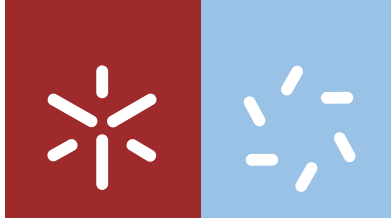


Universidade do Minho
Escola de Ciências

Sílvia Patrícia Gonçalves dos Santos **Characterization of metallized polypropylene films**

Sílvia Patrícia Gonçalves dos Santos

**Characterization of metallized
polypropylene films**



Universidade do Minho
Escola de Ciências

Sílvia Patrícia Gonçalves dos Santos

Characterization of metallized polypropylene films

Master thesis:
Master in Characterization Techniques and Chemical Analysis

Under orientation of:
Professor Doctor Maria Manuela Silva
Engineer José Aurélio Campos

March 2021

DIREITOS DE AUTOR E CONDIÇÕES DE UTILIZAÇÃO DO TRABALHO POR TERCEIROS

Este é um trabalho académico que pode ser utilizado por terceiros desde que respeitadas as regras e boas práticas internacionalmente aceites, no que concerne aos direitos de autor e direitos conexos.

Assim, o presente trabalho pode ser utilizado nos termos previstos na licença abaixo indicada.

Caso o utilizador necessite de permissão para poder fazer um uso do trabalho em condições não previstas no licenciamento indicado, deverá contactar o autor, através do RepositóriUM da Universidade do Minho.

Licença concedida aos utilizadores deste trabalho



Atribuição-NãoComercial-SemDerivações
CC BY-NC-ND

<https://creativecommons.org/licenses/by-nc-nd/4.0/>

ACKNOWLEDGMENT

My acknowledgment to all who gave me positive energy to overcome this challenge and never give up. All are important for me!

To my parents Rosário e Pedro, who gave me strength to believe all these years that it could be possible.

To my husband Carlos, who was always here, for all his patient and love. My daughter Inês and my son Simão, my life!

To my brother João Pedro and his wife Joana, even far from here, in France, always sending me comforting messages.

My all family and friends, for their patience and good times during this hard and strange year.

My teacher, Professor Doctor Maria Manuela Silva for being my supervisor, always available and very cooperating.

To Engineer José Aurélio Campos who gave me this opportunity, to never deny me a lesson or a meeting, always present and specially to believe in me.

To Professor Doctor Susana Costa and Dr. Daniela Carvalhal, always available to explain me all questions and doubts concerning the master.

To Vishay Electrónica Portugal, Lda which gave me the opportunity to make the master thesis. Thank you to Dr. Vítor Morais and Dr. Cláudia Cruz, Engineer Vítor Araújo, Engineer Geraldo Silva, Engineer José Lagido, Engineer Geert Stevens, Doctor Atul Killedar and Engineer Marc Vanoosthuyse.

To my colleagues from Process Engineer and Research & Development departments, as well as, Production, Quality, Logistics and Maintenance departments.

To all Vishay Electrónica Portugal team.

Thank you.

STATEMENT OF INTEGRITY

I hereby declare having conducted this academic work with integrity. I confirm that I have not used plagiarism or any form of undue use of information or falsification of results along the process leading to its elaboration. I further declare that I have fully acknowledged the Code of Ethical Conduct of the University of Minho.

Characterization of metallized polypropylene films

ABSTRACT

The main goal of a capacitor is to store energy and release it in a circuit when needed. There are in the market several types of capacitors, as film capacitors, ceramic capacitors and electrolytic capacitors. For this study, it will be presented a metallized film capacitor.

Film capacitors are electronic components with a high importance in a lot of electronic devices. They are used in different applications as, for instance, in televisions, radios, automotive industry, renewable energy devices, like solar panels and in hybrid and electrical cars. Different materials and components can be part of a film capacitor, as a plastic case, an epoxy, a thin plastic film metallized with metal alloys, solder, wires, and terminals.

The focus of this work is the body of the film capacitor, which is the plastic film, metallized with a very thin metal layer.

The intend of this work was to study six different polypropylene grades, from several suppliers, using different characterization techniques, as Differential Scanning Calorimetry, Fourier Transform Infrared Spectroscopy, Atomic Force Microscopy and Scanning Electron Microscopy. These techniques allow a better understanding of the behaviour and performance of each material, throughout the life of a film capacitor and prevent the failures during its life cycle. Thus, they had shown that there are some properties that influence the behaviour of each sample, as for instance, the surface roughness, the BOPP fabrication process (sequential stretching *versus* simultaneous stretching) and thickness, which influence crystals grains and the polymer chain orientation, that in turn, influence the crystallinity.

All these techniques gave an important contribute for the understanding of the different polypropylene samples, concluding that sample 5 seems to be the best material to be used in a film capacitor, due to its high crystallinity, constant roughness, high Young Modulus and low shrinkage.

Keywords: BOPP, film capacitors, metallized film capacitor, polypropylene.

Caracterização de bases de fita de polipropileno metalizadas

RESUMO

O principal objetivo de um condensador é armazenar energia e libertá-la num circuito quando for necessário. Existem no mercado vários tipos de condensadores, como condensadores de filme, condensadores cerâmicos e condensadores eletrolíticos. Para este estudo, será apresentado um condensador de filme metalizado.

Os condensadores de filme são componentes eletrônicos de grande importância em muitos dispositivos eletrônicos. São usados em diferentes aplicações como, por exemplo, em televisores, rádios, indústria automóvel, dispositivos de energia renovável, como painéis solares e em carros híbridos e elétricos. Diferentes materiais e componentes podem fazer parte de um condensador de filme, como uma caixa de plástico, um epóxi, um fino filme plástico metalizado com ligas metálicas, soldas, fios e terminais.

O foco deste trabalho é o corpo do condensador de filme, que é o filme plástico, metalizado com uma camada de metal muito fina.

O objetivo deste trabalho foi estudar seis amostras de polipropileno, de diversos fornecedores, utilizando diferentes técnicas de caracterização, como Calorimetria Diferencial de Varrimento, Espectroscopia de Infravermelho por Transformada de Fourier, Microscopia de Força Atômica e Microscopia Eletrônica de Varrimento. Estas técnicas permitem uma melhor compreensão do comportamento e desempenho de cada material, ao longo da vida de um condensador de filme e evitam falhas durante o seu ciclo de vida. Assim, elas mostraram que existem algumas propriedades que influenciam o comportamento de cada amostra, como por exemplo, a rugosidade superficial, o processo de fabricação do BOPP (estiramento sequencial *versus* estiramento simultâneo) e a espessura que influenciam os grãos dos cristais e a orientação da cadeia polimérica, que por sua vez, influenciam a cristalinidade. Todas estas técnicas deram um importante contributo para a compreensão das diferentes amostras de polipropileno, concluindo que a amostra 5 parece ser o melhor material para ser utilizado num condensador de filme, devido à sua elevada cristalinidade, rugosidade constante, elevado Módulo de Young e baixo encolhimento.

Palavras-chave: BOPP, condensador de filme metalizado, condensadores de filme, polipropileno.

CONTENT

ABSTRACT	v
RESUMO.....	vi
FIGURES CONTENT	ix
TABLE CONTENT	xii
ABBREVIATION LIST	xiv
CHAPTER 1- INTRODUCTION	1
1. Introduction	1
1.1. The company: Vishay 's history	1
1.2. Scope	1
1.3. Thesis organization	2
CHAPTER 2- STATE OF ART	3
2. State of art.....	3
2.1. Film Capacitor	3
2.2. Materials.....	7
2.2.1. Polypropylene film	8
2.2.2. iPP as a dielectric film.....	11
2.3. Characterization techniques	15
2.3.1. Differential Scanning Calorimetry.....	16
2.3.2. Fourier Transform Infrared Spectroscopy	17
2.3.3. Scanning Electron Microscopy.....	18
2.3.4. Atomic Force Microscopy	19
2.4. Mechanical tests.....	20
CHAPTER 3- MATERIALS AND METHODS.....	23
3. Materials and methods	23
3.1. Metallized PP film samples.....	23
3.2. Characterization Techniques	23
3.2.1. Differential Scanning Calorimetry analysis	23
3.2.2. Fourier Transform Infrared Spectroscopy analysis.....	25
3.2.3. Scanning Electron Microscopy and Energy Dispersive Spectroscopy analysis.....	26

3.2.4.	Atomic Force Microscopy analysis	26
3.2.5.	Mechanical tests analysis.....	27
3.2.6.	Film Shrinkage.....	28
CHAPTER 4- DISCUSSION AND RESULTS		29
4.	Discussion and results	29
4.1.	Differential Scanning Calorimetry.....	29
4.2.	Fourier Transform Infrared Spectroscopy.....	36
4.3.	Scanning Electron Microscopy.....	38
4.4.	Atomic Force Microscopy	53
4.5.	Mechanical tests	62
4.6.	Shrinkage	71
CHAPTER 5- CONCLUSION		73
5.	Conclusion	73
CHAPTER 6- FUTURE WORK		75
6.	Future work	75
BIBLIOGRAPHY		76
ANNEX I.....		80
ANNEX II.....		83
ANNEX III.....		86

FIGURES CONTENT

Figure 1- Vishay Electrónica Portugal, Lda [7]	1
Figure 2- Overview of the thesis chapters	2
Figure 3- Scheme of a film capacitor [9]	3
Figure 4- Steps for a film capacitor manufacturing [10]	4
Figure 5- Scheme of a film capacitor[10]	5
Figure 6- Failure modes, causes, effects and consequences which can occur in a metallized film capacitor [14]	6
Figure 7- Metallized film with electrode corrosion due to the presence of moisture [14].....	7
Figure 8- Metallized film which was demetallized due to corona arcing [14]	7
Figure 9- View of materials used in a film capacitor [15].....	8
Figure 10- Scheme of a metallized PP film [16].....	8
Figure 11- Classification of Polymers [17]	9
Figure 12- PP structure[18]	9
Figure 13- Polymerization of propene monomer by Ziegler-Natta Polymerization [18]	10
Figure 14- Tacticity of PP [21]	10
Figure 15- Semi-crystalline regions [24]	11
Figure 16- Tenter frame process to produce BOPP[28]	12
Figure 17- Diagram for a semi-crystalline polymer, relation between pressure, volume and temperature[30]	13
Figure 18- Influence of the orientation in the dielectric strength[33]	14
Figure 19- Influence of the shrinkage (specific volume versus temperature with no pressure) [30].....	14
Figure 20- Hazy film: Open fibrillar structure on the film surface (General electric)[33].....	15
Figure 21-DSC response of an organic polymer [30]	16
Figure 22- Infrared spectrum of n-heptane, by ATR- FTIR [41]	17
Figure 23- IR regions and respectively functional groups positions[42].....	18
Figure 24- Electromagnetic spectrum [47]	19
Figure 25- AFM scheme [50]	19
Figure 26- Example of a Ra and Rq measurement [51]	20
Figure 27- Stress- strain curve of a material [53].....	21

Figure 28- Examples of stress-strain curves for different polymers [30]	22
Figure 29- Metallized polypropylene film samples.....	23
Figure 30- PerkinElmer, model DSC 6000	24
Figure 31- Weight of a BOPP sample	24
Figure 32- PerkinElmer ATR-FTIR, model Frontier.....	25
Figure 33- JSM-6010PLUS/LA InTouchScope™ Multiple touch panel scanning electron microscope [54]	26
Figure 34- Identification of the three areas (A/B/C) used for the SEM-EDS analysis	26
Figure 35- Identification of the three areas (A/B/C), used for the surface roughness analysis	27
Figure 36- Section of the sample used to perform the mechanical tests.....	28
Figure 37- Thermograms of all Vishay BOPP samples, obtained with the program 1.	29
Figure 38- DSC result for sample 1, Vishay code nr. 80920622, using program 1	30
Figure 39- Percentage of crystallinity, using the program 1	31
Figure 40- DSC results from all Vishay BOPP samples, obtained with the program 2.....	32
Figure 41- DSC result for sample 1, Vishay code nr. 80920622, using program 2	32
Figure 42- Percentage of crystallinity, using the program 2.....	34
Figure 43- ATR-FTIR spectrum: overview of all BOPP samples	36
Figure 44- ATR-FTIR spectrum: examples of the comparison between two samples.....	37
Figure 45- Spectrum of a BOPP sample (sample 1).....	38
Figure 46- SEM- EDS images obtained in the Sample 1 – areas (A) and (C).	39
Figure 47- SEM- EDS images obtained in the Sample 1 - area (B).	40
Figure 48- SEM- EDS images obtained in the Sample 2 – areas (A) and (C).	41
Figure 49- SEM- EDS images obtained in the Sample 2 - area (B).....	42
Figure 50- SEM- EDS images obtained in the Sample 3 – areas (A) and (C)	43
Figure 51- SEM- EDS images obtained in the Sample 3 – area (B).....	44
Figure 52- SEM- EDS images obtained in the Sample 4 – areas (A) and (C)	45
Figure 53- SEM- EDS images obtained in the Sample 4 -area (B).....	46
Figure 54- SEM- EDS images obtained in the Sample 5 -areas (A) and (C)	47
Figure 55- SEM- EDS images obtained in the Sample 5 area (B).....	48
Figure 56- SEM- EDS images obtained in the Sample 6 -areas (A) and (C)	49
Figure 57- SEM- EDS images obtained in the Sample 6 -area (B).....	50

Figure 58- Optical AFM topography images and surface roughness of the sample 1, into three different areas: (A), (B) and (C). Image size: 5×5 μm	53
Figure 59- Optical AFM topography images and surface roughness of the sample 2, into three different areas: (A), (B) and (C). Image size: 5×5 μm	54
Figure 60 - Optical AFM topography images and surface roughness of the sample 3, into three different areas: (A), (B) and (C). Image size: 5×5 μm	55
Figure 61- Optical AFM topography images and surface roughness of the sample 4, into three different areas: (A), (B) and (C). Image size: 5×5 μm	56
Figure 62- Optical AFM topography images and surface roughness of the sample 5, into three different areas: (A), (B) and (C). Image size: 5×5 μm	57
Figure 63- Optical AFM topography images and surface roughness of the sample 6, into three different areas: (A), (B) and (C). Image size: 5×5 μm	58
Figure 64- Roughness surface: individual Rq values per sample and per area	59
Figure 65- Roughness surface: overview of Rq values per sample and per area	59
Figure 66- Roughness surface: individual Ra values per sample and per area	60
Figure 67- Roughness surface: overview of Ra values per sample and per area.....	60
Figure 68- Roughness surface: individual Rmax values per sample and per area.....	61
Figure 69- Roughness surface: overview of Rmax values per sample and per area	61
Figure 70- Tensile strain vs tensile stress graph for specimen of sample 1.....	63
Figure 71- Tensile strain vs tensile stress graph for specimen of sample 2.....	64
Figure 72- Tensile strain vs tensile stress graph for specimen of sample 3.....	65
Figure 73- Tensile strain vs tensile stress graph for specimen of sample 4.....	66
Figure 74- Tensile strain vs tensile stress graph for specimen of sample 5.....	67
Figure 75- Tensile strain vs tensile stress graph for specimen of sample 6.....	68
Figure 76- Overview of all characteristics performed in mechanical tests. per sample.....	69
Figure 77- Young Modulus obtained per sample.....	69
Figure 78- Tensile strength obtained per sample	70
Figure 79- Elongation at Break obtained per sample.....	70
Figure 80- Shrinkage overview, of each sample at different temperatures	72

TABLE CONTENT

Table 1- Example of materials used in a film capacitor	7
Table 2- Typical properties of oriented PP films [31].....	13
Table 3- Samples identification	23
Table 4- Program 1 used in the BOPP samples	25
Table 5- Program 2 used in the BOPP samples	25
Table 6- First run for the melting and crystallization, for the program 1, for each sample	30
Table 7- Second run for the melting and crystallization, for the program 1, for each sample...	31
Table 8- Crystallinity results for the first and second run during the heating	31
Table 9- First run for the melting and crystallization, for the program 2, for each sample	33
Table 10- Second run for the melting and crystallization, for the program 2, for each sample.	33
Table 11- Crystallinity results for the first and second run during the heating	34
Table 12- Identification and quantification of each element present in sample 1	40
Table 13- Identification and quantification of each element present in sample 2	42
Table 14- Identification and quantification of each element present in sample 3	44
Table 15- Identification and quantification of each element present in sample 4	46
Table 16- Identification and quantification of each element present in sample 5	48
Table 17- Identification and quantification of each element present in sample 5	50
Table 18- Overview of EDS Identification and quantification for each sample in graphs.....	52
Table 19- Surface roughness of the sample 1, into three different areas (A), (B) and (C)	53
Table 20- Surface roughness of the sample 2, into three different areas (A), (B) and (C)	54
Table 21- Surface roughness of the sample 3, into three different areas (A), (B) and (C)	55
Table 22- Surface roughness of the sample 4, into three different areas (A), (B) and (C)	56
Table 23- Surface roughness of the sample 5, into three different areas: (A), (B) and (C)	57
Table 24- Surface roughness of the sample 6, into three different areas (A), (B) and (C)	58
Table 25- Mechanical results obtained for sample 1	62
Table 26- Mechanical results obtained for sample 2.....	63
Table 27- Mechanical results obtained for sample 3.....	64
Table 28- Mechanical results obtained for sample 4.....	65
Table 29- Mechanical results obtained for sample 5.....	66
Table 30- Mechanical results obtained for sample 6.....	67

Table 31- Average (x) and standard deviation (S) overview for each sample	68
Table 32- Measurement, in percentage, of the length of each film	71

ABREVIATION LIST

A	Area
Al	Aluminium element
AFM	Atomic Force Microscopy
aPP	Atactic polypropylene
ASTM	American Society for Testing and Materials
ATR-FTIR	Attenuated Total Reflectance-Fourier Infrared Spectroscopy
BOPP	Biaxially oriented polypropylene
%C	Percentage of crystallinity
<i>D</i>	Distance
DSC	Differential Scanning Calorimetry
E	Young Modulus
EDS	Energy Dispersive Spectroscopy
EDX	Energy Dispersive X-Ray Spectroscopy
ε	Strain
ε_0	Vacuum permittivity
ε_r	Dielectric constant
ESR	Equivalent series resistance
<i>F</i>	Frequency
FTIR	Fourier Transformed Infrared Spectroscopy
ΔH_f	Enthalpy of fusion
HDPE	High density polyethylene
iPP	Isotactic polypropylene
ISO	International Standards Organization
IR or RIS	Insulation resistance
IUPAC	International Union of Pure and Applied Chemistry
LDPE	Low density polyethylene
MD	Machine direction
MDO	Machine direction orienter
MKP	Metallized Polypropylene film
MKT	Metallized Polyester film
MPa	Mega Pascal
PBT	Polybutylene terephthalate
PET	Polyethylene terephthalate
PP	Polypropylene
PPS	Polyphenylene sulfide
PS	Polystyrene
Ra	Roughness average
RIS	Resistance insulation
RMS or Rq	Root mean square
<i>s</i>	Standard deviation
SE	Secondary electron
SEM	Scanning Electron Microscopy
SnZn	Alloy of Tin and Zinc

SnCu	Alloy of Tin and Copper
sPP	Syndiotactic polypropylene
$\tan \delta$	Dissipation factor
TD	Transverse direction
TDO	Transverse direction orienter
T _g	Glass transition temperature
U	Voltage
$\bar{x}$	Average
X _c	Capacitive reactance
Zn	Zinc element
ZnAl alloy	Alloy of Zinc and aluminium
σ	Stress
%	Percentage

“To Dream

To Dare

To Do “

(By Dr. Felix Zandman)

CHAPTER 1- INTRODUCTION

1. Introduction

1.1. The company: Vishay 's history

Vishay Intertechnology, Inc. is a worldwide company which was growing through acquisitions of some companies as Roederstein, Dale, BCComponents and General semiconductor [1].

Discrete semiconductors and passive components represents the Vishay 's portfolio brands [2].

Its founder was Dr. Felix Zandman, who was born in 1928 and passed away in 2011. Doctorated in Mechanical Physics by University of Paris in 1953, he had a very important portfolio in the electronic industry [3].

Dr. Felix Zandman was a survivor of Holocaust horror. An example of strength, struggle, persistence, work and hope. His experience is described in his book "Never the Last Journey" [4].

Vishay Electrónica Portugal, Lda, is located in Portugal, near Vila Nova de Famalicão, in Calendário. It belongs to Vishay Intertechnology, Inc [5].

Roederstein was acquired by Vishay Intertechnology in 1993 and since that time it is Vishay Electrónica Portugal, Lda.

In 2019, Vishay Intertechnology celebrated the fiftieth anniversary. Vishay Electrónica Portugal, Lda celebrated the historical event in September 2019 [6].

In Portugal, Figure 1, it has around 200 workers and it produces film capacitors to electronic equipment's, as solar panels energy, televisions, radios and automotive industry. Vishay is growing and getting stronger and very competitive in the electric and hybrids automotive industry.



Figure 1- Vishay Electrónica Portugal, Lda [7]

1.2. Scope

The main goal of this work was to study and understand the different polypropylene (PP) films that are used in film capacitors.

To evaluate different suppliers and grades, different techniques, as Differential Scanning Calorimetry (DSC), Fourier Transformed Infrared Spectroscopy (FTIR), Scanning Electron Microscopy

(SEM), Atomic Force Microscopy (AFM) and mechanical measurements were applied. Furthermore, the knowledge of chemical and physics polypropylene properties was important too in this study.

1.3. Thesis organization

The thesis is divided in 6 chapters, as presented in the Figure 2.

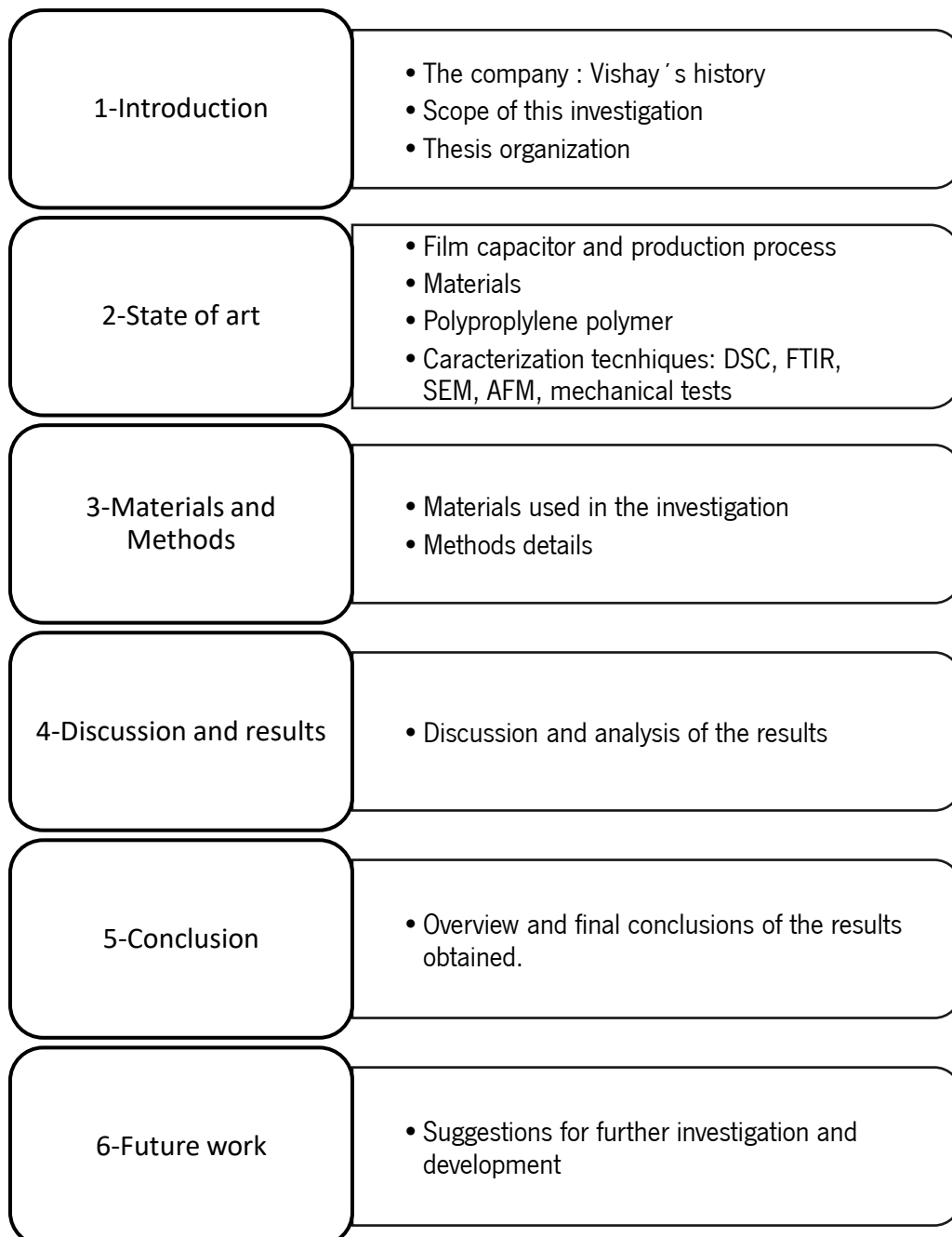


Figure 2- Overview of the thesis chapters

CHAPTER 2- STATE OF ART

2. State of art

2.1. Film Capacitor

A film capacitor is a passive component, meaning that it cannot introduce power into a circuit or either amplifying the flow of the electrical current or either capable of electrically controlling. It is able to store energy and release it in the circuit when needed[8].

The Figure 3 shows a scheme of a film capacitor. A capacitor consists in two electrodes (position 1) working as two conductive plates, separated by the dielectric (position 2), the insulating material. The distance between the electrodes is given by position 3.

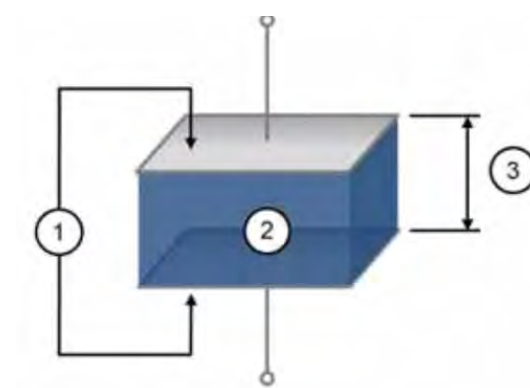


Figure 3- Scheme of a film capacitor [9]

For a better understanding of a metallized film capacitor, it will be described the steps involved to produce it.

The Figure 4 demonstrates the steps for manufacturing a film capacitor.



Figure 4- Steps for a film capacitor manufacturing [10]

The first step is the metallizing, where the metal layer is deposited in one side of the plastic film, under a vacuum deposition system. The result is a metallized film called as dielectric. After that, the metallized mother roll is cutted in several parts (film slitting) and packaged to be winding later on, in the same factory or to be sold.

Vishay Electrónica Portugal buys the metallized film from several suppliers.

The third step is the winding and here starts the production in Vishay Electrónica Portugal. They are winded the right and left metallized rolls with a high-speed machine. The product at this stage is called as cell.

Cells which are cylindric, are going to be flattened, in the flattening. After that, they will be involved in a masking paper and the sides (contact layer) are going to be sprayed with a metal layer, "schoopage".

Then, cells are tested in a machine, the healing step, to select the good ones and separate the cells with defects, as burnings. Some types, go to the impregnation, which is defined as giving a bath in a silicon oil tank. A wire or a terminal will be soldered in the contact layer (terminal welding step), before being casted with an epoxy, inside a box. Finally, the capacitor makes the electrical end test,

where it is tested in a specific electrical equipment which measures, for example, the capacitance and the dissipation factor.

Figure 5, presents a scheme of a film capacitor, after its manufacturing.

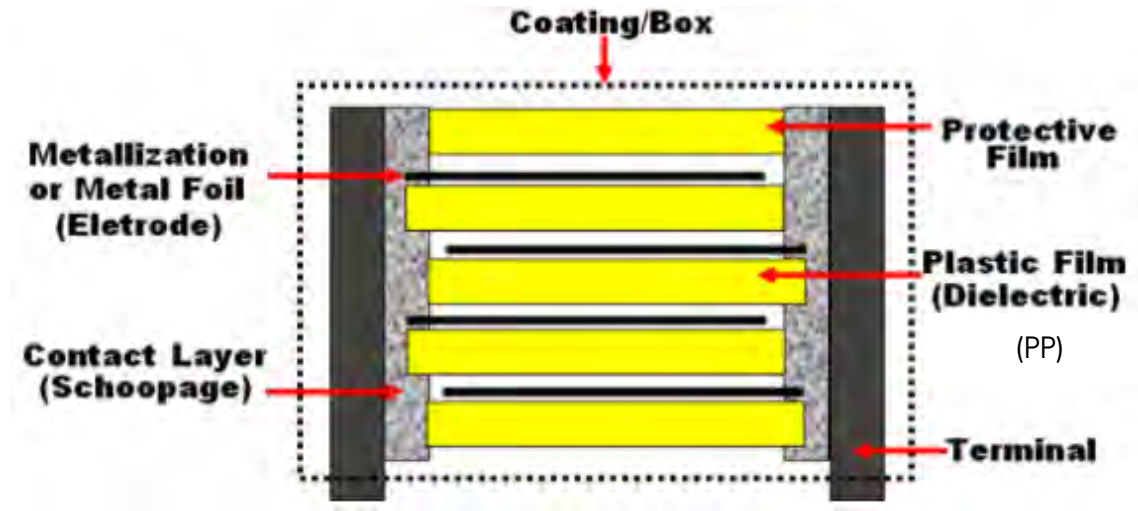


Figure 5- Scheme of a film capacitor[10]

Electrical tests are important to evaluate the good conditions of a capacitor. There are three important characteristics which are measured at the final production of a capacitor: the capacitance (C), the tangent delta ($\tan \delta$) and the insulation resistance (IR or RIS).

The capacitance is the capacity to store electric charges. It is expressed in Farad (F) and its value is proportional to the dielectric constant of the material, ϵ_r , to vacuum permittivity, ϵ_0 , and electrodes area, A , and inversely proportional to the distance between the electrodes, d , as expressed in Equation 1[11].

$$C = \frac{\epsilon_r \epsilon_0 A}{d} \quad \text{Equation 1}$$

The dissipation factor or tangent delta, $\tan \delta$, expressed in percentage (%) depends of the equivalent series resistance (ESR) and the capacitive reactance, X_c (Equation 2).

$$\tan \delta = \frac{ESR}{X_c} \quad \text{Equation 2}$$

The X_c , in ohms, is the impedance of the capacitor and it is inversely proportional to the capacitance and frequency, f . It is defined as the inertia against the flow of the current and it is given by Equation 3 [12], [11].

$$X_c = \frac{1}{2\pi f C} \quad \text{Equation 3}$$

The lower the ESR, the lower the tangent delta, the better the capacitor and the lower the losses. The value indicates losses of the resistance between the metallized film, metal spray layer and terminals, as well as, losses of the polarization in the dielectric[13].

The insulation resistance (IR), expressed in ohms (Ω) is related to the applied voltage, U , and the leakage current, I , given by Equation 4.

$$R_{IS} = \frac{U}{I} \quad \text{Equation 4}$$

An increase in I leads to a decrease in the IR, therefore, more heat is produced and the more the temperature increases leading in possible failures, as melting of the dielectric [14].

