

NATURAL OUTDOOR AND LABORATORY-CONTROLLED AGEING OF EPOXY ADHESIVES AND CFRP LAMINATES AFTER FOUR YEARS OF EXPOSURE

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ABSTRACT

Ageing of epoxy adhesive and carbon fibre reinforced polymer (CFRP) laminate was addressed in this work. The specimens were exposed to laboratory-controlled (20 °C/55% RH, and water immersion at 20 °C) and outdoor (ageing mainly promoted by carbonation, freeze-thaws, airborne chlorides, and elevated temperatures) conditioning for up to four years. The results from tensile tests showed water immersion of the epoxy adhesive as the most severe exposure condition, yielding to 66% and 75% decrease in tensile strength and elastic modulus, respectively. In contrast, CFRP laminate generally improved the same mechanical properties in outdoor environments, the highest being 16.1% and 10.4%, respectively.

KEYWORDS

Epoxy adhesive, CFRP laminate, Accelerated aging, Natural ageing, Tensile strength, Elastic modulus.

INTRODUCTION

Fibre reinforced polymer (FRP) composites have been widely known as one of the most effective composite materials in aerospace, automotive and construction industries, among others. This is mainly because of the properties of these materials including high strength-to-weight ratio, high fatigue resistance, and high corrosion resistance. Focusing on the construction industry, FRP materials are typically used in strengthening of RC structures, e.g., they have been largely applied to strengthen bridges, buildings, tunnels, silos, and tanks (Abbood et al., 2021), among others. FRP composite materials currently being used as construction composite materials includes carbon, glass, basalt, and aramid. The former (i.e., carbon FRP or CFRP) has been widely used (Correia et al., 2015)(Cruz et al., 2021)(Tatar & Hamilton, 2016)(Sena-Cruz et al., 2015) mainly because of its advantageous properties such as high fatigue strength, high conductivity, low density and conductivity, water resistance, and high elastic modulus (Abbood et al., 2021). CFRP composites can be bonded to each other or to other materials using a suitable adhesive. An ideal adhesive needs to possess bonding properties that provide strong hard-to-break bonds to the adherend, such as covalent bonds. According to a study by (Yamane et al., 2022), epoxy adhesives can be an effective bonding agent to CFRP since it mostly includes covalent bonds with very few weak hydrogen bonds. Epoxy adhesives have been widely used as a bonding agent in different existing studies, e.g., (Correia et al., 2015) (Cruz et al., 2022) (Cruz et al., 2023) (Tatar & Hamilton, 2016).

Knowledge about long-term durability of both the CFRP composites and epoxy adhesives is a key aspect towards having a more advanced understanding on their long-term structural and sustainability performance. Many relevant existing durability studies have been conducted using accelerated aging conditioning, where a single or more than one degradation agents are basically imposed to the material (e.g., epoxy adhesive or FRP) in order to degrade its properties. For example, starting from studies addressing the durability of the epoxy adhesive, the properties of the adhesive have been found to decrease significantly after being exposed to moisture (Fernandes et al., 2018) (Cruz et al.,

2023), or when they were fully immersed in water (Fernandes et al., 2018) (Cruz et al., 2023). Furthermore, exposure of the adhesive to wet-dry cycles decreased both its tensile strength and elastic modulus (Cui et al., 2021), high temperatures improved the adhesive properties through post-curing phenomenon (Grammatikos et al., 2016), high carbonation accelerated the curing of the epoxy resin (Hu et al., 2018), and chlorides exposure led to non-harmful effects (Fernandes et al., 2018). On the other hand, studies on the durability of CFRP composites show that exposure of the CFRP to many of degradation agents generally does not lead to significant detrimental effects. For example, CFRP laminate was found to resist water immersion exposure for up to 2 years (Cruz et al., 2023), and thermal cycles in air (Liu et al., 2019). However, exposure of the CFRP to freeze-thaw cycles can reduce its tensile strength and strain (Jiang et al., 2022), and some microcracks can form due to moisture attacking the matrix resin, and UV radiation can affect some microns from the CFRP surface (Helbling & Karbhari, 2007). Regarding the studies where durability is addressed using natural ageing conditioning, very few studies exist, e.g., in (Cruz et al., 2021) (Tatar & Brenkus, 2021).

