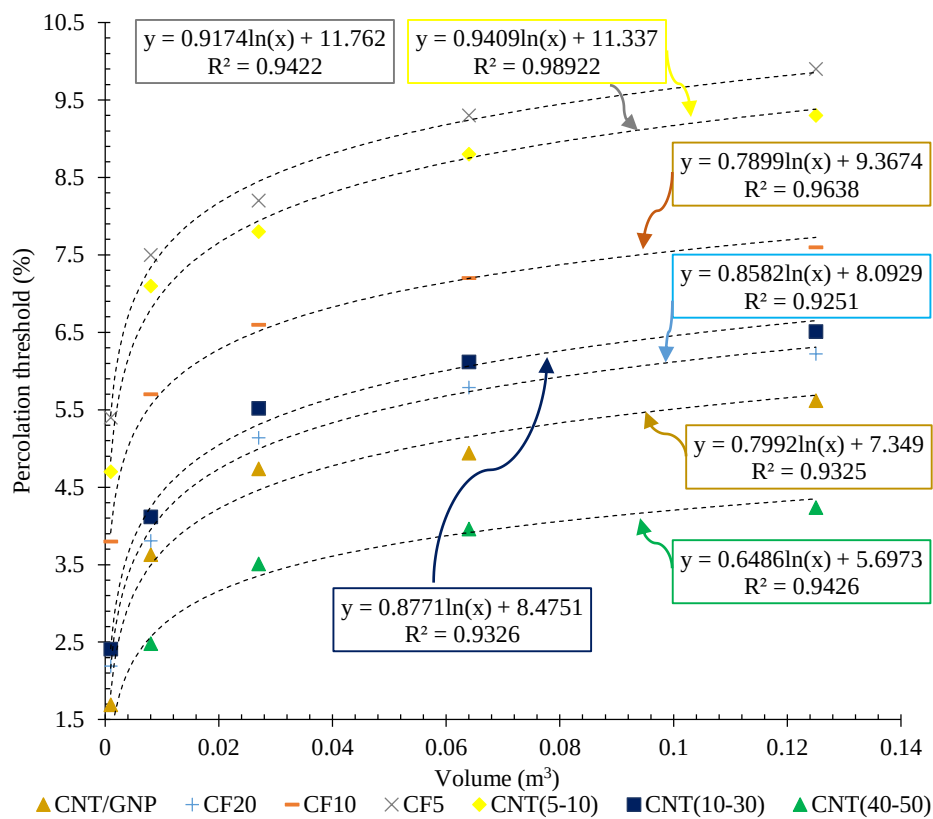


Figure 7-15. The percolation threshold vs matrix volume (surrounded between electrodes) for the different CF and CNT reinforced specimens.

The results clearly showed that increasing the matrix volume (surrounded between electrodes) of all types of conductive fillers consistently increased the electrical percolation threshold percentage. As can be observed, in an identical matrix volume, the lowest percolation threshold was obtained for the specimen composed of CNT (40-50). In fact, the prioritisation of conductive fillers in terms of their performance in reducing the electrical percolation threshold of cementitious geocomposites, from best to worst performance, was CNT (40-50), CNT/GNP, CF (20), CNT (10-30), CF (10), CNT (5-10), and CF (5).

Al-Dahawi et al [125] also assessed the effects of the aspect ratio of conductive fillers on the percolation threshold of a cementitious mortar. They incorporate the different content of MWCNT (20-30 nm of diameter and 10-30 μm of length), CF (7.5 μm of diameter and 12 mm of length), GNP (5 μm of diameter and 5-100 nm of thickness), and carbon black particles (CB, 20-100 nm of diameter) into a 15 cm length of the cubic specimens with water to cement ratio of 0.27. They measure the electrical resistivity of the specimens with two probes method and using a concrete resistivity meter with uniaxial configuration, which employs the alternating current (AC) technique. They reported the lower percolation threshold of 0.55%, 1.00%, 2.00%, and 2.00% for the CNT, CF, GNP, and CB respectively.

7.3.5. Piezoresistivity Investigations

Flexural loading

The percolation threshold of a $10 \times 10 \times 85 \text{ cm}^3$ beam was calculated using the obtained relation (Figure 7-13). The percolation threshold for this specimen was approximately 3.47% for the hybrid CNT/GNP. The piezoresistivity behaviour of this specimen under flexural cyclic loading is illustrated in Figure 7-16. The initial electrical resistivity of this specimen was approximately $3237 \text{ }\Omega\cdot\text{m}$. A proper correlation was obtained between the strain changes and the fractional change in electrical resistivity (FCR) which indicated the high potential of the SCG in terms of strain and/or stress detection. As shown in Figure 7-16, the FCR consistently increases with increasing load and decreases with decreasing load. Indeed, increasing the load and consequently, the strain, caused the destruction of the conductive paths formed by the conductive fillers (CNT/GNP) which led to an increase in the electrical resistivity of the composite. A similar trend was reported in the literature [46].

The gauge factor (GF) for this specimen was approximately 18.5, which demonstrates the excellent sensitivity of this composite to strain and stress. However, due to the noise of the signal, the mean of FCR values was used to calculate the sensitivity.

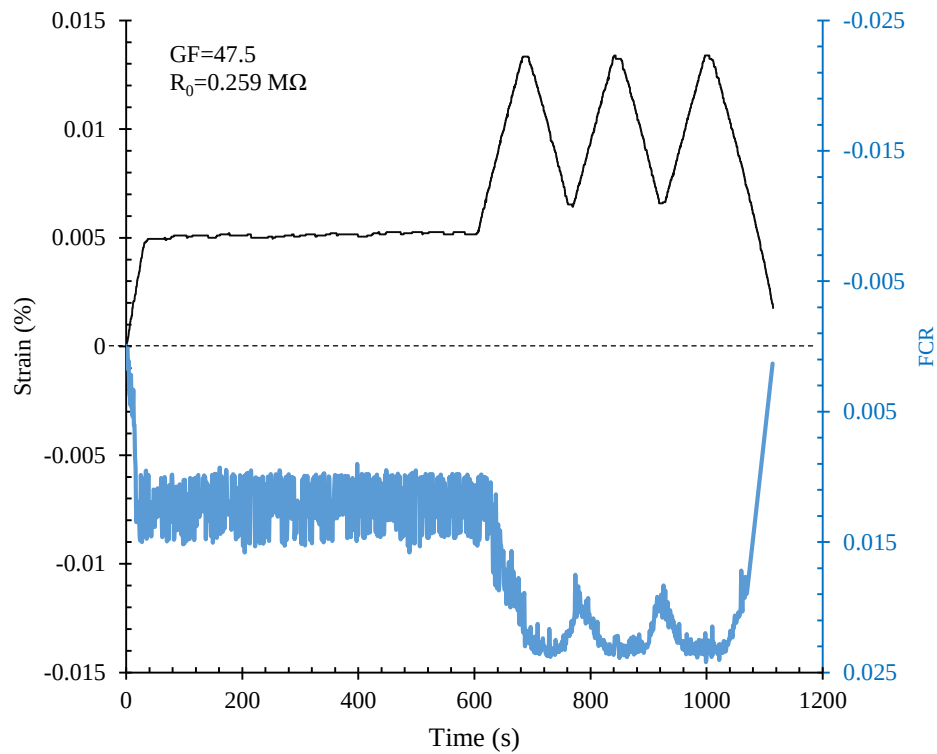


Figure 7-16 Piezoresistivity response of the beam under flexural cyclic loading.

Compression loading

In the compression loading mode, a cubic specimen with dimensions of 15 cm size was used. The percolation threshold for this volume was approximately 2.7%. The piezoresistivity responses of this specimen under compression cyclic loading are shown in Figure 7-17.

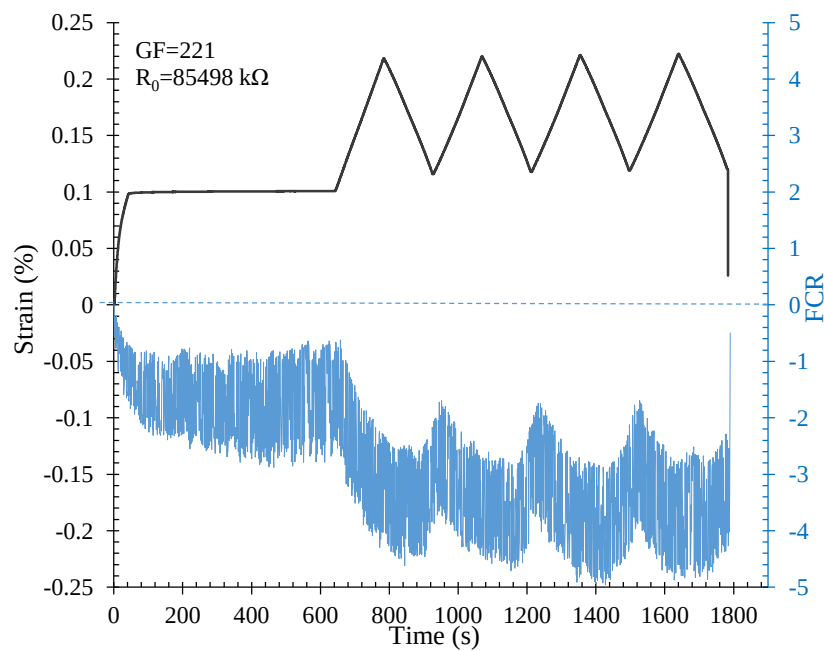


Figure 7-17 Piezoresistivity response of the cubic specimen under compression cyclic loading.

The electrical resistivity of this specimen was approximately $12750 \Omega.m$. In the compression loading mode, the results showed a proper correlation between the variation in FCR and strain. Negative values were measured for the FCR because of a decrease in the electrical resistance during compression loading relative to the initial electrical resistance of the composite [57]. In fact, the paths formed by conductive fillers got closer together during compression loading, which resulted in the decreased electrical resistivity and consequently enhanced the electrical conductivity of the composite. This specimen also indicated a high level of sensitivity to the compression loading in such a way that a gauge factor of approximately 221 was also obtained for this SCG.

7.3.6. Mechanical Investigations

Mechanical properties of the beam

The mechanical properties of the $10 \times 10 \times 85 \text{ cm}^3$ beam reinforced with 3.47% CNT/GNP (SCG 3.47%) are illustrated in Figure 7-18. The results did not show significant changes in the flexural and compression strengths of the specimen (SCG 3.47%) when compared to the plain specimen. However, reinforcing the plain specimen with 3.47% CNT/GNP caused an increase in the flexural elasticity modulus by approximately 25%. Furthermore, the incorporation of 3.47% hybrid CNT/GNP into the plain specimen increased the compression elasticity modulus by approximately 50%. A similar trend in terms of the ability of CNT/GNPs to increase the moduli has been reported in previous studies [46, 56].

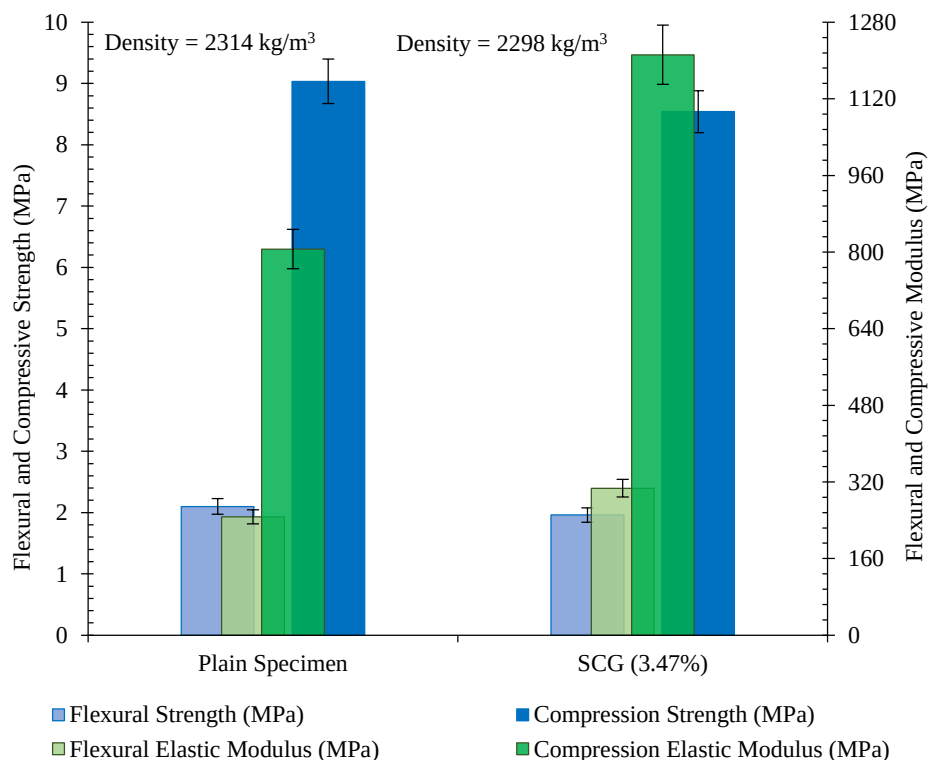


Figure 7-18. Mechanical properties of the $10 \times 10 \times 85 \text{ cm}^3$ beam reinforced with 3.47% of CNT/GNP (SCG 3.47%)

Mechanical properties of the cubic specimen

The compression strength of the 15 cm cubic specimen composed of 2.4% CNT/GNP (SCG 2.7%) is shown in Figure 7-19. The compressive strength of the specimen reinforced with 2.4% CNT/GNP increased by approximately 38% compared to that of the plain specimen. The compression elasticity modulus of the specimen (SCG 2.7%) obtained was also three times larger than that of the plain specimen.

Generally, reinforcing cementitious composites with CNTs and GNPs improved the physical and mechanical properties of the hardened composite through its filler function and bridging and/or through deviation of the cracks [46, 56, 145, 409, 516, 526]. A hybrid combination of one dimensional CNT and two dimensional GNP with different geometrical shapes and sizes can fill a wider range of pores and spaces between the cement particles. Then, by starting the cement hydration process, the nanoparticles buried among the hydration products can improve the physical properties and reinforce the microstructure of the composite [527]. The CNTs bridge across the cracks and the GNPs effectively divert them, thereby reducing the accumulation of cracks. These phenomena greatly increase the load-carrying capacity and forces needed for the development of crack and element failures. When a crack meets a buried CNT or GNP, it is blocked, and a new load increase is needed for further propagation. In addition, CNTs and GNPs could act as nucleation agents and increase the hydration rate in cementitious composites [208]. However, an excessive increase in CNM content caused agglomeration and consequently reduced the physical and mechanical performances of the composite [409].