During the manufacturing and the lifetime of a metallized film capacitor, there are some failures modes which can occur, as shown in Figure 6.

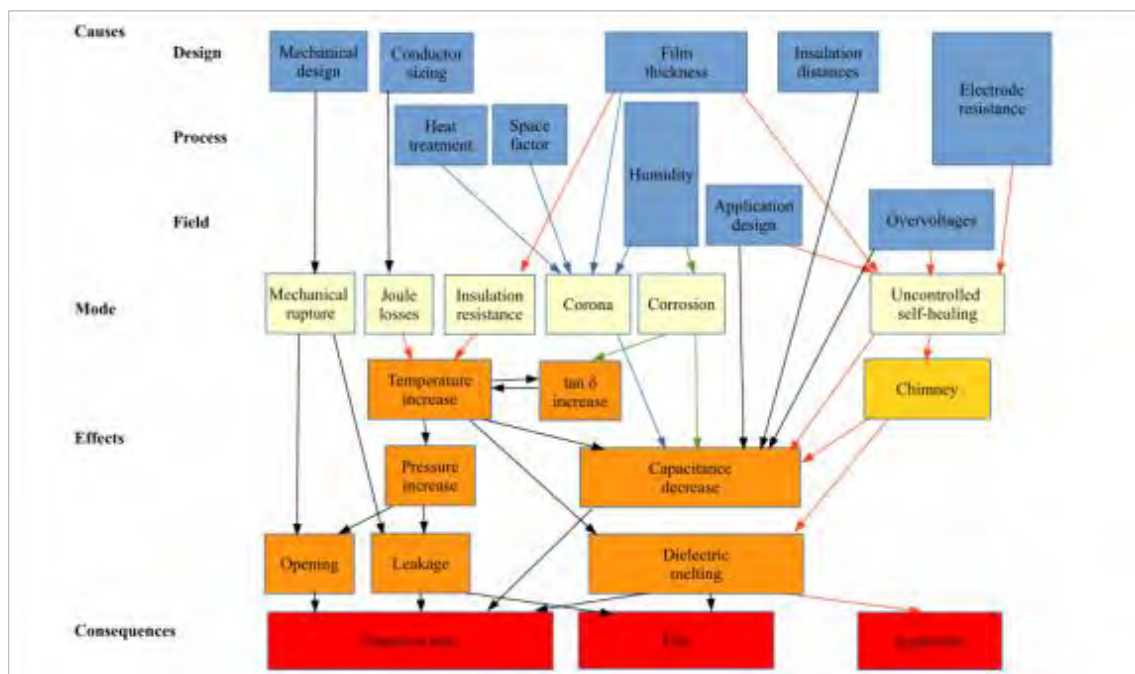


Figure 6- Failure modes, causes, effects and consequences which can occur in a metallized film capacitor [14]

Figures 7 and 8 show some failures in the metallized film, which can damage a metallized film capacitor.



Figure 7- Metallized film with electrode corrosion due to the presence of moisture [14]



Figure 8- Metallized film which was demetallized due to corona arcing [14]

2.2. Materials

Materials are selected according to the application of the film capacitor. The Table 1 shows some of the materials used to produce film capacitors.

Table 1- Example of materials used in a film capacitor

Material	To be used in
PP (or PET) metallized with ZnAl / Al	Cell
Cu tinned	Terminal or wire
ZnAl/ Al/SnZn/SnCu Alloy	Metal spray layer
PBT (or PPS) with glass fiber	Case
Epoxy resin/ Polyurethane	Resin

PP- Polypropylene; PET- Polyethylene Terephthalate; ZnAl- Zinc Aluminium; Al- Aluminium; Cu- Copper; SnZn- Tin Zinc; SnCu- Tin Copper; PBT- Polybutylene Terephthalate; PPS- Polyphenylene Sulfide.

The Figure 9, represents a typical internal construction of a film capacitor.

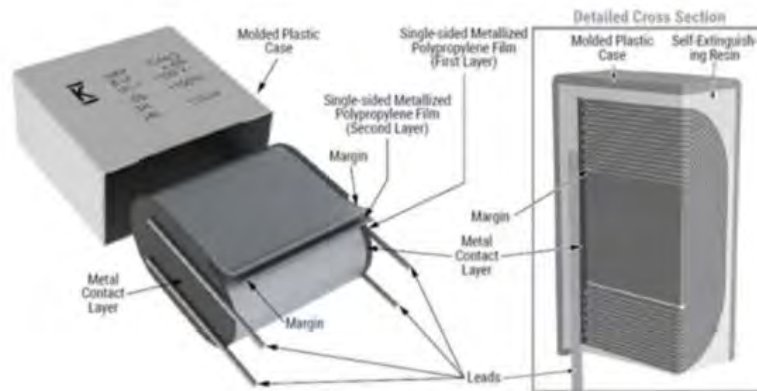


Figure 9- View of materials used in a film capacitor [15]

For this study the main polymer studied is the PP, metallized with a very thin metal layer, which is used in the cell, as shown in the Figure 10.

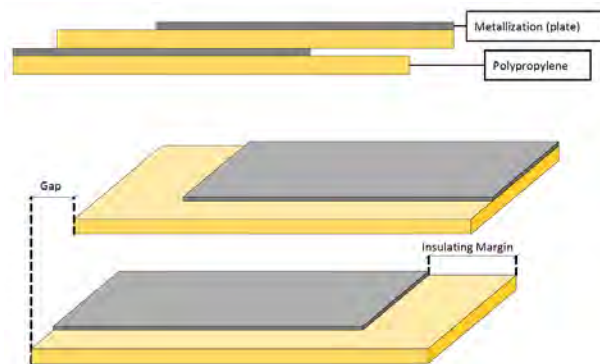


Figure 10- Scheme of a metallized PP film [16]

2.2.1. Polypropylene film

There are in the market a lot of polymers, normally they are classified according three groups, as Figure 11 [17].

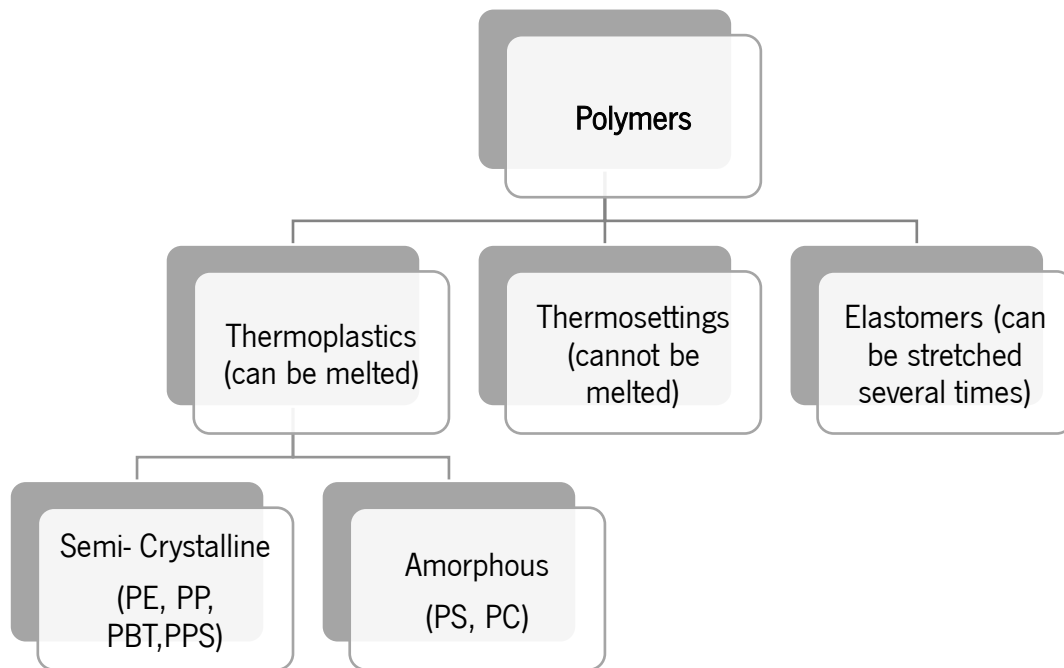


Figure 11- Classification of Polymers [17]

PP is a semi-crystalline thermoplastic and it is the most widely polymer used, from consumer foods to the automotive industry, due to its attractive price, as well as, its physical, mechanical and chemical properties. It belongs to the polyolefin family, as its molecular structure is carbon and hydrogen (hydrocarbon chain), as presented in the Figure 12.

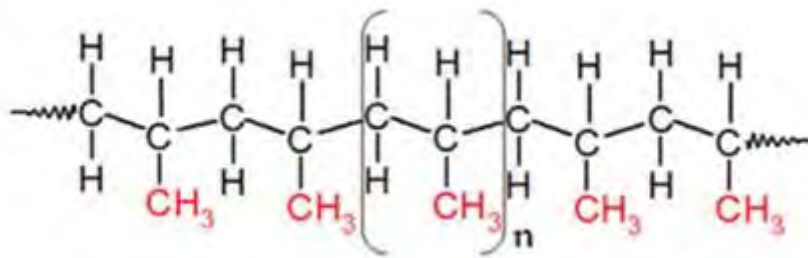


Figure 12- PP structure[18]

The polymerization of the propene monomer by Ziegler-Natta polymerization, for example, allows the production of the PP, as Figure 13 [19] .

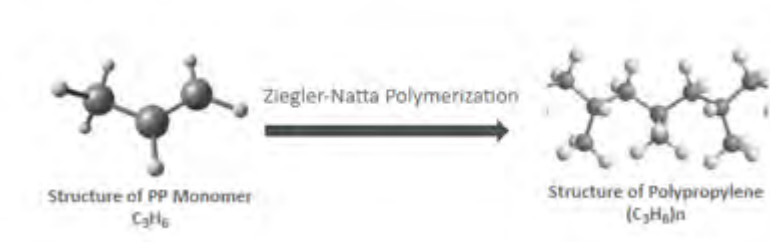


Figure 13- Polymerization of propene monomer by Ziegler-Natta Polymerization [18]

When polymerized, as propene is an asymmetrical molecule, can form three basic chain structures, called as syndiotactic polypropylene (sPP); atactic polypropylene (aPP) or isotactic polypropylene (iPP), depending on the position of the methyl groups (CH_3) on every second carbon atom, as demonstrated in Figure 14, named as tacticity.

Tacticity can affect the physical behaviour of a polymer chain, as well as, the ability of the polymer molecules to crystallize[20]. The iPP has got all methyl groups on one side of the carbon chain; in sPP, the methyl group, has got an alternating arrangement and in aPP, the methyl group, has got an irregular arrangement.

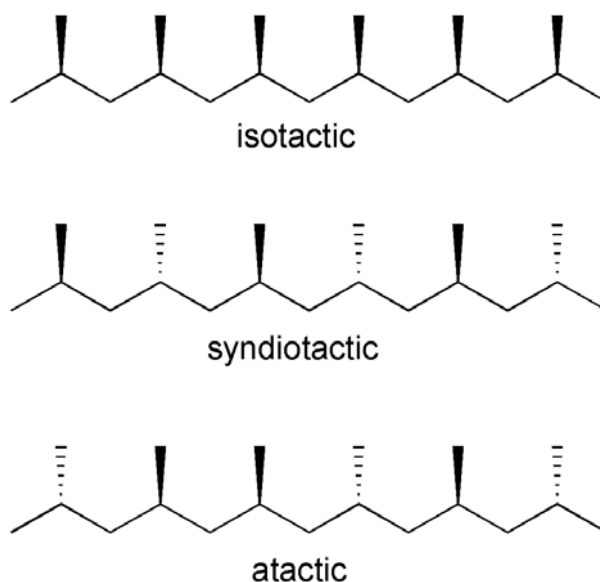


Figure 14- Tacticity of PP [21]

To produce film capacitors, it is used the iPP due to the high crystallinity[22].

Crystallinity is very important in the study of polymers behaviour. High crystallinity results in the increasing of melting point, tensile strength, stiffness, softening, resistance to oil and grease and it

increases the barrier to moisture and gases. As mentioned before, iPP is a semi-crystalline polymer, meaning that it has amorphous (disordered) and crystalline (ordered) regions, as shown in Figure 15 [23].

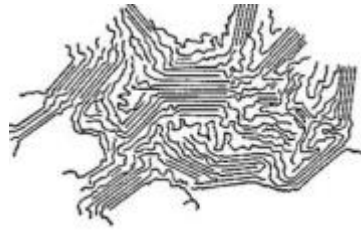


Figure 15- Semi-crystalline regions [24]

The crystallinity of a sample can be determined, for instance, by DSC and X-rays, but only DSC will be considered and described in this thesis [25].

2.2.2. iPP as a dielectric film

Film capacitors must withstand temperatures up to 105°C. Due to low volume, low cost and high dielectric performance, biaxially oriented polypropylene (BOPP) is a polymer suitable to be used in film capacitor. BOPP is manufactured by tenter- frame or tubular process (double- bubble process). Tubular process is less used than tenter frame process [26].

Figure 16 explains the production method tenter frame process, mostly used to produce BOPP for film capacitors. This method creates high dielectric strength and optimizes the film characteristics [27].

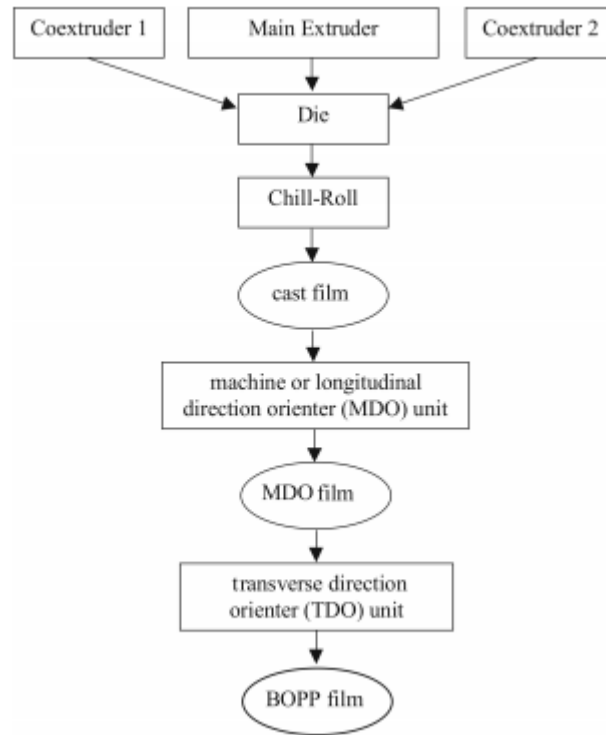


Figure 16- Tenter frame process to produce BOPP[28]

This technology consists in pass each raw material (PP, additives...) in an extruder and coextruders under certain temperature. Then, the melted raw materials flow to the die and in the chill-roll, the material is cooled very fast. In this stage, it is denominated as cast film, as it was reached the crystallization. After that, the material goes to the machine direction oriented (MDO) unit and it is heated until just before reach the melting point (called as softening point). Here the material is stretched in machine direction, called as MDO film. Finally, it goes to the transverse direction orienter (TDO) unit and again it is heated to the softening point and stretched, this time, in transversal direction, producing the BOPP [28].

Nowadays, a new technology can produce BOPP films in a simultaneous orientation, showing significant advantages in producing thinner films (less than $5\mu\text{m}$), leading in the reduction of the volume of a film capacitor and giving at the same time better electrical and mechanical properties, due to the crystal grains be more isotropically dispersed in longitudinal and transverse directions [29].

Film properties depend on the processing conditions. Considering a certain rate of cooling, Figure 17, demonstrates that for different pressures there is a relation between the volume and the temperature. By increasing the pressure, the crystallization temperature increases [30].

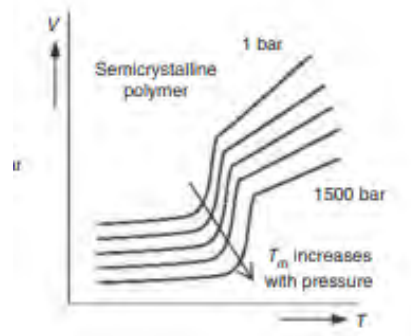


Figure 17- Diagram for a semi-crystalline polymer, relation between pressure, volume and temperature[30]

The Table 2 lists an example of some properties of oriented PP films.

Table 2- Typical properties of oriented PP films [31]

Property	ASTM Test Method	Uniaxial Orientation	Biaxial Orientation
Specific gravity (g/cm ³)	D1505	0.885–0.905	0.902–0.907
Tensile strength (psi)	D882	4500–7000	7500–40,000
Elongation (%)	D882	550–1000	352475
Tear strength (g/mil propagation)	D1922	25 MD 600 TD	3–10
Fold endurance	D2176	Very high	Excellent
24 h % water absorption	D570	0.005	0.005
Water vapor transmission rate (g/mil/100 in. ² /24 h at 100°F)	E96	0.7	0.25
Oxygen permeability (cm ³ /100 in. ² /mil/24 h/atm. at 77°F)	D1434	150–240	160
Haze (%)	–	1–2	1–2

The biaxial orientation enhances barrier properties to oxygen and water vapor. To improve the adhesion and the wettability of BOPP films, it is used a corona treatment method which modifies the surface free energy of the polymer and improves further the metallization process [32].

The biaxial orientation increases the stiffness, toughness, as well as, the dielectric strength and the mechanical properties as shown in Figure 18 [33].

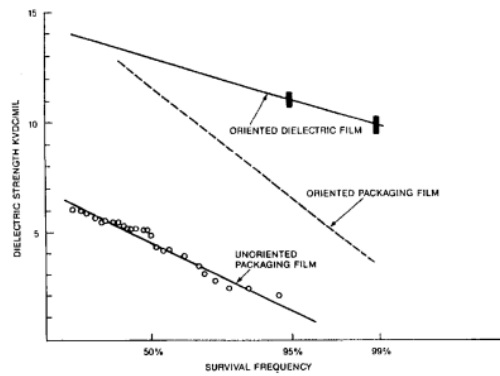


Figure 18- Influence of the orientation in the dielectric strength[33]

The Figure 18 demonstrates that the orientation plays an important rule on the dielectric strength improvement. An oriented dielectric film has higher dielectric strength, with a survival frequency of 99%, when compared with an oriented packaging film (or even with an unoriented packaging film, which cannot survive more than 95%).

To prevent the film from stretching, a higher Young Modulus value is preferred. Higher orientation level and higher modulus prevent the film from having permanently deformations.

The stretching causes wrinkles in the wounded roll and it is responsible for the non-desirable capacitance.

Furthermore, the shrinkage of the dielectric during the manufacturing can decrease the capacitance of the capacitor or it can lead to flashover shorts. For semi-crystalline polymers, as PP, the volume is reduced 20 to 25%, when cooled from the processing temperature to the room temperature, Figure 19 [30].

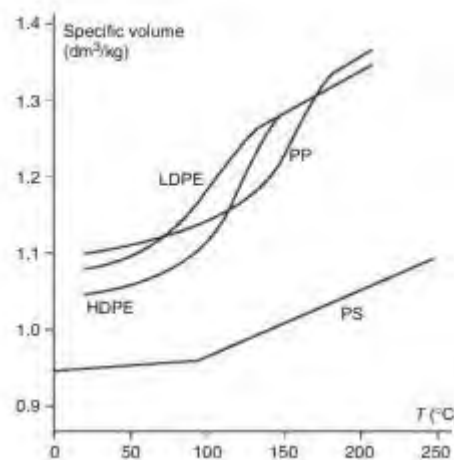


Figure 19- Influence of the shrinkage (specific volume versus temperature with no pressure) [30]

The shrinkage depends from the rate solidification and this can cause internal stress which can lead to void formation and tearing. A low shrinkage is desirable.

The surface roughness tends to affect the dielectric and physical strengths. To improve the impregnation and preserves the strength of the film, in a dielectric liquid impregnated electrical capacitor, for example, a morphological phenomenon, called as hazy PP film, is used as a dielectric medium. This provides an irregular surface on PP, but it is uniform in all film. As a BOPP is metallized with a thin layer of aluminium, for example, is desirable a strong adherence between the metal layer and the BOPP to improve the contact. The hazy film provides a good contact between the materials and improves the winding and the drying cycle of the material as it provides surface lack for removal of vapours [34].

Hazy film allows the dielectric oil penetrates into the capacitor due to one side of the film has an open fibrillar structure, Figure 20 [33].

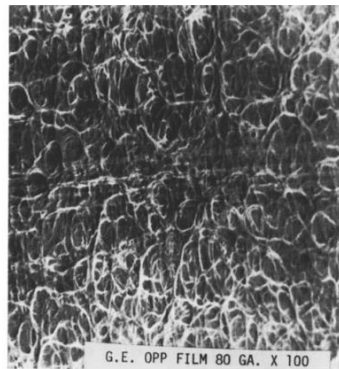


Figure 20- Hazy film: Open fibrillar structure on the film surface (General electric)[33]

In this case, the fibrillar surface depends of the resin characteristics and process parameters, while the oil cooperates to a good capacitance.

Other important point to consider, it is the contamination during manufacturing or present in the resin, which can lead to dissipation factors. During the manufacturing of the films is not allowed regrind material.

2.3. Characterization techniques

For the characterization of the polypropylene films, all samples were subjected to several techniques such as Differential Scanning Calorimetry (DSC), Fourier Transform Infrared spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and mechanical tests. It is going to be described a summary of each technique, which one, very useful to characterize materials.

2.3.1. Differential Scanning Calorimetry

DSC is a thermal analysis technique, very important in pharmaceutical and polymer fields, used in quality control methods, development and manufacturing of polymeric materials. It is able to measure the response of a material when heated or cooled, allowing to study physical properties, estimates the lifetime and curing processes for example, as well as, to study thermal and mechanical histories of a material [35].

The Figure 21 shows a typical thermogram for an organic polymer. It measures the transitions and enthalpy changes, occurred in a sample as a function of temperature and time.

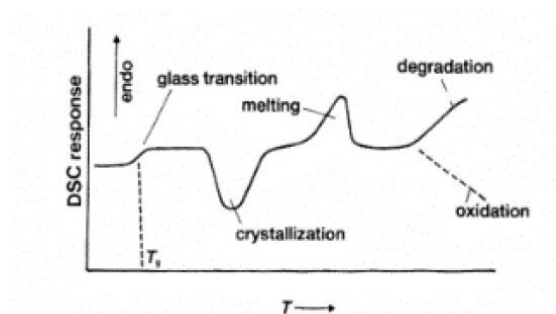


Figure 21-DSC response of an organic polymer [30]

At a constant heating and cooling rates, this technique allows the measuring of the glass transition (T_g), the melting, the crystallization, degradation, oxidation and the heat capacity of a material, providing a full thermal characterization of a material [30].

To study a polymer, it is made a first heating curve of a DSC measurement, to see the mechanical or thermal history, and then a second heating curve to determine the properties of the material, under certain conditions [36].

Many materials have crystalline and amorphous regions. The crystalline structure and the morphology (as spherulites dimensions) have a significant influence in the mechanical properties of semi-crystalline polymers, as PP [37]. For PP, there are four different types of spherulites [22].

The DSC can measure the degree of crystallization of a structural order of a solid, becoming possible the study of polymer properties. It is influenced by the thermal history and the chemical structure. By Equation 5, it can be obtained the relative percentage of crystallinity (%C) by measuring the enthalpy of fusion of a sample (ΔH_f) and comparing it with the enthalpy of the pure crystalline material (ΔH_f 100%). The enthalpy for 100% crystalline PP is 207J/g [38].

$$\%C = \frac{\Delta H_f}{\Delta H_f 100\%} \times 100 \quad \text{Equation 5}$$

The higher the percentage of crystallinity, more brittle is the material but stronger and stiffer is. Moreover, it influences the density, transparency and hardness of a material [37].

2.3.2. Fourier Transform Infrared Spectroscopy

FTIR is used in a wide range of industries, as an example, pharmaceuticals, food, chemicals, materials, forensic labs and at academics. It covers chemical applications for organic compounds and polymers. It allows, for instance, the investigation of adulterants in the food, analyze product formulation, identify product contaminants and solve manufacturing problems [39].

It is a non-destructive technique that measures the interaction of infrared light with the material, through the vibrational transitions of molecules.

It consists in passing an infrared radiation through a sample, while some radiation is transmitted and other is absorbed by the atoms. This absorption is detected by the detector, which gives a signal of the molecular “fingerprint” of the sample, the spectrum. This can be done by handling compressed alkali metal halide pellet method (using for example Bromine Potassium (KBr)) or use an Attenuated Total Reflectance- Fourier Transform Infrared Spectroscopy (ATR-FTIR).

By the measuring of the vibrations of atoms is possible to determine the functional groups and obtain an infrared spectrum, as shown in Figure 22 [40]. The frequency is measured as wavenumbers, over the range of 4000cm^{-1} and 600cm^{-1} , typically.

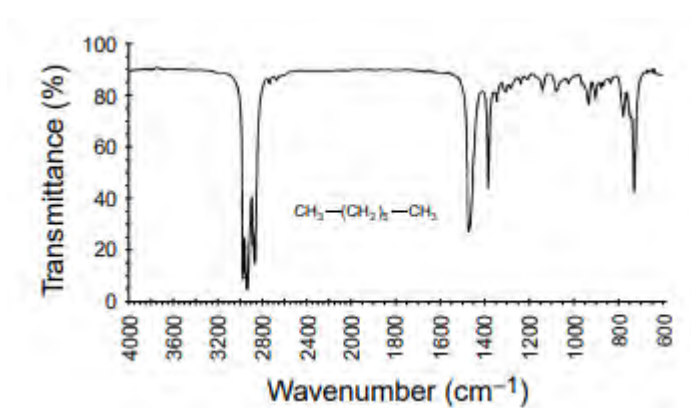


Figure 22- Infrared spectrum of n-heptane, by ATR- FTIR [41]

The main regions of the infrared spectrum is listed in Figure 23.

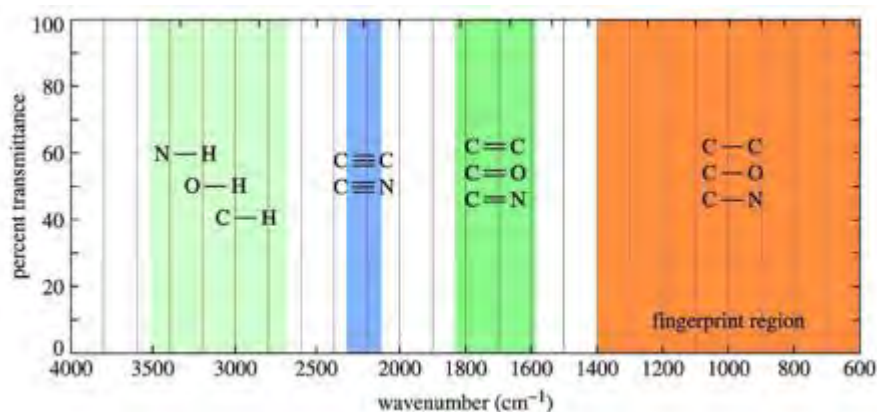


Figure 23- IR regions and respectively functional groups positions[42]

2.3.3. Scanning Electron Microscopy

Morphological aspects of a material can be determined by SEM, a non-destructive technique, very useful for the characterization of materials, as it is able to give detailed images of the sample which is being analyzed.

Electron microscope has the same principle of an optical microscope, but due to its electrons with very short wavelengths it allows higher resolution image than the optical microscope[43].

Atoms present in the surface of the sample interact with the electron beam producing several signals on the surface of the solid sample giving information about, for example, the chemical composition, morphology, crystallinity structure and orientation of the materials[44].

SEM can be coupled with energy dispersive spectroscopy (SEM-EDS). EDS can be also designated as energy dispersive X-ray spectroscopy (EDX). It allows the identification and the quantification of each element present in the sample and it is useful for instance, for contamination analysis, material defects and tin whiskers analysis[45].

EDX consists in bombarding, with X-ray radiation, the surface of a sample, in keV, through an electron beam. The X-rays emitted depend on the acceleration voltage of the electron beam, as well as, on the material which is being analyzed and the elements found near the surface of the sample. For example, metals like gold, zinc are very easy to be detected by this technique, while elements with low atomic number are more difficult to be detected [46].

Figure 24 presents the electromagnetic spectrum, showing the distribution of the electromagnetic radiation in function of frequency and wavelength. The higher the frequency the shorter the wavelength, as observed for X-rays [47].

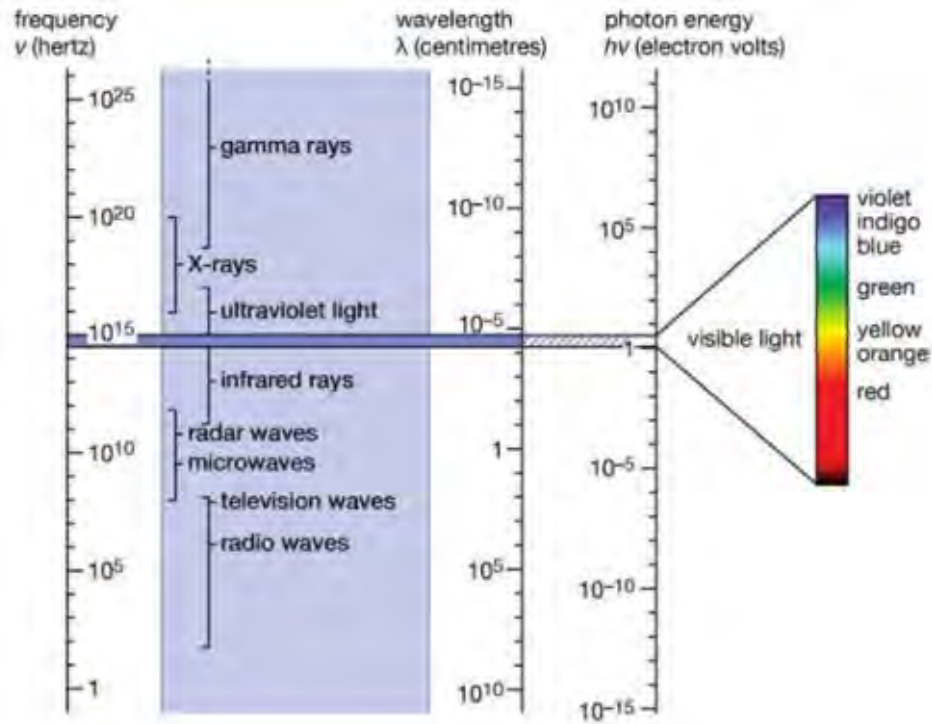


Figure 24- Electromagnetic spectrum [47]

2.3.4. Atomic Force Microscopy

AFM characterizes the surface topography of samples through, for example, the determination of the roughness [48]. As SEM, it allows the analysis of the surface with a high accuracy.

Figure 25 presents a scheme of an AFM system. The sample is placed in the AFM and, by a piezo actuator with displacement in XYZ positions, scans the surface of the sample. An optical system, with laser beam and a photo detector, determine the deflection level of the cantilever, according the surface topography. Finally, a specific software will translate the information and give the image of surface topography[49].