It can be noted from the above literature that the durability of both the epoxy adhesive and CFRP composites has been significantly addressed using accelerated ageing conditioning, with little knowledge of their behaviour when exposed to outdoor environments. This makes the durability of these materials still an open area for further research. Mainly, studies with natural outdoor exposures and comparative perspectives on both the accelerated and natural outdoor aging conditioning can play a major role in deciding the most appropriate methods to use when predicting the environmental conversion factors. In this context, the present work addressed the durability of two different epoxy adhesives (denoted as ADH1 and ADH2) and two CFRP laminates (denoted as L10 and L50). Both epoxy adhesives and CFRP laminates were exposed to the same environmental conditions (i.e., two different laboratory-based conditioning, and four different natural outdoor conditionings) for up to four years. Furthermore, durability tests were conducted yearly to examine the effects of degradation agents (in each studied environment) on the mechanical properties (tensile strength and elastic modulus) of each material.

METHODS

Properties of the epoxy adhesives and CFRP laminates

A total of 250 specimens of the epoxy adhesives were cast in dog-bone shapes as shown in Figure 1a. Two different types of the adhesives were used (adhesive denoted as ADH1 and the other denoted as ADH2). The adhesive ADH1 was produced by S&P® Clever Reinforcement Ibérica Lda. Company (Seixal, Portugal), while the adhesive ADH2 was produced by SIKA Schweiz AG (Zurich, Switzerland). The properties of both ADH1 and ADH2 are as shown in Table 1. Additionally, a total of 300 CFRP laminate specimens were produced. Examples of the L10 specimens are shown in Figure 1b. These laminates (L10 and L50) were produced by the same company that produced the adhesive ADH1, and their properties are as shown in Table 1. It can be noted that L10 and L50 solely differ by their thickness and widths, while other properties are the same.

Environmental exposures and testing times

Five specimens of each adhesive and six specimens of each CFRP laminate were first tested at the initial time (T0) to be considered as references (i.e., before exposure). The remaining 240 epoxy adhesive and 288 CFRP laminate specimens were then placed in six different environmental exposures. The first two environments were laboratory-based (one environment, denoted as E1, characterized by conditioning of the specimens around 20 °C/55% RH, and the other, denoted as E2, characterized by immersing the specimens in water with controlled 20 °C temperature, see Figure 1c). The remaining four environments were outdoor exposures, where the stations were selected in different regions of Portugal to mainly promote ageing due to carbonation (i.e., the environment denoted as E3), freeze-thaws attack (denoted as E4), elevated temperatures (denoted as E5), and airborne chlorides attack (denoted as E6). Example of outdoor environment (E4) is shown in Figure 1d. Every year, 10 epoxy adhesive specimens (i.e., 5 ADH1 and 5 ADH2), and 12 CFRP laminates (6 L10 and 6 L50) were collected from each of the mentioned environments (E1-E6) and tested. Four different testing times, corresponding to four different exposure times, were considered.

That is, the tests were performed at the testing times of one year (denoted as T1), two years (T2), three years (T3) and four years (T4) of exposures.

Table 1: Properties of the materials investigated

Property	Epoxy adhesive - ADH1	Epoxy adhesive - ADH2
Type of adhesive	Cold-curing S&P Resin 220	Sikadur-30
Flexural elastic modulus [GPa]	>7.1	-
Tensile strength [MPa]	19.9 (after 7 days of curing at 20 °C)	26, 29 ¹
Glass transition temperature [°C]	46.2 (after 7 days of curing at 23 °C)	52 (after 30 days of curing at 30 °C)
Density, at 23 °C [g/cm ³]	1.7-1.8	1.65
Compressive strength [MPa]	>70	>75, 90 ¹
Shear strength [MPa]	>26	>18
BS by pull-off, on concrete [MPa]	3 (after 3 days of curing at 20 °C)	>4 (after 7 days of curing at 23 °C)
Property	CFRP laminate - L10	CFRP laminate - L50
Type and trademark	S&P clever (CFK 150/2000)	S&P clever (CFK 150/2000)
Prefabricated by	Pultrusion	Pultrusion
Fiber orientation	Unidirectional	Unidirectional
Fiber content [%]	68	68
Fiber matrix	Vinyl ester resin	Vinyl ester resin
External surface	Black, smooth	Black, smooth
Elastic modulus [GPa]	>170	>170
Tensile strength [MPa]	>2000	>2000
Dimensions [mm]	1.4 (thickness), 10 (width)	1.2 (thickness), 50 (width)