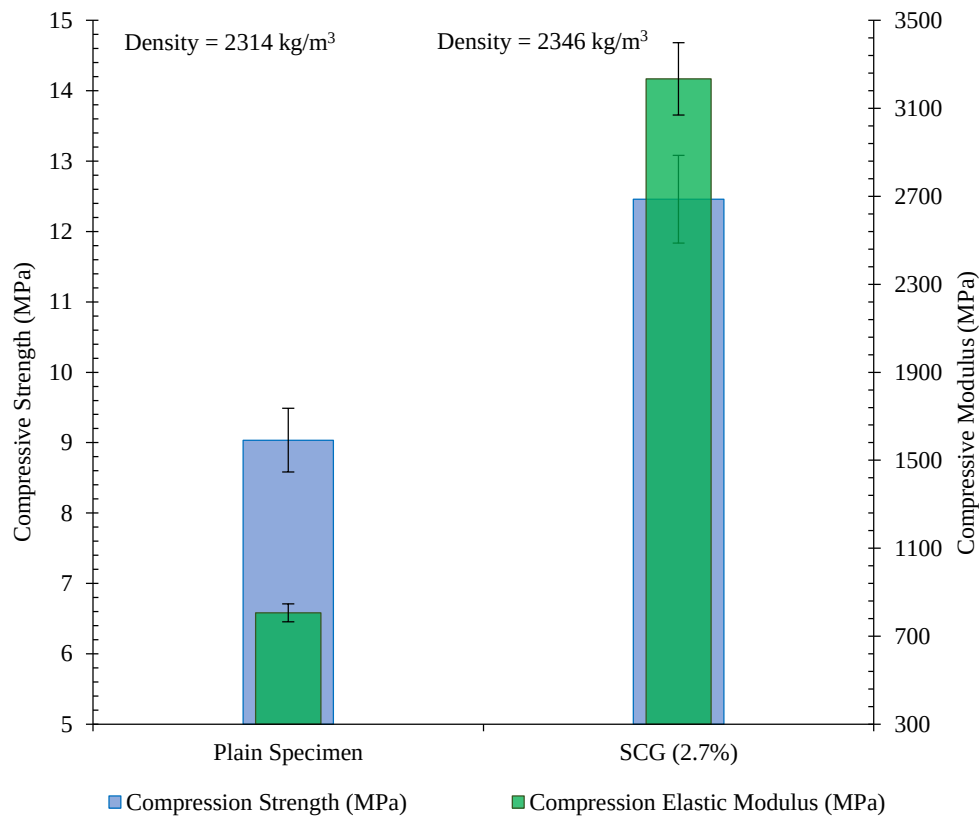


Figure 7-19. Mechanical properties of the 15 cm cubic specimen reinforced with 2.4% of CNT/GNP (SCG 2.7%)

7.4 Conclusions

This was an extensive study designed to clarify the co-effects of electrode space and surface area as well as filler scales on the electrical percolation threshold, which is one of the most important parameters involved in the design of self-sensing cementitious composites for the field applications. In this route, the electrical conductivity of several specimens of different shapes, sizes, and content of carbon conductive fillers such as CNT, GNP, and CF, were measured. The electrodes were installed on both ends of specimens to completely surround the matrix. In addition, conductive fillers with different aspect ratios were used to investigate the effects of filler scale on the electrical percolation threshold. In this study, stabilised sand with 10% cement was used, and the specimens were fabricated using the compaction method. Furthermore, the numerical relations between the electrical percolation threshold and matrix dimensions were expressed for different conductive fillers.

Finally, the electrical percolation threshold of two large-scale specimens with different shapes (a 10×10×85 cm³ beam and a 15 cm cube) were predicted through the obtained numerical relations, and their piezoresistivity performances were investigated under flexural and compression cyclic loading. In addition, the mechanical properties of these specimens were evaluated and the following conclusions were drawn:

The volumetric electrical resistance of conductive specimens with different dimensions showed that the changes in the length, width, and thickness of the matrix surrounded between electrodes had a significant effect on the changes in the electrical percolation threshold of the composite. Generally, increasing the volume of the surrounded matrix caused an increase in the percolation threshold.

The investigations showed that the effects of length changes of surrounded matrix in percolation threshold were greater than the effects of its width and thickness changes in such a way that increasing the length, width, and thickness of the surrounded matrix from 10 cm to 50 cm, caused an increase in electrical resistivity by approximately 124%, 101%, and 53%, respectively.

Generally, increasing the aspect ratio of the conductive fillers causes a reduction in the electrical percolation threshold of the cementitious composite.

In this study, the prioritisation of conductive fillers in terms of their performance in reducing the electrical percolation threshold of cementitious geocomposites from most to least effective was as follows: CNT (40-50), CNT/GNP, CF (20), CNT (10-30), CF (10), CNT (5-10), and CF (5).

The 10×10×85 cm³ beam and 15 cm cubic specimens with hybrid CNT/GNP content of 3.47 and 2.74%, respectively (obtained by the numerical relation presented in this study), showed a proper piezoresistive response under flexural and compression loading, respectively.

Incorporation 3.47% of CNT/GNP into the specimens (beam) did not cause a considerable change in the flexural and compression strengths of the specimen. However, it increased the flexural and compression elasticity modules by approximately 25% and 50%, respectively.

Reinforcing the cubic specimen with 2.4% CNT/GNP improved the compression strength by approximately 38%. The compressive elasticity modulus of this specimen was three orders of magnitude greater than the plain composite.

In summary, we believe that this novel approach could contribute to the new era of self-sensing geocomposite design for intelligent structures. Indeed, the results of the present study illuminate the path for passing from small scale laboratory investigations towards large scale self-sensing geocomposites.

Chapter 8. Effects of Temperature, Humidity Loading Type and Speed on Sensing Potential of Cementitious Geocomposite.

«Research is an art to see what everybody else has seen and to think what nobody else has thought. »

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

Based on:

- Abedi, Mohammadmahdi, Mohammad Jawed Roshan, Federico Gulisano, Javad Shayanfar, Mostafa Adresi, Raul Fanguero, and António Gomes Correia. "An Advanced Cement-Based Geocomposite with Autonomous Sensing and Heating Capabilities for Enhanced Intelligent Transportation Infrastructure." *Construction and Building Materials*. No. 411 (2024): 134577. <https://doi.org/10.1016/j.conbuildmat.2023.134577>

Abstract

In this study, an innovative smart cementitious-based geocomposite with self-sensing and self-heating capability has been developed for infrastructural health monitoring and traffic recognition. In this route, hybrid carbon nanotubes and graphene nanoplatelets were dispersed among the stabilized sand with 10% cement. Mechanical and microstructural properties of the composite were investigated with various tests. The self-heating potential of this cementitious geocomposite was assessed. The effects of temperature, moisture content, and loading shape, speed and magnitudes on the strain and stress sensing capability of the specimens were also investigated. In addition, the self-sensing performance of this cementitious geocomposite for the asphalt layer strain detection was evaluated in a hybrid section including the asphalt mixture layer and smart geocomposite. The 0.98 °C/s heating rate, 526 mW/ °C heating power, and 251 °C maximum temperature proved the high heating potential of this geocomposite for deicing, self-healing, and temperature sensing applications. The specimens showed a supreme correlation between strain changes and FCR values in different loading patterns. An appropriate correlation between gauge factor, modulus, and digital image correlation analysis was observed. The piezoresistive response of this geocomposite in the hybrid section, representative of the common structural layers of urban pavements, showed the ability of the smart geocomposite in terms of strain and stress detection of itself and even in the asphalt layer. The outcomes of this study can draw a bright horizon in smart materials development with applications in civil infrastructures including transportation and railway.

8.1 Introduction

Nowadays, with the advancement of intelligent cities and technologies, the world is witnessing increasing efforts to develop smart transportation and railway infrastructure, aligning with twenty-first-century sensibilities concerning technology, safety, security, and convenience [1, 528, 529]. Accordingly, this has caused a growing demand for multifunctional and smart composites in the civil construction industry. Sensing and heating performances are among the fundamental capabilities which are the bases of many other functions and mechanisms in transportation infrastructure such as stress, strain, and damage monitoring, as well as de-icing, and self-healing performances [1, 46, 81, 530-532]. Infrastructure monitoring is the automated condition assessment of structures through observation and analysis of data collected on-site by sensing systems [437, 533]. Infrastructure monitoring enables the early detection of damage that, if addressed, reduces the risk of sudden collapses and optimizes the service life of structures [1, 57, 534]. It also enables condition-based maintenance instead of prevention-based or breakdown-based maintenance [535]. Furthermore, infrastructure monitoring systems are useful platforms for extracting current data at an operational level which have many benefits in various areas of smart transportation, traffic flow, speed and weight monitoring, and driverless vehicle development. Indeed, smart infrastructures have enormous economic potential for societies and enhance the quality of life of members. Despite the significant research efforts made in recent years [536, 537] to address the advanced state of deterioration of infrastructural systems particularly in western countries, infrastructure monitoring is still far from being widely implemented [437]. Moreover, most monitoring systems and instrumentation, such as electric strain gauges, fibre Bragg grating sensors, and PZT-based piezoelectric sensors, involve the placement of a limited number of sensors in spot locations along infrastructure [3, 56, 535, 538]. The fundamental constraint of present infrastructure monitoring methods is the bottlenecks of their scalability to large-scale structures because of administration and maintenance concerns and due to the rapid increase of required sensors number with growing structural size for an accurate assessment [58, 539]. Among the common infrastructure monitoring technologies, self-sensing composites with intrinsic sensing capabilities based on piezoresistive behaviour provide the most integrated, real-time, and practical solution for infrastructure monitoring [540].

As mentioned, currently the heating potential of cementitious composites has attracted particular attention due to its function in snow-melting, de-icing, common healing mechanisms, and energy-efficient applications [541-544]. Built-in heating pipelines [545-547] Self-heating coating [548, 549] phase-change materials [550, 551] and super-hydrophobic coating [552] have been often used as active methods in previous studies to assign the heating performance to different civil structures. However, the cost of

production, the low energy efficiency, and the degradation of the host matrix has made some challenges for their wide implementation [553, 554]. Nowadays, self-heating concretes are being developed by taking advantage of advancements in material science and nanotechnology in the construction industry. In addition to compatibility with cementitious matrix, these self-heating composites offer improvement in mechanical and durability performance, a flexible design, and an easy production process [555-557]. Self-sensing and self-heating cementitious composites are based on the incorporation of the conductive phases such as short fibers, as well as micro- and nano-particles, mostly carbon-based materials [82] into non-conductive host matrices, to provide electrical conductivity [125, 518, 558, 559]. Indeed, the conductive phase forms random conductive paths based on percolation and electron tunnelling effects which enables the transduction of a mechanical strain into a measurable variation of the electrical resistance. Furthermore, passing a certain amount of electrical current through these conductive paths causes an increase in the temperature of the composite [532, 560-562].

Despite recent advances and various research carried out on different self-sensing and self-heating composites including, cementitious [107, 187, 392, 563-565], bitumen [390, 566-569], and polymer composite [570-574], the lack of studies on geomaterial as an integral part of structural layers of transportation and railway infrastructures is noticeable. Furthermore, allocating sensing and heating capabilities simultaneously to structural elements and infrastructure is still challenging. A novel self-sensing cementitious geocomposite composed of stabilized sand and hybrid carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) has recently been developed by the authors. In this route, an effective and compatible dispersion method was first developed [76]. Thereafter the effects of different conductive fillers on the mechanical, microstructural, durability and piezoresistivity of the cementitious composite were assessed [200]. The effects of different measurement methods, electrode layout and section scale on the sensing capacity of this composite as an initial step to developing a field-applicable, smart cementitious geocomposite, were investigated.

Following this route, the effects of temperature and water content as two crucial effective environmental factors on the piezoresistivity capacity of the composite were evaluated in this study. Furthermore, the effects of different loading patterns including loading shape, speed and magnitudes with the respect to different traffic loading simulations were assessed. The self-heating capability of this composite was also investigated in detail. In addition, the self-sensing performance of this cementitious geocomposite for the asphalt layer strain detection was evaluated in a hybrid section including the asphalt mixture layer and smart geocomposite. The damage diagnosis ability of the composite was also assessed using digital image correlation analysis. The outcomes of this study could provide a great contribution to the development of

new smart structures with applications in civil engineering related to structural layers in transportation and railway infrastructures.

8.2 Materials and Methods

8.2.1. Raw materials

The multiwall carbon nanotube (MWCNT) and multilayer graphene nanoplatelets used in this study were supplied by Graphenest and IoLiTec companies. The X-Ray spectra, morphology, and characteristics of these carbon nanomaterials (CNMs) are presented in Figure 8-1, Figure 8-2 and Table 8-1.

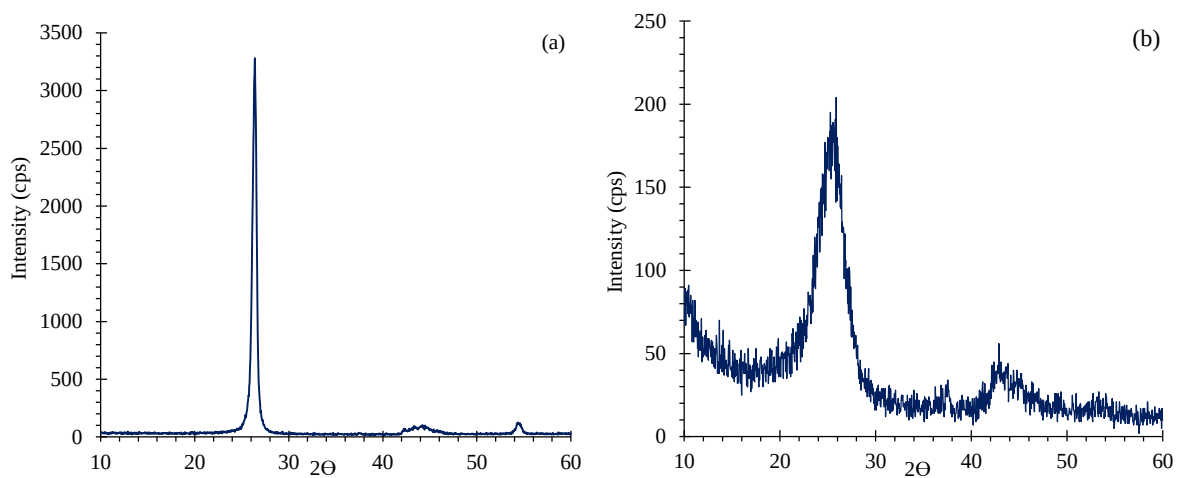
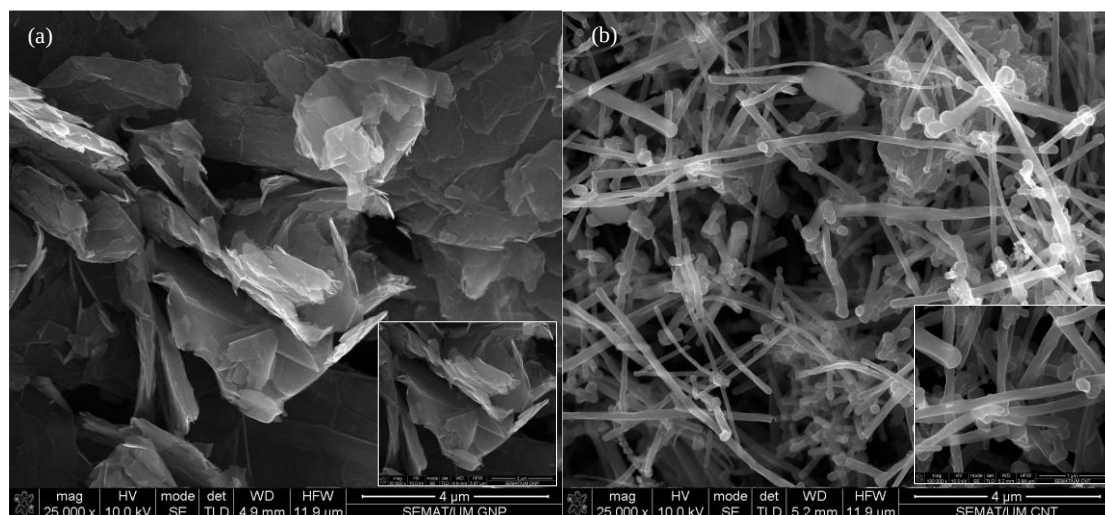


Figure 8-1. The X-Ray spectra of carbon nanomaterials: a) GNPs, b) MWCNTs.



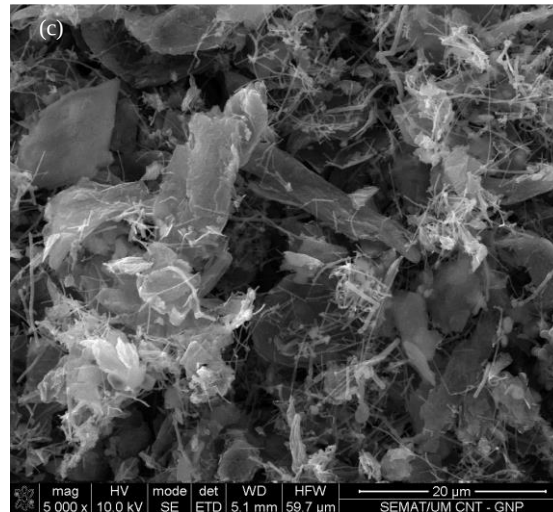


Figure 8-2. The morphology of carbon nanomaterials: a) GNPs, b) MWCNTs, c) the mix of CNT/GNPs.

Table 8-1. The main characteristics of GNP and MWCNT used in this study.