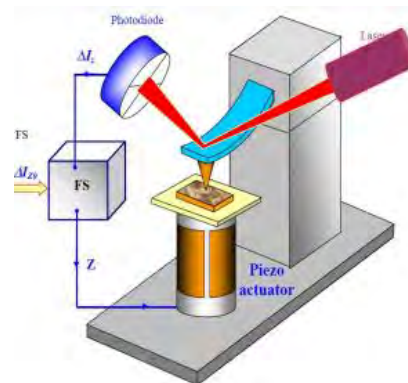


Figure 25- AFM scheme [50]

There are height parameters able to characterize the surface topography. Figure 26 shows some of principle parameters, where M is the mean line and L is the sampling length (L)[51].

Most common parameters are the roughness average (Ra), which is defined as the arithmetic average of the absolute values of the profile heights, measured from the mean line; the root mean square deviation (RMS or Rq), which corresponds to the standard deviation of the surface height measured from the mean line[52].

The maximum roughness depth (Rmax) is the difference between the highest and the lowest vertical distance point of the profile.

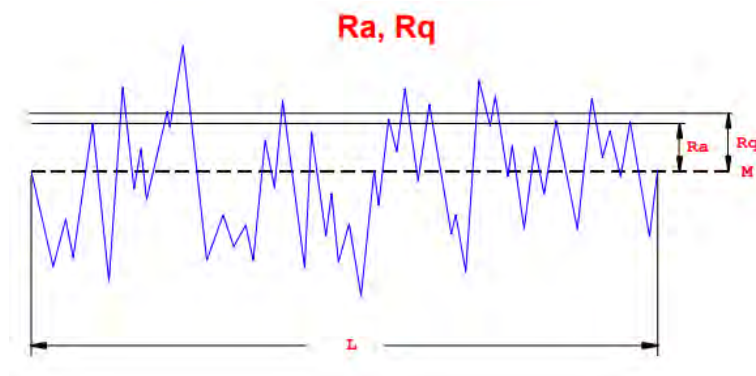


Figure 26- Example of a Ra and Rq measurement [51]

2.4. Mechanical tests

During the development and research of plastics materials, it is very important to study and know more about the properties and the behavior of each material to predict future problems as ruptures or breaks.

There are some characteristics as Young Modulus (or Tensile Modulus), tensile strength and elongation at breaks which are very useful to understand how the material reacts under compression or even tension. Of course, these properties can be influenced by the process used later on, but normally they are given by the raw material supplier.

The Young Modulus, E , is measured in Pascal (Pa) and evaluates the elasticity of solid materials [53].

The Young Modulus is determined by the Equation 6. It gives a measure of the resistance to deformation of a material and it is defined as the deformation ε (strain), of a material and the force σ (stress), needed to deform it [30].

$$E = \frac{\sigma}{\varepsilon} \quad \text{Equation 6}$$

Tensile strength, measured in MPa, is the maximum stress that a material can handle. It is the maximum value just before the rupture.

The elongation at breaks, measured in percentage (%), is the elongation that a material can withstand before its break.

The Figure 27 shows a stress-strain curve of a material. This analysis provides information about the mechanical behavior. A sample, with a known dimension, is deformed at a given rate while the strength needed to induce the deformation is recorded.

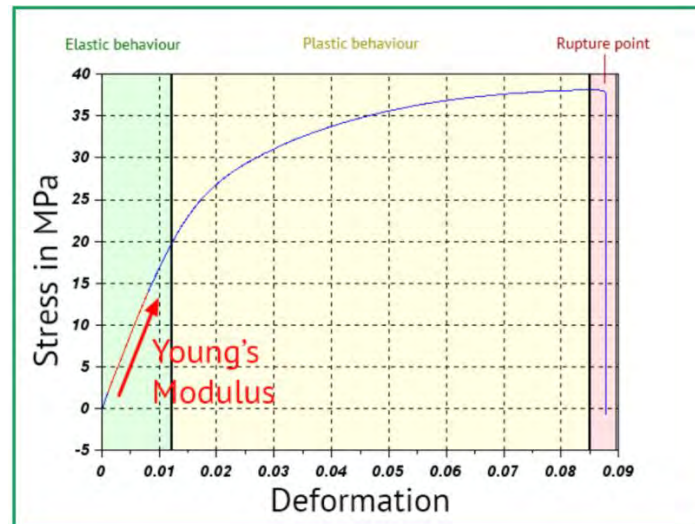


Figure 27- Stress- strain curve of a material [53]

The elastic behavior is the first yield point, where the material can return to the initial state if released the applied force.

Then the second yield point is the plastic behavior where the material will have a permanent deformation.

The third yield point is the rupture point, which means that the strength applied was too high. After the rupture point, the material will break.

Figure 28 shows a scheme of stress-strain curves, depending on the polymer.

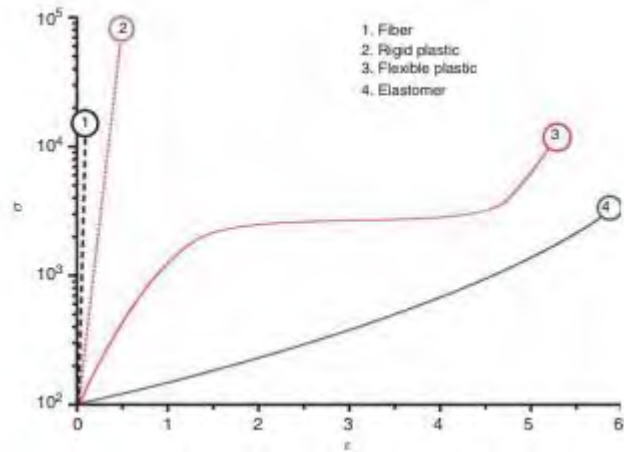


Figure 28- Examples of stress-strain curves for different polymers [30]

In conclusion, the elongation at break is related to the polymer plasticity and its ability to extend before breaking. Young Modulus is related to the elasticity while the tensile strength is related to the mechanical strength of a polymer [47].

The stiffer the material, the greater the Young Modulus value.

CHAPTER 3- MATERIALS AND METHODS

3. Materials and methods

3.1. Metallized PP film samples

PP film rolls with a thin metallization layer presenting 36.0mm width, with different thicknesses (2.4 μ m to 3.0 μ m) were the samples used for the investigation, as presented in Figure 29.



Figure 29- Metallized polypropylene film samples

All samples were identified from 1 to 6, according to Vishay code number, as listed in the Table

3.

Table 3- Samples identification

Sample	Vishay Code nr.
1	80920622
2	80920625
3	80921546
4	80933978
5	80932744
6	80920623

3.2. Characterization Techniques

3.2.1. Differential Scanning Calorimetry analysis

DSC curve of the samples was recorded with a differential scanning calorimeter from PerkinElmer, model 6000, equipped with an intracooler (Figure 30).

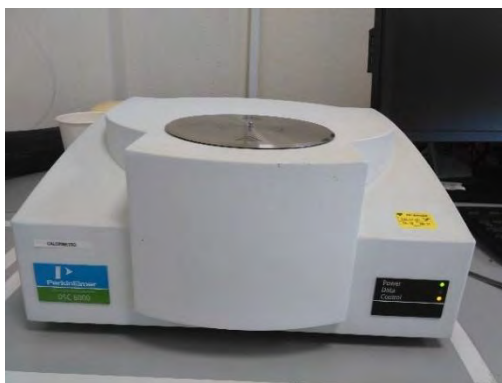


Figure 30- PerkinElmer, model DSC 6000

The equipment was purged and calibrated. The purge was made with dry high purity nitrogen gas with a constant flow rate of 20mL/min. The calibration was made with pure indium, checking the melting point and comparing with the limits allowed for this element.

For each sample of Table 3, it was weighted around 0,0064g ($\pm 0,0001$ g) of BOPP film (Figure 31). It was introduced in an aluminum cap, placed in the DSC equipment and the thermogram was recorded. It was used two different programs: one with a heating and cooling rate of 10°C /min and the other with a heating rate of 10°C /min and a first cooling rate of 100°C/min.



Figure 31- Weight of a BOPP sample

The first program had a heating rate of 10 °C/min, as well as, the cooling rate as shown in the Table 4, holding 10 minutes after the first running and holding 5 minutes in the end of the program.

Table 4- Program 1 used in the BOPP samples

1) Heat from -20.00°C (±0,01°C) to 280.00°C(±0,01°C) at 10°C /min
2) Cool from 280.00°C(±0,01°C) to -20.00°C(±0,01°C) at 10°C/min
3) Hold for 10.0 min at -20.00°C(±0,01°C)
4) Heat from -20.00°C(±0,01°C) to 280.00°C(±0,01°C) at 10°C/min
5) Cool from 280.00°C (±0,01°C) to -20.00°C(±0,01°C) at 10°C/min
6) Hold for 5,0 min at -20.00°C(±0,01°C)

The second program had a heating rate of 10°C/min and a cooling rate of 100°C /min in the same running. as shown in the Table 5, holding 1 minute after the first running and holding 1 minute in the end of the program.

Table 5- Program 2 used in the BOPP samples

1) Heat from -20.00°C(±0,01°C) to 280.00°C(±0,01°C) at 10°C /min
2) Cool from 280.00°C(±0,01°C) to -20.00°C (±0,01°C) at 100°C/min
3) Hold for 1.0 min at -20.00°C(±0,01°C)
4) Heat from -20.00°C(±0,01°C) to 280.00°C(±0,01°C) at 10°C/min
5) Cool from 280.00°C(±0,01°C) to -20.00°C(±0,01°C) at 10°C/min
6) Hold for 1.0 min at -20.00°C(±0,01°C)

3.2.2. Fourier Transform Infrared Spectroscopy analysis

The FTIR spectra of samples (Table 3) were registered with a PerkinElmer spectrometer. The equipment used was an ATR-FTIR with a diamond crystal, model Frontier (Figure 32). The equipment was calibrated with a PET standard film sample. Before starting the scanning, it was made the background and, then, each sample was placed on the crystal and made the spectrum. Again, a software from PerkinElmer available in the computer, for this specific equipment acquired all the results. Each spectrum was recorded with a range of 4000cm⁻¹ and 600cm⁻¹.



Figure 32- PerkinElmer ATR-FTIR, model Frontier

3.2.3. Scanning Electron Microscopy and Energy Dispersive Spectroscopy analysis

For this analysis, it was used a SEM equipment from JEOL, series JSM-6010LV (Japan), similar to the Figure 33, coupled with an EDS - INCAx-Act, PentaFET Precision (Oxford Instruments).



Figure 33- JSM-6010PLUS/LA InTouchScope™ Multiple touch panel scanning electron microscope [54]

It was analysed the surface morphology of all samples, at high vacuum mode with an accelerating voltage of 10keV, by detecting secondary electron (SE). By the analyses of EDS, it was possible to see the distribution and the identification of the elements present in each sample. Therefore, each sample was divided in 3 areas as shown in Figure 34.

By the quantitative analysis, is possible to calculate the concentration of each element. For the analysis it was used gold as the conductive coating.

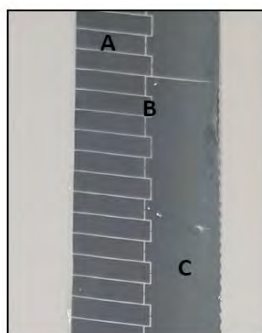


Figure 34- Identification of the three areas (A/B/C) used for the SEM-EDS analysis

These analyses were performed in the 3B's Research Institute.

3.2.4. Atomic Force Microscopy analysis

For this analysis, the equipment used was a *Dimension Icon, Bruker, USA*.

To measure the surface roughness, it was used ScanAsyst® mode, which is a PeakForce Tapping® based image optimization technique. Using a single- touch scanning, this equipment allows a high resolution AFM images [55].

ScanAsyst® mode was performed by using a silicon nitride tip with the nominal radius of 20 nm.

Parameters settings were the following: ScanAsyst-Air, Bruker, nominal frequency 150 kHz, nominal spring constant of 0.7N/m; Linear scanning rate: 0.5 and 1 Hz; Scan resolution: 512 samples/line.

The surface roughness analysis, for each sample, was measured in three points as demonstrated in the Figure 35.

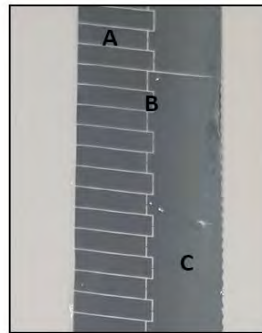


Figure 35- Identification of the three areas (A/B/C), used for the surface roughness analysis

Surface roughness was expressed as root mean square roughness (R_q) and roughness average (R_a) and the surface analysis was performed on the height images.

These analyses were performed in the 3B's Research Institute.

3.2.5. Mechanical tests analysis

Mechanical tests were performed by the 3B's Research Institute, according *ISO 527-3 Plastics – Determination of tensile properties – Part 3: Test conditions for films or sheets* [56].

The equipment used, for the tensile test, was Instron 4505 Universal Mechanical Testing Equipment using a 50 N load cell, crosshead speed of 5 mm.min⁻¹, using a distance between grips of 100 mm.

The PP film was cutted in strips of 25mm width and 150mm length. For each condition, they were tested at least 7 specimens.

For the elastic modulus, it was determined from the initial linear region of the stress/strain curve. The results will be presented in chapter 4 and includes the average, $\bar{x}$, (Equation 7) and the standard deviation, s (Equation 8).

$$\bar{x} = \frac{\sum xi}{n} \quad \text{Equation 7}$$

$$s = \sqrt{\frac{\sum (xi - \bar{x})^2}{n-1}} \quad \text{Equation 8}$$

The Figure 36 shows the section of the sample used to perform the mechanical tests under tensile load.

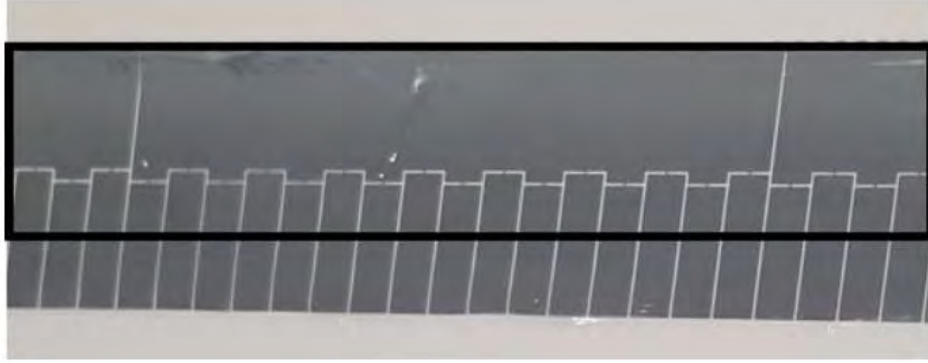


Figure 36- Section of the sample used to perform the mechanical tests

3.2.6. Film Shrinkage

For the shrinkage evaluation, of each sample, they were cutted 5 strips of each base film (as Figure 36) and measured the film length (machine direction) at 25°C, with a ruler of an optical microscope, in mm.

The strips were allocated between two pre-dried papers and inserted in the oven *Nahita* Model 632 Plus, at 85°C for 30 minutes. The film length was measured again, with a ruler of an optical microscope, in mm.

The process was repeated at 105°C and 120°C.

For the ΔL calculation, it was considered the average of the 5 specimens, using Equation 7, for each sample, considering the average length of final temperature, at 85°C, 105°C and 120°C (ΔL_{T_2}) and the average length of initial temperature, at 25°C (ΔL_{T_1}). To calculate the percentage of shrinkage, it was used Equation 9.

$$\text{Shrinkage} = \frac{\Delta L_{T_2} - \Delta L_{T_1}}{\Delta L_{T_1}} \times 100\% \quad \text{Equation 9}$$

CHAPTER 4- DISCUSSION AND RESULTS

4. Discussion and results

Chapter 4 is dedicated to present and discuss the results obtained, for the six samples investigated.

4.1. Differential Scanning Calorimetry

DSC thermograms of all samples shown in Figure 37.

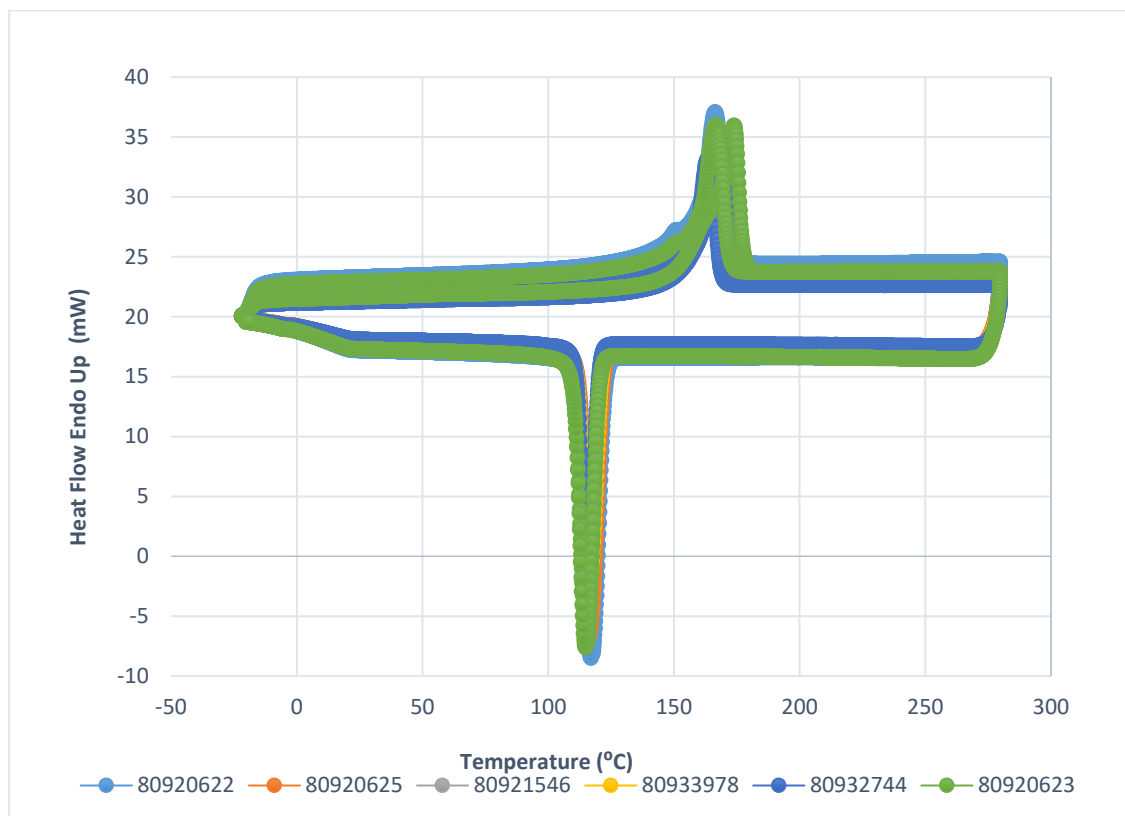


Figure 37- Thermograms of all Vishay BOPP samples, obtained with the program 1.

The DSC thermograms show two endothermic peaks corresponding to the melting temperature of the polymer around 170 °C and one exothermic peak corresponding to the crystallization peak around 120 °C.

For example, considering sample 1, 80920622, it was determined the thermogram of figure 38.

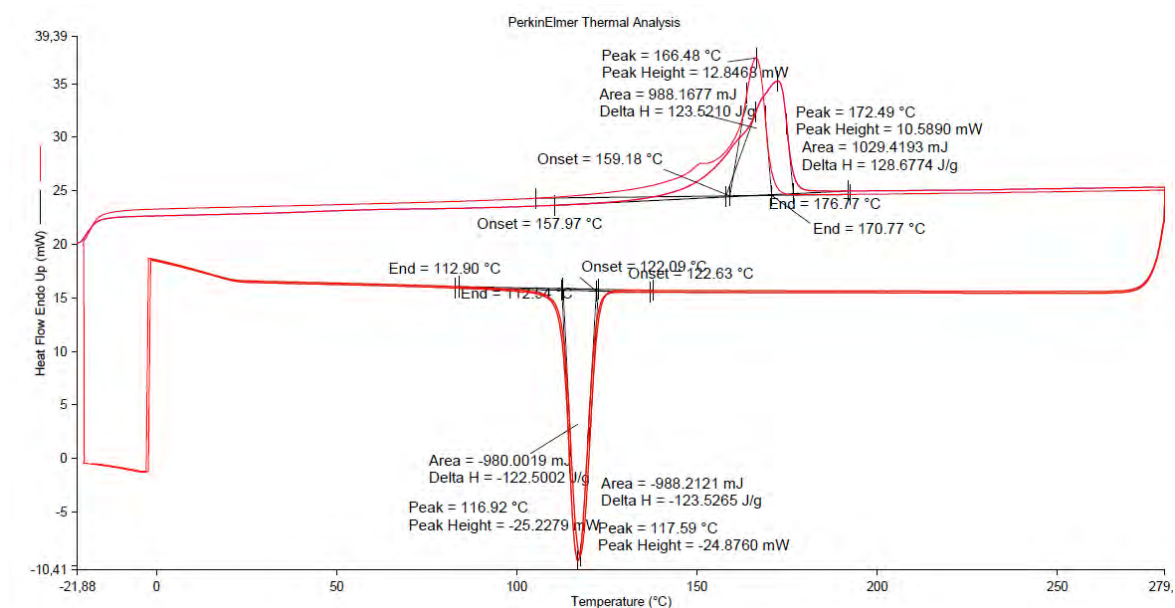


Figure 38- DSC result for sample 1, Vishay code nr. 80920622, using program 1

All thermograms, using program 1, are presented in annex I, of this thesis.

To have an overview of results, Table 6 and Table 7 present the melting and the crystallization peaks of each sample, using program 1, during the first and the second run, respectively. As it can be seen, there were some differences for T_m , ΔH_m and consequently, crystallinity percentage variation (table 8) of the structure. Increasing the onset melting temperature in BOPP films can be the result of transition of structures of stacked lamellae into fibrillar structures which are oriented along transverse [57], but the DSC technique is not sensitive to detect these changes.

Table 6- First run for the melting and crystallization, for the program 1, for each sample

First run	Heating (melting)					Cooling (crystallization)				
BOPP sample	Onset ($\pm 0,01^\circ\text{C}$)	End ($\pm 0,01^\circ\text{C}$)	T_{melting} ($\pm 0,01^\circ\text{C}$)	Area (mJ)	ΔH_m (J/g)	Onset ($\pm 0,01^\circ\text{C}$)	End ($\pm 0,01^\circ\text{C}$)	$T_{\text{crystallization}}$ ($\pm 0,01^\circ\text{C}$)	Area (mJ)	ΔH_c (J/g)
1-80920622	157.97	176.77	172.49	1029.42	128.68	122.63	112.90	117.59	988.21	123.53
2-80920625	170.09	176.49	172.46	830.12	105.08	121.48	112.57	117.18	863.25	109.27
3-80921546	160.08	174.23	170.79	917.82	99.76	119.78	110.13	114.51	909.01	98.81
4-80933978	167.05	176.06	173.05	784.84	120.74	121.15	111.52	116.28	767.16	118.02
5-80932744	163.62	174.42	171.47	726.97	111.84	120.19	111.34	115.79	747.71	115.03
6-80920623	167.00	177.48	173.87	991.65	116.66	120.39	110.61	115.23	973.90	114.58

Table 7- Second run for the melting and crystallization, for the program 1, for each sample

Second run	Heating (melting)					Cooling (crystallization)				
BOPP sample	Onset (±0,01°C)	End (±0,01°C)	T _{melting} (±0,01°C)	Area (mJ)	ΔH _m (J/g)	Onset (±0,01°C)	End (±0,01°C)	T _{crystallization} (±0,01°C)	Area (mJ)	ΔH _c (J/g)
1-80920622	159.18	170.77	166.48	988.17	123.52	122.09	112.54	116.92	980.00	122.50
2-80920625	160.52	171.04	166.54	864.16	109.39	122.15	112.80	116.54	870.62	110.21
3-80921546	157.29	169.71	164.55	850.58	92.45	119.97	110.33	114.34	912.26	99.16
4-80933978	158.05	169.97	165.04	775.73	119.34	121.46	111.65	115.95	767.31	118.05
5-80932744	157.47	168.49	163.40	716.89	110.29	120.42	111.45	115.46	748.92	115.22
6-80920623	158.74	171.56	166.67	917.01	107.88	120.24	110.52	114.90	978.78	115.15

With this information (Tables 6 and 7) is possible to calculate the crystallinity of each BOPP sample, by Equation 5, considering ΔH_f of PP is 207 J.g^{-1} and presented in table 8.

Table 8- Crystallinity results for the first and second run during the heating

BOPP sample	$\Delta H_m(\text{J/g})$ 1st run	% C	$\Delta H_m(\text{J/g})$ 2nd run	% C
1-80920622	128.68	62.16	123.52	59.67
2- 80920625	105.08	50.76	109.39	52.84
3-80921546	99.76	48.19	92.45	44.66
4-80933978	120.74	58.33	119.34	57.65
5-80932744	111.84	54.03	110.29	53.28
6-80920623	116.66	56.36	107.88	52.12

Figure 39 shows the percentage of crystallinity of the six samples studied with the program 1.

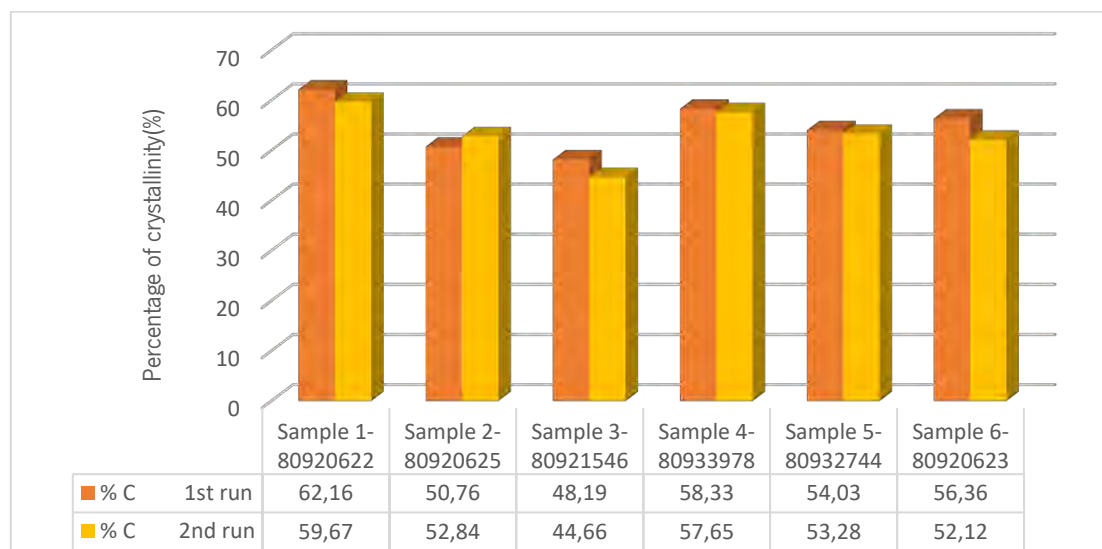


Figure 39- Percentage of crystallinity, using the program 1

Sample 1 has the highest percentage of crystallinity, followed by sample 4. Samples 5 and 6 have similar crystallinity, while the lowest crystallinity is observed in sample 3.

Polymers which are more ordered tend to lead to high degree of crystallinity.

The first run and the second run have the same rate of cooling, so it is not seen significant differences between the runs, in terms of crystallinity.

Considering the program 2, Figure 40 presents the thermograms of all samples, from Table 3.

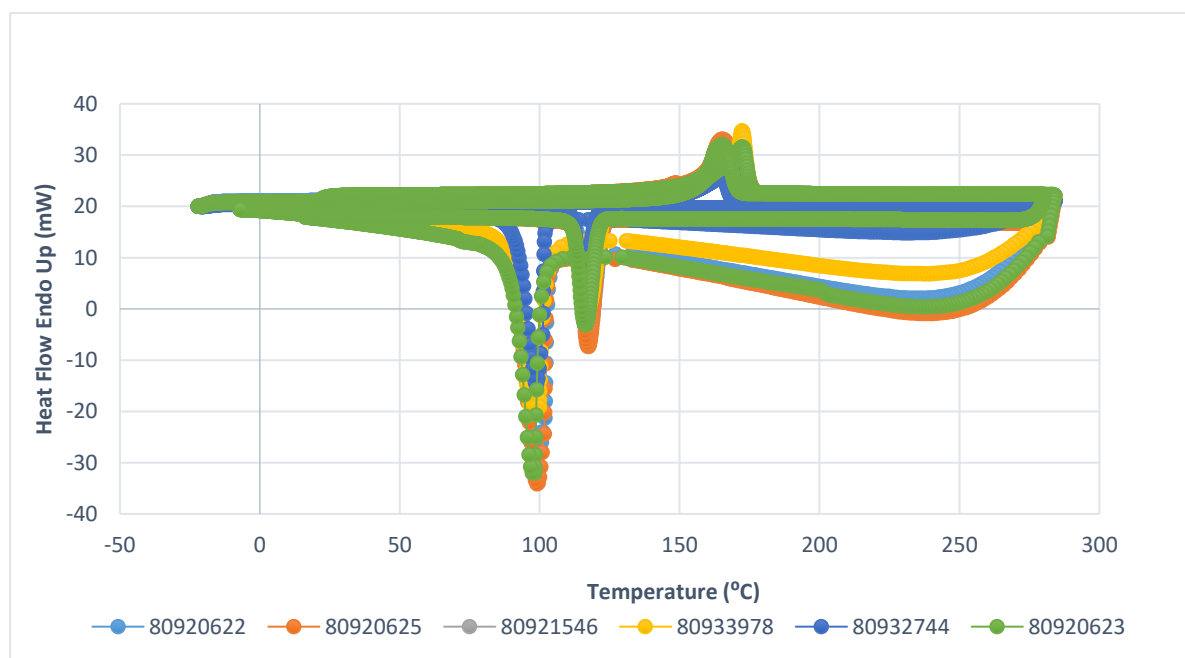


Figure 40-DSC results from all Vishay BOPP samples, obtained with the program 2

For example, considering sample 1, 80920622, it was determined the thermogram of Figure 41.

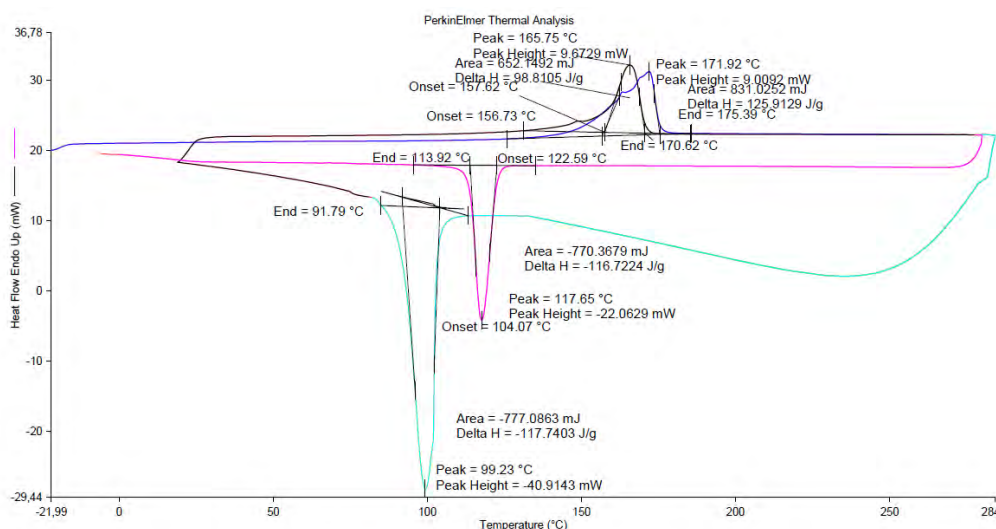


Figure 41- DSC result for sample 1, Vishay code nr. 80920622, using program 2

All thermograms, using program 2, are presented in annex II, of this thesis.