Notes: ¹ after 7 days of curing at +10 °C., BS: bond strength

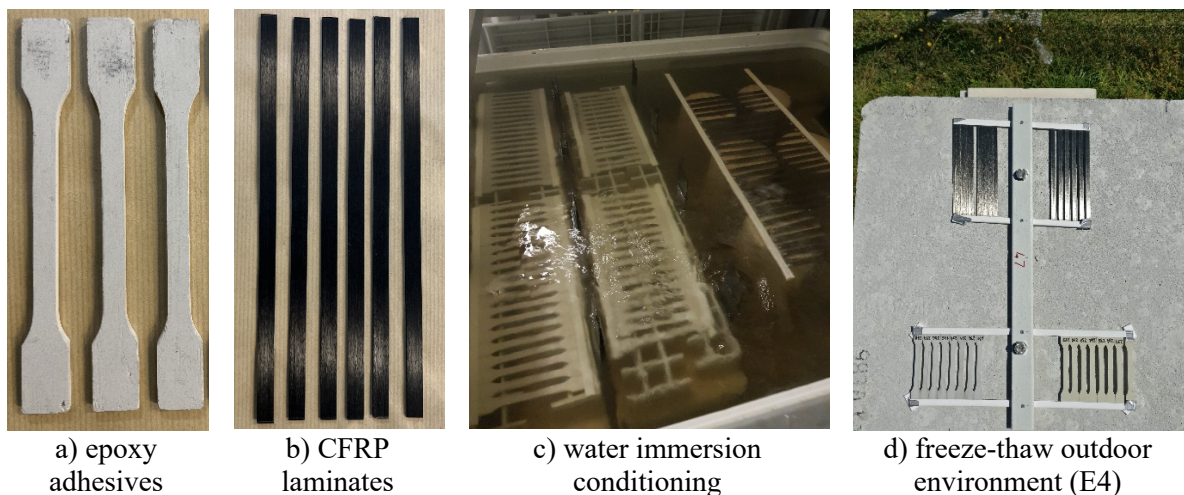


Figure 1: Materials and example of environments studied

Tensile tests for epoxy adhesives and CFRP laminates

Tensile tests for both the epoxy adhesive and CFRP laminate specimens were carried out using an MTS testing machine of 100 kN maximum capacity. Example of the tensile test of the epoxy adhesive is shown in Figure 2.a, while that of CFRP laminate is shown in Figure 2b. Testing of the epoxy adhesive followed recommendations as per EN ISO 527-2:2012 (ISO, 2012) and the elastic modulus was determined as recommended by the same standard, using the slope of the secant line on the stress-strain curve from 0.05% to 0.25% of the strains. On the other hand, the CFRP laminates were tested according to ISO 527-5:2009 (ISO, 2009) using the same test configuration as that of the adhesives. Typical failure modes observed for the adhesive and CFRP tests are shown in Figure 2c and Figure 2d, respectively. The adhesive failed by abrupt breaking within the region of interest, with a minimal sound produced. For the case of the CFRP laminate, an abrupt progressive brittle failure was observed, where the fibres at the width edges were the ones to start breaking, and then progressively moving towards the fibres in the centre of the laminate until the complete failure of all fibres occurred thereafter producing a massive sound.

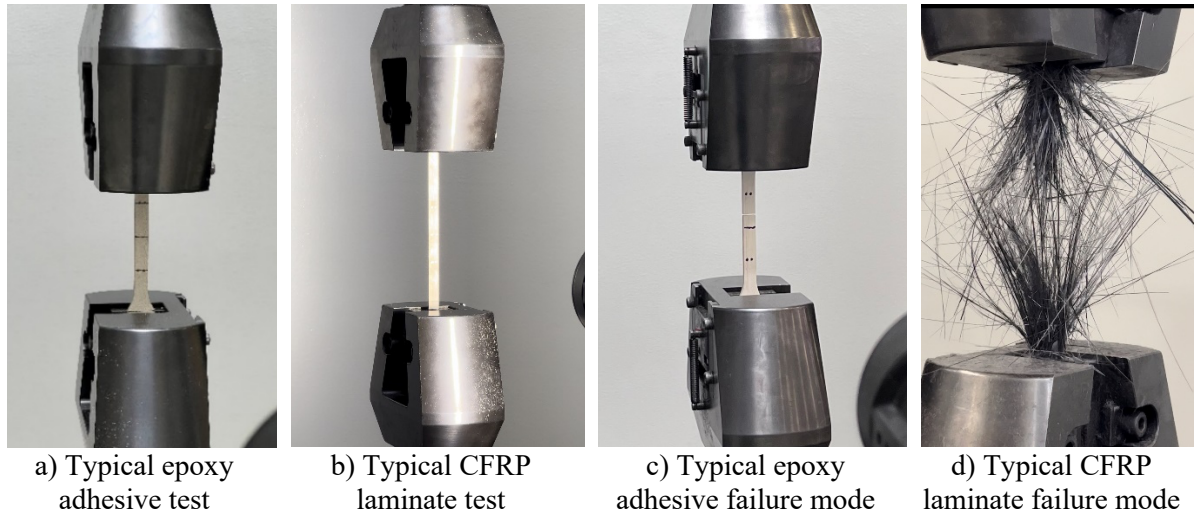


Figure 2: Tensile tests of the epoxy adhesive and CFRP laminate.