GNP									
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Carbon content (%)	Tensile modulus (Gpa)	PH Value (30 °C)	Tensile strength (GPa)	Layers	Dimension		Form
120-150	2.25	> 99.5	1000	7-7.65	5	< 20	Thickness 4-20nm	Diameter 5-10μm	Gray Powder
MWCNT									
Surface area (m ² g ⁻¹)	Density (g/cm ³)	Color	Outside Diameter (nm)	Length (μm)	Ash (wt%)	Carbon content (%)			
≥380	2.17	Black	1-2	5-30	< 1.5	> 90			

The stabilized cemented sand, which was made using ordinary Portland cement type I (CEM I 42.5 R) was adopted as the matrix in this study. The matrix was also composed of CEN Standard sand with siliceous and clean particles, which was classified as well-graded sand following the unified soil classification system (USC). The particle size distribution curve and the physical properties of the sand and cement are presented in Figure 8-3, Table 8-2, respectively. The noncovalent surfactant Pluronic F-127 was used to disperse the CNMs into the aqueous suspension. Pluronic F-127 is a non-ionic triblock copolymer surfactant including two hydrophilic chains of polyoxyethylene on either side of a core hydrophobic chain of polyoxypropylene. These molecular configurations resemble those of polycarboxylate, which is frequently employed as a superplasticizer in cementitious composites. Additionally, to further prevent pore formation by the surfactant, tributyl phosphate (TBP, 97%) was utilized as an antifoam at a weight ratio of half that of the surfactant.

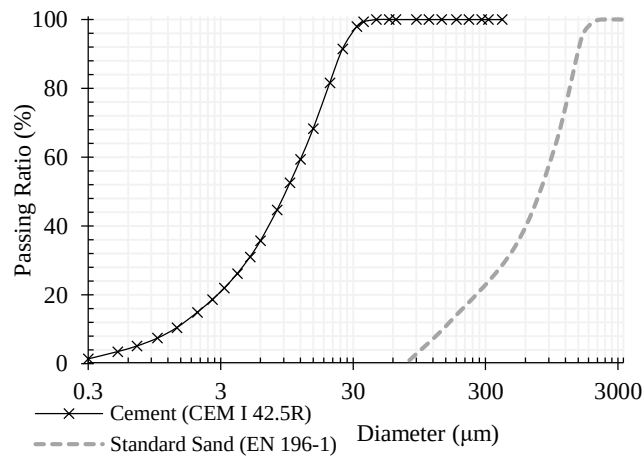


Figure 8-3. Particle size distribution of the used cement and sand.

Table 8-2. The main physical characteristics of the sand.

Mesh Size (mm)	0.08	0.16	0.5	1	1.6	2
Cumulative retained (%)	99±1	87±5	67±5	33±5	7±5	0
Specific gravity G_s	2.67		Cu^a	7.5	Cc^b	1.8

^a Uniformity coefficient, ^b Curvature coefficient

8.2.2. CNMs dispersion

The proper dispersion of CNMs in the composite is crucial for maintaining their distinctive features [575, 576]. Indeed, the high tendency of CNMs to agglomerate is caused by the presence of strong surface van der Waals forces between CNMs due to their high specific surface area and aspect ratio [107, 577, 578]. An effective, compatible and applicable dispersion method was recently developed by the authors to overcome this challenge, which was also used in this study. In this route, TBP was first dissolved into water, and then 10% of Pluronic F-127 (by weight of CNMs) was added to the suspension and mixed using the magnetic stirrer. Based on the previous studies, the optimal concentration of hybrid CNT/GNP (1:1) required for the development of the smart cementitious composite is approximately 1.0 % (wt of the sand), which was estimated by considering the physical and piezoresistivity behaviour. Hence, the same concentration was used in this study. The CNMs were added to the suspension and stirred continuously. The suspensions were then placed in a bath sonicator with output power and frequency of 80 W and 45 kHz, respectively, for 3 h. Additionally, a digital temperature regulator comprising a circulation system, radiator, and sensors, was used to adjust the temperature during ultrasonication at 40 °C. Under these specific dispersion conditions, negligible structural damage was expected for the CNMs. The scheme of the dispersion method used for CNMs is shown in Figure 8-4.

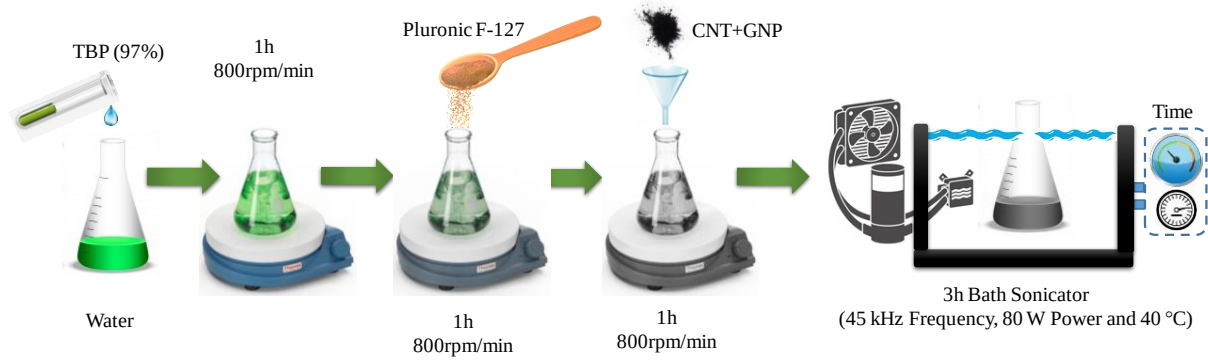


Figure 8-4. CNMs dispersion process used in this study.

8.2.3. Specimen fabrication

Due to the desired strength of the cemented sand, the cement percentage in stabilized sands typically varies by around 10% [148, 579]. For the composite preparation in this study, 10% of cement (by weight of the dry sand) was also employed. Based on previous studies the 8% water (by weight of the dry sand) also was used for the specimen fabrication concerning physical properties and dispersion issues. The prismatic specimens with dimensions of $40 \times 40 \times 160$ mm were made using the compaction method. Split moulds were filled with the wet mixture in three equal-height layers. The required amount of the well-mixed wet mixture was calculated based on a density of 2 g/cm^3 (to an accuracy of 0.01 g), poured into the moulds, and then compacted carefully by a metal tamper to the desired height (controlled by a calliper to an accuracy of 0.02 mm). After the moulds were filled with the compacted mixture, they were sealed at both ends and extracted after a 24 h period of hardening; then, the specimens were cured for 28 days. Four copper plates also were embedded during the fabrication process to pass electrical current along the specimen as illustrated in Figure 8-5.

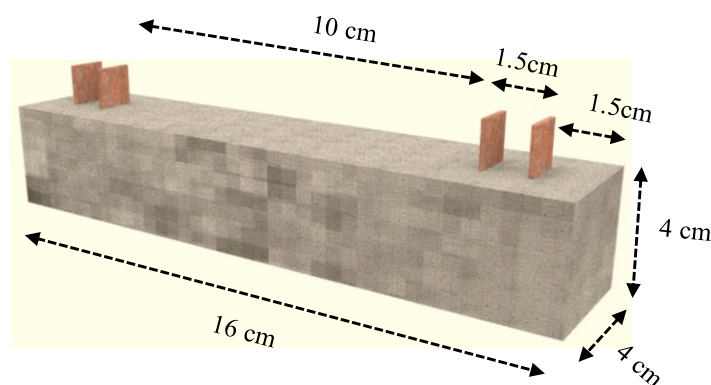


Figure 8-5. The specimen configuration and electrode layout.

8.2.4. Mechanical and microstructural investigations

Flexural and compressive strength of the specimens were assessed following EN 196-1 standard after 28 days of hydration period and curing [68]. The fracture surfaces of the specimens were investigated using Scanning Electron Microscopy (SEM-FEG, Nano SEM NOVA 200, FEI). The sample surfaces were first

coated with an Au-Pd thin film (30 nm) using a high-resolution sputter coater (Cressington 208HR) and the morphology of the specimens was assessed using an acceleration voltage of 10 kV and a secondary mode of electrons. Diffraction analysis of X-ray powder was also done using CuK α radiation over a 5–80° angular range at a 3°/min scanning speed.

8.2.5. Self-heating assessment

The self-heating performance of the composite was investigated using CPX200 D Dual 180-Watt DC supply source from Power Flex and Testo 890 thermography camera as shown in Figure 8-6. For this purpose, the thermography camera was first calibrated and adjusted at the proper distance and different DC currents (3, 6, and 9 A) were applied to the outer electrodes during the measurement. The self-heating power was calculated by Eq. 1, which is the power in mW/°C that caused an increase in the internal temperature of the specimen by 1°C.

$$P = \frac{I \times V}{T - T_{max}} \quad \text{Eq.1}$$

where I is the current, V is the corresponding voltage, T is the room temperature and T_{max} is the maximum internal temperature of the specimen during the test.

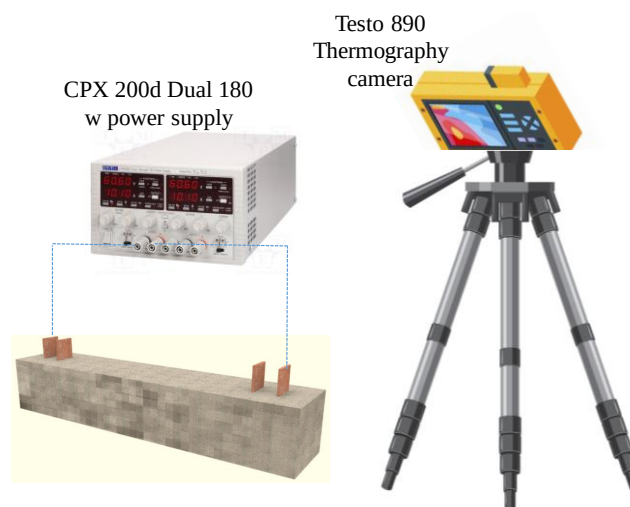


Figure 8-6. The test setup used for self-heating assessment.

8.2.6. Self-sensing investigations

Effects of loading protocols

The sensing capability of the composite was investigated under different compression cyclic loading with various patterns, amplitude and rates. In this route, as shown in Figure 8-7(a), the 40*40*60 mm specimens were cut and a two-electrode method with Agilent 34461A digital multimeter was used. The loading patterns used for this purpose are shown in Figure 8-8. As shown in Figure 8-7(b), the

Chapter 8. Effects of Temperature, Humidity Loading Type and Speed on Sensing Potential of Cementitious Geocomposite.

piezoresistivity response of the composite was also assessed in a hybrid section including a common asphalt mixture layer (Stone Mastic Asphalt, SMA11, [580]) with equal dimension on top which was installed on the cementitious geocomposite with a bitumen binder to simulate the in-situ interlayer bonding. SMA mixtures have been widely used in wearing course of the road pavement structure, due to their high durability [581].

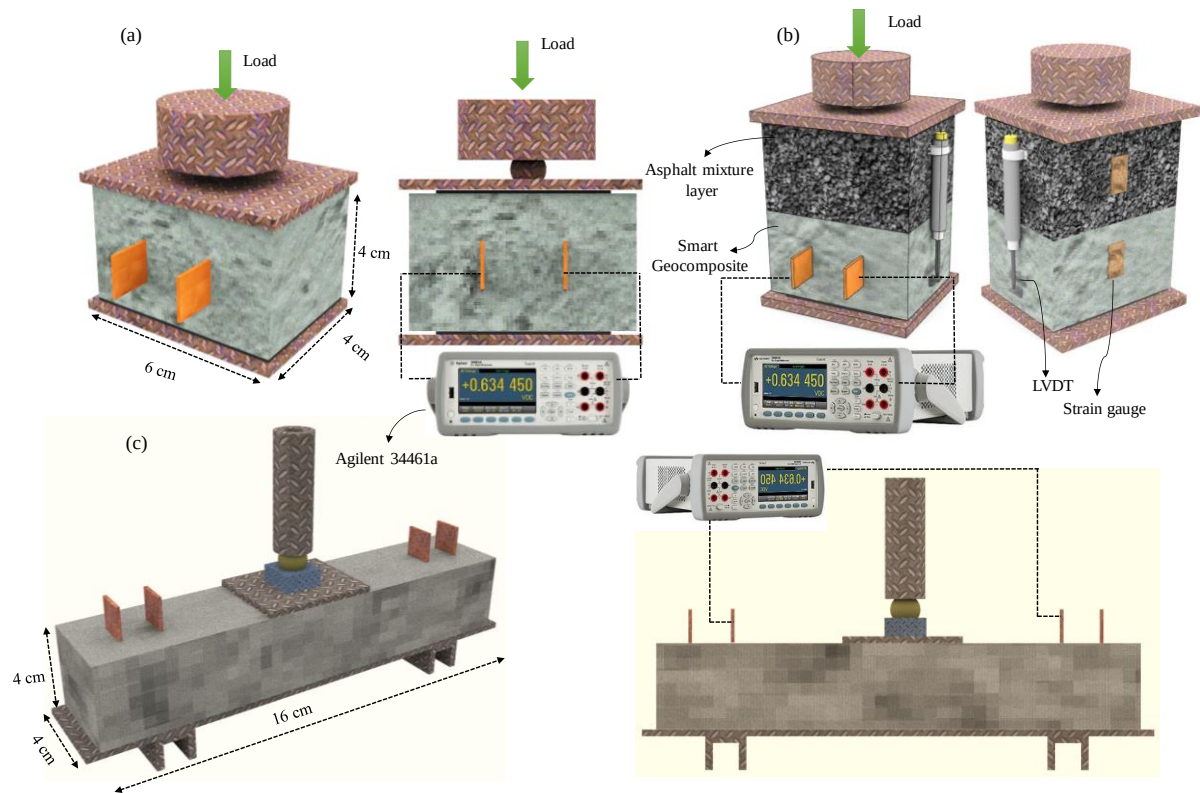
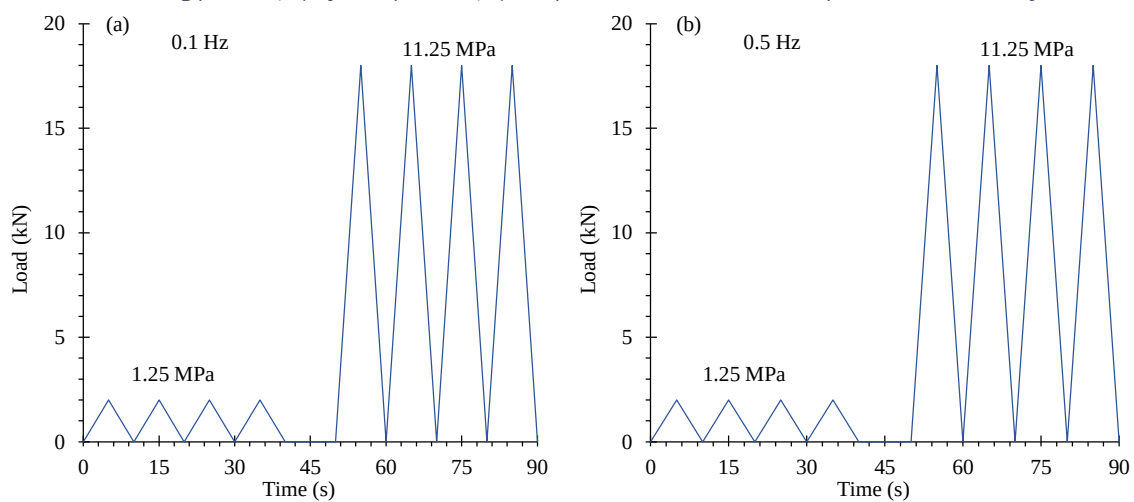


Figure 8-7. Specimen and electrical circuit configuration in piezoresistivity investigations: a) setup used for evaluation of different loading patterns, b) hybrid specimen, c) setup used for evaluation of temperature and humidity effects.