To have an overview of results, Tables 9 and 10, present the melting and the crystallization peaks of each sample, using program 2, during the first and the second run, respectively.

Table 9- First run for the melting and crystallization, for the program 2, for each sample

First run	Heating (melting)					Cooling (crystallization)				
BOPP sample	Onset ($\pm 0,01^\circ\text{C}$)	End ($\pm 0,01^\circ\text{C}$)	T_{melting} ($\pm 0,01^\circ\text{C}$)	Area (mJ)	ΔH_m (J/g)	Onset ($\pm 0,01^\circ\text{C}$)	End ($\pm 0,01^\circ\text{C}$)	$T_{\text{crystallization}}$ ($\pm 0,01^\circ\text{C}$)	Area (mJ)	ΔH_c (J/g)
1-80920622	157.62	175.39	171.92	831.03	125.92	104.07	91.79	99.23	777.09	117.74
2-80920625	157.84	175.87	171.70	888.19	120.03	103.19	91.57	99.04	938.62	126.84
3-80921546	156.92	174.14	170.34	782.28	110.18	102.41	90.04	97.24	806.65	97.24
4-80933978	168.64	175.10	172.34	733.56	114.62	105.30	89.62	97.99	769.38	120.22
5-80932744	157.62	175.39	171.92	831.03	125.92	104.07	91.79	99.23	777.09	117.74
6-80920623	157.84	175.87	171.70	888.19	120.03	103.19	91.57	99.04	938.62	126.84

Table 10- Second run for the melting and crystallization, for the program 2, for each sample

Second run	Heating (melting)					Cooling (crystallization)				
BOPP sample	Onset ($\pm 0,01^\circ\text{C}$)	End ($\pm 0,01^\circ\text{C}$)	T_{melting} ($\pm 0,01^\circ\text{C}$)	Area (mJ)	ΔH_m (J/g)	Onset ($\pm 0,01^\circ\text{C}$)	End ($\pm 0,01^\circ\text{C}$)	$T_{\text{crystallization}}$ ($\pm 0,01^\circ\text{C}$)	Area (mJ)	ΔH_c (J/g)
1-80920622	156.73	170.62	165.75	652.15	98.81	122.59	113.92	117.65	770.37	116.72
2-80920625	157.62	170.56	165.40	830.37	112.21	121.93	113.67	117.38	819.64	110.76
3-80921546	156.58	169.40	163.27	694.51	97.82	121.25	112.25	115.77	723.15	101.85
4-80933978	157.04	170.45	164.79	605.48	94.61	122.37	112.20	117.38	732.39	114.44
5-80932744	156.73	170.62	165.75	652.15	98.81	122.59	113.92	117.65	770.37	116.72
6-80920623	157.62	170.56	165.40	830.37	112.21	121.93	113.67	117.38	819.64	110.76

It can be checked that, for program 2, there is almost no variation in the onset temperature, possible meaning that the lamellae crystals are more uniform between them.

With the information, from Tables 9 and 10, is possible to calculate the crystallinity of each BOPP sample, by Equation 5, considering ΔH_f of PP is 207 J.g^{-1} and presented in the Table 11.

Table 11- Crystallinity results for the first and second run during the heating

BOPP sample	ΔH_m (J/g) 1st run	% C	ΔH_m (J/g) 2nd run	% C
1-80920622	125.92	60.83	98.81	47.73
2- 80920625	120.03	57.98	112.21	54.21
3-80921546	110.18	53.23	97.82	47.26
4-80933978	114.62	55.37	94.61	45.70
5-80932744	98.04	47.36	83.61	40.39
6-80920623	131.34	63.45	118.76	57.37

Figure 42 shows the percentage of crystallinity of the six samples studied with the program 2.

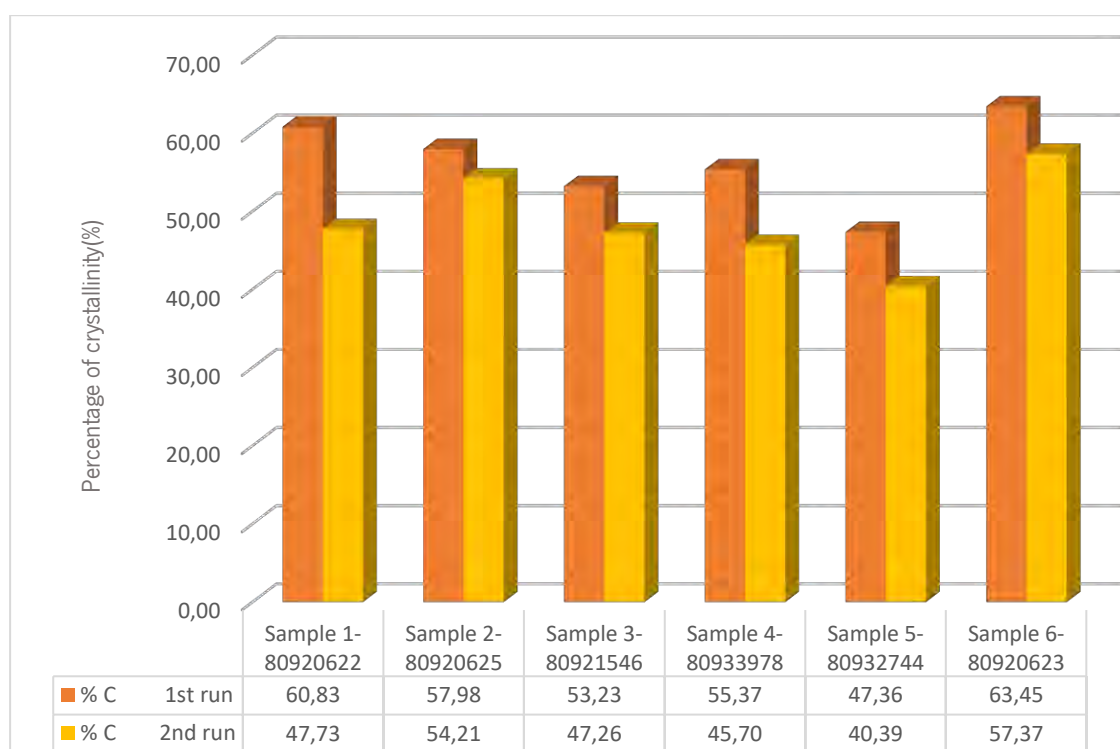


Figure 42- Percentage of crystallinity, using the program 2

With program 2, the percentage of crystallinity is different, when compared with the results of program 1. Sample 6 has the highest percentage of crystallinity, followed by the samples 1, 2 and 4, while the lowest crystallinity is observed in sample 5.

By the DSC, the first heating, which gives the information about the material as it is, shows that the polymer is an iPP, as the melting point of a perfect iPP is 171°C. During the first heating are visible two peaks that can give an indication of two different crystal morphologies of the iPP. Normally commercial iPP has a melting point around 160-166°C, and this can be observed in the second heating (which gives information about the behavior of the material). The program used can influence the

crystallinity of the polymer, as proved by using different cooling's at program 1 and program 2. The first cooling allows the molecular orientation, gives information about the recrystallization of the iPP and delete the thermal history of the material[58].

The cooling run provides information about the manufacturing process, where it can be seen a crystallization peak which proves that manufacturing process was too fast and gave no time to occur the crystallization of the iPP.

Molecules cannot have time to organize themselves and this can lead to thermal and mechanical failures. Samples 4 and 5 have the lowest area in the melting point region, during the endothermic reaction.

The crystallinity of the PP is around 40-60%, as the results show, samples 1 and 6 have the highest crystallinity and therefore more brittle is the sample[58].

The higher the crystallinity the stiffer is the sample and the better the material resistance.

Fast cooling can lead to crystallinity problems because the material cannot have time to crystallize in total. The program 1 has a slow cooling, 10°C/min, which can influence the crystallinity and can give time for the molecular chain organization, as well as, crystalline structures. This is shown by the high percentage of crystallinity, during the second run. For example, sample 1 has a % C of 62.16/59.67, during first and second run in program 1, respectively, while the same sample, with program 2, has a %C of 60.83/47.73 during first and second run, respectively.

The processing conditions, as cooling/heating rate and time, and the thickness of samples can influence the results obtained, as shown with different cooling rates. Other important factor that can affect the crystallinity is the thickness of PP film, the higher the thickness the lower the crystallization.

This can explain the low crystallinity observed in sample 3, which is the thicker sample.

In the first heating run, it is visible two different melting peaks which can be justified as two different crystal morphologies of the polymer. The first melting peak disappears in the second heating run, probably due to high energy and imperfect crystals.

Considering the total area during the heating, it can be observed that the area in first run is higher than in the second run, for both programs.

As samples were not normalized, it cannot be said that the lower the area, the less quantity of material was melted. However, it can be observed that the second melting peak is thinner than the first melting peak.

This technique demonstrates that the polymer studied is a PP and samples have different crystallinities, which can influence the mechanical properties of each sample.

4.2. Fourier Transform Infrared Spectroscopy

FTIR measurements were used to evaluate the vibrational characteristic bands of the samples and the Figure 43 shows the spectrum obtained for all samples, which confirms that it is a PP sample.

As PP is an alkane, it has only C-C and C-H bonds. From the ATR-FTIR spectrum, and checking literature, it can be observed that samples have four peaks between 3000cm^{-1} and 2800cm^{-1} , which are related to the CH_3 asymmetric stretching vibration (2950cm^{-1}), CH_3 symmetric stretching vibration (2867cm^{-1}), CH_2 asymmetric stretching vibration (2918cm^{-1}), CH_3 symmetric stretching vibration (2838cm^{-1}), respectively.

The peak at 1453cm^{-1} can be attributed to the CH_2 scissor vibrations or to CH_3 asymmetric deformation vibrations, while the peak at 1375cm^{-1} is attributed to CH_3 asymmetric deformation vibrations.

Between 1200cm^{-1} to 750cm^{-1} there are several small peaks. The peak at wavenumber around 1167cm^{-1} can be justified by the CH_3 asymmetric rocking vibration or C-H wagging vibration or even due to the C-C asymmetric stretching vibration. The peak at wavenumber around 998cm^{-1} can be attributed to CH_3 asymmetric rocking vibrations. The peak at wavenumber around 972cm^{-1} is due to the C-C asymmetric stretching and the CH_3 asymmetric rocking vibrations, while the peak at 899cm^{-1} is due C-C asymmetric and symmetric stretching vibrations and CH_3 asymmetric rocking vibrations. Finally, the peaks at 848cm^{-1} and 809cm^{-1} are related to CH_2 rocking vibrations. [59]

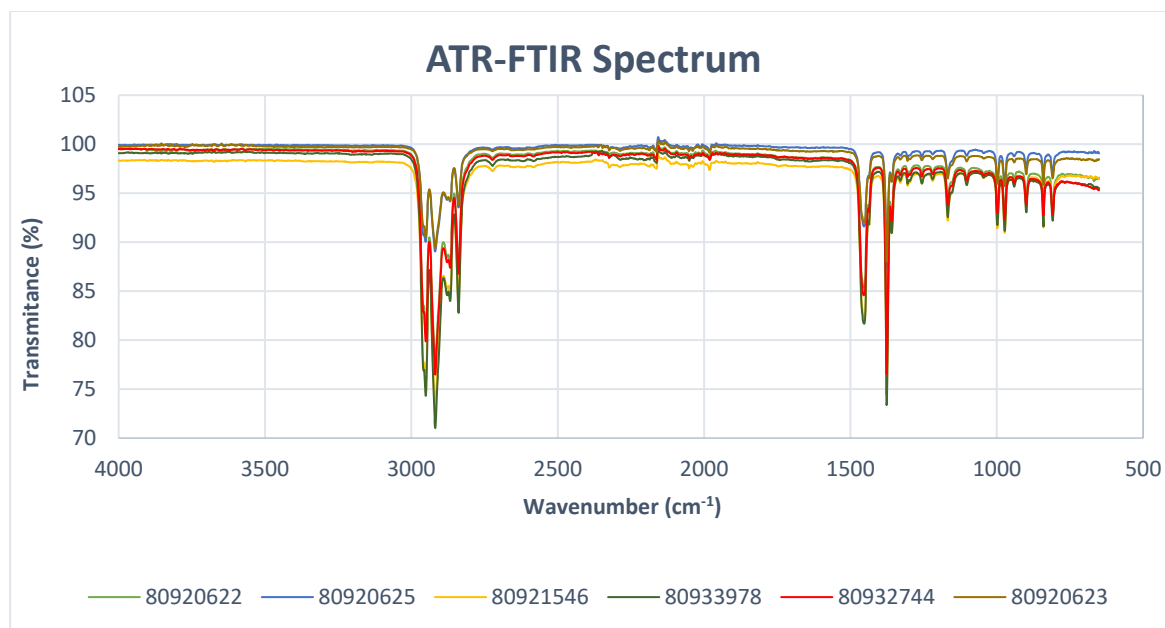


Figure 43- ATR-FTIR spectrum: overview of all BOPP samples

Splitting the samples of Figure 43, it can be observed that they have the same spectrum, with different transmittances. For example, in Figure 44, sample 1 (80920622) has the same transmittance of sample 5 (80932744). Sample 2 (80920625) has the same transmittance of sample 6 (80920623). Sample 3 (80921546) has the same transmittance of sample 4 (80933978). The comparison, for example, of sample 1(80920622) with sample 3 (80921546) shows high transmittance for sample 3. This happened because the spectrums were not normalized by equalizing the height of the absorption peak at 2918cm⁻¹.

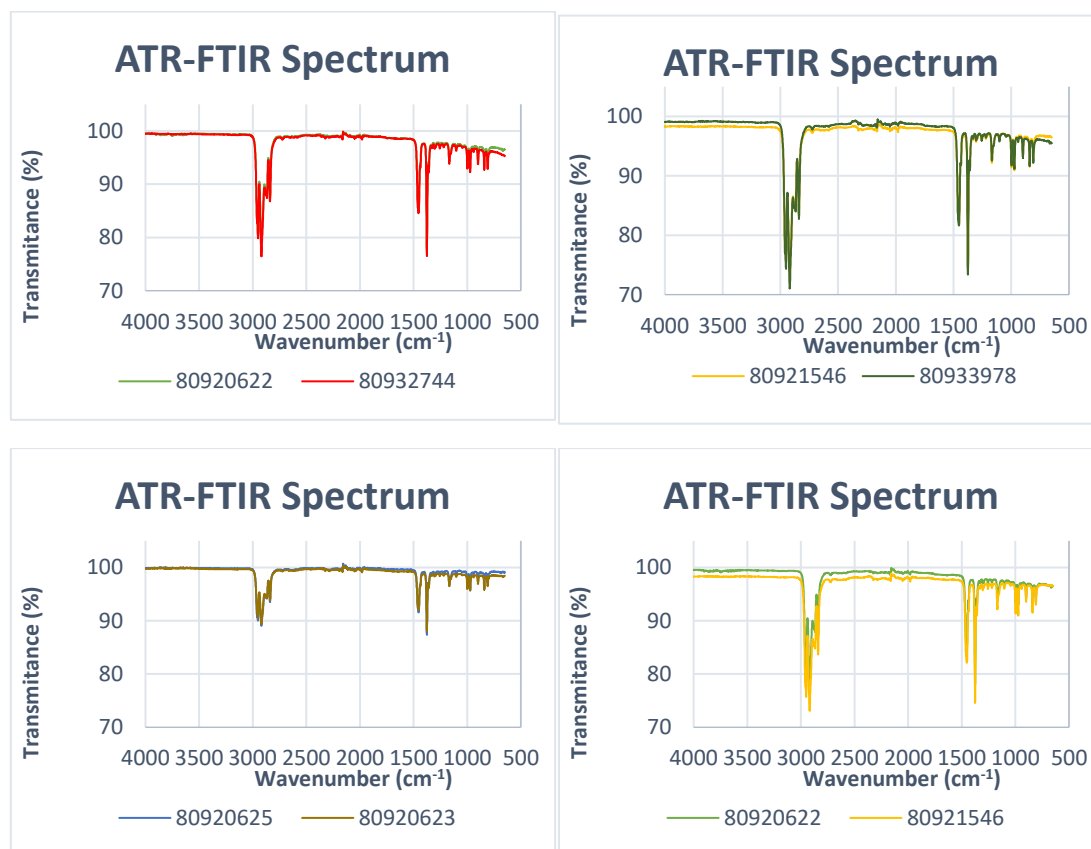


Figure 44- ATR-FTIR spectrum: examples of the comparison between two samples

For sample 1, it is shown in Figure 45, the spectrum taken from the FTIR software.

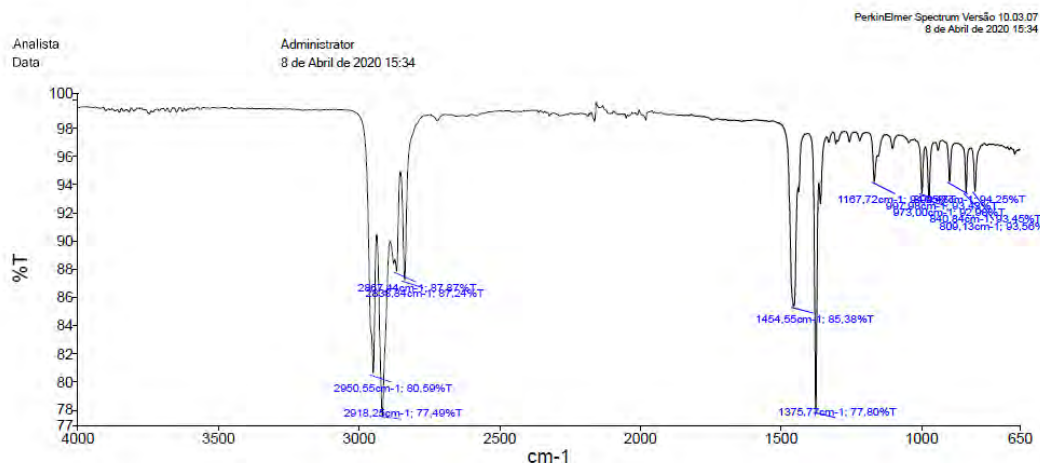


Figure 45- Spectrum of a BOPP sample (sample 1)

All samples spectrum are presented in annex III, of this thesis.

4.3. Scanning Electron Microscopy

Figures 46 to 57 show surface morphological studies of samples, per area, evaluated by SEM. Element mapping of the particles was performed by SEM-EDS and the chemical composition results are summarized, per sample and per area, in Tables 12 to 17.

The pictures (SEM) evidence different surface morphologies depending on the samples and suppliers. The surface of layer in the BOPP film is very smooth, and wrinkles and other defects are few, as shown in the picture in Figure 46 and 47, and for low magnification.

Sample 1- Areas A and C

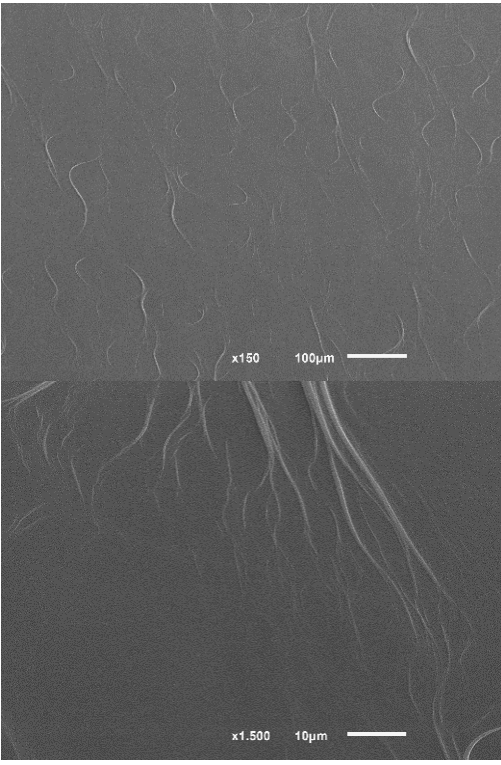
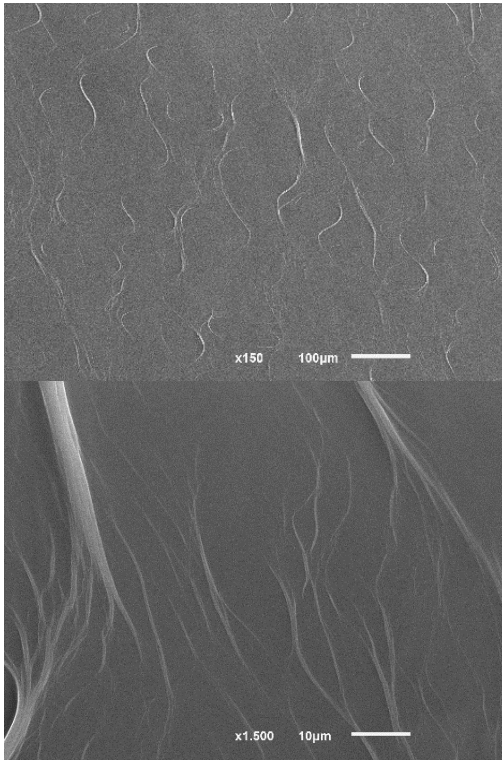
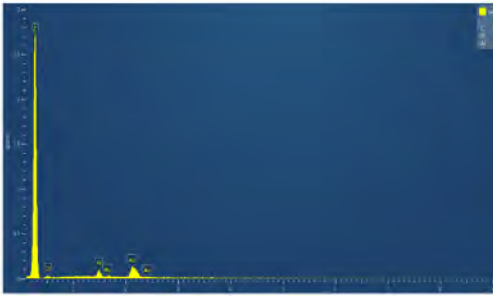
Area	A (between segments)	C (not segmented)
SEM image		
EDS image		

Figure 46- SEM- EDS images obtained in the Sample 1 – areas (A) and (C).

Sample 1- Area B

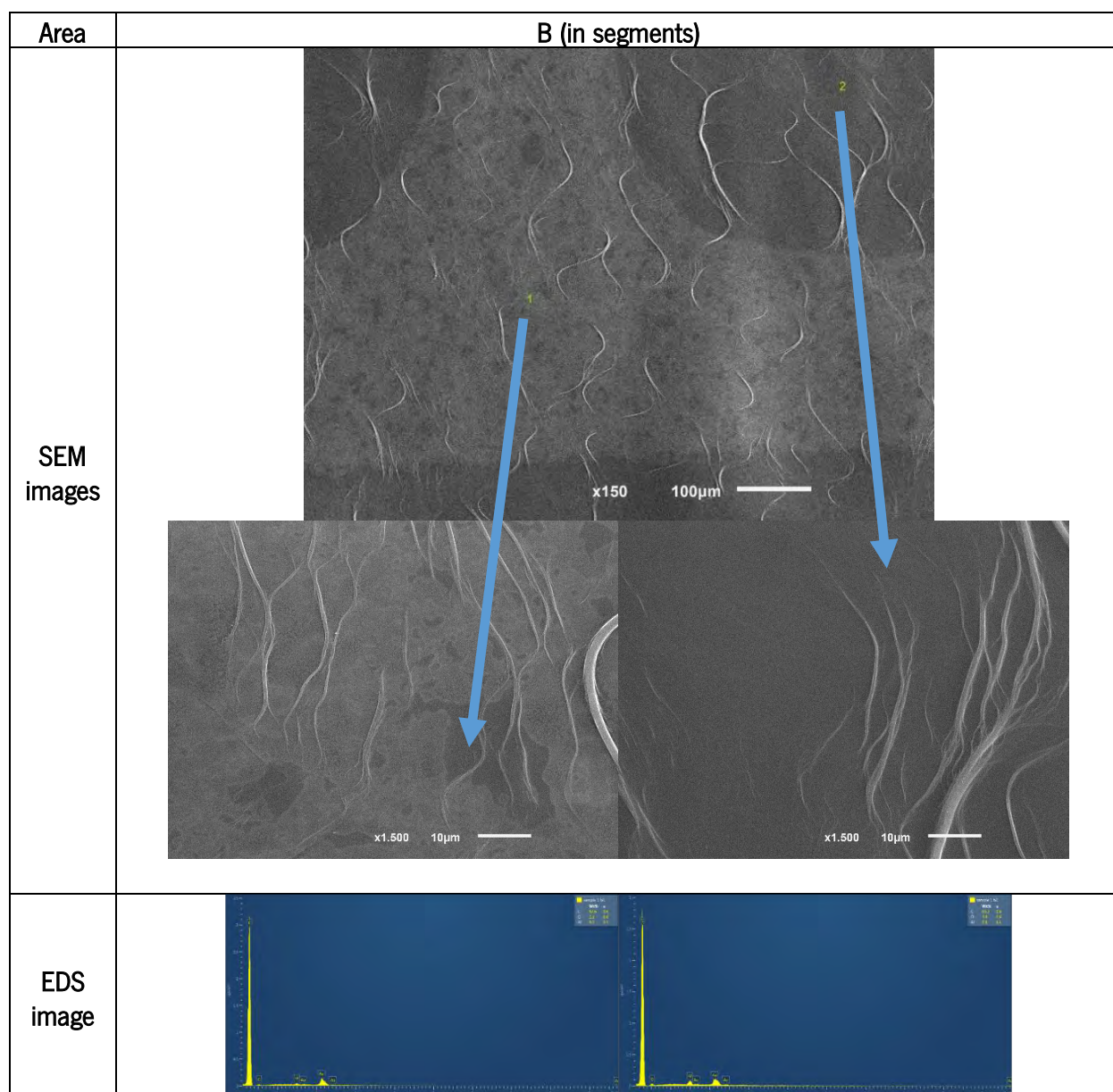


Figure 47- SEM- EDS images obtained in the Sample 1 - area (B).

The high-magnification SEM picture (Figures 46 and 47) show that PP lamellae grow to the fiber axis. However, there exists a single lamella, which grows directly into the area where the fan-shaped PP crystal started.

Table 12- Identification and quantification of each element present in sample 1

Element	Element weight (%)			
	A Area	B1 Area	B2 Area	C Area
C	95.8	97.6	95.3	94.3
Zn	0.0	0.0	0.0	0.0
O	3.3	2.1	3.9	3.3
Al	0.9	0.3	0.8	2.4

Sample 2 - Areas A and C

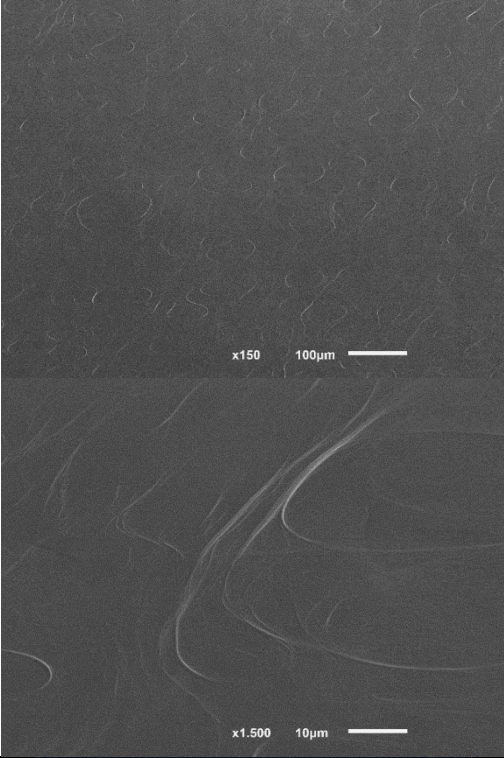
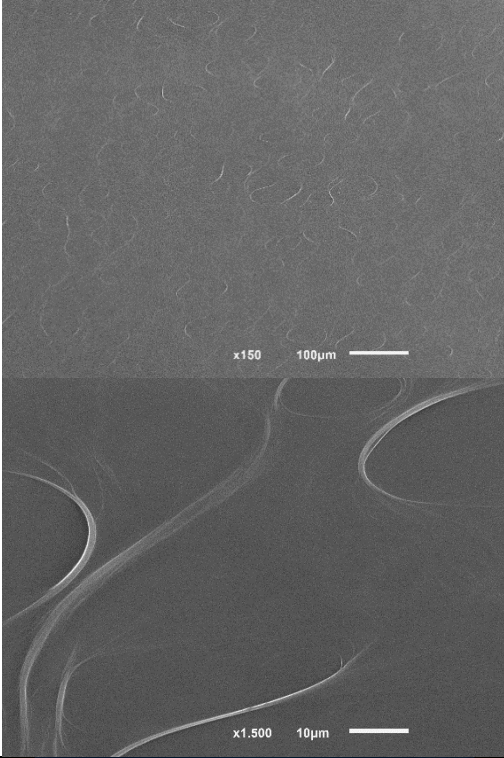
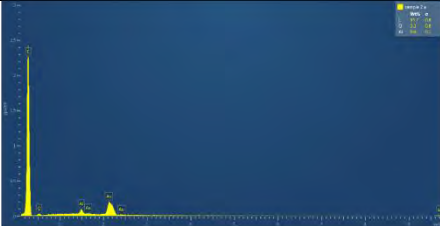
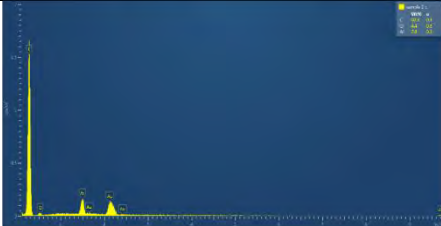
Area	A (between segments)	C (not segmented)
SEM images		
EDS image		

Figure 48- SEM- EDS images obtained in the Sample 2 – areas (A) and (C).