RESULTS AND DISCUSSION

Results from tensile tests of the epoxy adhesives

The tensile strength (f_a) and elastic modulus (E_a) of the two studied epoxy adhesives (ADH1 and ADH2) are shown in Figure 3 and Table 2. The results for each type of adhesive are discussed as follows.

Epoxy adhesive ADH1

The evolutions of the tensile strength and elastic modulus of the epoxy adhesive (ADH1) are shown in Figure 3a,b. The highest degradation of both f_a and E_a can be seen in E2 environment with retentions of 0.37 and 0.26, which corresponds to 66.3% and 75.4% decreases, respectively - it should be highlighted that the specimens of E2 environment were tested in wet state, i.e. just after being removed from the water. This substantial decrease can be attributed to the effects of plasticization, already known to lead to degradation of the adhesive properties (Fernandes et al., 2018). During the first year of exposure, ADH1 slightly increased both its f_a and E_a in outdoor environments (except E6), the highest increase being observed in E5 with approximately 1.10 and 1.15 retentions, respectively. The observed increase can be attributed to elevated temperatures in E5 that led to post-curing phenomenon thereby improving the adhesive properties. Post-curing is already known to improve adhesive properties, e.g., in (Grammatikos et al., 2016). However, in the latter years, it can be seen that ADH1 generally decreased both its f_a and E_a properties. This decrease may be thought to result from the degradations agents that dominated the post-curing effects, thereby progressively damaging the adhesive properties. The highest degradations in outdoor environments were found in E4 and E5, which can mainly be due to freeze-thaw cycles in the former and high humidity in the latter. This agrees with existing studies, where water and moisture significantly deteriorated the adhesive properties (Fernandes et al., 2018).

Epoxy adhesive ADH2

The f_a and E_a for ADH2 are plotted in Figure 3c,d. A pronounced decrease in both f_a and E_a for the specimens in E2 can be noted, just in a similar trend as that of ADH1. This decrease can be attributed to the effect of plasticization as previously explained. In E2, the f_a decreased to nearly 60% and the E_a to nearly 70% after 3 years. Similar behavior was observed for ADH1. Hence, the E_a seems to degrade faster than the f_a . Apart from the specimens in E2, all specimens in other environments showed a pronounced increase during T1 as a result of higher post-curing rate (than that observed for the case of ADH1) that dominated the negative effects from degradation agents in each environment. The highest f_a and E_a improvements were obtained from outdoor environments, particularly from E3 and E5, with approximately 1.39 and 1.4 f_a retentions, and approximately 1.38 and 1.5 E_a retentions, respectively. The post-curing might have been boosted by elevated temperatures in both E3 and E5; besides, the carbonation is inevitable in outdoor environments, hence the ingress of atmospheric CO_2

might have also participated in the post-curing, as it was found to increase the curing rate of the resin matrix in (Hu et al., 2018). After the post-curing phenomenon, a decreasing phase in the following consecutive years in E3, E5, E6 can be noted, which indicates that the rate the continuation of post-curing reduced with time whereas the effects of degradation agents became more dominant. For the case where there is a decrease in one year followed by an increase in the next year (e.g., the E_a in E4 from T1 to T3), this may depend on a number of factors. Taking example of E4 where the specimens were mainly exposed to natural outdoor freeze-thaw attacks, the observed fluctuations might indicate the possibility of effects of reversible reactions taking place or some physical phenomena such as plasticization mechanisms. These reactions (or mechanisms) can be inferred to have mainly depended on the type of exposure (freeze-thaw as the main exposure type and other exposures such as UV radiations and presence of some carbonation), exposure duration (e.g., how many months of freezing within a year), and exposure severity (e.g., how severe was the freezing). These three factors can vary from year to year, mainly due to the climate change effects, hence causing conditioning dissimilarity from year to year despite the specimens being in the same environment. The same thought was also reported in (Dushimimana et al., 2022). This can also show how accelerated aging test protocols might be somehow misleading, as such tests generally do not include the effects of the mentioned conditioning dissimilarity in the consecutive periods. However, the controlled laboratory environments (E1 and E2) also show some fluctuations from one year to the other. Therefore, the observed fluctuations may additionally result from other factors different from exposure type, severity, and duration. Examples of those factors may include the testing accuracy, measurement errors, and the standard deviations from year to year (see Table 2).