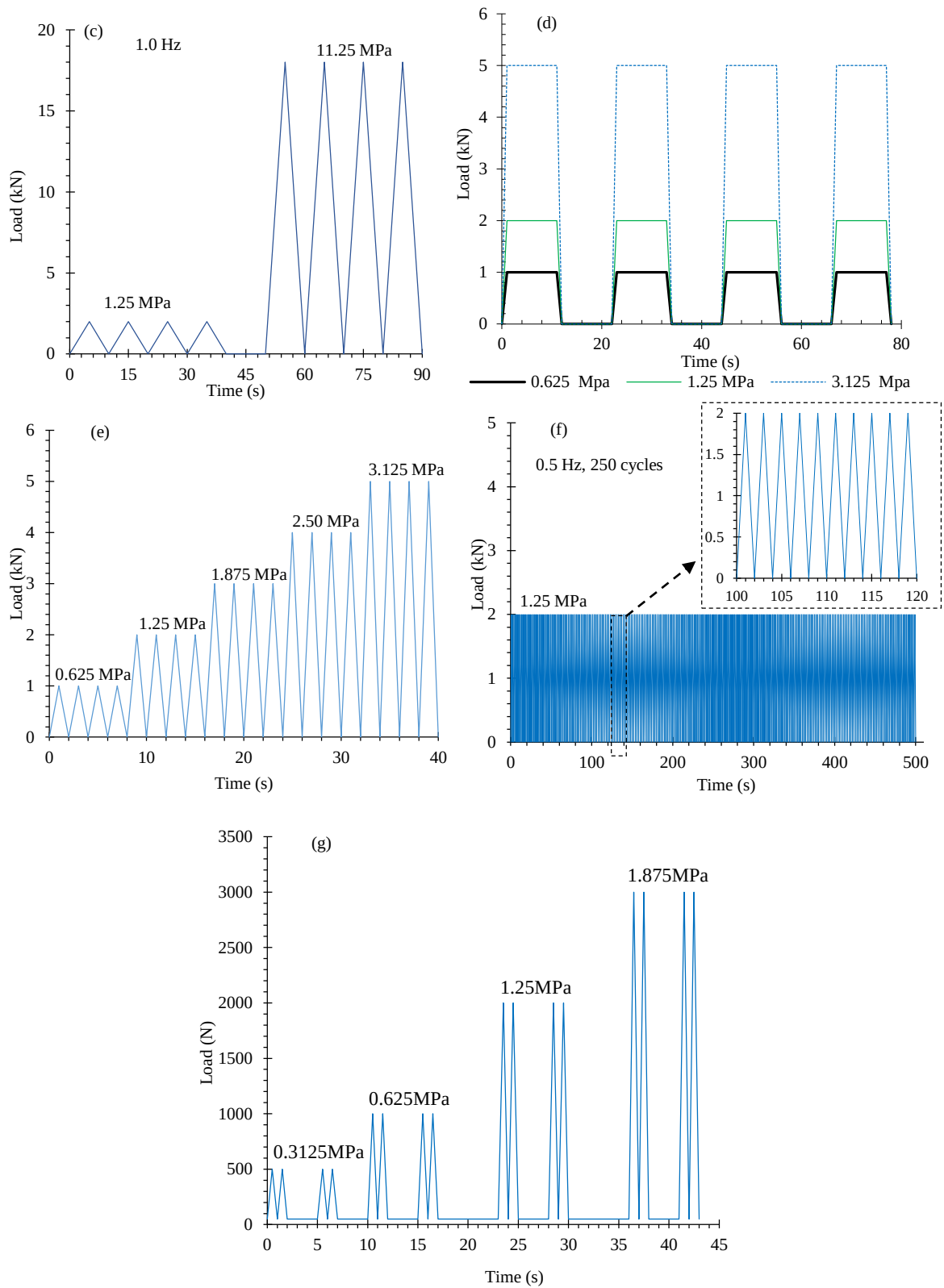


Figure 8-8. The loading patterns used for the piezoresistivity investigation: a) 0.1 Hz, b) 0.5Hz, c) 1.0Hz, d) creep loading, e) multiple loading, d) fatigue loading, e) pattern used for hybrid specimen.

Damage detection

The damage diagnosis ability of the composite was also assessed in both monotonic and cyclic loading (Figure 8-9 (b)). A similar electrical circuit and specimens with a similar dimension was used. As shown in Figure 8-9 (a), a hole of 5mm diameter was drilled at the centre of the specimen. It was intended to facilitate mixed-mode crack initiation from the drilled hole. In order to visualize the crack propagation digital image correlation (DIC) method was employed. For this purpose, the specimen surface was air sprayed with a speckle pattern of black and white paint. A camera with a resolution of 512×1024 pixels was used to record the video during specimen loading and FCR measurement. The camera was synchronized with the loading machine and multimeter.

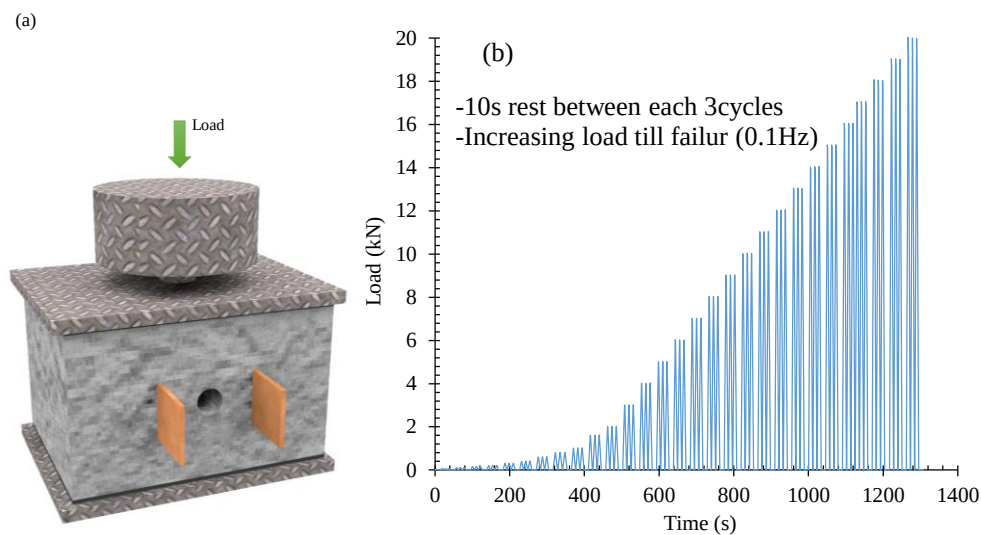


Figure 8-9. a): Specimen configuration and b): compressive cyclic loading pattern used for damage detection.

Effects of temperature

The thermocouples were installed into the specimens to evaluate the temperature effects on the electrical and sensing capability of the composite. For this purpose, the specimens were dried first a hole with a length of 10 mm was drilled into the specimen. the holes were covered with cement past again after the thermocouple installation. The electrical resistance of the composite was measured along with temperature during a phase reduction process from 120°C to ambient temperature. The effects of temperature on the sensing capability of the composite also were measured under cyclic loading shown in Figure 8-10(a). To prevent the interference of the drilled hole and thermocouple with the loading area, the load was applied in the middle of the specimen as depicted in Figure 8-7(c).

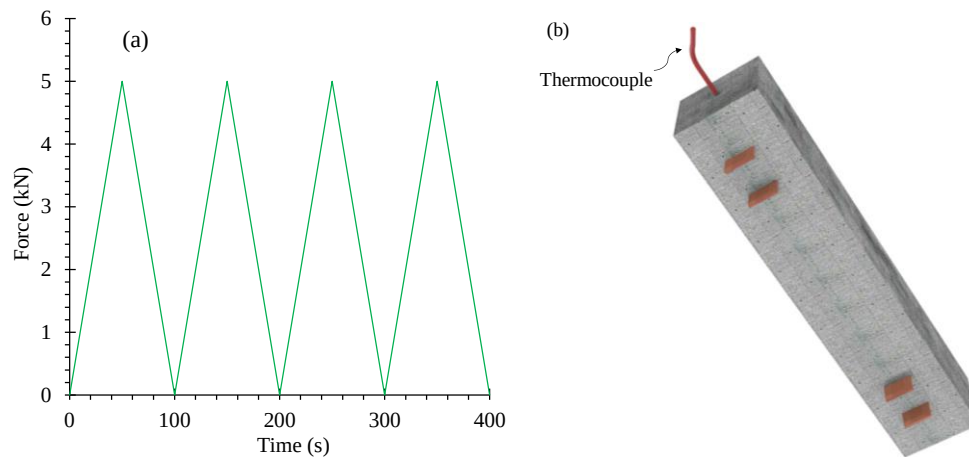


Figure 8-10. a) Loading pattern used for assessment of temperature and humidity effects, b) specimen configuration.

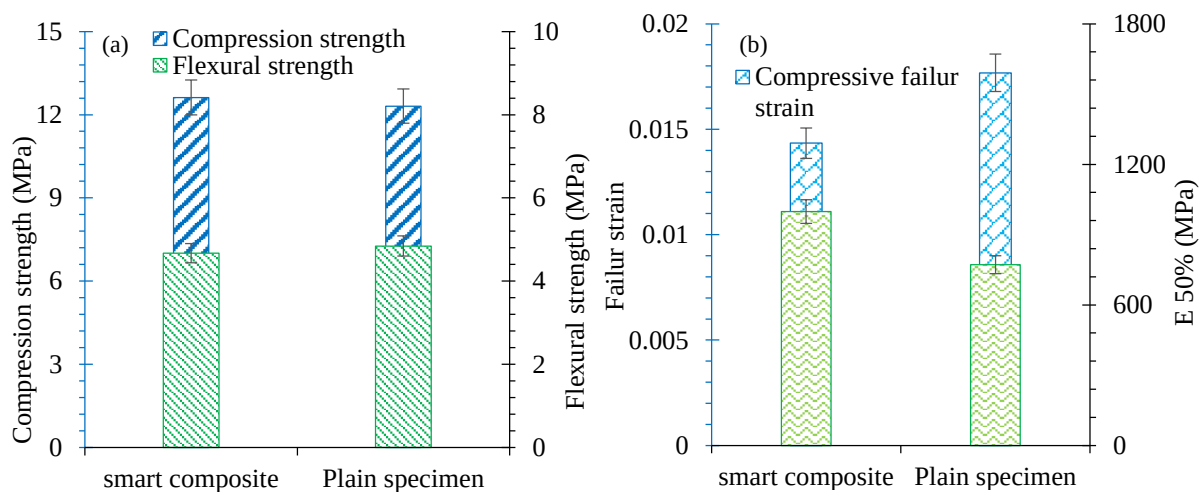
Effects of water content

A similar electric circuit (Figure 8-7 (c)) and loading protocol (Figure 8-10 (a)) were used for the investigation of water content effects on the piezoresistivity performance of the composite. Dried specimens were placed into the water, and the water content of the specimens was calculated through weight measurement in each step.

8.3 Results and discussion

8.3.1. Mechanical assessment

The main mechanical characteristics of the smart and plain composite including compression and flexural strength, compression failure strain and elastic modulus, are reported in Figure 8-11.



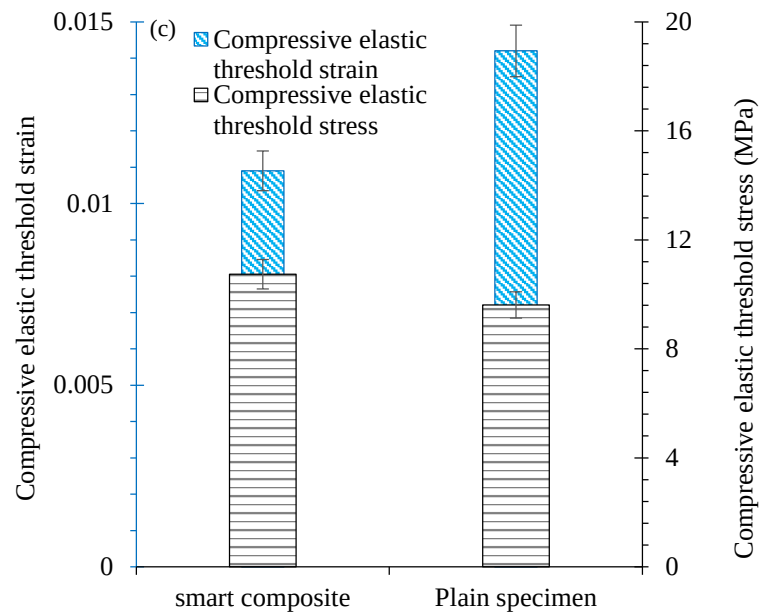


Figure 8-11. Main mechanical characteristics of the smart and plain cementitious geocomposite: a) compressive and flexural strength, b) compressive failure strain and compressive elastic modulus ($E_{50\%}$), c) compressive elastic threshold strain and stress.

Since the modulus of elasticity for cementitious geomaterials is typically expressed by the modulus at 50% of the peak stress [410, 582], the tangent $E_{50\%}$ of the specimens was calculated at 50% of the maximum compression stress. As can be observed, the incorporation of 1.0% CNMs into the cementitious stabilized sand, didn't cause significant changes in the compression and flexural strength of the composite. However, as shown in Figure 8-11(b), the compression elastic modulus of the smart composite was increased by around 29%. Meanwhile, the compression failure strain of the composite was reduced by approximately 20% influenced by the inclusion of the nanomaterials. A similar trend has been reported in the literature about reinforcing cementitious composite reinforced with CNMs [180, 583].

8.3.2. Microstructural investigation

The morphology of the smart composite containing CNMs is illustrated in Figure 8-12. Clearly can be observed that the incorporation of CNTs and GNPs into the cementitious composite prevent crack propagation through crack bridging and/or its path deviation. However, in some cases, the inclusion of a high concentration of CNMs required for some necessities of sensing issues caused the agglomeration formation which consequently reduces the physical and mechanical performances Figure 8-12 (d). The CNTs bridge across the cracks and the GNPs effectively divert them, thereby reducing the accumulation of cracks. These phenomena greatly increase the load-carrying capacity and forces needed for the development of crack and element failures. When a crack meets a buried CNT or GNP, it is blocked, and a new load increase is needed for further propagation. Generally, the inclusion of an optimum

concentration of CNMs (from the mechanical point of view) into the cementitious composite, improves the physical and mechanical properties of the composite through filler effects, crack bridging and deviation [584-587], and hydration rate enhancement [588-590].

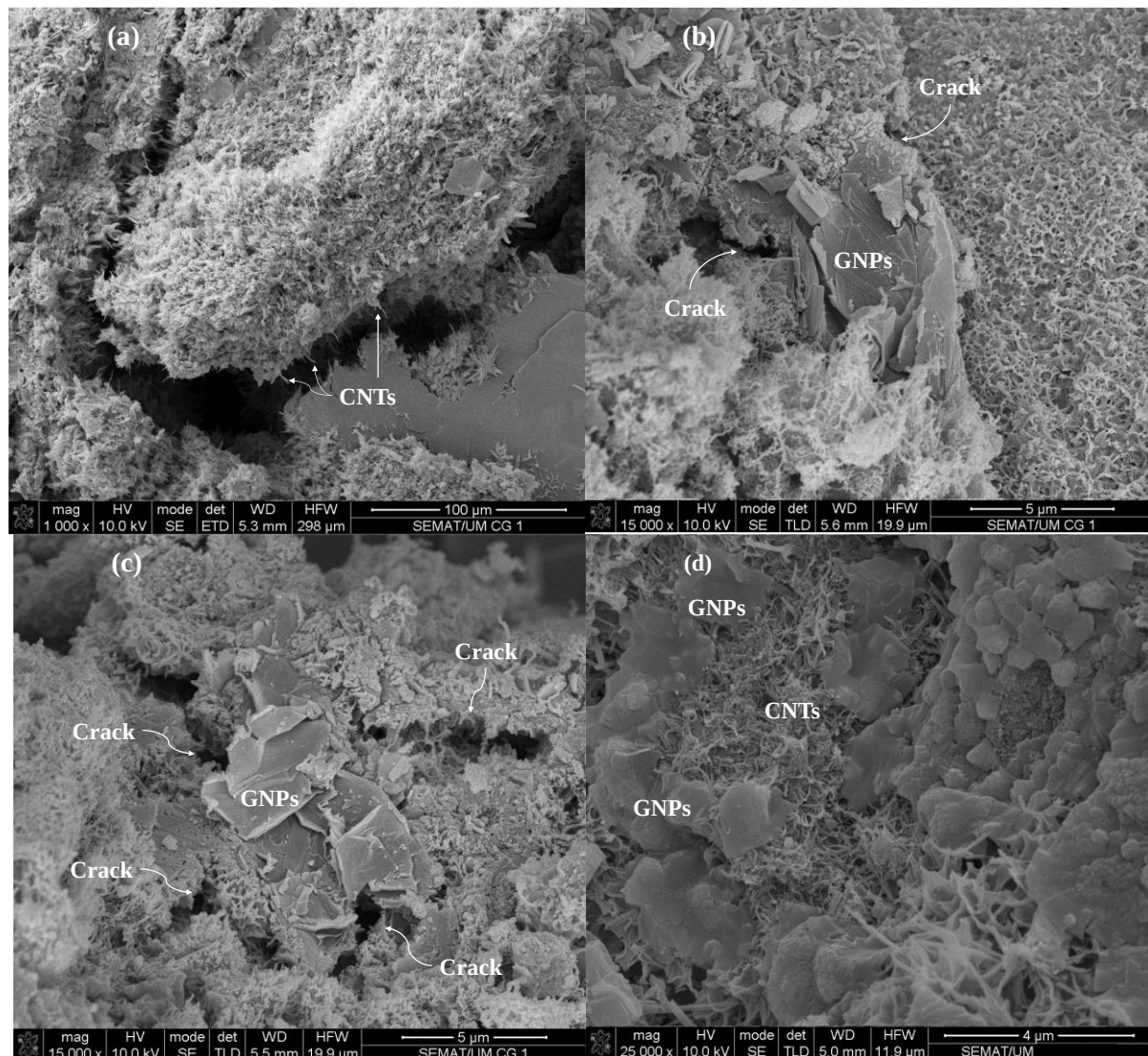


Figure 8-12. SEM photographs of the smart cementitious composite: a), b), and c) crack bridging and divination by CNTs and GNPs, d) CNTs and GNPs agglomerations.