Sample 2- Area B

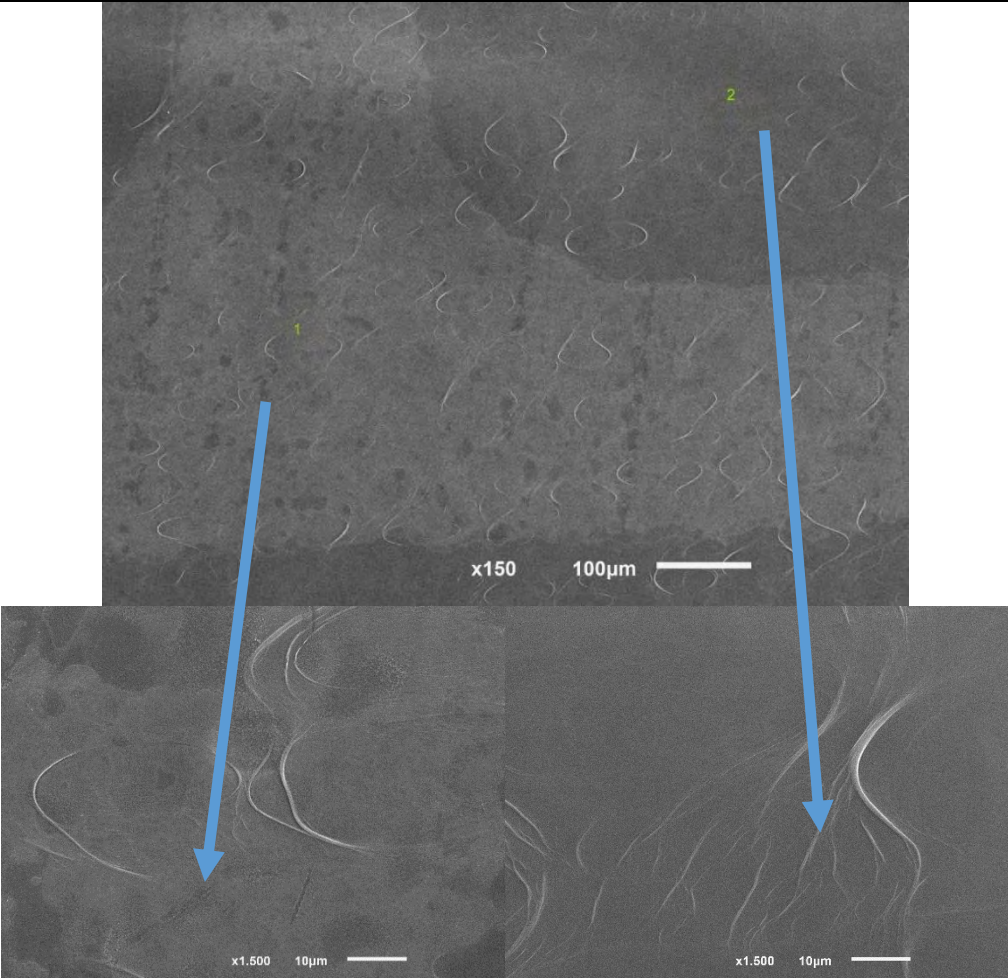
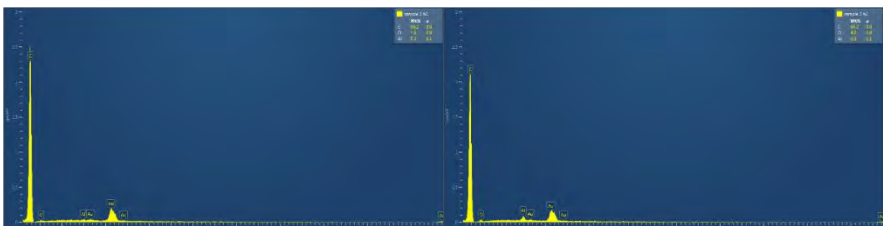
Area	B(in segments)
SEM images	
EDS image	

Figure 49- SEM- EDS images obtained in the Sample 2 - area (B)

The high-magnification SEM picture (Figure 49) show that PP lamellae grow to the fiber axis.

Table 13- Identification and quantification of each element present in sample 2

Element	Element weight (%)			
	A Area	B1 Area	B2 Area	C Area
C	95.7	98.2	95.2	92.8
Zn	0.0	0.0	0.0	0.0
O	3.3	1.6	4.0	4.4
Al	0.9	0.2	0.9	2.8

Sample 3- Areas A and C

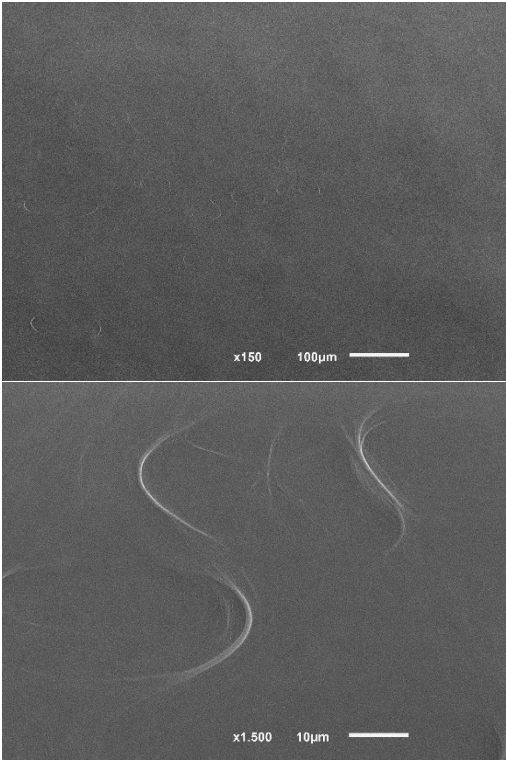
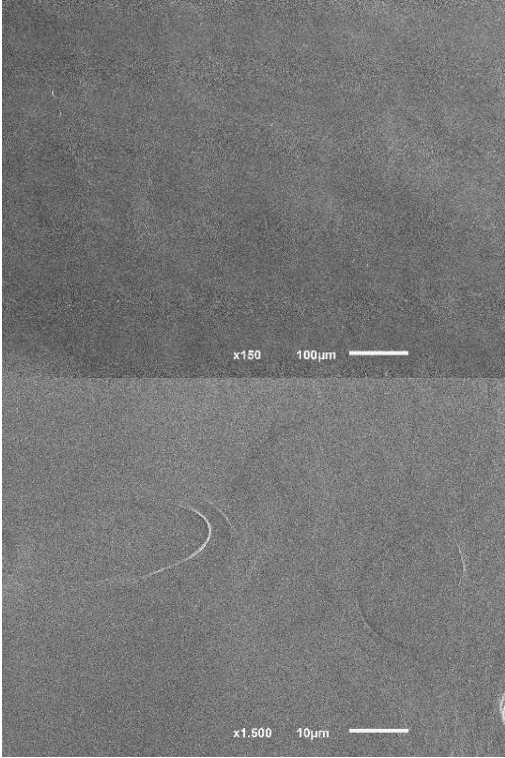
Area	A (between segments)	C (not segmented)
SEM images		
EDS image		

Figure 50- SEM- EDS images obtained in the Sample 3 – areas (A) and (C)

Sample 3- Area B

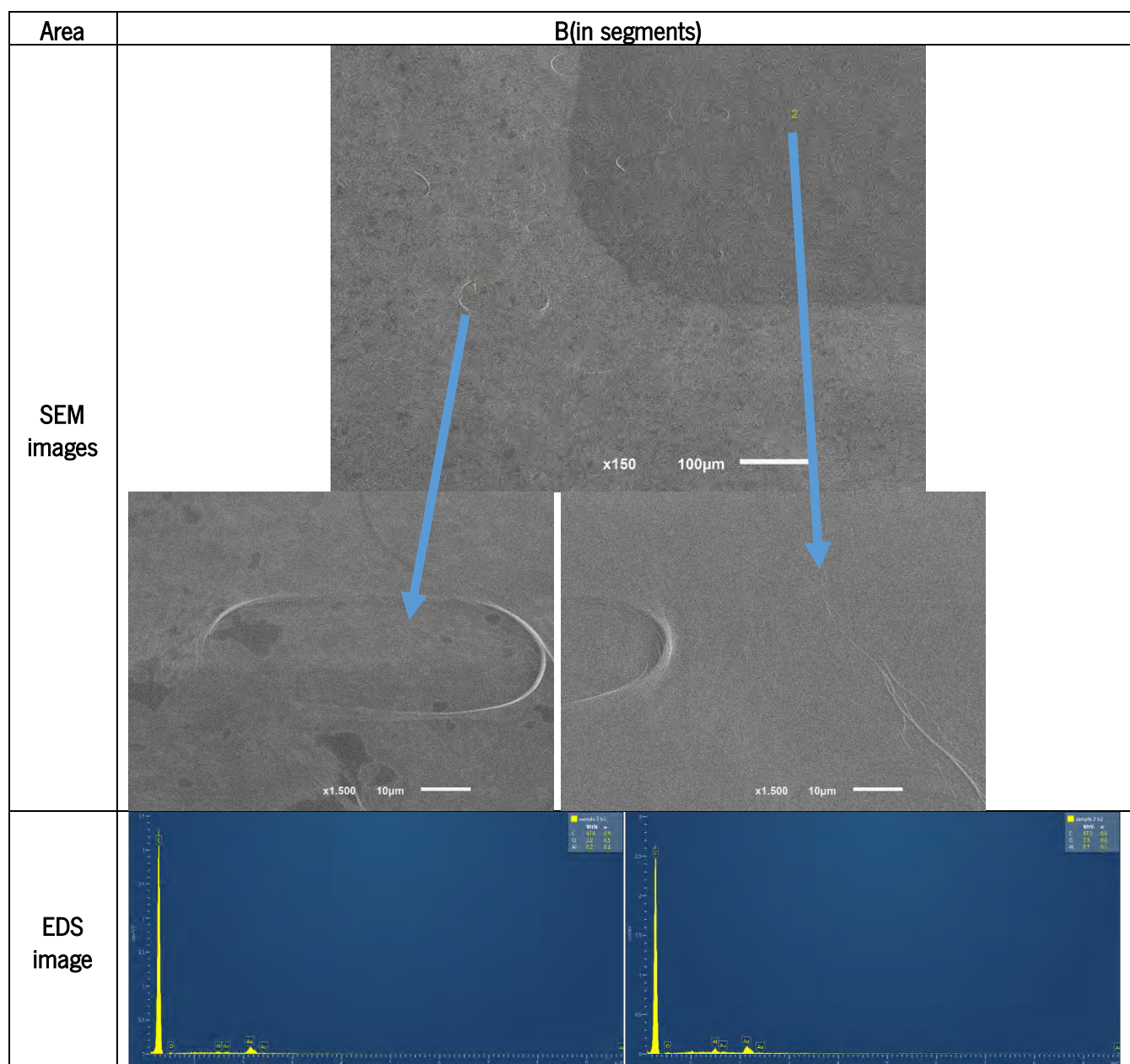


Figure 51- SEM- EDS images obtained in the Sample 3 – area (B)

SEM images of sample 3 (Figures 50 and 51) show surface homogeneity with no obvious phase separation.

Table 14- Identification and quantification of each element present in sample 3

Element	Element weight (%)			
	A Area	B1 Area	B2 Area	C Area
C	96.8	97.6	97.0	85.3
Zn	0.0	0.0	0.0	10.9
O	2.6	2.2	2.3	3.0
Al	0.7	0.2	0.7	0.9

Sample 4 - Areas A and C

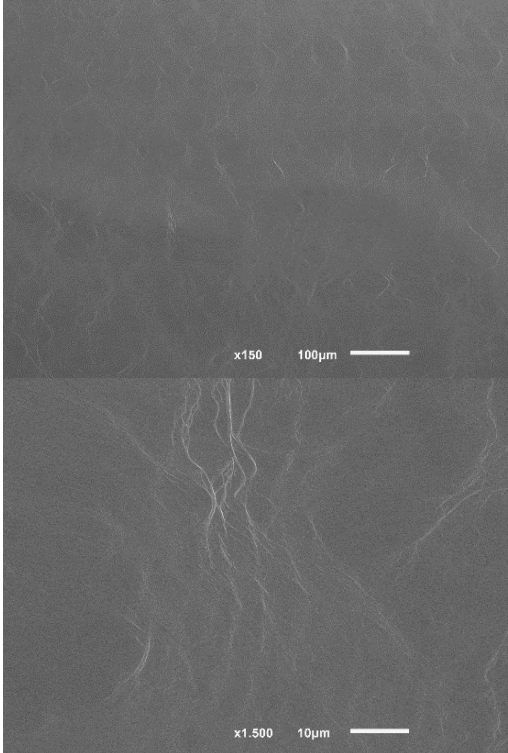
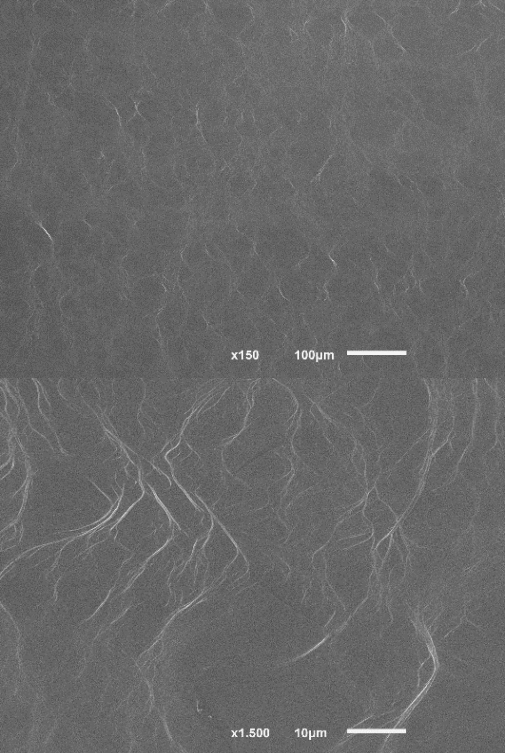
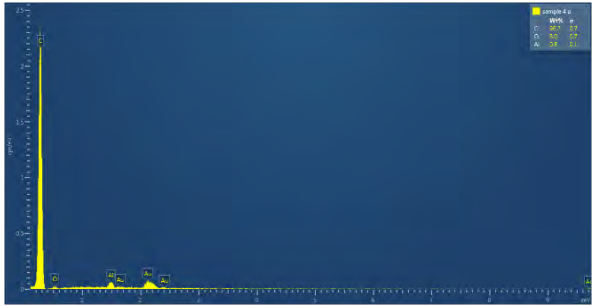
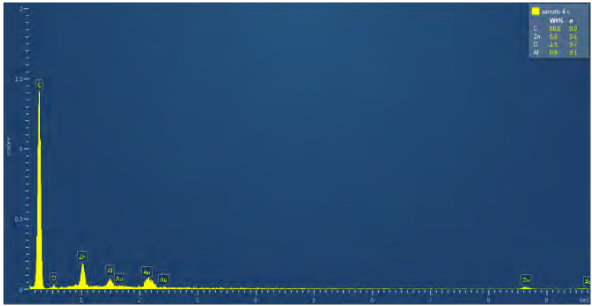
Area	A (between segments)	C (not segmented)
SEM images		
EDS image		

Figure 52- SEM- EDS images obtained in the Sample 4 – areas (A) and (C)

Sample 4- Area B

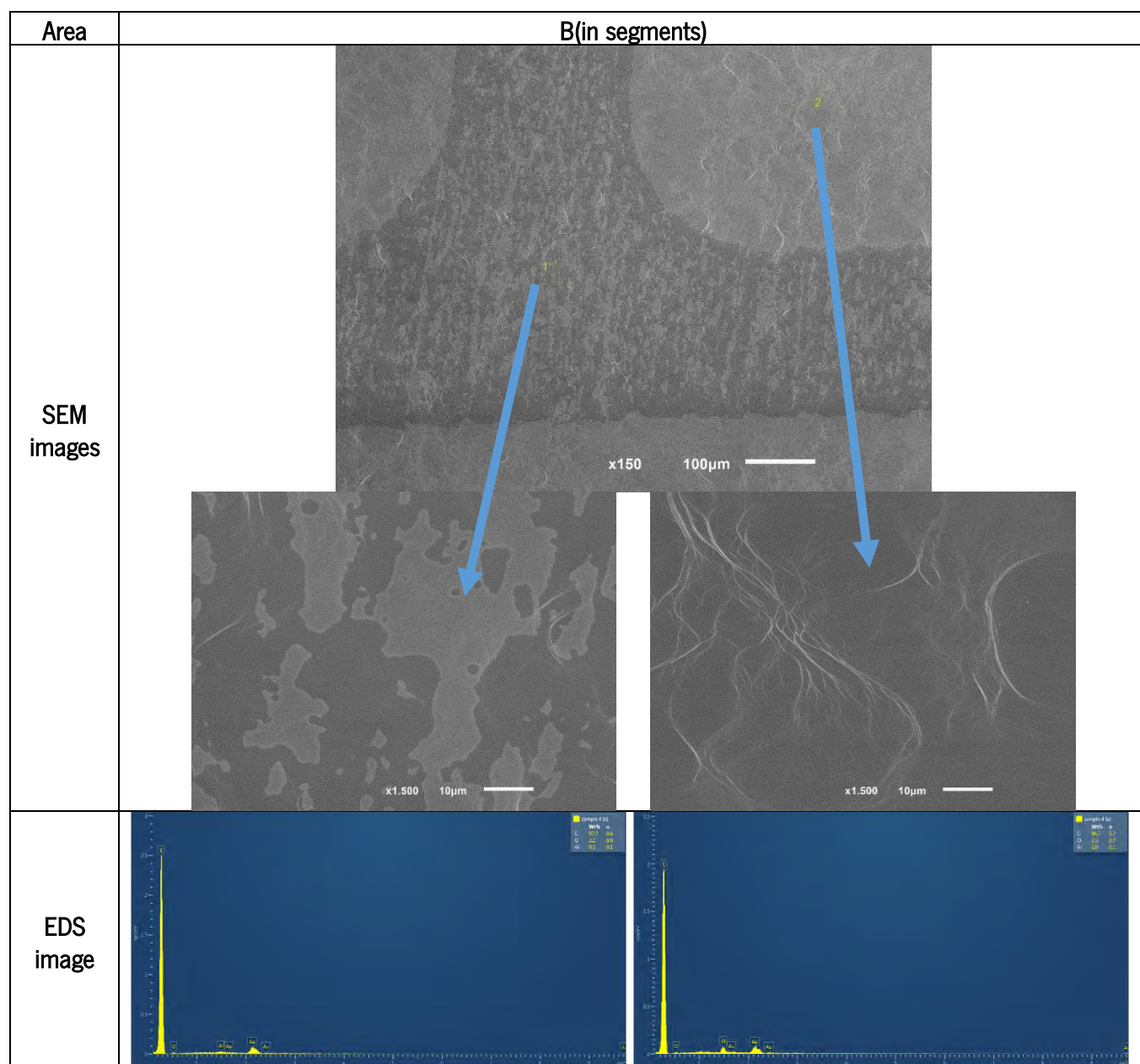


Figure 53- SEM- EDS images obtained in the Sample 4 -area (B)

The high-magnification SEM picture (Figure 53) shows that PP lamellae grow to the fiber axis.

Table 15- Identification and quantification of each element present in sample 4

Element	Element weight (%)			
	A Area	B1 Area	B2 Area	C Area
C	96.2	97.7	96.7	88.6
Zn	0.0	0.0	0.0	6.9
O	3.0	2.2	2.3	3.5
Al	0.8	0.1	1.0	0.9

Sample 5 – Areas A and C

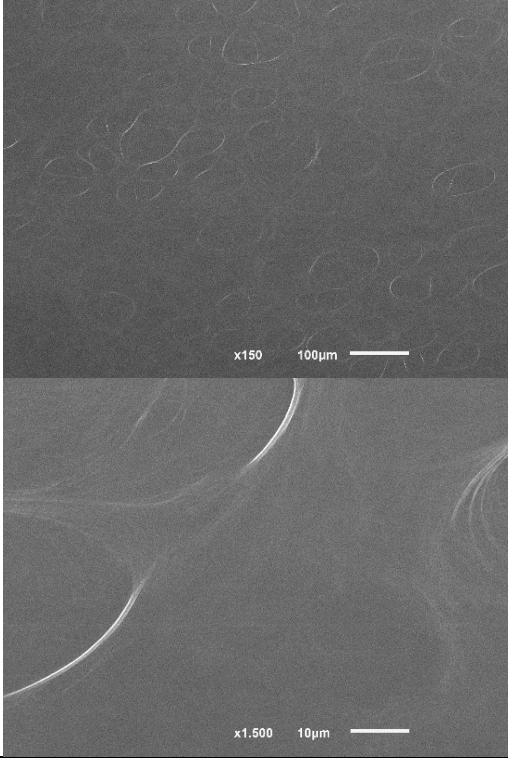
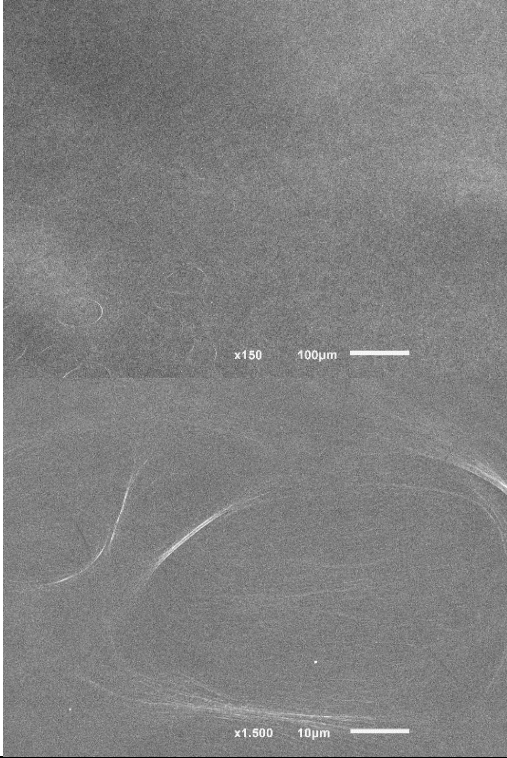
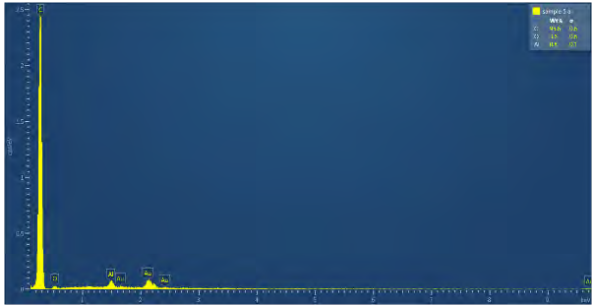
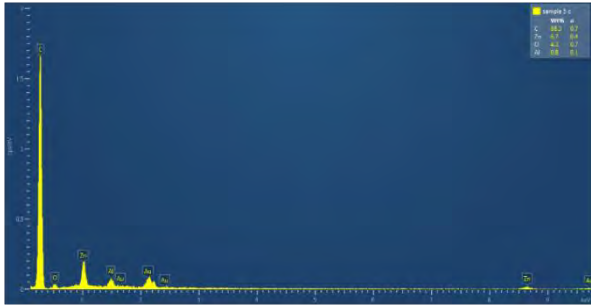
Area	A (between segments)	C (not segmented)
SEM images		
EDS image		

Figure 54- SEM- EDS images obtained in the Sample 5-areas (A) and (C)

Sample 5- Area B

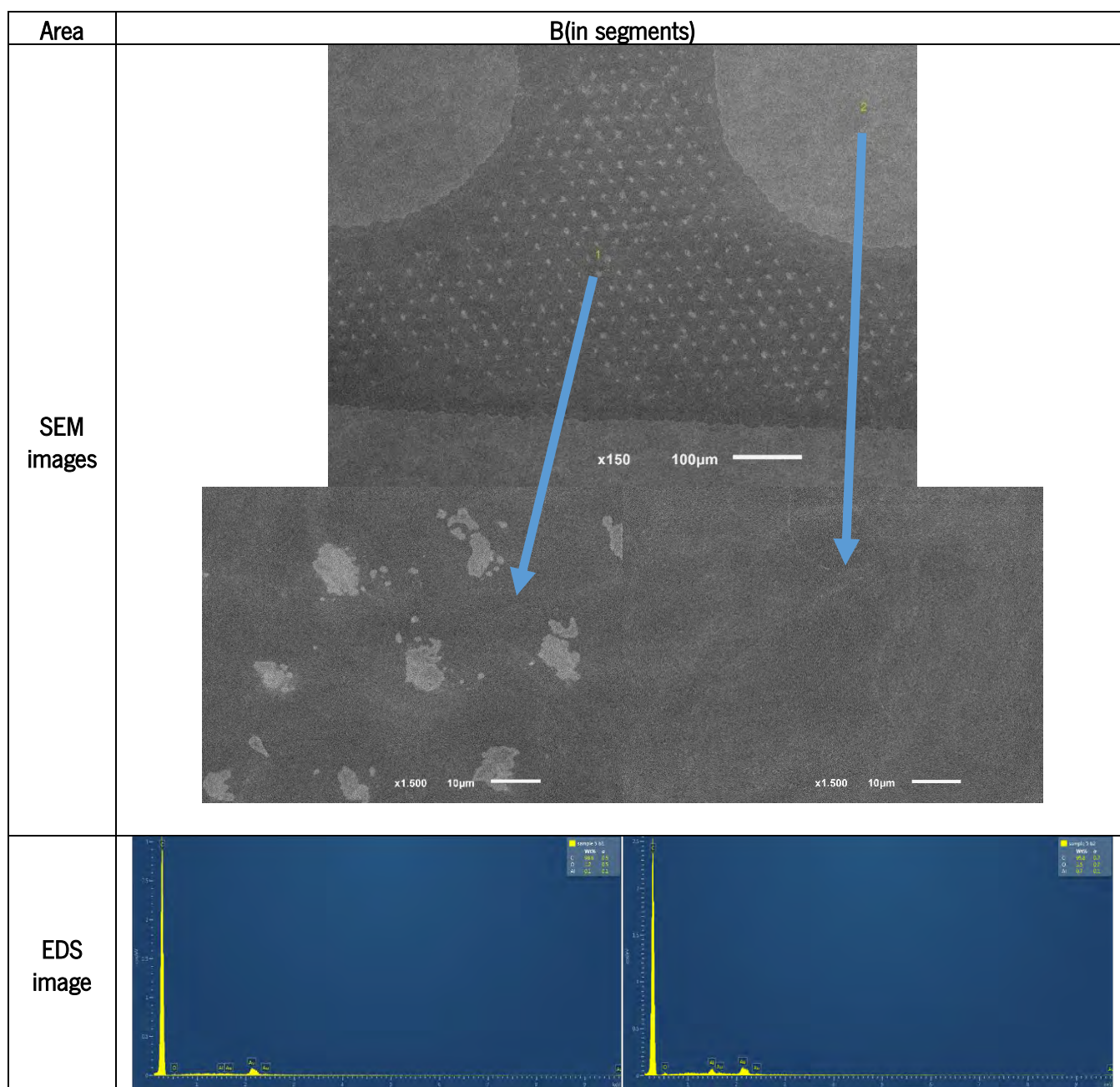


Figure 55- SEM- EDS images obtained in the Sample 5 area (B)

In Figure 55, SEM analyses of the cross sections (B) confirmed uniform distribution of clusters inside the sample.

Table 16- Identification and quantification of each element present in sample 5

Element	Element weight (%)			
	A Area	B1 Area	B2 Area	C Area
C	95,6	98,8	95,8	88,3
Zn	0	0	0	6,7
O	3,6	1,2	3,5	4,2
Al	0,8	0,1	0,7	0,8

Sample 6 - Areas A and C

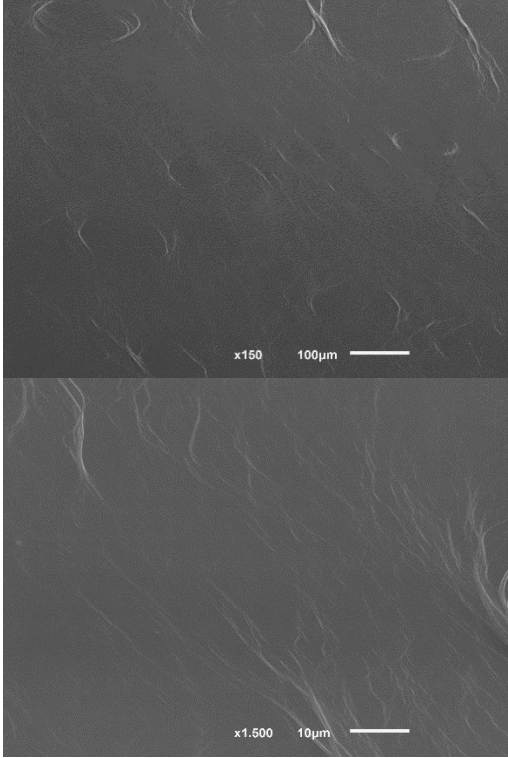
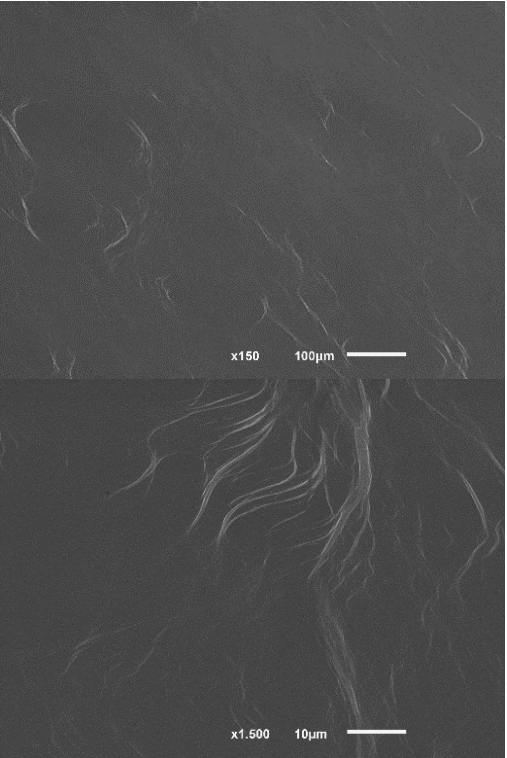
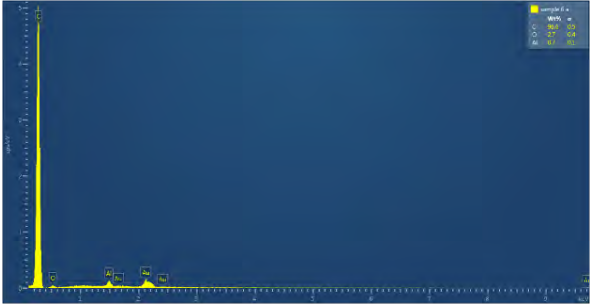
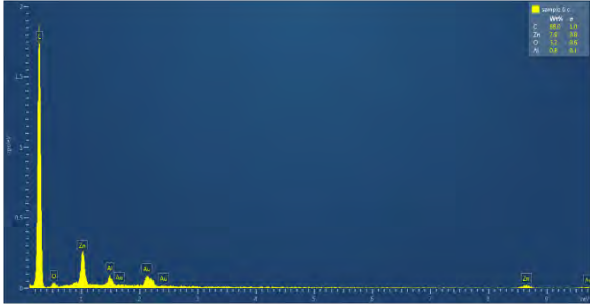
Area	A (between segments)	C (not segmented)
SEM images		
EDS image		

Figure 56- SEM- EDS images obtained in the Sample 6 -areas (A) and (C)

Sample 6- Area B

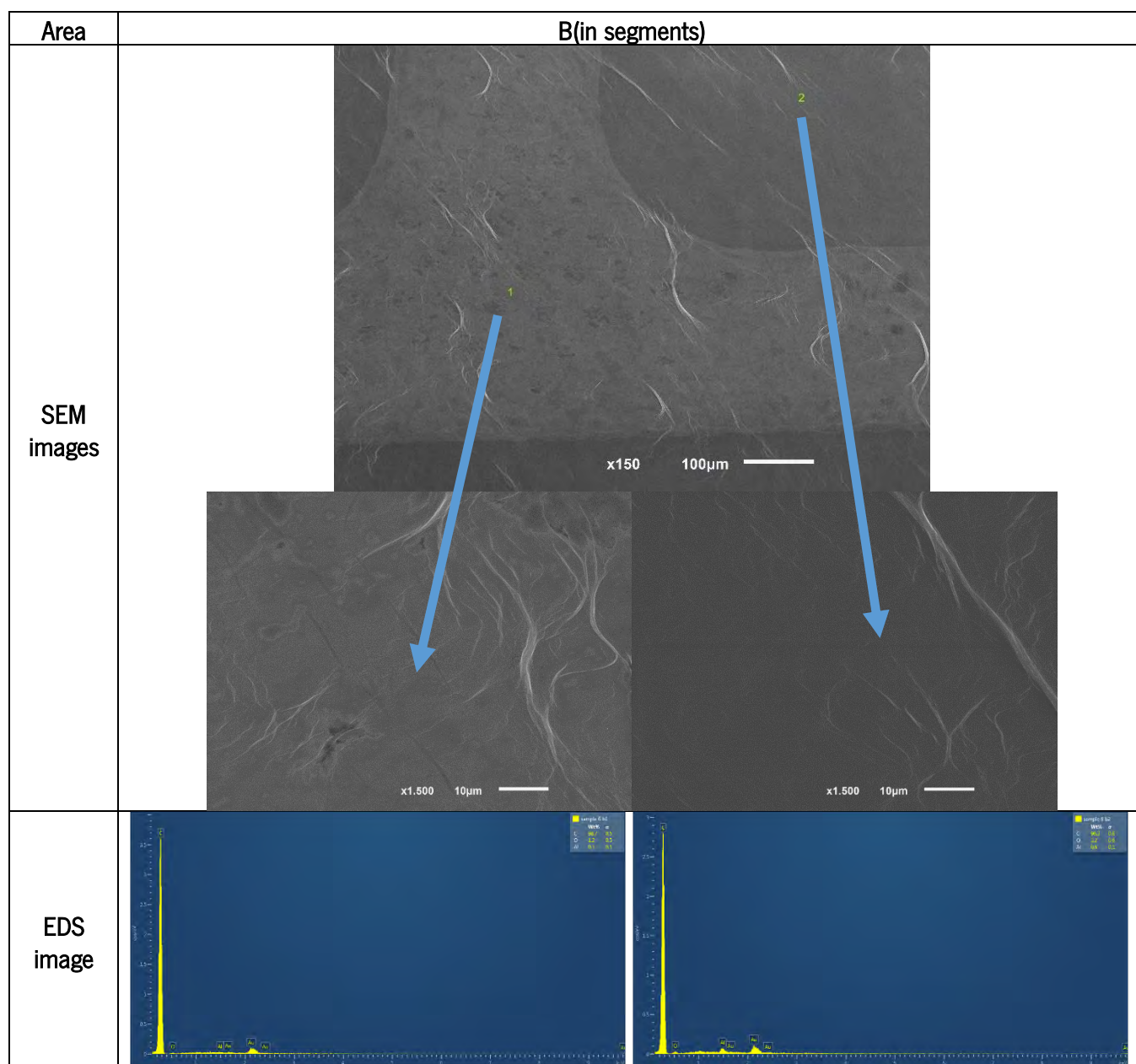


Figure 57- SEM- EDS images obtained in the Sample 6 -area (B)

The high-magnification SEM picture (Figures 56 and 57) show that PP lamellae grow to the fiber axis.