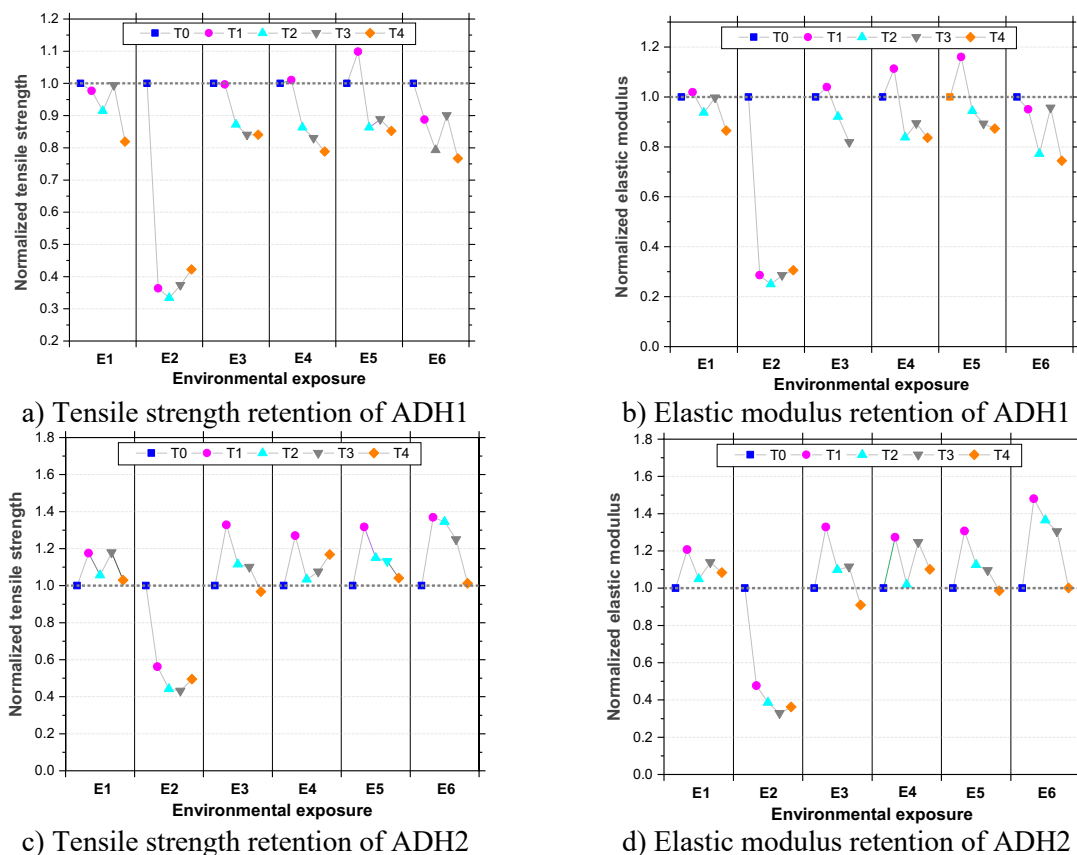


Figure 3: Epoxy adhesive test results

Overall, it can be noted that post-curing for both ADH1 and ADH2 is an important phenomenon. However, the post-curing of the former is minimal and hence the degradation agents are able to attack and degrade the ADH1 properties within a shorter period of exposure. In fact, only after T1, the degradation agents led to ADH1 f_a and E_a values lower than those at initial time (T0). Contrary, for the case of ADH2, the post-curing was substantial which positively imposed longer times for the

degradation agents to start degrading its properties (i.e., the f_a and E_a values after four years are generally still higher than those at T0).

Results from tensile tests of the CFRP laminates

The tensile strength (f_f) and elastic modulus (E_f) of the two studied CFRP laminates (L10 and L50) are shown in Figure 4 and Table 2. The results for each CFRP laminate are discussed as follows.