CNM inclusion can promote cement's pozzolanic reaction, particularly in the early ages. The CNTs and GNPs in fresh cement are absorbed by the cement grains, while the majority are dispersed throughout the pore solutions [591]. There is compelling evidence that the CNMs, whether presented in agglomerate or individual forms, act as nucleation sites for cement hydrates to form and grow [592]. Following the hydration reaction, C-S-H gels form around CNTs preferentially. As time passes, the C-S-H layer covering the CNTs becomes thicker, and after roughly three hours, a network of C-S-H gel will be formed [593, 594]. The growth of cement hydrates products on GNPs has also been reported in the literature. Due to the high surface energy of GNPs and the hydrophilic groups, the C-S-H deposits on the GNPs sheets can form on the surfaces [592]. Indeed, the GNPs surfaces act as a nucleating substrate for cement hydrates

products, especially C-S-H, which preferentially grows on the GNP rather than solely on the surface of the adjacent cement grains. In this study, thermography and XRD measurements were conducted to further evaluation of CNMs' effects on cement hydration rate in cementitious geocomposite and the results are shown in Figure 8-13 and Figure 8-14. In thermal analysis, generally, the decomposition of cement hydrates can be divided into four main phases. The first step in the temperature range from 25 to 105 °C represents the evaporable surface adsorbed water. The second endothermic decay between 105 and 400 °C is attributed to the dehydration of C-S-H and ettringite and calcium aluminate hydrate (AFt and AFm) [595]. The CaO: SiO₂ ratio that is present in the hydrated cement matrix determines the temperature at which these compounds begin to lose water. The third peak between 400 and 600 °C corresponds to the dehydroxylation of Portlandite (decomposition of Ca(OH)₂, (CH) formed during hydration).

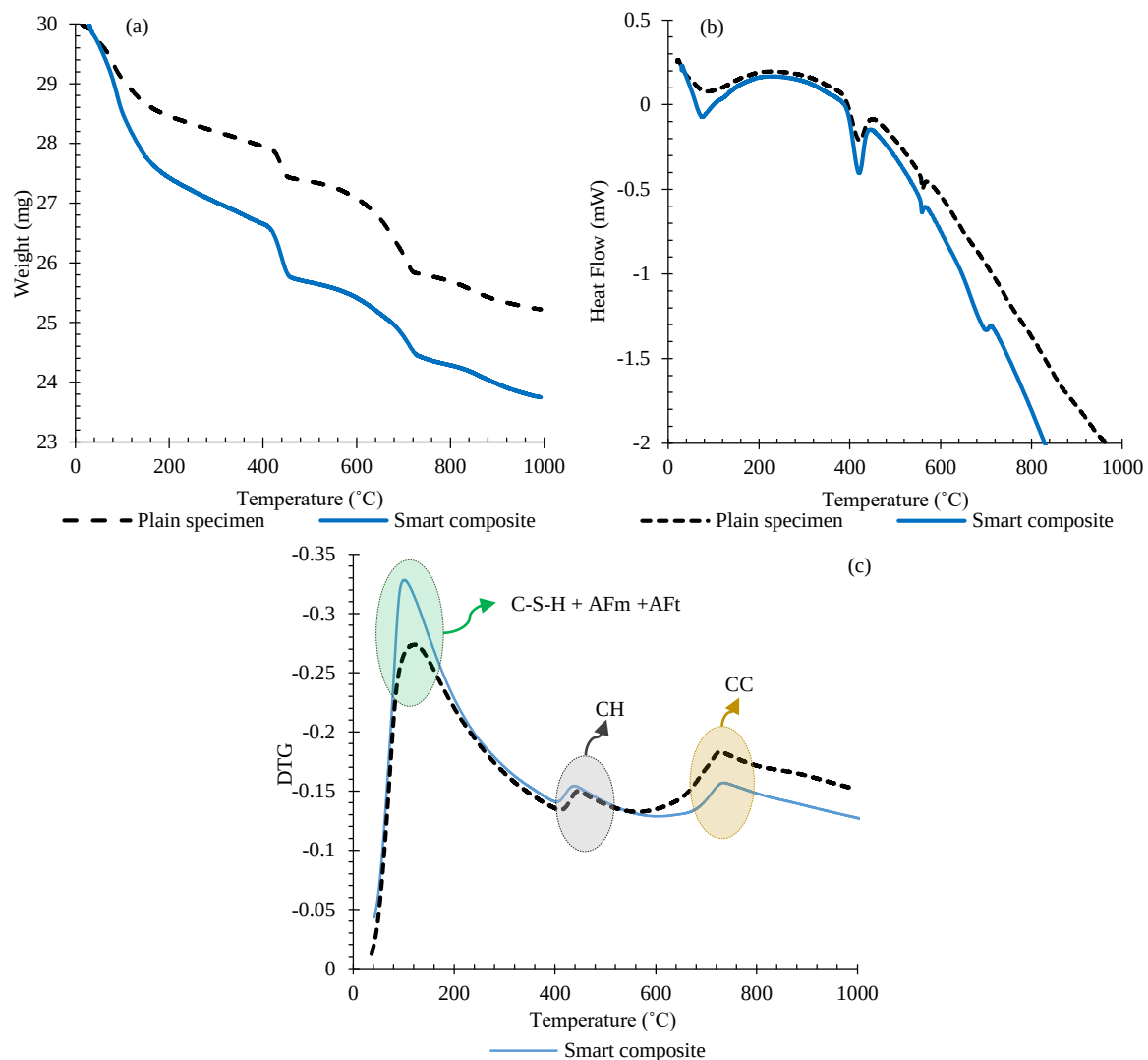


Figure 8-13. Thermography analysis of smart and plain composite: a) TGA, b) DSC, c) DTG.

Finally, the last endothermic peak represents the decarbonisation of calcium carbonate in the hydrated compound (CaCO_3 , CC) recorded at approximately between 600 and 800 °C [596, 597]. The curves all indicated the higher hydration degree of the CNM-reinforced composite. As can be observed the XRD patterns (Figure 8-14) also showed a similar trend.

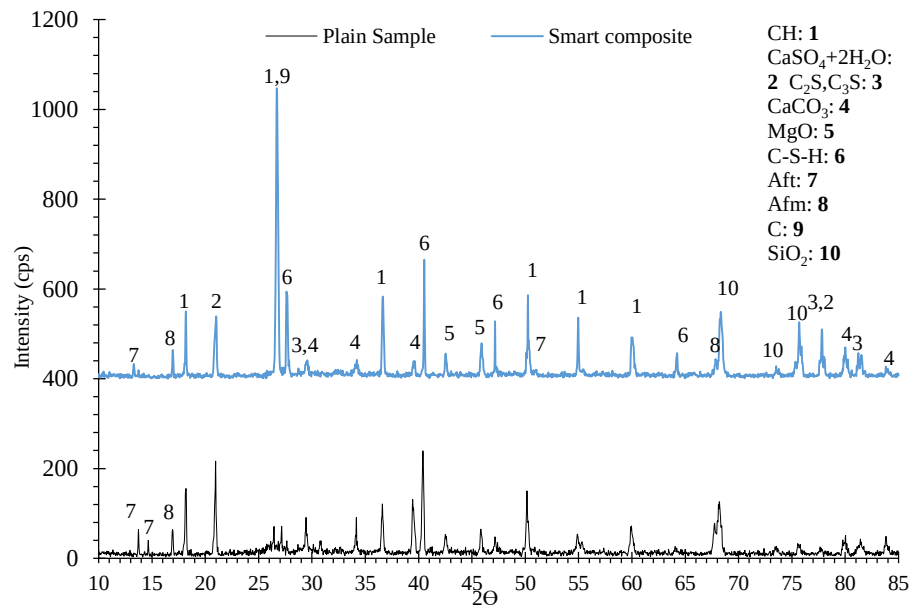


Figure 8-14. XRD spectra of the smart and plain composite.

The intensities of the characteristic peaks with respect to CH and C-S-H in the smart composite containing the CNMs were both higher, while the intensity of the C_2S and C_3S phases corresponding to un-hydrated cement grains, in this composite was lower than the plain sample.

8.3.3. Self-heating performance

The Joule effect, or the typical heating mechanism of an electric resistance, which is produced by the dissipation of the kinetic energy of the moving electrons within an electric field when they interact with the atoms of the material, is responsible for the self-heating function in carbon-cement composites [561, 562, 598]. By implementing this idea to structural materials and layers of the infrastructures, it is feasible to use the structural material itself to cause the ice to melt (or to prevent its formation) and to facilitate the healing process in the top layers of bitumen or polymer composites. The heating rates of the smart cementitious composite in different currents are presented in Figure 8-15. The results showed a significant increase in temperature and heating rate with increasing currents. The heating rate and self-heating power for this composite with 9A and 12V, 6A and 8V, 3A and 4V circuits were approximately 0.98 and 526, 0.83 and 476, 0.45 °C/s and 272 mW/ °C respectively.

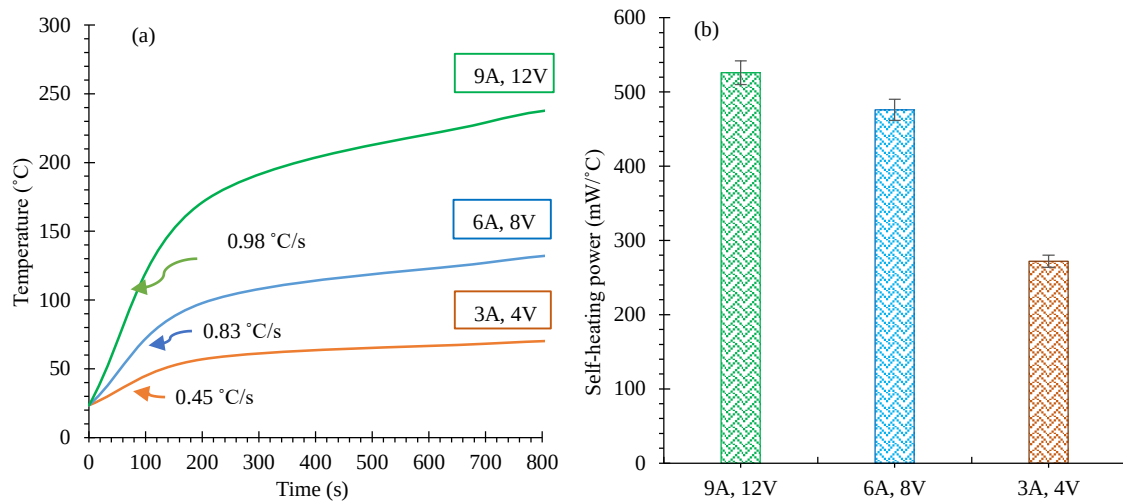


Figure 8-15. Self-heating performance of the smart cementitious geocomposite: a) the heating rate, b) self-heating power. Thermographic images of the smart cementitious composite in different currents are also illustrated in Figure 8-16 to show the maximum temperature and heat distribution after 800s in the specimen. As can be seen, the temperature of the specimen increased to approximately 62, 117, and 250 °C by applying 3, 6, and 9 A currents during the test, respectively. The results indicate the high potential and ability of the composite in terms of temperature adjustment in a wide range according to the intended application.

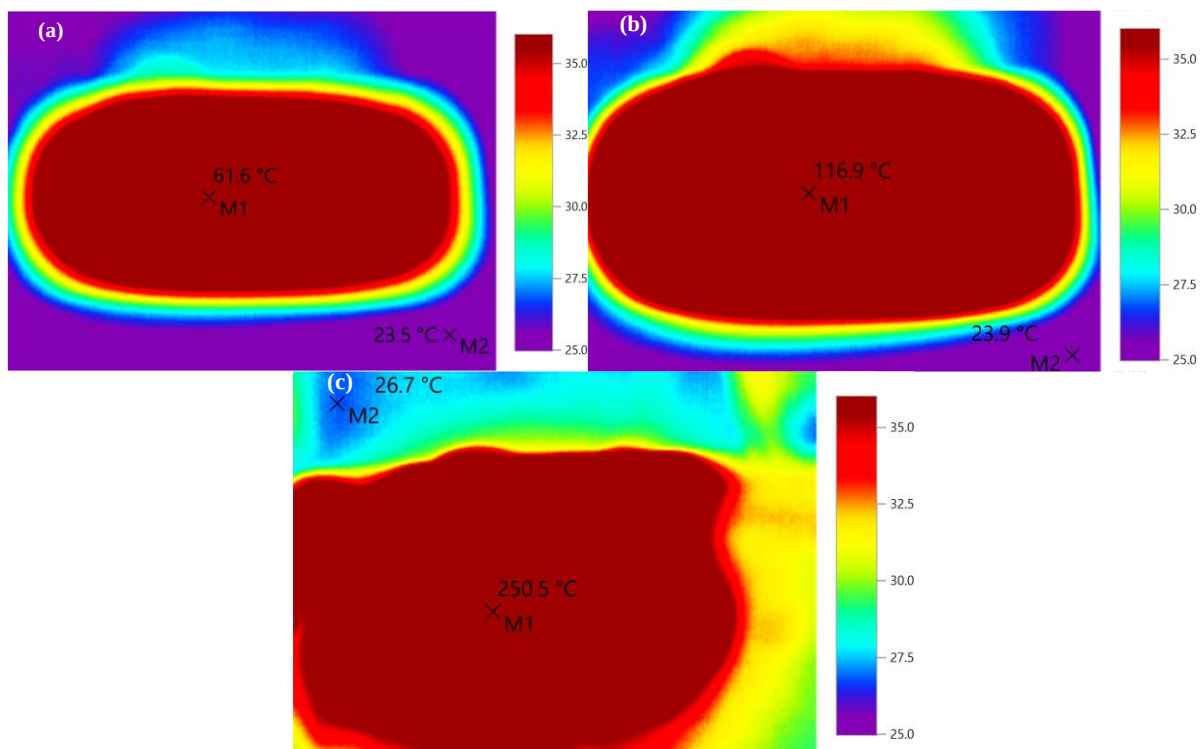


Figure 8-16. Thermography images of smart cementitious geocomposite: a) 3A and 4V, b) 6A and 8V, c) 9A and 12V.

Self-heating performance of different cementitious composites containing different conductive fillers is presented in Table 8-3. The comparison of the result proved the adequacy of the smart composite potential in terms of self-heating performance.