Table 17- Identification and quantification of each element present in sample 6

Element	Element weight (%)			
	A Area	B1 Area	B2 Area	C Area
C	96.6	98.7	96.3	88.0
Zn	0.0	0.0	0.0	7.9
O	2.7	1.2	3.2	3.2
Al	0.7	0.1	0.6	0.8

Considering the Table 18, with the EDS identification and quantification of each element, it can be observed, that sample 1 and sample 2 don't have zinc (Zn) in C area, meaning that they are metallized only with Al. Samples 3 to 6 have Zn in C area, meaning that they were metallized in ZnAl.

Observing the images from the morphological aspect, they suggest that all samples are from different suppliers, as they have different morphological aspect. The same conclusion was observed with DSC and ATR-FTIR techniques, as all samples shown different thermograms and spectrums, respectively.

PP lamellae grow to the fiber axis and they are visible in almost all films, however samples 1 and 2 seems to have more than the other samples. Then appears sample 6 and 3, however the most homogeneous samples seem to be samples 5 and 4.

Considering B area, sample 4 has almost no PP lamellae in B1 area and sample 5 has no PP lamellae in B1 and B2 areas.

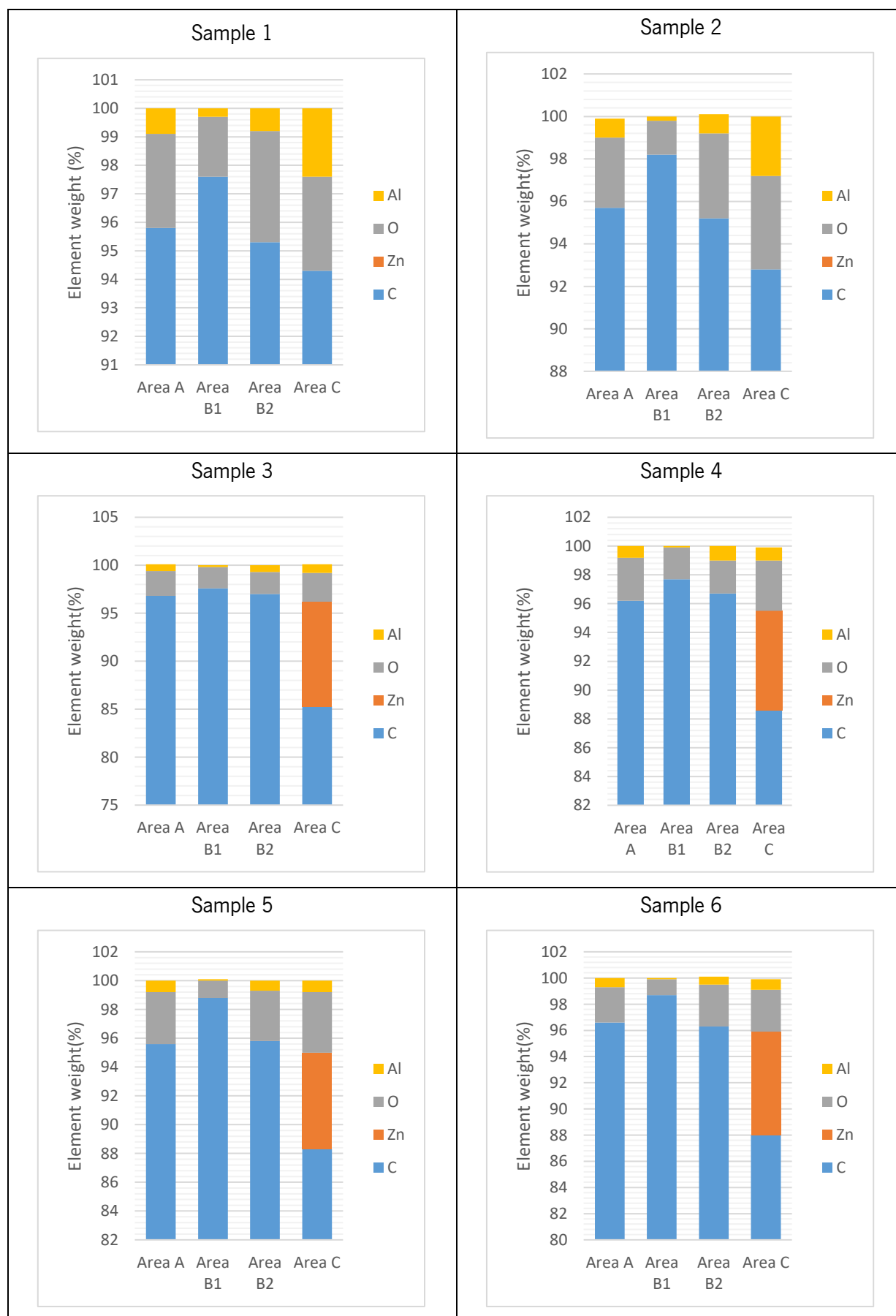
Samples 4 and 5 show a special treatment or process in area B. This difference can give an indication about different metallization processes. It is interesting to observe B1 area, specially sample 5, as it has clusters, very well defined and uniform. It would be interesting to check the element composition in this area (dark and white part).

Sample 1 and 2 seem to have the same metallization process as the EDS is very similar. Samples 4 to 6 have the lowest Al percentage in B1 area (0.1%) against samples 2 and 3 (0.2%) and sample 1 (0.3%), which can give an indication about the cleaning of the segments.

Comparing samples 3 to 6 metallized with ZnAl, it can be observed that sample 3 has the highest weight of Zn in C area (10.9%), against the lower quantity of Zn presented in sample 5 (6.7%). Considering the weight of Al in these samples, it is visible that the Al is more uniform in sample 5 in all the extension of the film, from the A area to the C area (0.7% and 0.8%) and almost no Al in B1 area (0.1%). This can suggest that the process is controlled. For example, in sample 4, the Al weight from A area to C area vary between 0.8% and 1.0%.

The deterioration of Al caused by oxidation is not desirable, hence, to prevent the oxidation and improve the humidity resistance of the metallized film it is used Zn [60], [61].

Table 18- Overview of EDS Identification and quantification for each sample in graphs



4.4. Atomic Force Microscopy

Figures 58 to 63 and Tables 19 to 24, present AFM images of each sample area, as well as, the surface roughness values of each sample area, respectively.

Tests were performed in transverse direction of the BOPP film.

- **Sample 1**

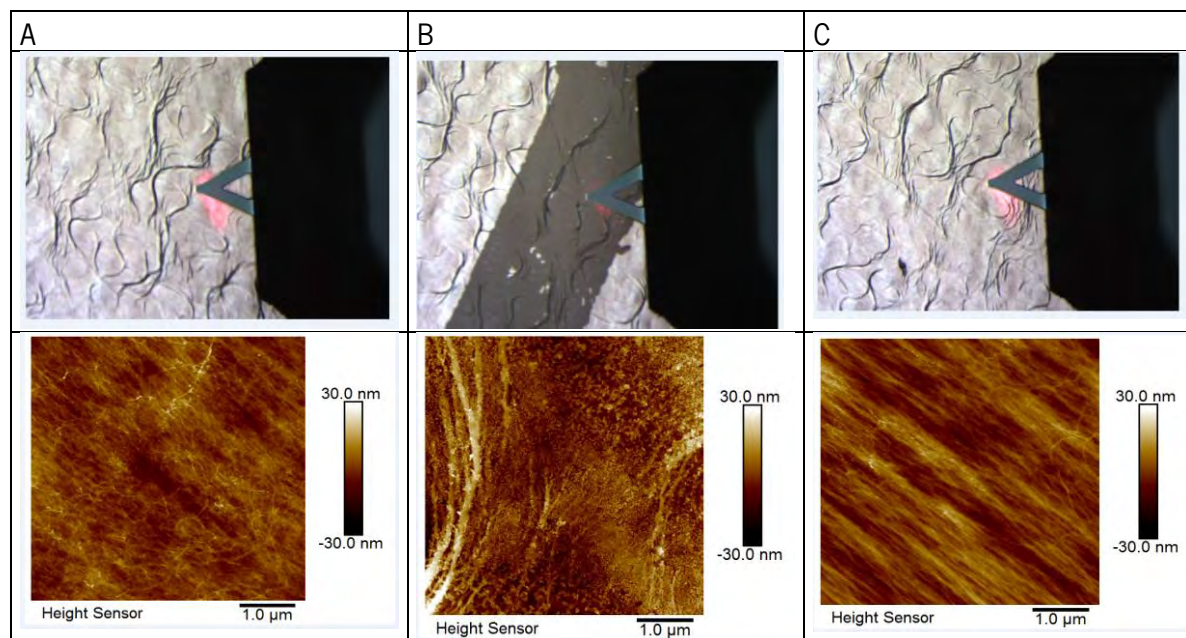


Figure 58- Optical AFM topography images and surface roughness of the sample 1, into three different areas: (A), (B) and (C). Image size: 5×5 μm

Table 19- Surface roughness of the sample 1, into three different areas (A), (B) and (C)

S1	A	B	C
Rq (nm)	4.18	7.91	4.92
Ra(nm)	3.28	6.22	3.98
Rmax (nm)	42.80	70.80	39.40

Sample 2

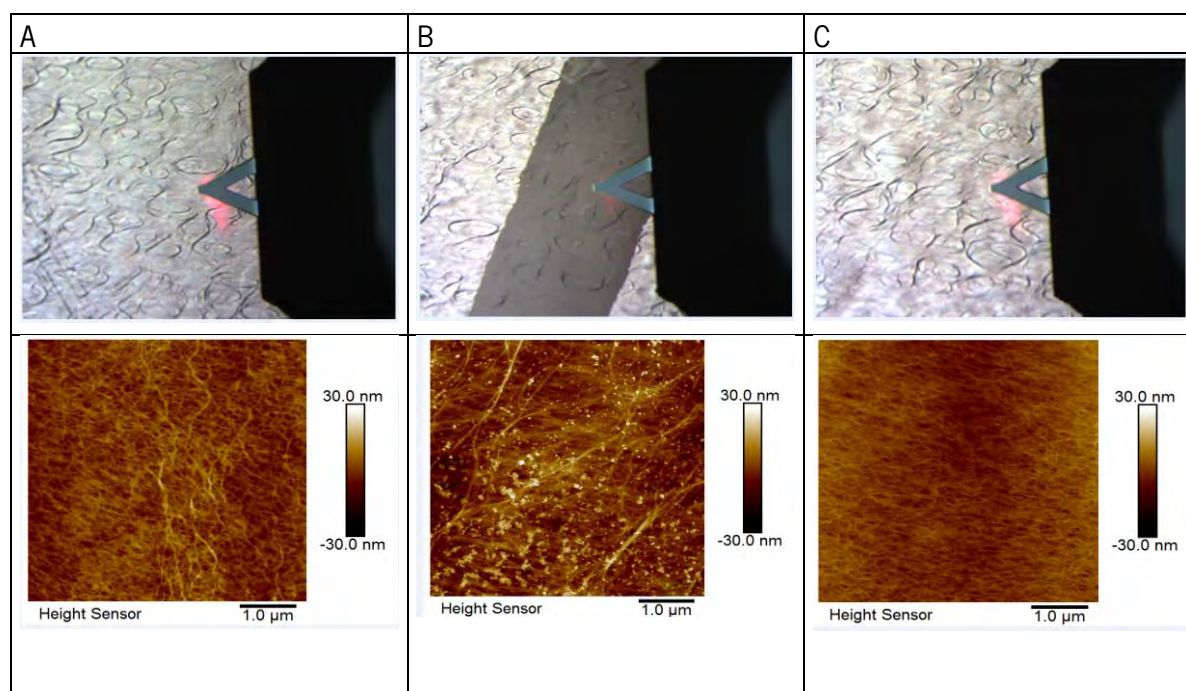


Figure 59- Optical AFM topography images and surface roughness of the sample 2, into three different areas: (A), (B) and (C). Image size: 5×5 μm

Table 20- Surface roughness of the sample 2, into three different areas (A), (B) and (C)

S2	A	B	C
Rq (nm)	3.97	7.37	3.51
Ra(nm)	3.16	6.04	2.83
Rmax (nm)	36.50	61.00	30.70

- Sample 3

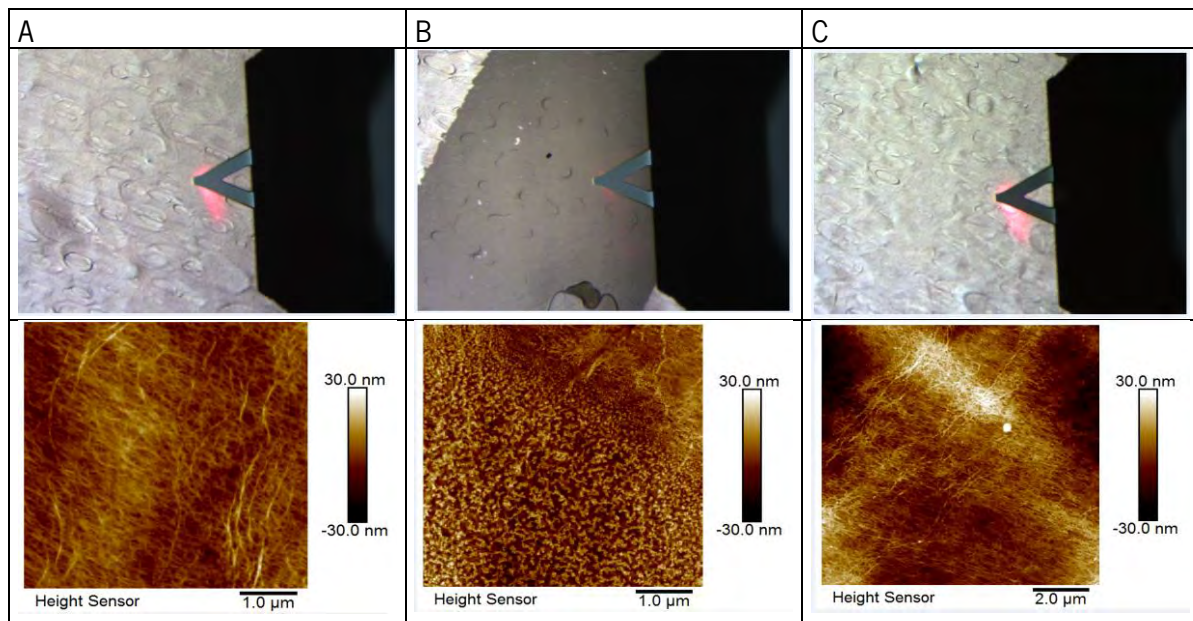


Figure 60 - Optical AFM topography images and surface roughness of the sample 3, into three different areas: (A), (B) and (C). Image size: 5×5 μm

Table 21- Surface roughness of the sample 3, into three different areas (A), (B) and (C)

S3	A	B	C
Rq (nm)	4.78	7.57	9.32
Ra(nm)	3.82	6.22	7.23
Rmax (nm)	46.60	55.80	91.00

- Sample 4

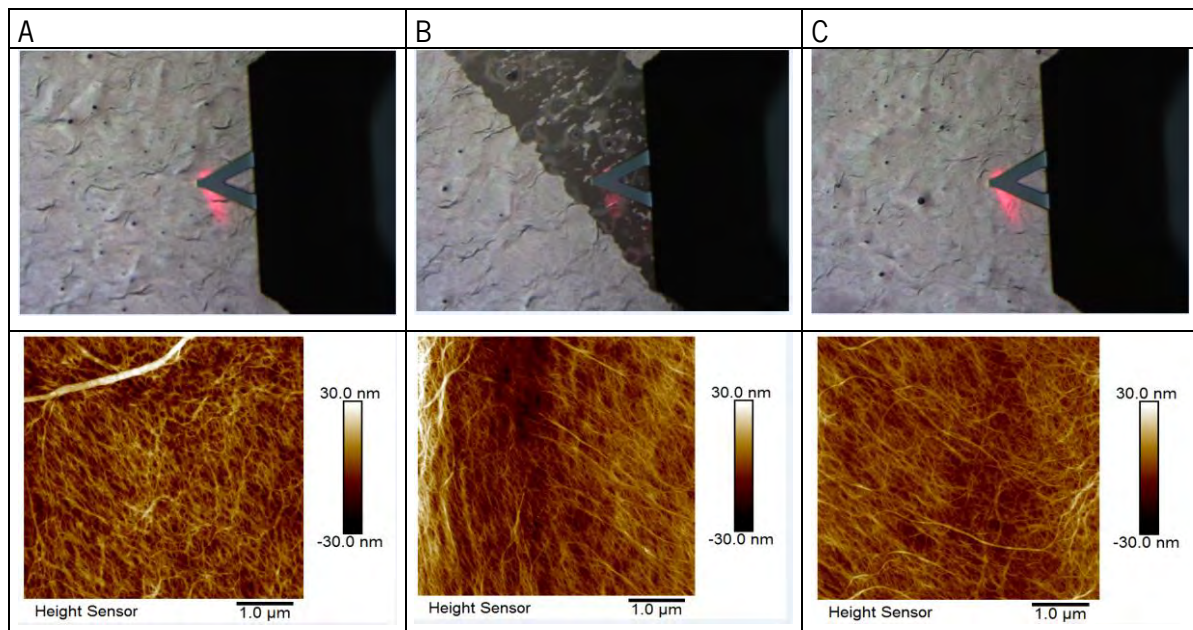


Figure 61- Optical AFM topography images and surface roughness of the sample 4, into three different areas: (A), (B) and (C). Image size: 5×5 μm

Table 22- Surface roughness of the sample 4, into three different areas (A), (B) and (C)

S4	A	B	C
Rq (nm)	6.46	7.28	4.83
Ra(nm)	4.93	5.54	3.79
Rmax (nm)	64.50	71.20	45.50

- Sample 5

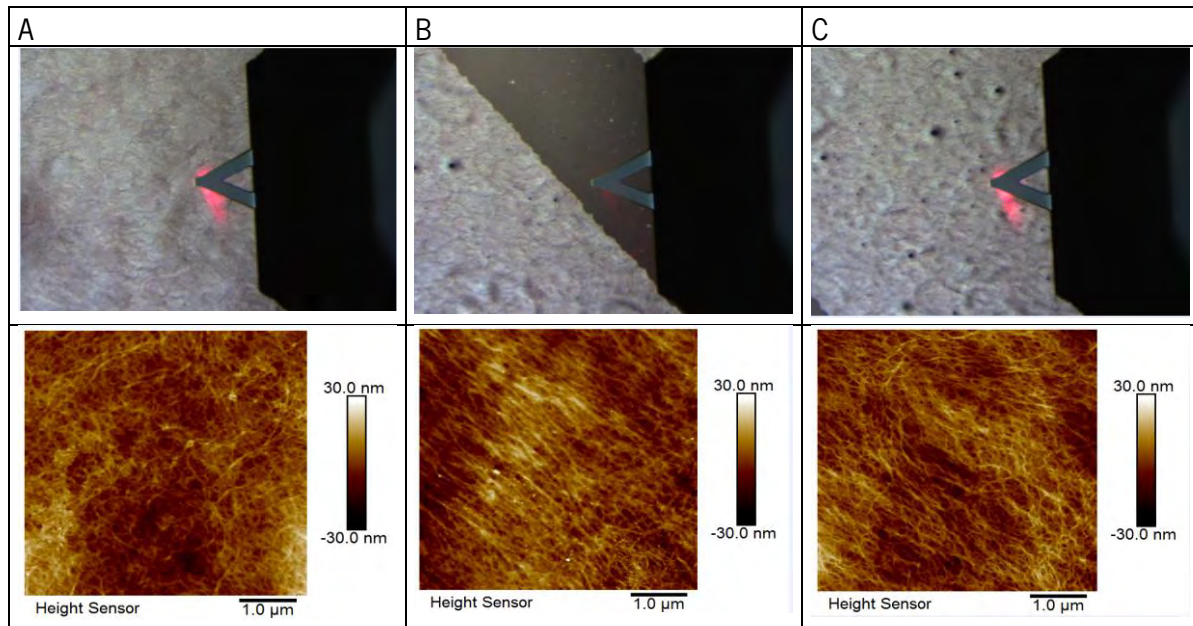


Figure 62- Optical AFM topography images and surface roughness of the sample 5, into three different areas: (A), (B) and (C). Image size: 5×5 μm

Table 23- Surface roughness of the sample 5, into three different areas: (A), (B) and (C)

S5	A	B	C
Rq (nm)	5.82	6.02	5.72
Ra(nm)	4.56	4.81	4.55
Rmax (nm)	47.60	58.20	49.10

- Sample 6

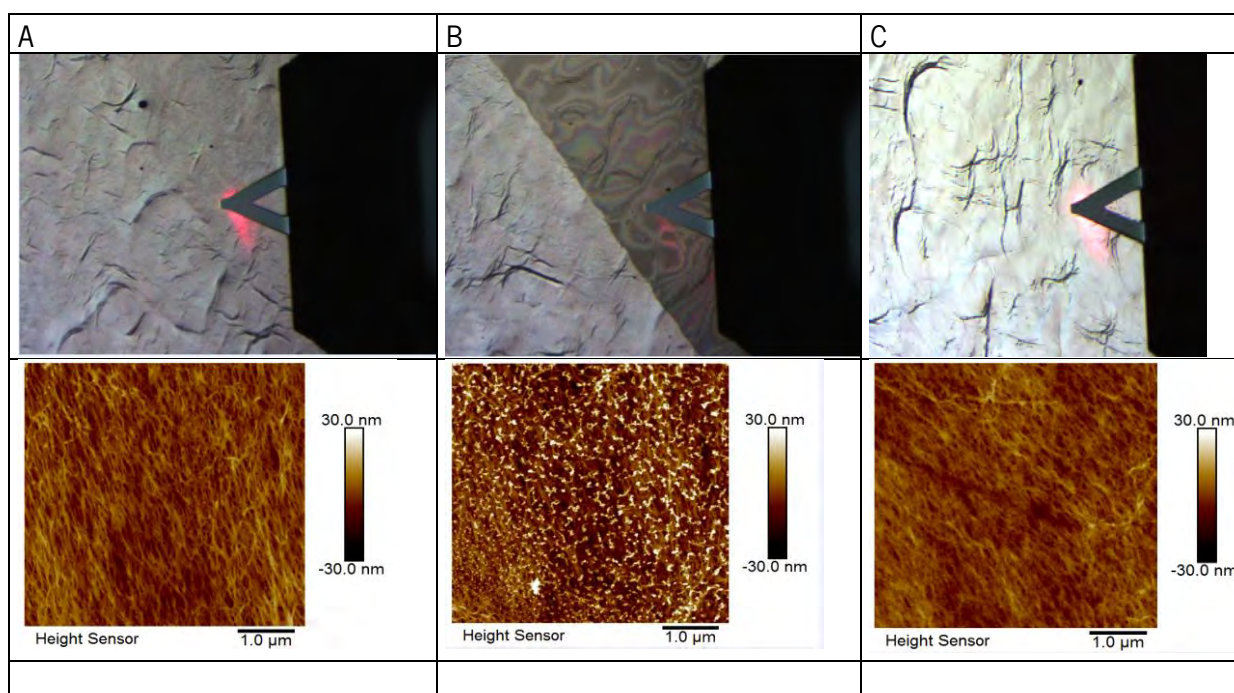


Figure 63- Optical AFM topography images and surface roughness of the sample 6, into three different areas: (A), (B) and (C). Image size: 5×5 μm

Table 24- Surface roughness of the sample 6, into three different areas (A), (B) and (C)

S6	A	B	C
Rq (nm)	4.71	9.32	4.03
Ra(nm)	3.74	7.10	3.19
Rmax (nm)	41.40	87.40	35.70

Considering the roughness surface results of all samples and analyzing the below Figures 64 and 65, with the interaction for Rq, it is possible to observe that the Rq values can vary between 3nm and 10nm.

Sample 2 has the lowest Rq value on A and C areas. Sample 5 has no big differences between A/B/C areas, values are very similar between the three areas. Sample 3 has an increase on Rq from A to C area.

B area has the highest Rq values, for all samples.

Considering the A area, sample 4 has the higher Rq. Sample 6 shows the highest Rq for B area, while sample 3 has the higher Rq in C area.

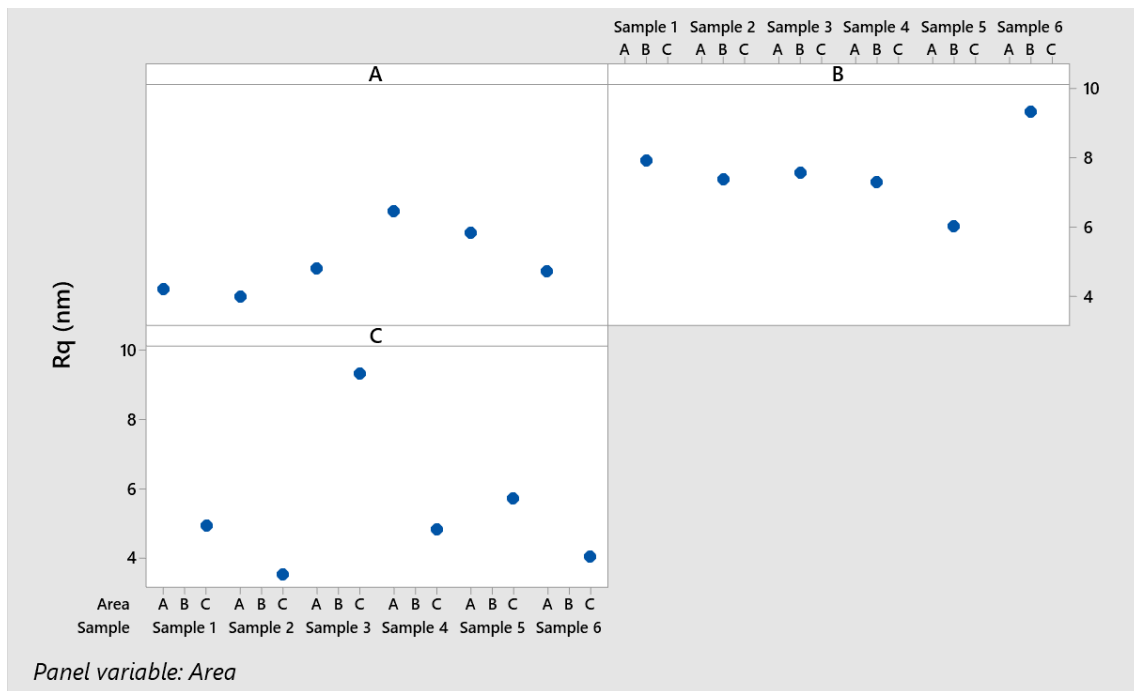


Figure 64- Roughness surface: individual Rq values per sample and per area

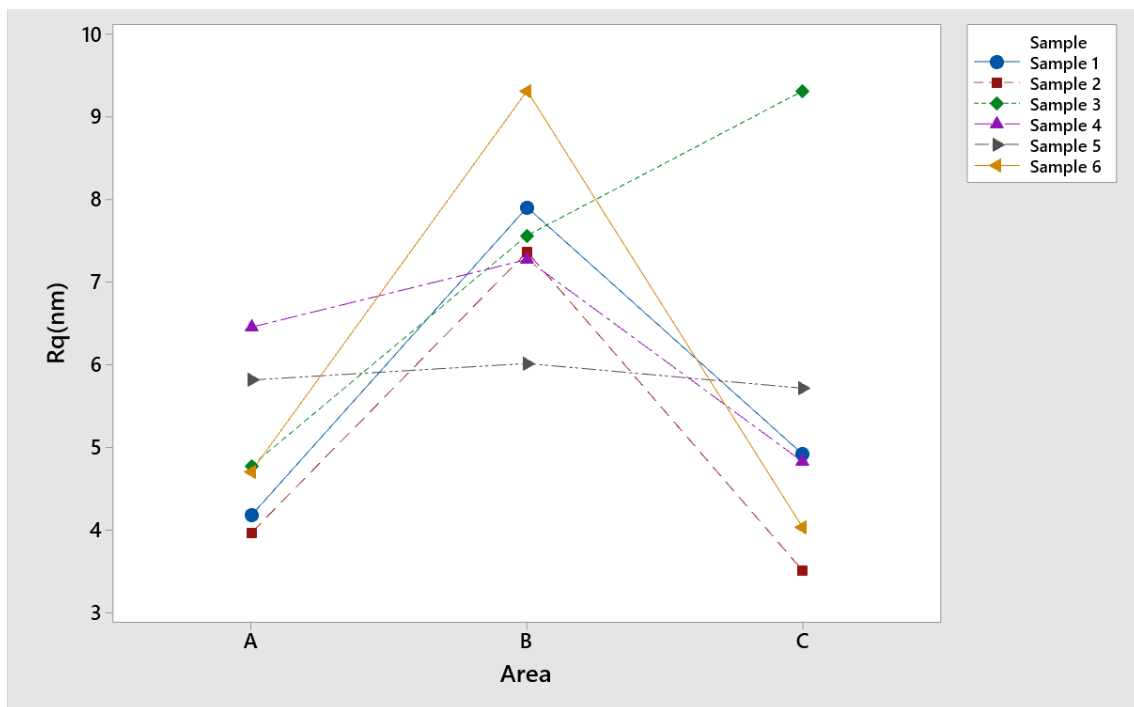


Figure 65- Roughness surface: overview of Rq values per sample and per area

Considering the roughness surface results from all samples and analyzing the following Figures 66 and 67, with the interaction for Ra of each area, it is possible to observe that the Ra values can vary between 3nm and 7nm.