Results from tensile tests of the CFRP laminate L10

The f_f and E_f for the CFRP laminate L10 are presented in Figure 4a, and Figure 4b, respectively. The results show that the L10 did not have any degradation over four years for all types of environmental exposures, instead, there was a significant increase in both the f_f and E_f retentions within the first year (T1) of exposure, which can be attributed to the post-curing of the fibre matrix. In general, the retentions ranged between 1.0-1.15 and 1.0-1.10 for f_f and E_f , respectively. Like what was observed for the epoxy adhesives, post-curing of the CFRP resin matrix might have been the reason for the increased f_f and E_f observed in E1. After T1, a general decrease in both f_f and E_f in E1 can be noted, which can result from the diminished rate of post-curing, nevertheless, the f_f and E_f values were still higher than those recorded at the initial time (T0). In E2, since water immersion does not lead to detrimental effect (Cruz et al., 2021), the improved properties can be attributed to the post-curing of the epoxy resin; however, the fluctuations observed after T1 may result from the competing effects between the continuation of post-curing and the plasticization of the fibre resin matrix, nevertheless, the values after four years were also still higher than that at T0. The specimens in E3 shows the highest f_f increase within T1, which can be attributed to the combined effect of the high carbonation (known to fasten the curing of the resin matrix (Hu et al., 2018) and high temperatures (Grammatikos et al., 2016)). In E4, the rate of post-curing within the first year T1 was high, which significantly increased both the f_f and E_f . However, in the later years, the competing mechanisms between the continuation of post-curing (with lower rate than during T1) and the freeze-thaw attacks can be attributed to the decreasing trend in f_f and E_f . That is, the freeze-thaw attacks dominated the post-curing phenomenon (i.e., more microcracks at the fibre matrix level were formed due to freeze-thaws (Jiang et al., 2022)), which led to a progressive decrease in the f_f and E_f . In E5, high temperatures combined with the effects of carbonation continued to improve the E_f until the end of T3 thereafter showing an abrupt decrease in T4 (but still higher than the E_f at T0); the f_f showed increase until T2 thereby decreasing during T3 and then an increase again in T4. This decrease in one year followed by an increase in the following year can reveal the effect of exposure type, exposure severity, and exposure duration as previously explained for the case of the adhesives. In E6 both f_f and E_f increased during T1 as a result of high post-curing, which was followed by a progressive decrease during the next two years (T2 and T3) and the tendency to increase again in T4, this tendency being attributed to the effects of exposure duration and severity as previously explained. Existing studies show that exposure of CFRP to chlorides does not affect the CFRP properties (Cruz et al., 2021), hence it can be inferred that the observed decrease resulted from the post-curing which was further boosted by the presence of carbonation (i.e., as it has the ability to make the resin cure faster (Jiang et al., 2022)).

Results from tensile tests of the CFRP laminate L50

The f_f and E_f results for CFRP L50 are presented in Figure 4c,d. It can be seen that the CFRP laminate L50 had different trend than that of L10. This leads to the believe that the dimensioning of the CFRP used in outdoor applications might influence its degradation. Most importantly, the E_f for L50 showed a significant decrease in all environments, contrary to L10 that showed a significant increase. The f_f also followed the same trend as that of L10 with increase in T1; however, after T1, the f_f reduction rate for the L50 was much faster than that of L10. From this, it can be noted that the CFRP with smaller width may present higher durability features than that of larger width, particularly when considering the E_f .