Table 8-3. Self-heating performance of the different cementitious composite.

Type of the composite	Conductive filler		Current and/or voltage	Temperature (°C)	References
	Type	percentage (wt%)			
Mortar	Graphite powder	8.75	40V	39	[562]
Mortar	Expanded graphite	5	10V	92	[598]
Cement paste	Graphite powder	5	40V	76	[561]
Concrete	Carbon fiber	1(by volume)	118V	45	[599]
Cement paste	CNT	2	10V	65	[600]
			8V	55	
			3A/4V	62	
Cementitious stabilized sand	CNT/GNP	1	6A/8V	117	Present study
			9A/12V	250	

8.3.4. Self-sensing performance

Effects of temperature

The effects of temperature changes on the electrical performances of the smart cementitious geocomposite are shown in Figure 8-17. As can be observed, the electrical resistance of the composites reduced with increasing temperature. Indeed, increasing the temperature, raise the kinetic energy and consequently the potential of electrons for jumping from the valence band to the conduction band, creating free movement between the two bands, thus, increasing the conductivity. As shown in Figure 8-17 (b), there is a well-defined relation between electrical resistance and temperature which proved the high potential and capability of this composite for temperature sensing applications in the field. The piezoresistivity behaviour of the smart composite in different temperatures is also shown in Figure 8-17 (c). The results indicated an improvement in the sensing performance of the composite with increasing temperature. It can also be seen that increasing the temperature caused an increase in the amount of the strain, which can be one of the reasons in terms of piezoresistivity enhancement. Meanwhile increasing the kinetic energy of the electrons and electrical conductivity of the specimen with rising the temperature can boost the electron tunnelling effects and consequently improve the piezoresistivity response of the composite against stress. A similar trend was also observed in the previous studies [601, 602]. At temperatures of 35.6, 59, and 102 °C, the amount of GF was 165, 116, and 155, respectively.

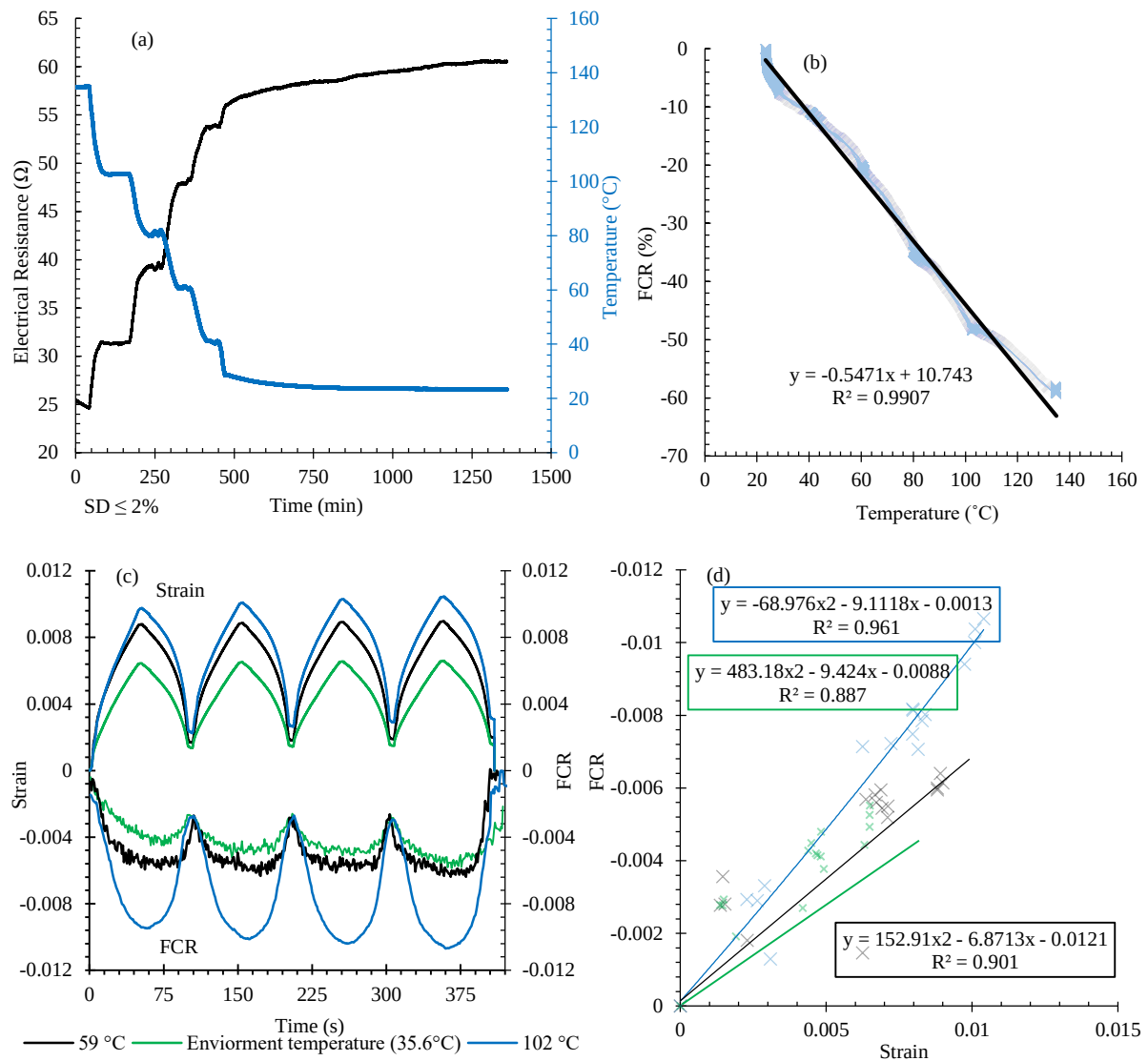


Figure 8-17. Effects of temperature on the electrical performance of the cementitious geocomposite: a) variations of electrical resistance against temperature, b) temperature coefficient of resistance, c) effects of temperature on piezoresistivity behaviour, d) variations of FCR against the strains in different temperatures.

Effect of moisture content

The results of piezoresistivity behaviour for the smart cementitious geocomposite in different moisture content are shown in Figure 8-18. As can be observed increasing the water content reduced the piezoresistivity response of the composite. Although there are contradictory results regarding the effect of water content in reducing or increasing the electrical resistance of the cementitious composites [601], most of them indicate a decrease in FCR and piezoresistivity response with an increase in the amount of moisture content in the specimen [602]. It seems that the effects of water content on the electrical resistance of the cementitious composites are very dependent on the percentage of nanomaterials. Indeed, in the composite reinforced with a CNMs percentage lower than the percolation threshold, the number of free ions might be increased with the augment of the water content, thus resulting in the

enhancement of ionic conduction and streaming current [602]. However, in the composite containing a high amount of CNMs (more than the percolation threshold) the CNMs particle are enclosed with the water layers due to the water content increasing which boosts the electric polarization effect leading to the reduction of electrical conductivity [229, 602-604]. Furthermore, in the cementitious composites with a high concentration of CNMs, the conductivity and the piezoresistivity (FCR changes) are mainly due to the direct contact between CNMs particles, while a thin film of water reduces the direct connection of the particles and reduces its electrical conductivity [601]. In this circumstance, the conductivity of the ions had dominance over the electrons' conductivity and led to a higher electrical resistivity [114]. Since the CNMs particles are totally inside the water layers, the applied load initially caused the deformation of the composite and then transferred to the deformation of water layers, and finally delivered to the location alterations of CNMs particles [601]. Hence, the GF of the specimens decreased from 164 to 98.7 and 46.9 as water contents increased from 7.7% to 12.2 and 15.4%, due to the enclosing of the CNM particles, interference of ions, and electric polarization.

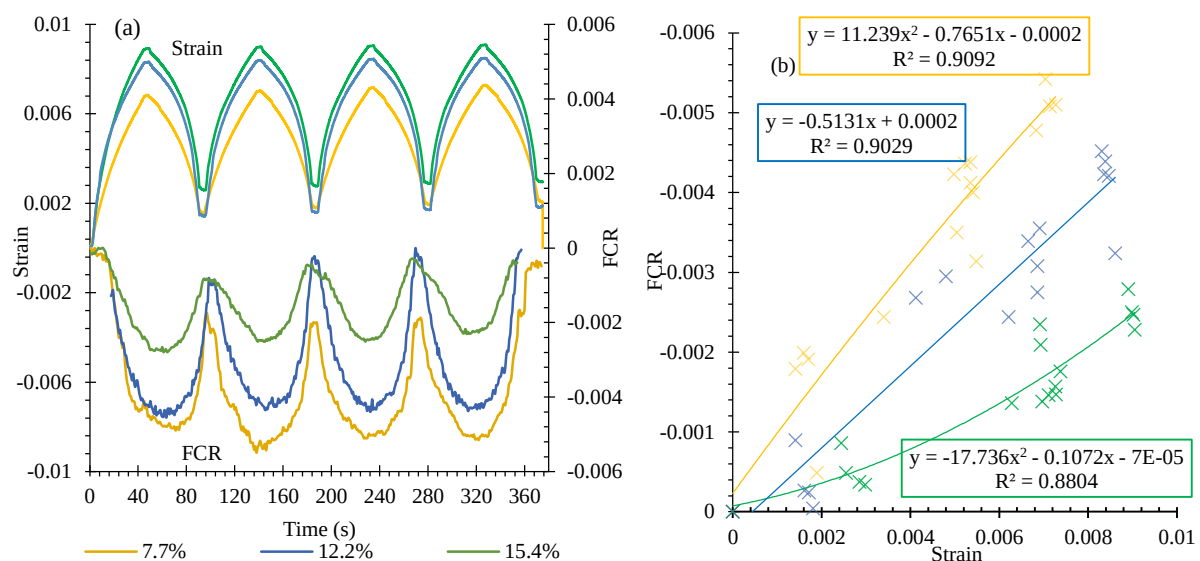


Figure 8-18. Effects of water content on the sensitivity performance of the cementitious geocomposite: a) piezoresistivity behaviour, d) variations of FCR against the strains in different water contents.

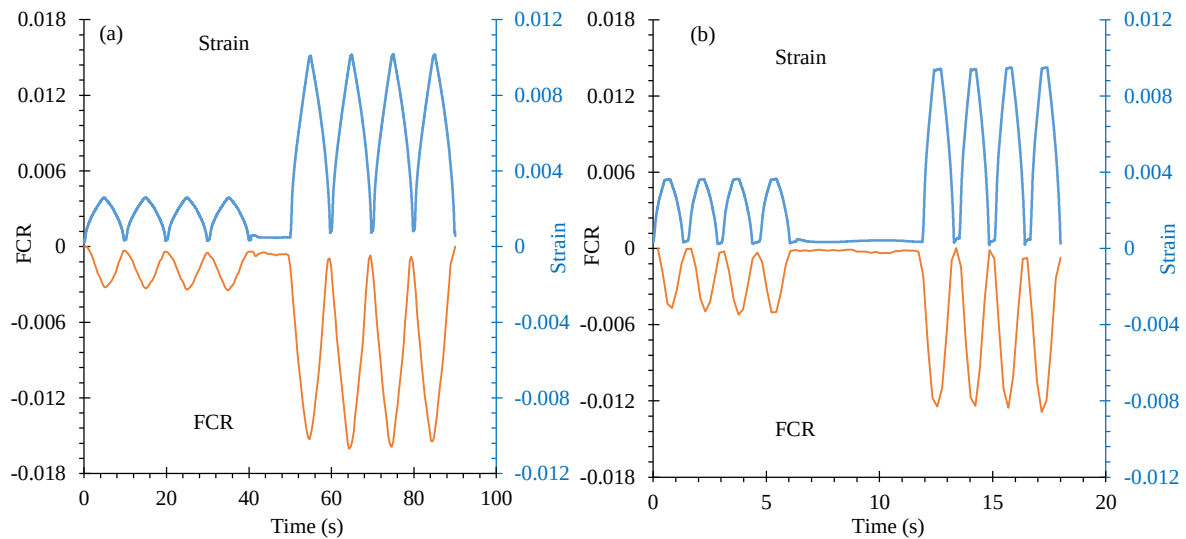
Effect of loading speed

The piezoresistivity responses of the smart cementitious geocomposite in different load amplitude and speeds are presented in Figure 8-19. The negative values were measured for FCR, due to a decrease in the electrical resistance during compression loading relative to the primary electrical resistance of the specimen. Indeed, the conductive paths and fillers get closer together during compression loading, resulting in more conductive pathways formation, which reduces the electrical resistivity and consequently enhances the electrical conductivity of the specimen.

As mentioned before (Figure 8-8), two different load magnitudes, one corresponding to the elastic region and another beyond of elastic threshold were used for this purpose. As can be observed, increasing the loading speed caused an increase in FCR corresponding to the loading less than elastic threshold stress. However, in the case of loading further from the elastic region, increasing the loading speed decreased the FCR values. A similar trend was also observed in the strain values which is aligned with the achievements of the previous studies [605].

Indeed, deformations occur quickly with loading in the elastic region (especially in preliminary steps) and increasing the loading speed, deprives the microstructure of the opportunity to return the deformations. Hence increasing the loading speed in this region (particularly for the low amount of loading) increase the amount of the strain and consequently FCRs. In the elastoplastic region, deformations occur for a relatively longer time, hence increasing the loading speed causes a decrease in the strain and FCR values.

Generally increasing the loading frequency from 0.1Hz to 0.5 and 1.0 Hz, increased the FCR values by around 54 and 109% for the loading corresponding to the elastic region (1.25 MPa), respectively. However, increasing the frequency to the same values, reduced the FCR by approximately 20 and 32% for the loading beyond the elastic threshold (11.125 MPa).



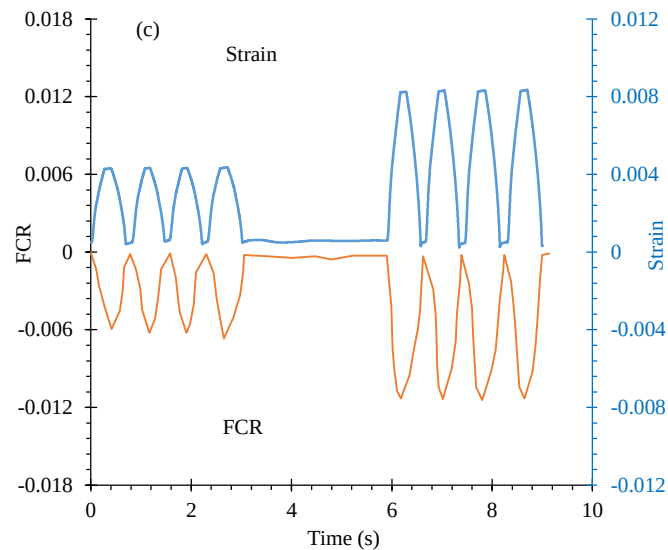


Figure 8-19. piezoresistivity behaviour of the smart geocomposite in different frequencies and load amplitude: a) 0.1 Hz, b) 0.5Hz, c) 1.0 Hz.

Effects of loading shape

In this section, the sensing performance of the smart cementitious geocomposite was assessed under different loading patterns to prove the detection capability of this composite for various sensing applications.

The piezoresistive behaviour of the smart cementitious geocomposite under the rectangular compressive cyclic loading with different magnitudes is shown in Figure 8-20. The results indicated a high correlation between the strain and FCR. Furthermore, increasing the stress magnitudes was accompanied by increased stability of the FCR values.

Chapter 8. Effects of Temperature, Humidity Loading Type and Speed on Sensing Potential of Cementitious Geocomposite.