Sample 2 has the lowest Ra value on A and C areas. Sample 5 has no big differences between A/B/C areas, values are very similar between the three areas. Sample 3 has an increase on Ra from A to C area.

B area has the highest Ra values, for all samples.

Considering the A area, sample 4 has the higher Ra. Sample 6 shows the highest Ra for B area, while sample 3 has the higher Ra in C area.

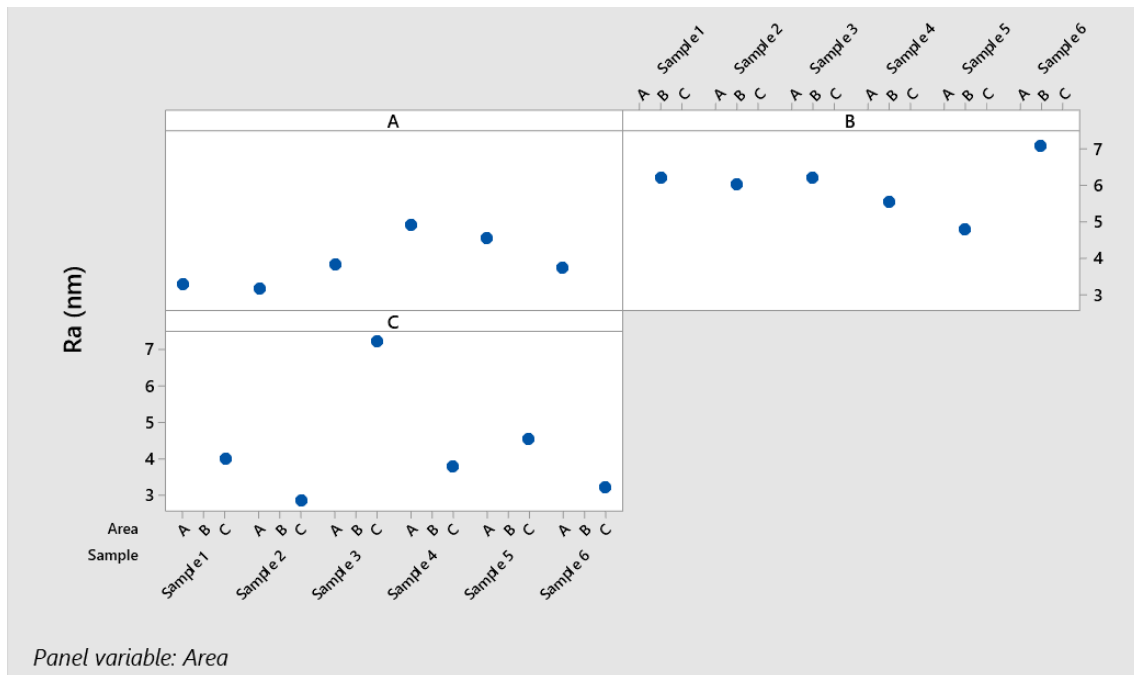


Figure 66- Roughness surface: individual Ra values per sample and per area

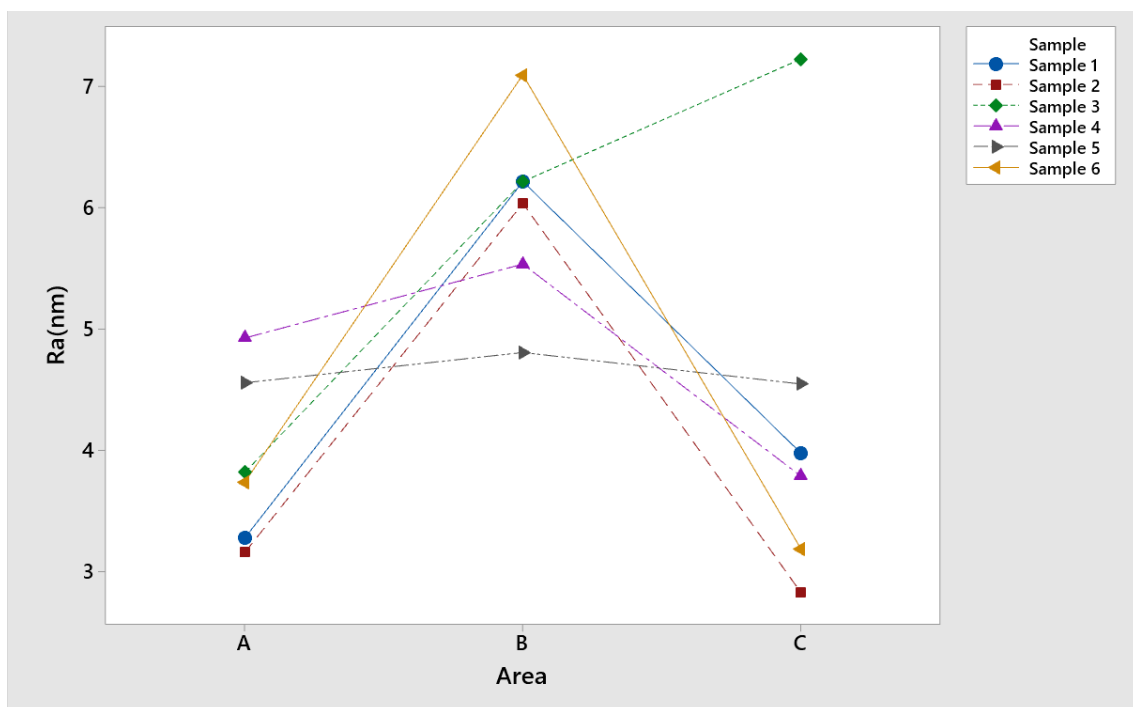


Figure 67- Roughness surface: overview of Ra values per sample and per area

Same behavior, as Rq and Ra, is observed for Rmax, as shown in Figures 68 and 69.

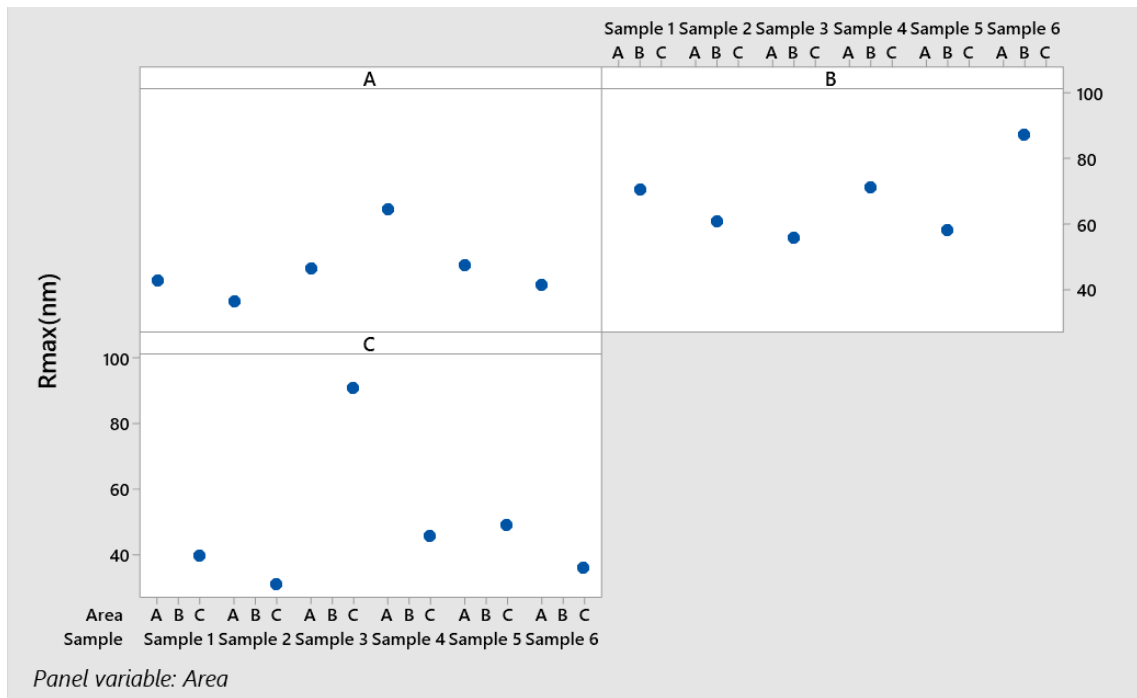


Figure 68- Roughness surface: individual Rmax values per sample and per area

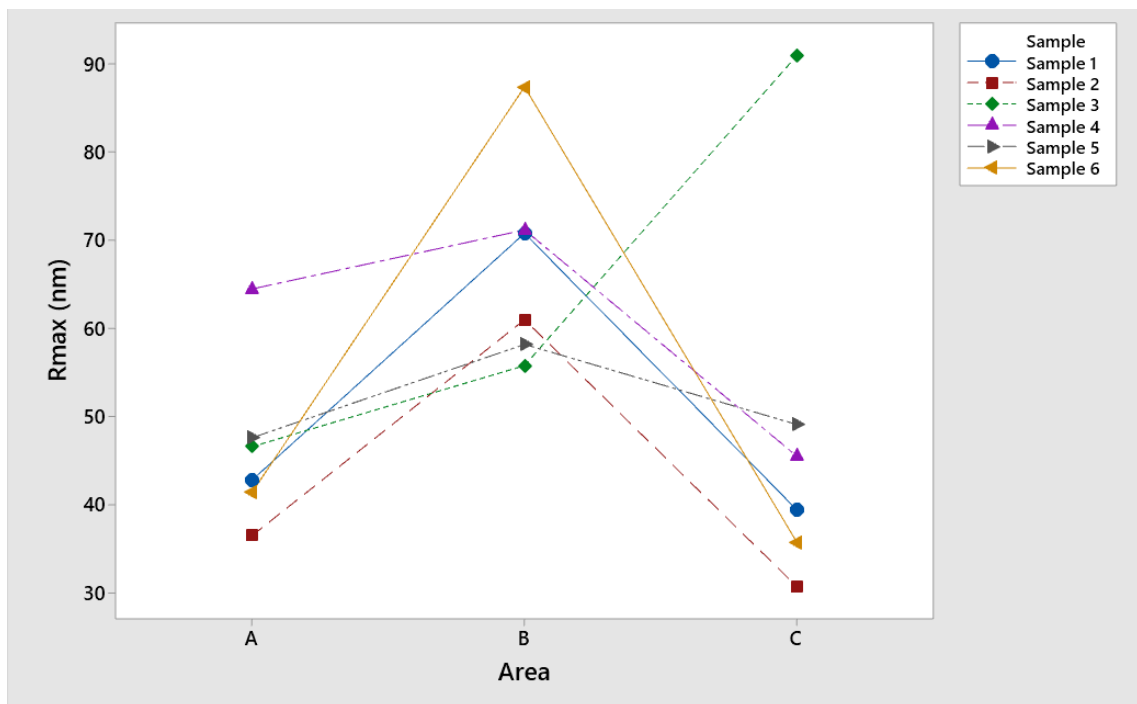


Figure 69- Roughness surface: overview of Rmax values per sample and per area

Surface roughness improves the adhesion from the thin metal spray layer (metallization) to the iPP, and the contact can affect the dielectric and the physical strengths. In general, comparing all samples, sample 5 is the one which presents a more constant and uniform roughness in all the

extension of the film (around 4.6nm for Ra; 6.0nm for Rq and 55 Rmax). Roughness is needed in a PP film, but high roughness is not desirable, as this can lead in electrical failures. Considering the results seems that a constant Ra among all film, of around 4,6nm is desirable for a metallized PP film.

Analyzing AFM and SEM results, sample 5 is the most homogeneous sample in both techniques, this can be explain by the possible simultaneous stretching of the sample which lead to fiber orientation in transverse and longitudinal directions.

4.5. Mechanical tests

From Table 25 to Table 31, it is presented the results of the mechanical tests obtained for sample 1 to 6, respectively, including the average and the standard deviation. All tests were performed in transverse direction of the film.

The Table 25 show the results in sample 1, for each mechanical test.

Table 25- Mechanical results obtained for sample 1

Specimen	Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	3124.04	32.19	126.71	48.13	48.13
2	3588.76	38.77	95.83	21.86	21.85
3	3651.71	36.79	127.03	40.73	40.73
4	3983.11	42.88	149.24	53.73	53.73
5	3814.16	43.44	98.00	21.16	21.16
6	3188.59	41.47	92.87	22.92	22.92
7	3787.97	40.78	137.42	50.00	50.00
$\bar{x}$	3591.19	39.47	118.16	36.93	36.93
s	323.01	3.95	22.48	14.52	14.52

Figure 70 presents the strain-stress curve for the 7 specimens of sample 1.

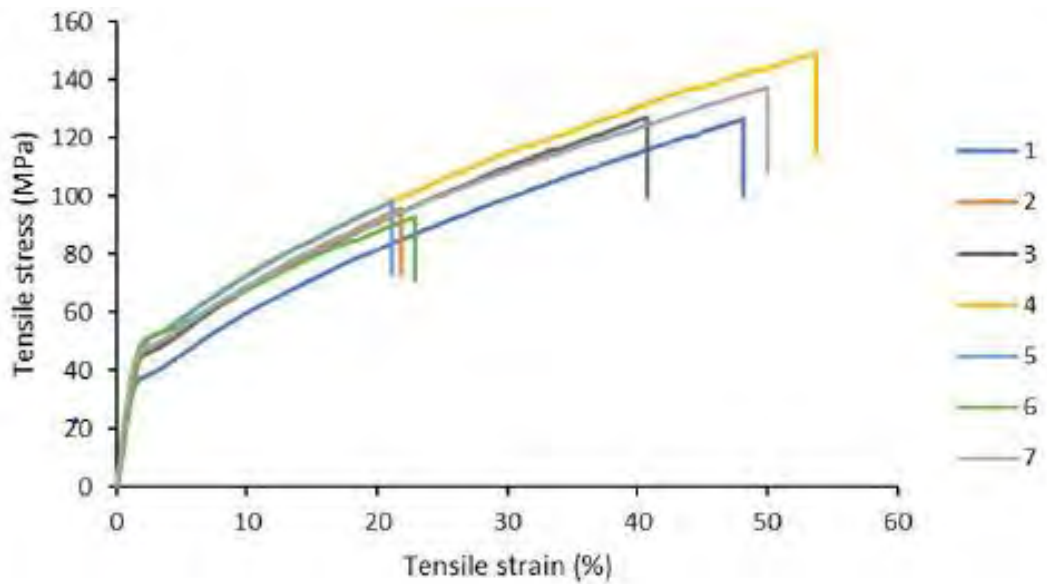


Figure 70- Tensile strain vs tensile stress graph for specimen of sample 1

The Table 26 shows the results in sample 2, for each mechanical test.

Table 26- Mechanical results obtained for sample 2

Specimen	Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	3335.00	34.57	66.92	18.50	18.50
2	3629.26	34.71	138.26	87.03	87.03
3	3033.38	34.62	135.53	88.24	88.24
4	3286.39	39.70	124.48	67.70	67.70
5	3155.33	35.26	128.26	81.49	81.49
6	2906.95	35.94	125.10	78.56	78.53
7	2597.86	34.55	80.50	31.20	31.20
$\bar{x}$	3134.88	35.62	114.15	64.67	64.67
s	331.28	1.87	28.36	28.26	28.26

Figure 71 presents the strain-stress curve for the 7 specimens of sample 2.

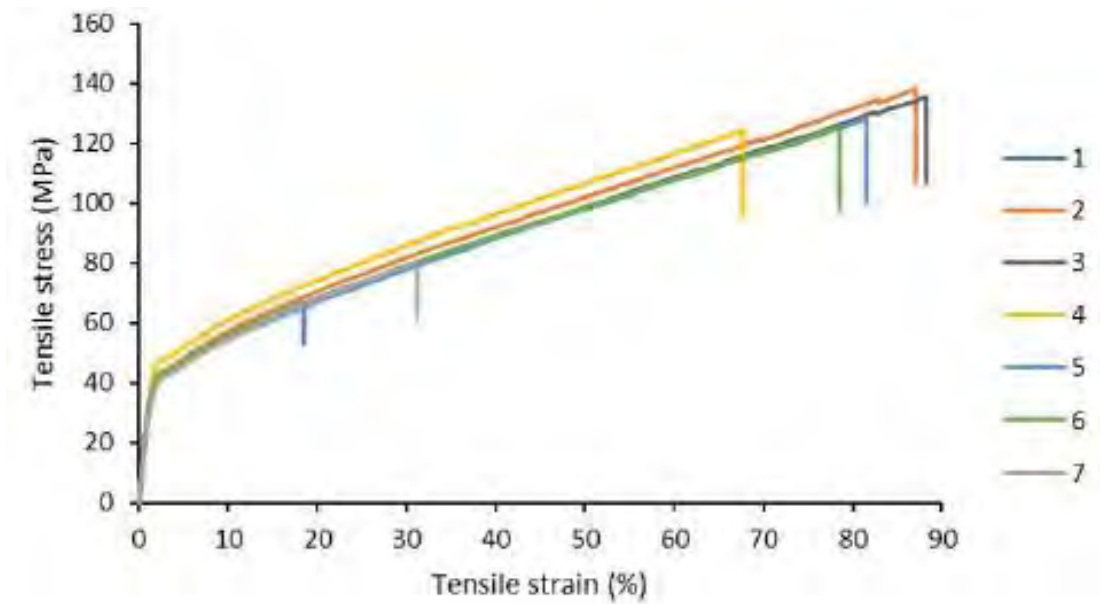


Figure 71- Tensile strain vs tensile stress graph for specimen of sample 2

The Table 27 shows the results in sample 3, for each mechanical test.

Table 27- Mechanical results obtained for sample 3

Specimen	Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	2958.44	26.95	61.22	30.83	30.83
2	2638.43	34.9	94.54	71.3	71.29
3	3453.38	31.37	81.75	50.69	50.66
4	3174.76	35.43	113.02	90.83	90.83
5	3185.16	27.31	102.94	90.86	90.86
6	3498.51	40.6	136.71	109.13	108.78
7	2915.1	28.89	115.26	104.95	104.92
$\bar{x}$	3117.68	32.21	100.78	78.37	78.31
s	305.95	5.02	24.57	28.98	28.92

Figure 72 presents the strain-stress curve for the 7 specimens of sample 3.

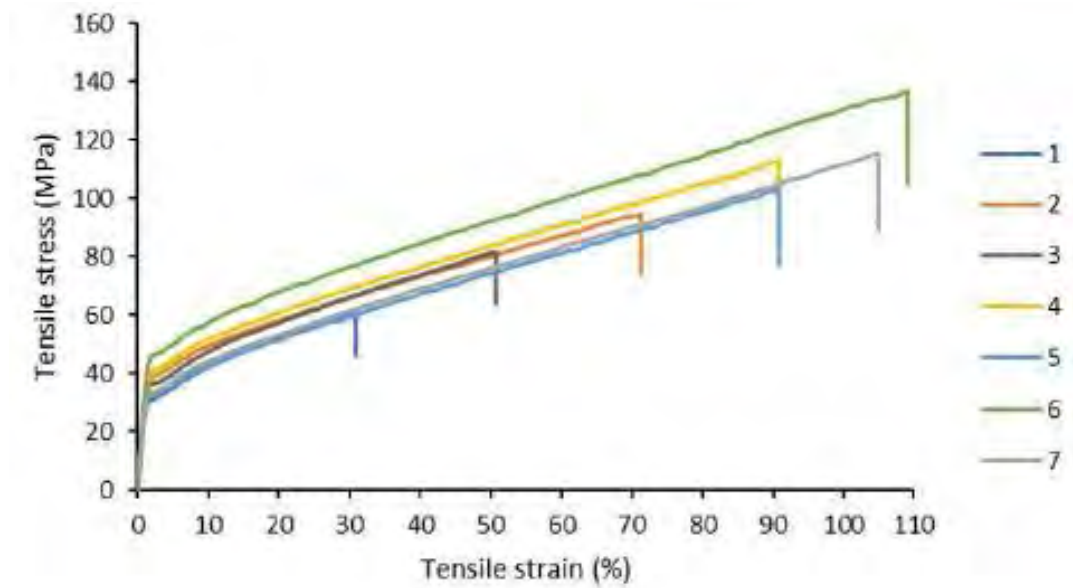


Figure 72- Tensile strain vs tensile stress graph for specimen of sample 3

The Table 28 shows the results in sample 4, for each mechanical test.

Table 28- Mechanical results obtained for sample 4

Specimen	Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	4000.51	41.03	65.2	13.41	13.39
2	3635.76	26.87	48.92	12.89	12.87
3	3464.92	44.45	153.2	105.87	105.87
4	3526.07	33.08	66.64	25.15	25.15
5	3128.08	39.71	61.56	14.32	14.32
6	3901.33	32.37	67.19	24.42	24.41
7	3799.9	32.37	80.15	37.96	37.94
$\bar{x}$	3636.65	35.70	77.55	33.43	33.42
s	297.06	6.17	34.60	33.18	33.18

Figure 73 presents the strain-stress curve for the 7 specimens of sample 4.

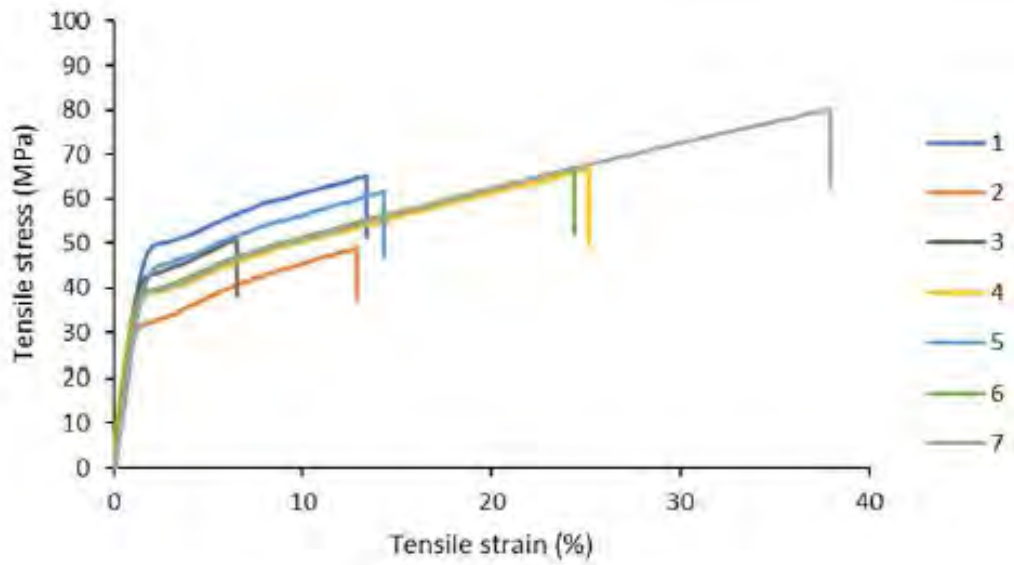


Figure 73- Tensile strain vs tensile stress graph for specimen of sample 4

The Table 29 shows the results in sample 5, for each mechanical test.

Table 29- Mechanical results obtained for sample 5

Specimen	Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	3979.96	38.38	64.79	8.18	8.16
2	3581.4	36.75	123.07	37.79	37.79
3	3644.13	28.81	78.78	20.16	20.15
4	3935.7	36.69	126.15	40.18	40.18
5	4487.97	44.37	125.22	32.69	32.69
6	3925.9	31.98	111.73	32.84	32.82
7	3762.43	46.26	163.88	57.71	57.7
$\bar{x}$	3902.50	37.61	113.37	32.79	32.78
s	299.97	6.22	32.93	15.63	15.63

Figure 74 presents the strain-stress curve for the 7 specimens of sample 5.

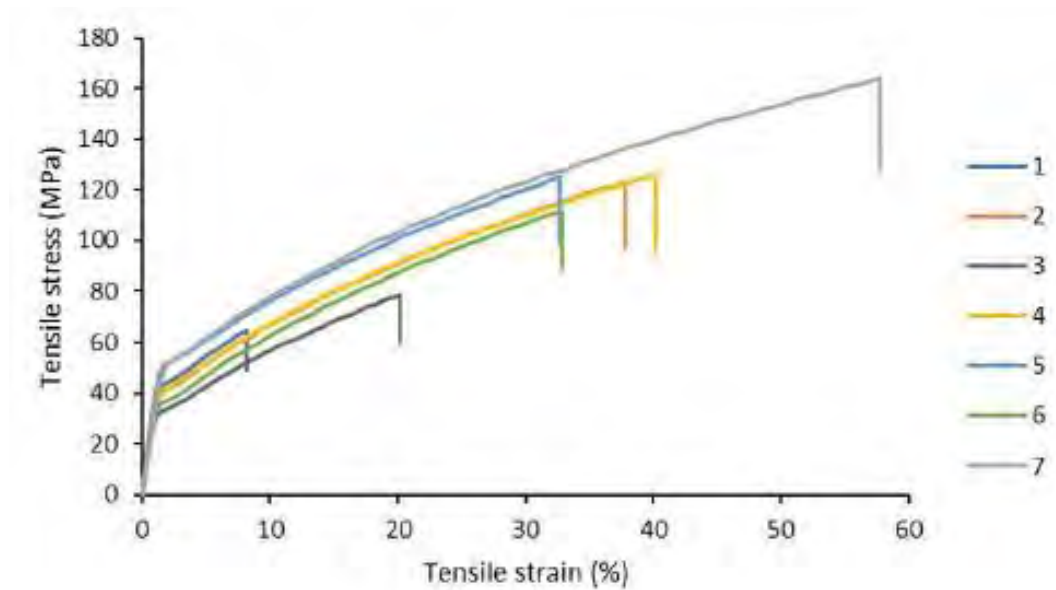


Figure 74- Tensile strain vs tensile stress graph for specimen of sample 5

The Table 30 shows the results in sample 6, for each mechanical test.

Table 30- Mechanical results obtained for sample 6

Specimen	Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	3578.22	34.71	56.08	11.56	11.52
2	3658.58	38.81	124.92	80.57	80.57
3	3409.16	37.91	119.22	79.98	79.98
4	3076.15	32.24	55.99	18.73	18.73
5	3699.15	35.72	100.76	54.61	54.61
6	3454.75	33.67	77.5	36.46	36.45
7	3176.68	32.63	96.06	56.36	56.36
$\bar{x}$	3436.10	35.10	90.08	48.32	48.32
s	236.93	2.53	27.96	27.43	27.44

Figure 75 presents the strain-stress curve for the 7 specimens of sample 6.

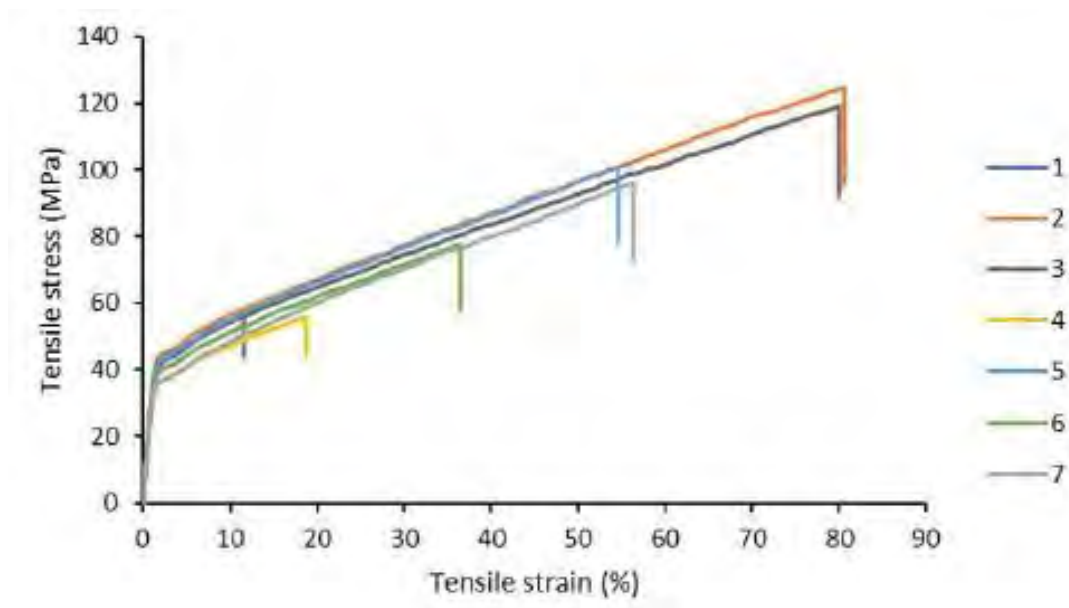


Figure 75- Tensile strain vs tensile stress graph for specimen of sample 6

An overview of the average and standard deviation of each sample is presented in the Table 31.

Table 31- Average ($\bar{x}$) and standard deviation (s) overview for each sample

Sample	Vishay Code nr.		Tensile Modulus or Young Modulus (MPa)	Tensile stress at Yield (Offset 0.2 %) (MPa)	Tensile strength (MPa)	Strain at maximum load or elongation at break (%)	Strain at break (%)
1	80920622	$\bar{x}$	3591.19	39.47	118.16	36.93	36.93
		s	323.01	3.95	22.48	14.52	14.52
2	80920625	$\bar{x}$	3134.88	35.62	114.15	64.67	64.67
		s	331.28	1.87	28.36	28.26	28.26
3	80921546	$\bar{x}$	3117.68	32.21	100.78	78.37	78.31
		s	305.95	5.02	24.57	28.98	28.92
4	80933978	$\bar{x}$	3636.65	35.70	77.55	33.43	33.42
		s	297.06	6.17	34.60	33.18	33.18
5	80932744	$\bar{x}$	3902.50	37.61	113.37	32.79	32.78
		s	299.97	6.22	32.93	15.63	15.63
6	80920623	$\bar{x}$	3436.10	35.10	90.08	48.32	48.32
		s	236.93	2.53	27.96	27.43	27.44

Figures 76 to 78 show the variation and the comparison of all samples.