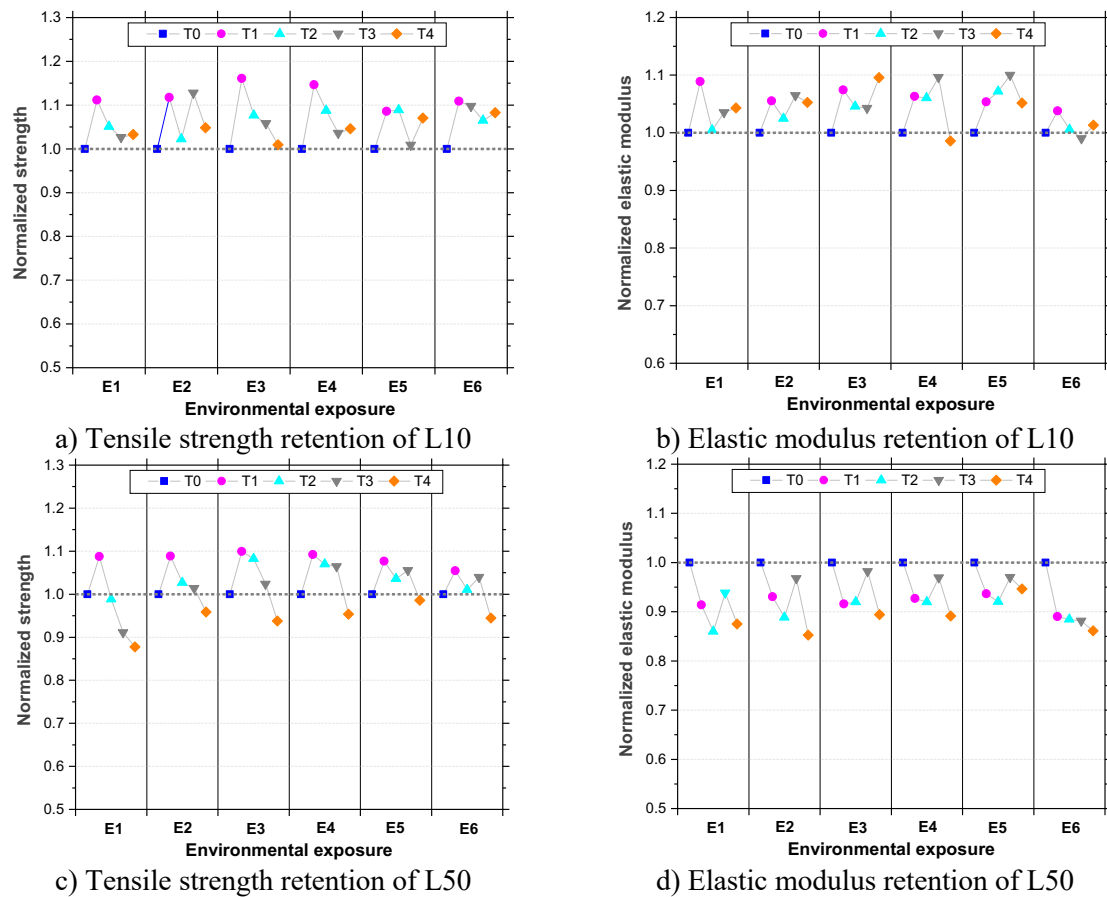


Figure 4: CFRP laminate test results

Overall, after 0 year of exposure, CFRP with smaller width (L10) showed significant post-curing effects, mainly because the agents promoting the post-curing were able to reach a significant region of the specimens, while for the CFRP with larger width (L50), the diffusion of the agents may have not reached significant regions, hence leading to minimal post-curing phenomenon. In the latter years, a general decrease in both CFRP f_f and E_f can be noted (except for L10 in E5 where post-curing seems to be ongoing thereby leading to further increase in both f_f and E_f), which can be attributed to the effects of degradation agents being more dominant than those of continuation of post-curing. The decrease from one year to another followed by an increase (or vice-versa) can be thought to result from unequal distributions of exposure duration and exposure severity from year to year. Comparing with the CFRP E_f and f_f recorded at the initial time (T0), the L10 specimens in E5 and E3 showed the highest CFRP E_f improvement with 10.4% (at T3) and 9.8% (at T4) increase, respectively, while those in E3 and E4 showed the highest CFRP f_f improvement with 16.1 % (at T1) and E4 with 14.6% (at T1) increase, respectively. On the other hand, the lowest L50 f_f retentions were approximately 0.86, 0.85 and 0.87 in E1, E2 and E6 respectively.

CONCLUSIONS

In this work, two different epoxy adhesives (with different properties) and two different CFRP laminates (with same properties but with different widths) were investigated after being exposed to different ageing environments (accelerated and natural ageing) for a period of four years. The testing times were of one year interval; hence four series of testing were performed, In addition to the initial testing at initial time. The key findings observed from these testing series in terms of the tensile strength and elastic modulus of both the adhesives and CFRP laminates are highlighted as follows.