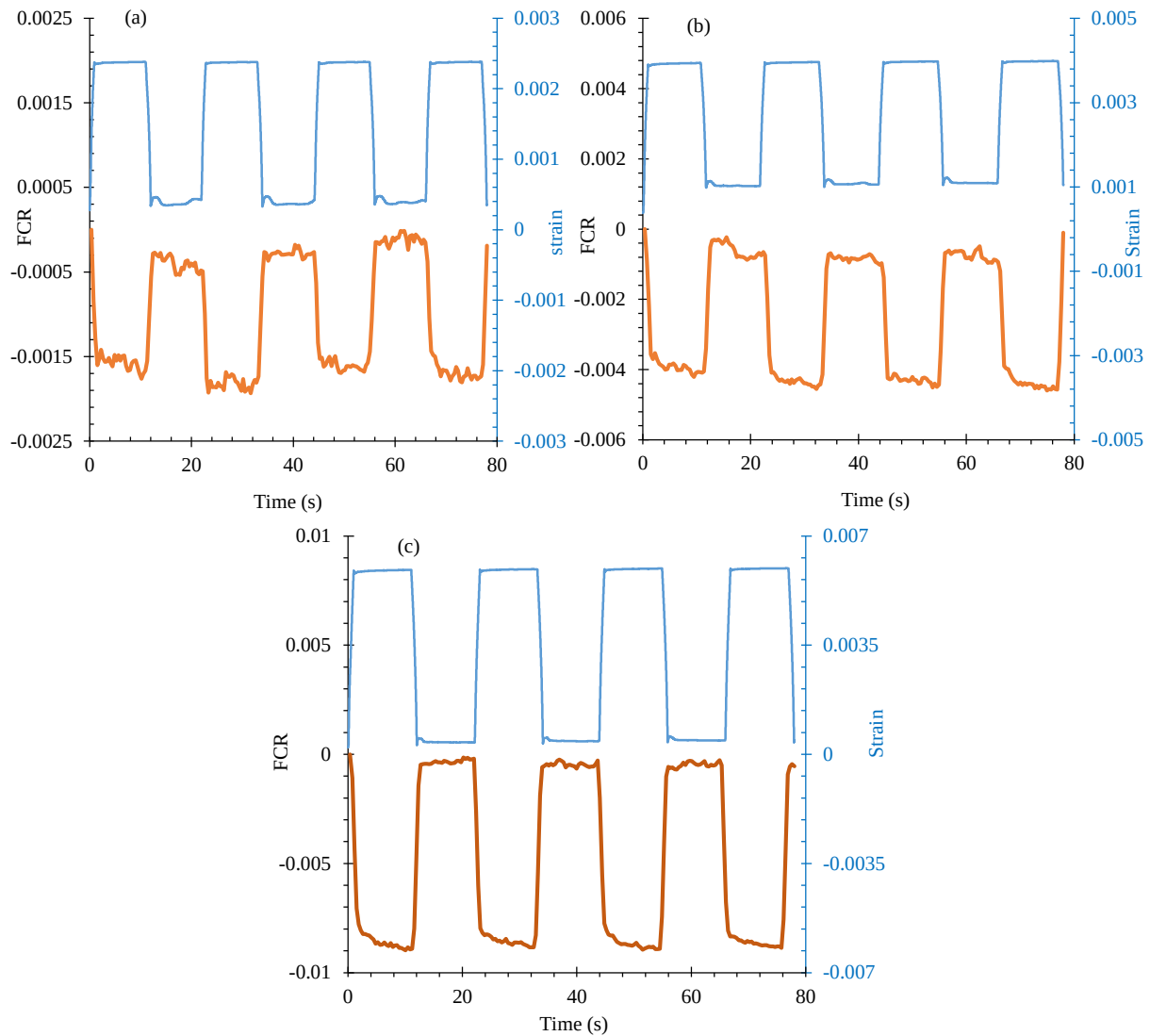


Figure 8-20. Rectangular loading cycle: a) 0.625 MPa, b) 1.25 MPa, c) 3.125 MPa.

The piezoresistive response of the smart cementitious geocomposite under continuous incremental and long-term cyclic loading is presented in Figure 8-21. The results indicate a unique correlation between strain changes and FCR values (Figure 8-21 (a)). The results indicate a unique correlation between strain changes and FCR values which proves the high potential of this composite for traffic monitoring. The significant changes in FCR values (approximately 42%) affected by the low amount of strain (0.0008) and load changes (1kN) showed the supreme capability of this cementitious geocomposite for high-resolution traffic recognition and monitoring.

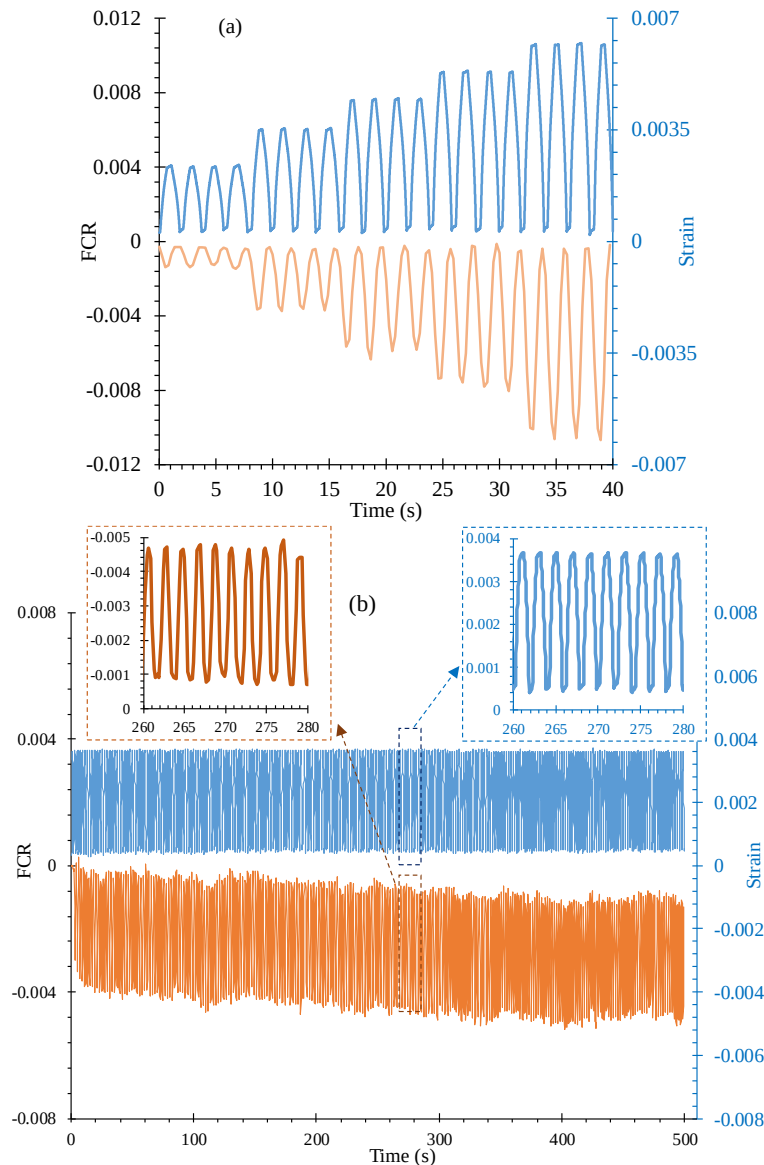


Figure 8-21. The piezoresistive response of the smart cementitious geocomposite under the continuous cyclic loading: a) continuous incremental compressive cyclic loading, b) long-term continuous compressive cyclic loading (250 cycles, 0.5Hz). As illustrated in Figure 8-21 (b), the stability of the FCR values and their correlation with strain changes after 250 compression cycles, also showed the proper sensing performance of this composite for long-term detection. The results also showed a low amount of polarization effects during long-term sensing.

Damage detection

The trend of FCR during the monotonic load increasing up to the failure point is illustrated in Figure 8-22. The slopes of the stress-strain and FCR-strain curves in each stage, indicate the modulus and gauge factor respectively. The typical strain distribution in the last captured DIC images at different loading stages is also shown in Figure 8-23. An appropriate correlation between GF and modulus can be observed which can be a bright horizon to detect the mechanical behaviour and consequently damage levels of the self-sensing cementitious composite through FCR analysis. The loading process of the specimen increased

linearly with strain increments up to 0.011. In this stage, the conductive fillers and paths are closer together under compression which reduces the electrical resistivity. By this point, the stress has reached 65% of its maximum capacity.

The amount of modulus and FCR in this stage are roughly 710 MPa and 4 respectively. With increasing the load in the next stage, the amount of strain becomes higher than the elastic threshold and the mechanical behaviour of the composite enters the non-linear region. At this stage, nanoscale cracks begin to form.

As the load increases to 93%, these cracks progress and propagate until the first microcrack appears on the specimen surface (Figure 8-23 (b)). The amount of modulus and FCR in this stage are roughly 576 MPa and 21 respectively. The formation and propagation of the cracks in the microstructure of the composite, reduce the stiffness of the specimen and consequently leads to the approaching of the conductive paths.

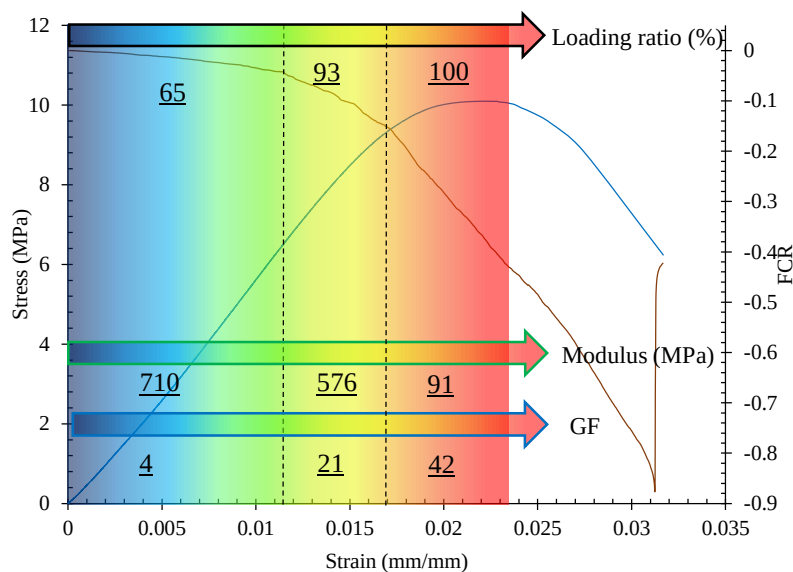


Figure 8-22. The trend of FCR-strain together with stress-strain during the monotonic load increasing.

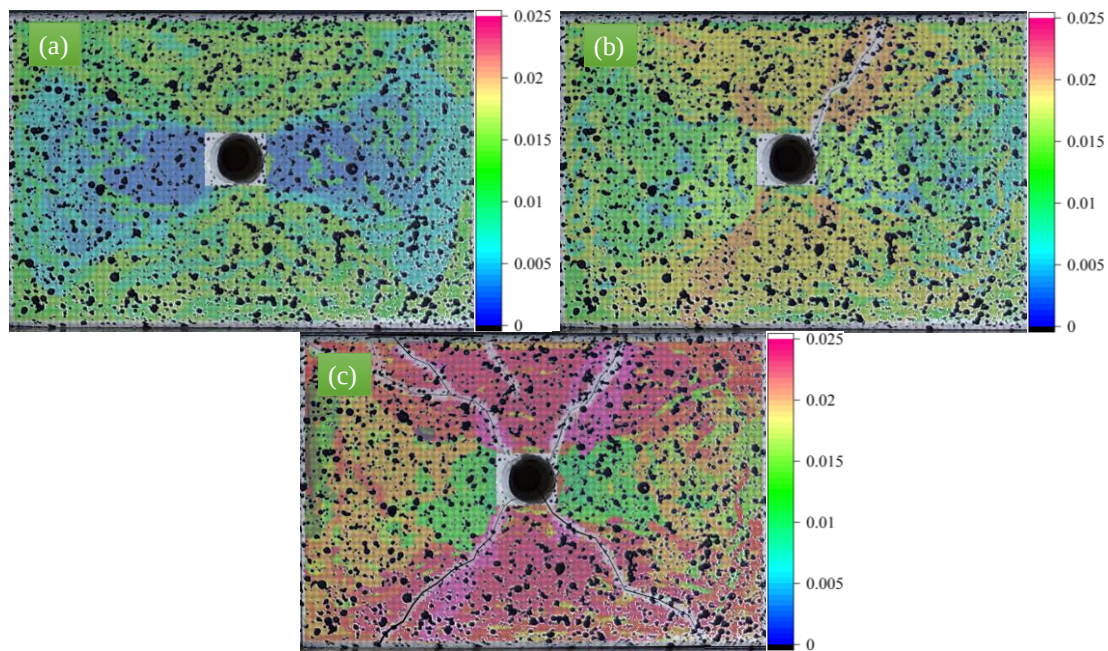


Figure 8-23. The typical strain distribution at different stages of monotonic loading: a) after 65% loading, b) after 93% loading c) after 100% loading (a moment before the failure).

Once the strain increases in the next stage, the specimen's loading force exhibits a roughly steady trend since the energy is released by the generation and propagation of cracks till failure (Figure 8-23 (c)). The average amount of modulus and FCR in this stage are roughly 91 MPa and 42 respectively.

The trend of FCR and strain changes during an increasing cyclic compressive loading till the failure point is shown in Figure 8-24. An appropriate correlation between FCR and mechanical behaviour was also obtained under cyclic compressive loading. A similar trend was also observed in the case of cyclic incremental compressive loading. The typical strain distribution at different stages of cyclic compressive loading is illustrated in Figure 8-25. The first microcrack appeared on the surface after approximately 91% of the loading of the specimen (Figure 8-25 (b)). As shown in Figure 8-24, the mechanical behaviour of the composite enters the non-linear region after 63% loading which caused the residual strain at the end of each cycle. The residual strain increased significantly as the loading rate and crack propagation increased.

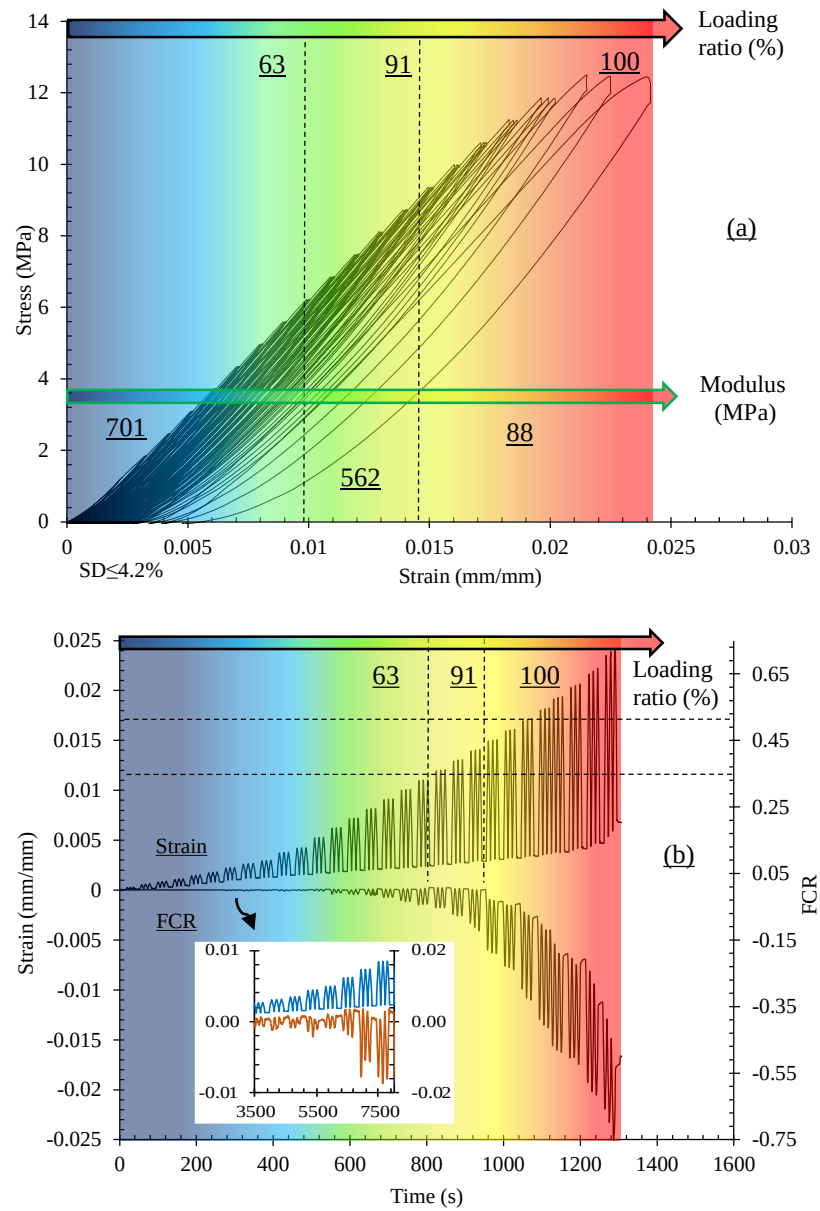


Figure 8-24. The trend of FCR and strain changes during the cyclic compressive loading: a) stress-strain curve, b) FCR together with strain over time.