1. One type of adhesive (ADH2) showed a substantial post-curing rate within the first year of exposure thereby leading to a significant increase in the adhesive f_a and E_a , and a progressive decrease in the later years until the fourth year of exposure. However, the f_a and E_a decreases were still higher than those from non-exposed specimens. This decrease in the latter years is thought to have resulted from the decrease of post-curing rate, which gave way to dominance of the degradation agents. On the other hand, another type of epoxy adhesive (ADH1) showed insignificant post-curing within the first year of exposure, which led to a substantial decrease in both f_a and E_a in the latter years with progressive decrease until the four years. Because of the marginal post-curing, lower f_a and E_a values were observed as compared to the values of non-exposed specimens. This shows that high post-curing can delay the degradation of adhesive properties. Both adhesives showed significant losses of f_a and E_a after being immersed in water, where one adhesive (ADH1) decreased its f_a and E_a by approximately 66% and 75%, respectively. In general, the adhesive E_a degraded faster than its f_a .
2. The CFRP laminate with smaller width (10 mm) significantly increased both f_f and E_f after environmental exposure, while that of larger width (50 mm) increased the f_f but with significant decreases in E_f . Hence, the width of CFRP laminate is a considerable parameter that can affect its durability performance, hence a careful selection of the CFRP laminate width is crucial. Furthermore, exposure of the CFRP with the 10 mm width to high temperatures and carbonation led to significant post-curing, thereby resulting in the highest improvements in E_f of approximately 10.4% and 9.8%, respectively. Furthermore, carbonation and freeze-thaw attacks led to the highest improvement of f_f with approximately 16% and 15% respectively. Overall, the post-curing of the fibre matrix can increase the f_f and E_f of smaller CFRP laminate width while CFRP laminate with larger width can still benefit from the post-curing by increasing the f_f but with little or no benefits on their E_f .

Table 2: Mean values of tensile strength of epoxy adhesives and CFRP laminates after 0 (T0), 1 (T1), 2 (T2), 3 (T3), and 4 (T4) years of different environmental exposures (E1 to E6)

Environment	T0	T1	T2	T3	T4	T0	T1	T2	T3	T4
	ADH1: Tensile strength [MPa]					ADH2: Tensile strength [MPa]				
REF	19.9 (3.0)					24.8 (7.0)				
E1	-	19.5 (1.8)	18.2 (2.8)	19.8 (4.9)	16.3 (14.6)	-	29.2 (3.8)	26.2 (5.7)	29.3 (4.6)	25.6 (10.7)
E2	-	7.2 (3.1)	6.7 (2.7)	7.4 (7.1)	8.4 (3.6)	-	14.0 (2.5)	11.0 (7.6)	10.7 (4.5)	12.3 (10.6)
E3	-	19.9 (3.1)	17.4 (5.3)	16.7 (5.9)	-	-	33.0 (3.6)	27.7 (5.8)	27.4 (4.3)	24.0 (12.5)
E4	-	20.1 (3.4)	17.2 (4.3)	16.5 (9.8)	15.7 (20.1)	-	31.5 (1.8)	25.7 (5.7)	26.7 (11.0)	29.0 (3.6)
E5	-	21.9 (5.2)	18.0 (3.6)	17.7 (6.5)	17.0 (5.8)	-	32.7 (4.6)	28.6 (4.0)	28.1 (5.6)	25.9 (6.1)
E6	-	17.7 (6.4)	15.8 (4.3)	18.0 (4.2)	15.3 (2.6)	-	34.0 (3.8)	33.4 (3.8)	31.0 (5.7)	25.1 (9.1)
Environment	L10: Tensile strength [MPa]					L50: Tensile strength [MPa]				
REF	2405 (3.8)					2527 (11)				
E1	-	2674 (2.72)	2528 (4.4)	2469 (6.4)	2484 (3.0)	-	2748 (2.6)	2497 (1.7)	2302 (3.9)	2217 (5.7)
E2	-	2688 (3.4)	2460 (7.1)	2713 (4.5)	2522 (7.1)	-	2750 (2.0)	2594 (2.8)	2562 (3.2)	2422 (5.6)
E3	-	2792 (3.7)	2590 (5.4)	2546 (5.1)	2427 (6.8)	-	2778 (2.1)	2735 (1.8)	2587 (3.6)	2369 (3.6)
E4	-	2757 (2.9)	2617 (4.5)	2492 (5.0)	2516 (3.9)	-	2760 (2.5)	2703 (3.4)	2690 (2.9)	2409 (3.5)
E5	-	2611 (5.0)	2619 (5.3)	2427 (4.1)	2575 (3.9)	-	2720 (3.9)	2618 (3.6)	2667 (4.6)	2491 (8.1)
E6	-	2667 (3.0)	2640 (2.9)	2561 (2.8)	2605 (5.1)	-	2665 (2.2)	2554 (4.6)	2626 (1.3)	2386 (2.6)

Note: all values in parentheses express *coefficient of variation* in percentage; L10: CFRP with a 1.4 mm × 10 mm cross section; L50: CFRP with a 50 mm × 1.2 mm cross section (tested coupons of 15 mm × 1.2 mm); REF: Reference values from the specimens tested at the beginning i.e., at T0.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest associated with the work presented in this paper.

DATA AVAILABILITY

Data on which this paper is based is available from the authors upon reasonable request.

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