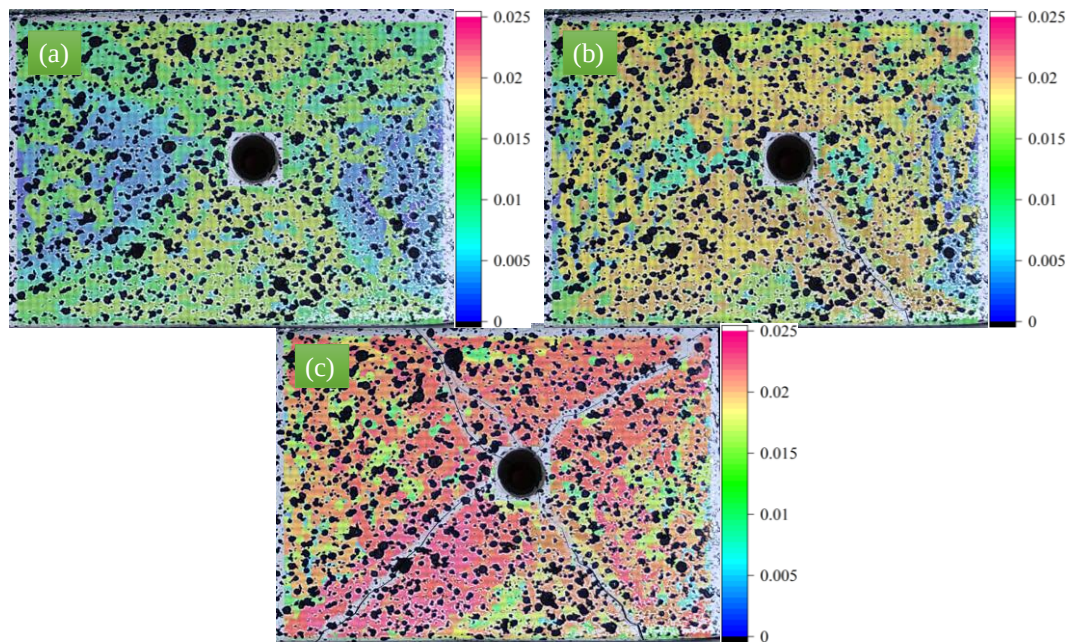


Figure 8-25. The typical strain distribution at different stages of cyclic compressive loading: a) after 63% loading, b) after 91% loading c) after 100% loading (a moment before the failure).

Hybrid section

The sensing capability of the smart cementitious geocomposite in a hybrid section, representative of one of the common structural layers of urban pavements, was investigated [606, 607]. This section was including a typical SMA11 asphalt mixture layer on top which was installed on the cementitious geocomposite with bitumen binder (Figure 8-7 (b)). The purpose of conducting this experiment was just to provide an idea to investigate the ability of composite sensing in a quasi-real pavement structure and also to assess the sensing potential of the smart geocomposite for the asphalt mixture layer strain detection. Hence, further detailed research is left to future studies. Nowadays, developing self-sensing asphalt composite has attracted the attention of many researchers [569, 608, 609]. However, the generation of an applicable smart asphalt composite is still faced with many challenges due to visco-elastic behaviour, conductive filler dispersion challenges, heterogeneity, cost of production and its complexity, low durability, and the direct effects of environmental factors [609].

Hence using smart cementitious geocomposite as an assistant layer for the asphalt composite in order to strain and stress detection might be a proper idea to overcome the existing challenges, particularly due to the high self-heating and temperature sensing capability of the smart cementitious geocomposite which can be employed for the self-healing applications in asphalt composite.

The piezoresistive behaviour of the smart cementitious geocomposite in the hybrid section is presented in Figure 8-26. The results indicated a proper correlation between strain and FCR values of the smart cementitious geocomposite. Furthermore, the unique correlation between the strain of the asphalt mixture

layer and FCR values of the smart cementitious geocomposite showed the ability of the smart geocomposite in terms of strain and stress detection in the asphalt mixture layer and proved the possibility of this concept. However, more comprehensive studies are still required.

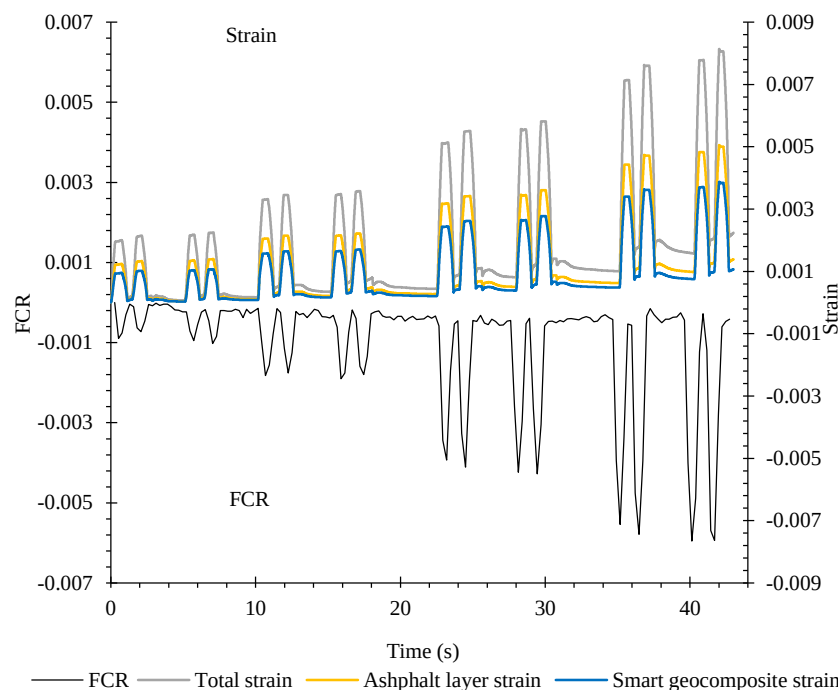


Figure 8-26. Piezoresistive response of the smart cementitious geocomposite in hybrid section.

8.4 Conclusions

In this study, the self-heating capability of a novel smart cementitious geocomposite was investigated in detail. In addition, the effects of temperature and moisture content on the piezoresistivity performance of the composite were also evaluated. Furthermore, the effects of different loading patterns including different loading speeds, magnitudes and shapes with the respect to different traffic loading simulations were assessed. The damage diagnosis capability of the composite was also assessed in both monotonic and cyclic loading through DIC analyses. Moreover, the ability of the geocomposite sensing in a quasi-real pavement structure and also its sensing potential for the asphalt layer strain detection were evaluated in a hybrid section including asphalt mixture layer and smart cementitious geocomposite and the following conclusions were drawn:

- Although the incorporation of 1.0% CNT/GNPs into the cementitious stabilized sand, didn't cause significant changes in the compression and flexural strength of the composite, the compression elastic modulus of the smart composite was increased by around 29%. Meanwhile, the

compression failure and elastic threshold strain of the composite was reduced by approximately 20% and 24% respectively.

- Thermography and XRD analysis showed a significant enhancement in cement hydration rate influenced by the inclusion of the CNT/GNP into the cementitious geocomposite. The SEM photographs indicated that the incorporation of an optimum concentration of CNMs (from the mechanical point of view) into the cementitious composite, improves the physical and mechanical properties of the composite through filler effects, crack bridging and deviation.
- The self-heating assessment and thermographic images of the smart cementitious geocomposite proved the high heating potential of the composite for affordable field application. The temperature of the specimens increased to approximately 62, 117, and 250 °C by applying 3, 6, and 9 A currents during the test, respectively. The heating rate for this composite with 9A and 12V, 6A and 8V, 3A and 4V circuits were approximately 0.98, 0.83, and 0.45 °C/s respectively. In addition, the self-heating power in the same supply circuit was also obtained by around 526, 476, and 272 mW/ °C respectively.
- The piezoresistivity behaviour of the smart composite in different temperatures indicated an improvement in the sensing performance of the composite with increasing temperature. At temperatures of 35.6, 59, and 102 °C, the amount of GF was 165, 116, and 155, respectively. A well-defined relation between electrical resistance and temperature was achieved which proved the high potential and capability of this composite for temperature sensing applications in the field.
- The results of piezoresistivity behaviour for the smart cementitious geocomposite in different moisture content showed the negative effects of moisture content on the sensing compatibility of the composite. Hence, the GF of the specimens decreased from 164 to 98.7 and 46.9 as water contents increased from 7.7% to 12.2 and 15.4%, due to the enclosing of the CNM particles, interference of ions, and electric polarization.
- Generally increasing the loading frequency from 0.1Hz to 0.5 and 1.0 Hz, increased the FCR values by around 54 and 109% for the loading corresponding to the elastic region (1.25 MPa), respectively. However, increasing the frequency to the same values, reduced the FCR by approximately 20 and 32% for the loading beyond the elastic threshold (11.125 MPa).

- The supreme correlation between strain changes and FCR values in different loading patterns including different loading shapes, speeds and magnitude, proved the high sensing capability of this smart geocomposite for traffic monitoring and recognition as well as health monitoring.
- Piezoresistive behaviour of the smart cementitious geocomposite in the hybrid section, representative of the common structural layers of urban pavements, showed the ability of the smart geocomposite in terms of strain and stress detection of itself and even in the asphalt mixture layer.
- An appropriate correlation between GF, modulus, and DIC analysis was observed in both monotonic and cyclic loading. This can be a bright horizon to detect the mechanical behaviour and consequently damage levels of the self-sensing cementitious composite through FCR analysis.

Chapter 9. Conclusions and Future Research Line.

«Every end is a new beginning. »

"مدعی خواست که از خاک کند ریشه ما غافل از آنکه خدا هست در اندیشه ما"

General Objectives	Chapters	Topics
	Chapter 1	Introduction
Research foundation, guiding methodology, choosing an appropriate conductive phase and dispersion method	Chapter 2	State of the art
Proof of The Concept	Chapter 3	Dispersion method development
	Chapter 4	Self-sensing cementitious mortar development
Self-sensing cementitious geocomposite development	Chapter 5	Self-sensing cementitious geocomposite development
	Chapter 6	Effects of the fibers on physical, mechanical, and piezoresistivity performances of cementitious geocomposite
Addressing the affecting factors	Chapter 7	Effects of size and electrodes layout on sensing potential of cementitious geocomposite
	Chapter 8	Effects of temperature, humidity loading type and speed on sensing potential of cementitious geocomposite
	Chapter 9	Conclusions and future research line

9.1 Introduction

In an era of advancing technologies and smart infrastructure, the need for innovative materials to monitor and enhance the performance of transportation and railway systems is paramount. This thesis presents a comprehensive exploration of self-sensing cementitious geocomposites, a promising class of smart materials, for intelligent infrastructure monitoring.

The journey of this paper-based thesis has been dedicated to exploring the innovative realm of self-sensing cementitious geocomposites for smart infrastructure monitoring, with a specific focus on transportation and railway systems. This work has centred on the potential of hybrid carbon nanotubes (CNT) and graphene nanoplatelets (GNP) as fillers in cementitious composites, uncovering their transformative capabilities for intelligent infrastructure applications. Through an in-depth analysis of laboratory outcomes, each chapter has contributed valuable insights into the dispersion and fabrication process, mechanical properties, microstructural characteristics, durability, piezoresistive behaviour, and effecting factors of these advanced materials.

9.2 Conclusions

As we approach the conclusion of this thesis, the following key conclusions emerge from the research presented in the preceding chapters. Through a systematic exploration of various laboratory tests, the study establishes a cohesive understanding of the immense promise offered by self-sensing cementitious geocomposites. Optimized dispersion techniques have demonstrated their importance in achieving superior microstructural characteristics and mechanical behaviour in these smart geocomposites. The hybrid CNT/GNP fillers play a pivotal role in reinforcing the mechanical properties of cementitious geocomposites, leading to significant improvements in flexural and compressive strengths, alongside enhanced elastic moduli.

Beyond their mechanical prowess, these self-sensing geocomposites exhibit impressive durability and carbonation resistance, effectively withstanding harsh environmental conditions such as freeze-thaw cycles. Their denser microstructures and efficient dispersion of CNT/GNP contribute to prolonged service life, positioning them as promising candidates for sustainable and long-lasting infrastructure projects.

One of the most notable findings lies in the piezoresistive behaviour of smart geocomposites. The inclusion of hybrid CNT/GNP fillers imparts self-sensing capabilities to the cementitious geocomposites, allowing them to gauge fractional changes in electrical resistance under cyclic loading. This piezoresistive response

serves as a reliable indicator of strain and stress, opening up avenues for real-time traffic and structural health monitoring in transportation and railway systems.

Additionally, the investigation into the self-heating capability of these smart cementitious geocomposites reveals a unique potential for temperature-sensing applications in the field. The materials showcase self-heating properties under applied electrical current, paving the way for innovative temperature monitoring, de-icing, and self-healing approaches in smart infrastructure.

Considering cost, sensing and physical performance, 1% hybrid CNT/GNP (1:1) is effective for developing smart cementitious geocomposites. Higher CNM concentrations can enhance sensing and electrical performance, but they can significantly challenge the physical and mechanical properties.

The developed dispersion technique serves as an efficient method for cementitious geocomposite development. However, an alternative dispersion method is required when dealing with lower water content or higher CNM concentration.

The electrode layout, type of current, and overall electrical circuit significantly impact the composite's sensing ability. Therefore, for large-scale field applications aimed at long-term health monitoring, it is recommended to divide the layer into smaller sections with electrodes placed at the boundaries and outside the loading region, using either AC or DC current. For speed and weight (traffic) detection, the electrodes should be placed within the loading area, utilizing DC current.

Environmental factors like temperature and humidity indeed influence the sensing potential of the composite. However, since both long-term health monitoring and traffic detection rely on fractional or differential changes, the impacts of environmental factors can be disregarded.

In conclusion, the research presented in this thesis has contributed significantly to the understanding and advancement of self-sensing cementitious geocomposites for smart infrastructure monitoring. The integration of multi-functional properties, long-term performance evaluation, and further optimization of composite characteristics will pave the way for the real-world implementation of these innovative materials. As we move forward, the application of smart geocomposites in intelligent structures promises to revolutionize the monitoring and maintenance of transportation and railway systems, ensuring the sustainability and resilience of critical infrastructure.

9.3 Future Research Directions:

As we conclude this thesis, the research presented opens the doors to several promising future research directions in the field of self-sensing cementitious geocomposites for smart infrastructure monitoring. The following areas warrant further investigation: **Long-term Field Performance:** Conducting long-term field studies to assess the performance and durability of self-sensing cementitious geocomposites in real-world infrastructure applications is crucial for validating their practicality and reliability. **Multi-functional Properties:** Exploring the integration of additional functionalities into self-sensing geocomposites, such as self-healing capabilities, could further enhance their potential for smart infrastructure applications. **Environmental Impact Assessment:** Conducting comprehensive environmental impact assessments to ensure the eco-friendliness and sustainability of self-sensing cementitious geocomposites, addressing concerns related to waste generation and end-of-life disposal. **Integration in Pavement Systems:** Investigating the practical implementation of self-sensing geocomposites in real pavement systems and assessing their ability to monitor traffic loading, pavement performance, and damage detection in transportation infrastructure. **Smart Infrastructure Monitoring Systems:** Developing integrated smart infrastructure monitoring systems that utilize self-sensing cementitious geocomposites as part of a comprehensive monitoring and maintenance strategy for transportation and railway systems. **Scaling-Up Production and Cost-Effectiveness:** Exploring scalable and cost-effective production methods for self-sensing geocomposites to facilitate their widespread use in large-scale infrastructure projects.

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