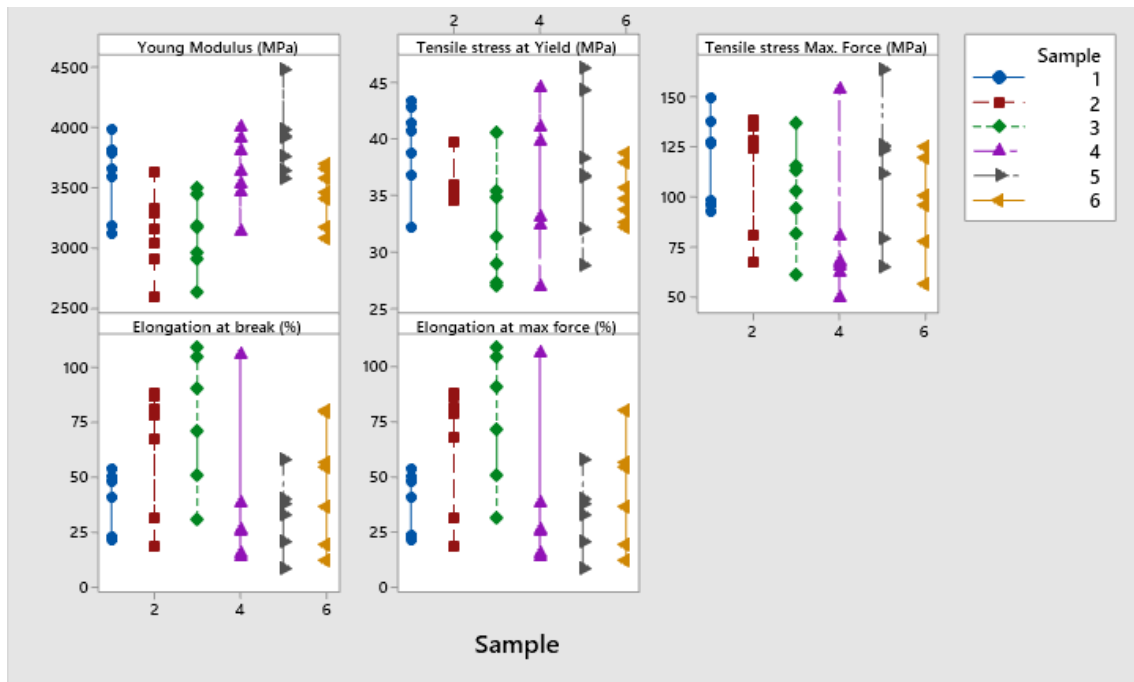


Figure 76- Overview of all characteristics performed in mechanical tests. per sample

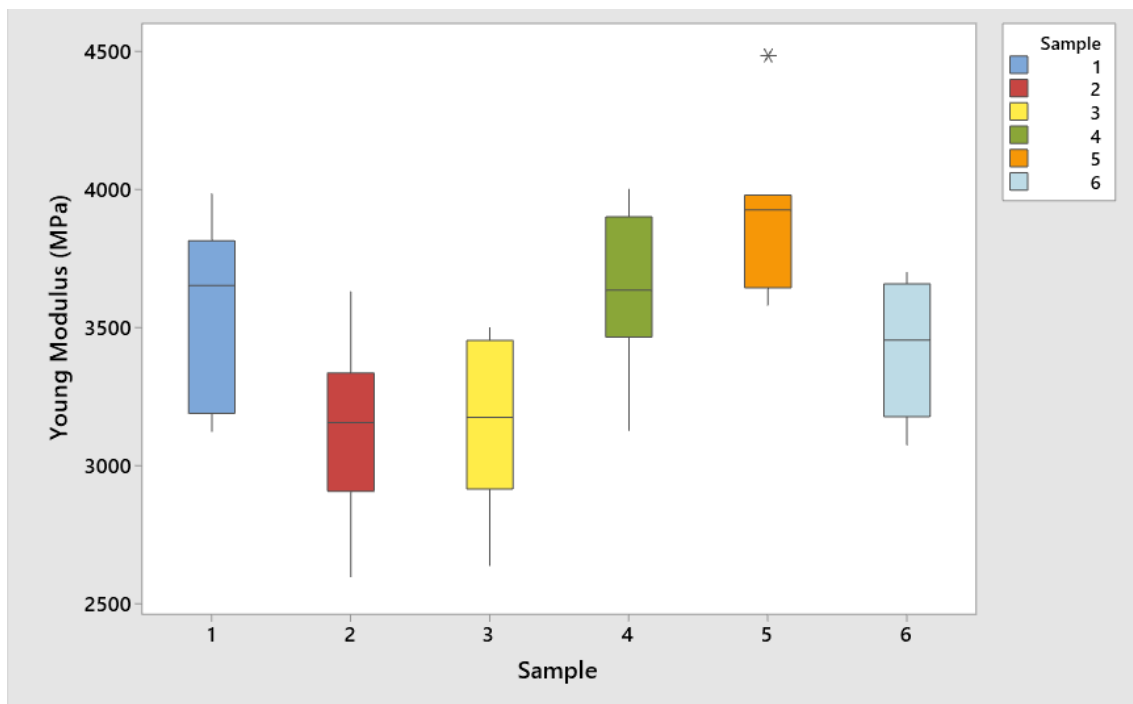


Figure 77- Young Modulus obtained per sample

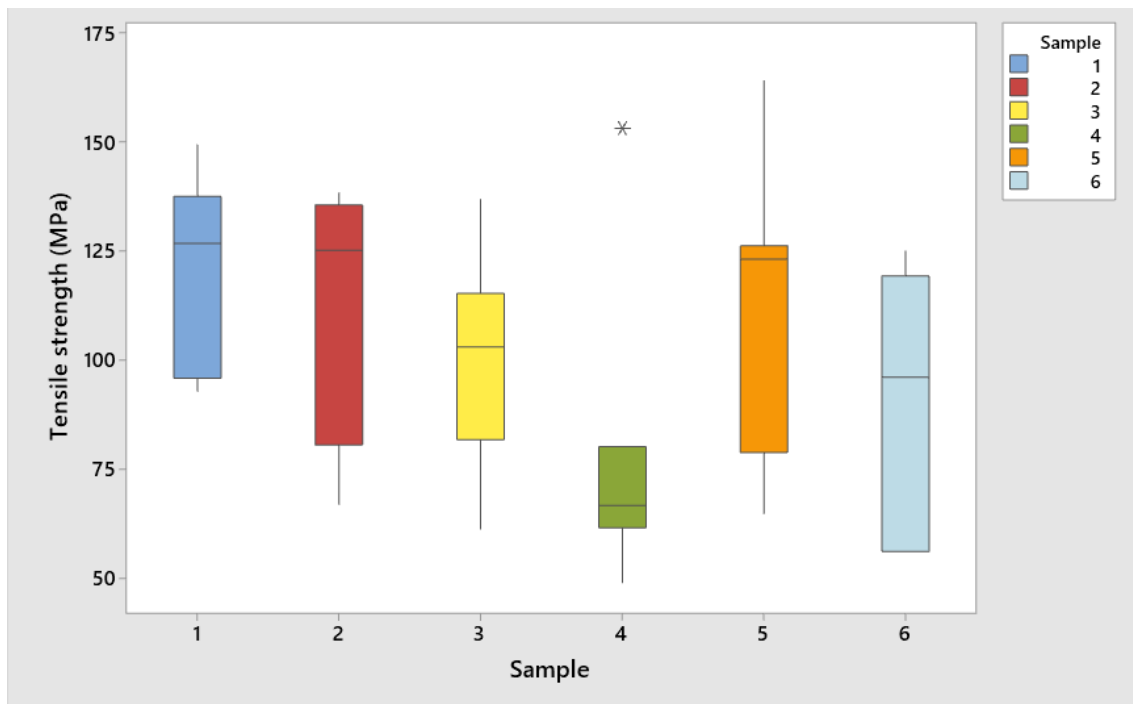


Figure 78- Tensile strength obtained per sample

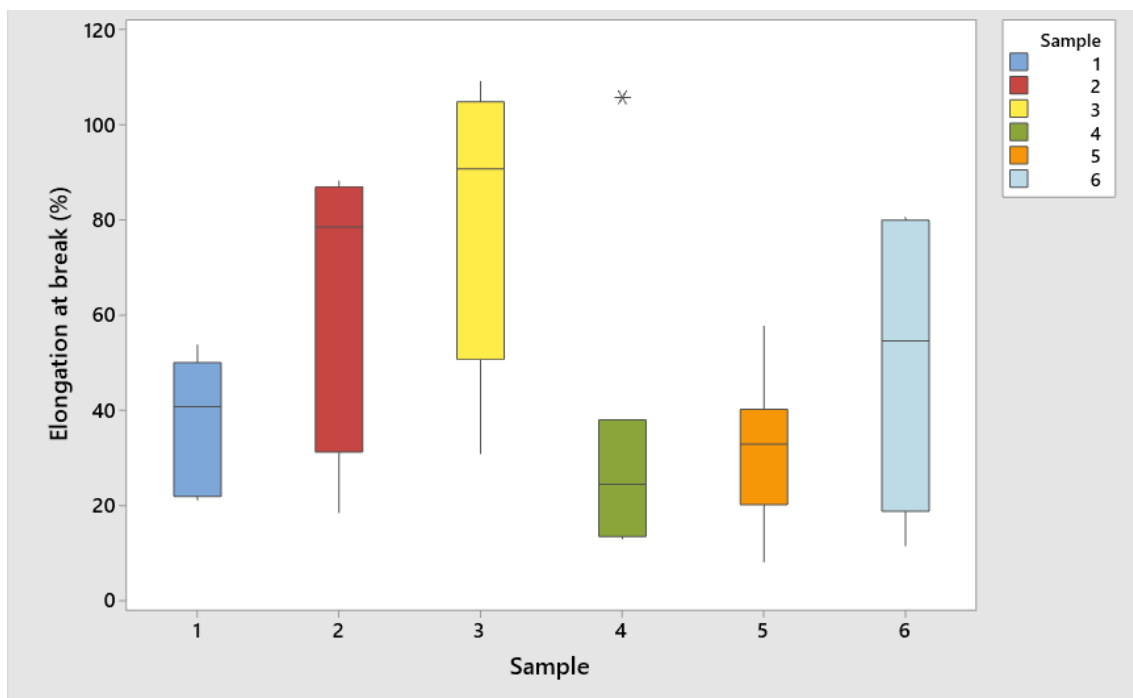


Figure 79- Elongation at Break obtained per sample

High Young Modulus is desirable, as it prevents deformations and cause less stretching, which in turn, can cause wrinkles and problems in capacitance of a capacitor. The higher the Young Modulus the less the material deforms and the higher the degree orientation. It depends from the crystal

orientation of the material. High elongation at break and high tensile strength leads to high toughness and a ductile material [62].

Samples 1, 4 and 5 have the highest Young Modulus, verified by the highest degree of crystallinity, as shown with DSC results, with program 1. Moreover, they have the lowest elongation at break. Samples 1, 2 and 5 have the highest tensile strength values. Sample 3 has the lowest Young Modulus, high tensile strength and the highest elongation at break values. The crystallinity and the thickness of the samples can explain the results obtained.

4.6. Shrinkage

Regarding the shrinkage of films, in machine direction, results are presented in Table 32, using Equation 9 and considering 0% of shrinkage at 25°C.

Table 32- Measurement, in percentage, of the length of each film

Sample	Temperature			
	25 °C	85 °C	105 °C	120 °C
1- 80920622	0%	-0,95%	-2,08%	-3,34%
2- 80920625	0%	-0,81%	-1,57%	-2,71%
3- 80921546	0%	-0,74%	-2,70%	-3,37%
4- 80933978	0%	-0,92%	-1,49%	-2,04%
5- 80932744	0%	-0,74%	-1,21%	-1,76%
6- 80920623	0%	-0,47%	-1,06%	-1,68%

The Figure 80 shows the shrinkage of all samples for the different temperatures. Shrinkage depends on the materials properties and polymer composition. The lower the shrinkage the better the capacitance of the capacitor and the less the internal stress of the film.

Sample 6 has the lowest shrinkage followed by sample 5 and 4, respectively. The highest shrinkage was observed in sample 3, which can be explained again by the orientation and the crystallinity of the polymeric chains. It is confirmed by other techniques like DSC, which had the low percentage of crystallinity, using program 1 and the high elongation at break in the mechanical tests.

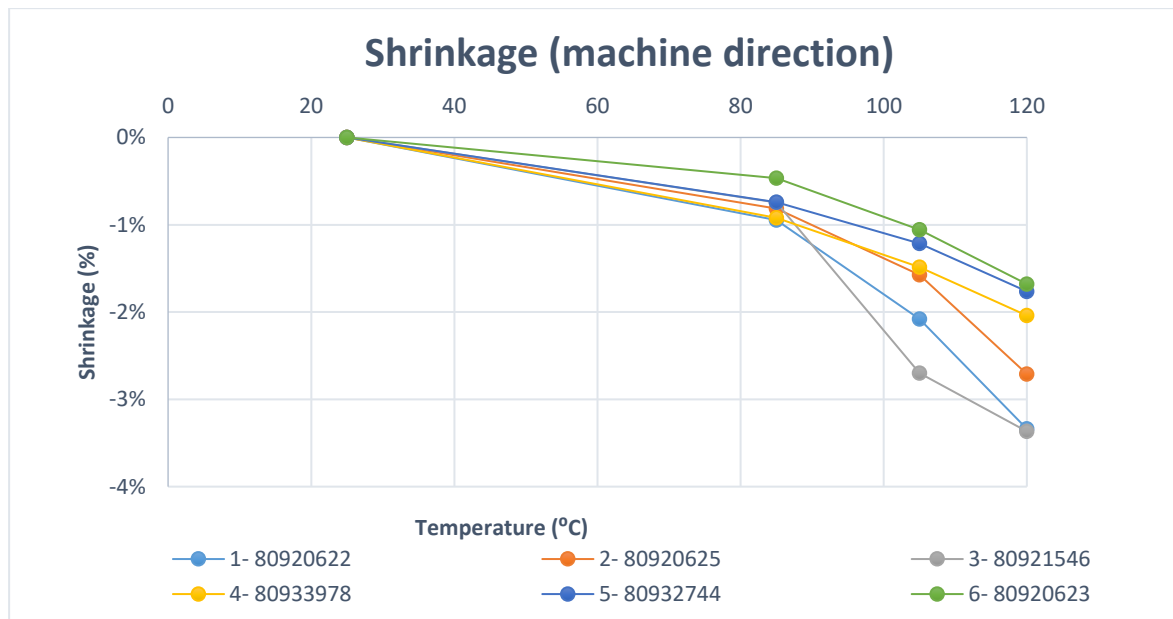


Figure 80- Shrinkage overview, of each sample at different temperatures

CHAPTER 5- CONCLUSION

5. Conclusion

As presented in this thesis there are a lot of techniques which allow to understand and characterize the materials. In this particular work, it was possible to apply DSC, FTIR, SEM, AFM and mechanical tests to conclude about the behaviour of different PP films.

The molecular organization and the polymer stereochemistry determine the properties of the polymeric material, which is reflected in the crystallinity and melting temperature.

The film thickness increasing seems to influence in the degree of orientation, leading to low crystallinity.

Considering each technique, making an overview and a ranking in each, as follow:

- DSC

- Ranking regarding the percentage of crystallinity during the second heating run:

Program 1: Sample 1> Sample 4> Sample 5> Sample 2> Sample 6> Sample 3

Program 2: Sample 6> Sample 2> Sample 1> Sample 3> Sample 4> Sample 5

- FTIR

- Ranking regarding the peaks: no differences were observed, as they have all same peaks.

- SEM

- Ranking regarding PP lamellae, observing the images:

Sample 1> Sample 2> Sample 4> Sample 6> Sample 3> Sample 5

- Ranking regarding the uniformity of the Al composition among the metallized PP, for films metallized with ZnAl:

Sample 5> Sample 6> Sample 4 > Sample 3

(Sample 1> Sample 2 are only metallized with Al)

Special treatment or process in B area was observed in Sample 5 and Sample 4. Samples 1/2/3/6 didn't show any difference in the B area (segments)

- AFM

- Ranking regarding the roughness uniformity among the metallized film:

Sample 5> Sample 4> Sample 1> Sample 2> Sample 6> Sample 3

- Mechanical tests

- Ranking regarding the high Young Modulus:

Sample 5> Sample 4> Sample 1> Sample 6> Sample 3> Sample 2

- Ranking regarding the high tensile strength:

Sample 1> Sample 2> Sample 5> Sample 6> Sample 3> Sample 4

- Ranking regarding the high elongation at break:

Sample 3> Sample 2> Sample 6> Sample 1 > Sample 5 > Sample 4

- Shrinkage

- Ranking in terms of high shrinkage:

Sample 3> Sample 1> Sample 2 > Sample 4 Z Sample 5> Sample 6

The higher the crystallinity the higher the orientation of the polymeric chains the stiffer the material the higher Young Modulus. In this case, sample 5 seems to be the best polymeric material.

Regarding the roughness of samples and the shrinkage, seems that the higher variation in roughness (among the metallized film) the higher is the shrinkage. Sample 3 has the highest variation in terms of roughness and the highest shrinkage, on the other hand, sample 5 has the most uniform roughness among the film.

Considering SEM images, and samples with Zn, PP lamellae are seen in sample 5 which has the lowest Zn weight.

Concluding, considering all characterization techniques and all properties of the films studied, seems that a high Young Modulus, low elongation at break, uniform roughness among the film, low shrinkage and more uniform Al weight composition among the film are important to have a good PP film.

CHAPTER 6- FUTURE WORK

6. Future work

As this thesis demonstrates, the good performance of a film capacitor needs continuing investigation and development, as well as, a good understanding of the BOPP films and their metallization processes. The relationship between polymeric and the dielectric properties can be studied deeply. Further work can be done by understanding the biaxial stretching behaviour, studying deeply the crystallization and producing capacitors, evaluating the dielectric breakdown and other electrical measurements, as capacitance and IR, for instance. It can be possible to enhance and optimize the performance of a film capacitor knowing at the very beginning some properties and behaviour of the materials.

BIBLIOGRAPHY

- [1] "VISHAY INTERTECHNOLOGY INC.," *Experimental Techniques*, vol. 21, no. 5, pp. 13–13, Sep. 1997.
- [2] "Film | Capacitors | Vishay." [Online]. Available: <http://www.vishay.com/capacitors/film/>. [Accessed: 15-Mar-2020].
- [3] "Vishay - Dr. Felix Zandman." [Online]. Available: http://www.vishay.com/landingpage/zandman/co_drz_memorial.html. [Accessed: 15-Mar-2020].
- [4] F. Zandman, D. Chanoff, and Mazal Holocaust Collection., *Never the last journey*. Schocken Books, 1995.
- [5] "VISHAY PORTUGAL - Famalicão Made In." [Online]. Available: https://www.famalicaomadein.pt/_vishay_portugal. [Accessed: 15-Mar-2020].
- [6] "Vishay Celebra 50 Anos Da Sua Unidade Industrial Em Famalicão - FamaTV." [Online]. Available: <https://www.famatv.pt/video/4/801/vishay-celebra-50-anos-da-sua-unidade-industrial-em-famalicao/>. [Accessed: 15-Mar-2020].
- [7] "Visita guiada à Vishay Electrónica Portugal, Lda." [Online]. Available: <https://www.ccila-portugal.com/pt/eventos/eventos-detalhes/visita-guiada-a-vishay-electronica-portugal-lda>. [Accessed: 16-Jan-2021].
- [8] F. Richert, "Introduction to electronic components: Active vs. passive components," 2020.
- [9] Würth Elektronik, "ABC of Capacitors: Basic Principles, Characteristics and Capacitor Types-First Edition", 2020
- [10] D.Montanari, K.Saarinen, F.Scagliarini "Film Capacitors for Automotive and Industrial Applications", KEMET Electronics Corporation, USA, 2009
- [11] R.S.Beerasha, A. M. Khan and H.V.M. Reddy "Design and EM-simulation of MIM capacitor", *2017 International Conference on Energy, Communication, Data Analytics and Soft Computing (IECCDS)*, Chennai, India, pp.1644-1649, 2017.
- [12] D.A.DeAngelis and G.W.Schulze, " The Effects of Piezoelectric Ceramic Dissipation Factor on the performance of Ultrasonic Transducers", *Physics Procedia*, Vol.63,pp. 28-36, 2015.
- [13] TDK, "Capacitors, Part 3 'Ceramic Capacitors [2]", USA, 2010
- [14] R. Gallay, "Metallized Film Capacitor Lifetime Evaluation and Failure Mode Analysis", *Conference CAS-CERN Accelerator School:Power Converters*, Geneva, Switzerland, May 2014.
- [15] KEMET Electronics, "Failure Analysis of Capacitors and Inductors"
- [16] Martin Christian, "Description of polypropylene film capacitor", University Lyon, 2018.
- [17] D. Whisnant, "Polymer Chemistry: Classification of Polymers," *Engineering LibreTexts*. 2020.
- [18] "Polypropylene (PP) Plastic: Types, Properties, Uses & Structure Info." [Online]. Available: <https://omnexus.specialchem.com/selection-guide/polypropylene-pp-plastic>. [Accessed: 05-Oct-2020].

- [19] Chemistry Libre texts, "Olefin Polymerization with Ziegler-Natta Catalyst," pp. 1–4, 2018.
- [20] D. E. Heath and S. L. Cooper, "Polymers: Basic Principles," *Biomaterials Science: An Introduction to Materials: Third Edition*, no. December 2013, pp. 64–79, 2013.
- [21] H. Hagen, J. Boersma, and G. Van Koten, "Homogeneous vanadium-based catalysts for the Ziegler-Natta polymerization of α -olefins," *Chemical Society Reviews*, vol. 31, no. 6, pp. 357–364, 2002.
- [22] D.W. Van der Meer, "Structure-Property Relationships in Isotactic Polypropylene", Ph.D. thesis, University of Twente, 2003.
- [23] Y.Kong, J.N. Hay, "The enthalpy of fusion and degree of crystallinity of polymers as measured by DSC", in *European Polymer Journal*, Vol.39, Issue 8, pp 1721-1727
- [24] B. Magalhães, "Comparação entre as técnicas difração de raios-x e DSC na obtenção do grau de cristalinidade do PP e do PEAD", Belo Horizonte, Brasil, 2017.
- [25] J. C. Mierzwa, "Estudo da morfologia e cristalinidade em polipropileno isotático submetido a deformação uniaxial em temperatura ambiente," Federal do Rio Grande do Sul, 2002.
- [26] T. Calafut, "Polypropylene Films," in *Plastic Films in Food Packaging*, Elsevier, 1998, pp. 17–20.
- [27] "Tervakoski Film Industry and Infrastructure - Tervakoski Film." [Online]. Available: <http://tervakoskifilm.com/applications/industry-and-infrastructure/>. [Accessed: 22-Nov-2020].
- [28] F. J. Díez *et al.*, "Influence of the stretching in the crystallinity of biaxially oriented polypropylene (BOPP) films," *Journal of Thermal Analysis and Calorimetry*, vol. 81, no. 1, pp. 21–25, Jul. 2005.
- [29] J. Xiong, X. Wang, X. Zhang, Y. Xie, J. Lu, and Z. Zhang, "How the biaxially stretching mode influence dielectric and energy storage properties of polypropylene films," *Journal of Applied Polymer Science*, vol. 138, no. 11, p. 50029, Mar. 2021.
- [30] B. S. Sroan and F. MacRitchie, "Mechanism of Gas Cell Stability in Breadmaking", in *Bubbles in Food 2* chapter 29, pp 299-306, 2008.
- [31] T.Calafut, "Polypropylene Films", *Plastic Films In Food Packaging*, pp. 17-20, 1998.
- [32] D. L. Williams and T. M. O'Bryon, "Cleanliness Verification on Large Surfaces," in *Developments Surface Contamination and Cleaning*, Elsevier, 2013, pp. 163–181.
- [33] J. L. Nash, "Biaxially oriented polypropylene film in power capacitors," *Polymer Engineering & Science*, vol. 28, no. 13, pp. 862–870, Jul. 1988.
- [34] E. L. Hobbs, Stanley Y.; Carley, "CA1053339A - Capacitor having a hazy polypropylene film - Google Patents," 1976.
- [35] R. B. Menczel, Joseph D.; Prime, " *Thermal Analysis of Polymers: Fundamentals and applications* ", Wiley, USA, 2014.
- [36] G.W.H.Hohne, W.F.Hemminger and H.-J.Flammersheim, "Differential Scanning Calorimetry, Second Edition", Springer, 2003
- [37] M. Gahleitner, C. Bachner, E. Ratajski, G. Rohaczek, and W. Neißl, "Effects of the catalyst

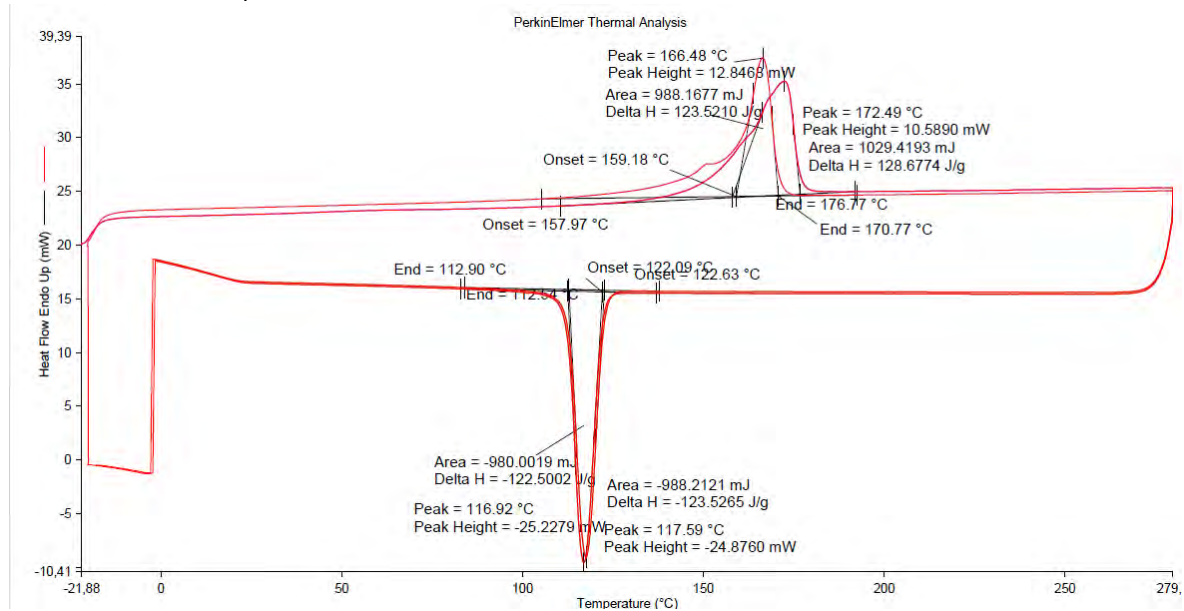
- system on the crystallization of polypropylene," *Journal of Applied Polymer Science*, vol. 73, no. 12, pp. 2507–2516, 1999.
- [38] Visakh P. M. and Poletto M., "Polypropylene-Based Biocomposites and Bionanocomposites", Wiley, 2018.
- [39] J.R.Ferraro and K.Krishnan, "Practical Fourier Transform Infrared Spectroscopy", Industrial and Laboratory Chemical Analysis, California, 1990.
- [40] Chemistry Libre texts, "Infrared Spectroscopy," 2020.
- [41] J. Coates, "Interpretation of infrared spectra, A practical approach," *Encyclopedia of Analytical Chemistry: Applications, Theory and Instrumentation*, 2006.
- [42] "Infrared spectroscopy (IR)." [Online]. Available: https://personal.utdallas.edu/~scortes/ochem/OCHEM_Lab1/recit_notes/ir_presentation.pdf. [Accessed: 29-Nov-2020].
- [43] N. Raval, R. Maheshwari, D. Kalyane, S. R. Youngren-Ortiz, M. B. Chougule, and R. K. Tekade, "Importance of Physicochemical Characterization of Nanoparticles in Pharmaceutical Product Development," in *Basic Fundamentals of Drug Delivery*, Elsevier, 2019, pp. 369–400.
- [44] Ni C., "Scanning Electron Microscopy (SEM)," in *Encyclopedia of Tribology*, pp. 2977–2982, 2013.
- [45] A. Nanakoudis, "EDX Analysis with SEM: How Does it Work? - Accelerating Microscopy," ThermoFisher Scientific, 2019.
- [46] R. W. Welker, "Size Analysis and Identification of Particles," in *Developments in Surface Contamination and Cleaning*, Elsevier, 2012, pp. 179–213.
- [47] Aakanksha Gaur, "Electromagnetic spectrum," *The editors of Encyclopedia Britannica*, 2019.
- [48] G. Rodrigues and D. A. Silva, "Microscopia de força atômica na análise de rugosidade e da dimensão fractal de membranas celulares," Federal de Alagoas, Brasil, 2016.
- [49] P. S. P. Herrmann *et al.*, "Microscopia de Varredura por Força: uma Ferramenta Poderosa no Estudo de Polímeros", *Polímeros* vol.7 no. 4, São Carlos, 2017.
- [50] S. S. Gajghate, "Introduction to Atomic Force Microscopy (AFM) by National Institute of Technology Agartala," August, 2017.
- [51] E. Guevara, "Surface roughness terminology and parameters." 2018.
- [52] M. Raposo, Q. Ferreira, and P. a Ribeiro, "A Guide for Atomic Force Microscopy Analysis of Soft-Condensed Matter," *Modern research and educational topics in microscopy*, pp. 758–769, 2007.
- [53] "Young's Modulus and Tensile Strength: All you need to know." [Online]. Available: <https://www.sculpteo.com/en/3d-learning-hub/3d-printing-materials-guide/youngs-modulus-and-tensile-strength-3d-printing/>. [Accessed: 15-Nov-2020].
- [54] "JSM-6010PLUS/LA InTouchScope™ Multiple touch panel scanning electron microscope | Products | JEOL Ltd." [Online]. Available: https://www.jeol.co.jp/en/products/detail/JSM-6010PLUS_LA.html. [Accessed: 29-Nov-2020].

- [55] D. Puhan, "Triboemission-lubricant interactions", Ph.D.thesis, London, October 2017
- [56] ISO, "ISO - ISO 527-3:2018 - Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets," 2018.
- [57] H. Bayazian, A. Yadgari, J. Morshedian, A. Jamshidi, and M. Razavi-Nouri, "Evaluation of morphology and crystallinity of biaxially oriented polypropylene films," 2019, vol. 2065, p. 040015.
- [58] Q. T. H. Shubhra and A. K. M. M. Alam, "Mechanical properties of polypropylene composites A review" in *Journal of Thermoplastic Composite Materials*, chapter 26, pp.362-391, April 2013.
- [59] R. Morent, N. De Geyter, C. Leys, L. Gengembre, and E. Payen, "Comparison between XPS- And FTIR-analysis of plasma-treated polypropylene film surfaces," *Surface and Interface Analysis*, vol. 40, no. 3–4, pp. 597–600, 2008.
- [60] A. Mertke and R. Feser, "Influence of organic sealants on the corrosion protection of thermally sprayed ZnAl 15 coating," Germany, 2016.
- [61] H. Takeoka, T.Saito,K.Shiota and T.Sasaki, "Development of polypropylene film capacitor using double metallizing technology," *Properties and Applications of Dielectric Materials*, vol.3, July 2003.
- [62] Y.H.Zhao, E.J.Lavernia, "The mechanical properties of multi-scale mettalic materials", *Nanostructured Metal and Alloys, Processing, Microstructure, Mechanical Properties Applications*, Elsevier, Chapter 13, 2011.

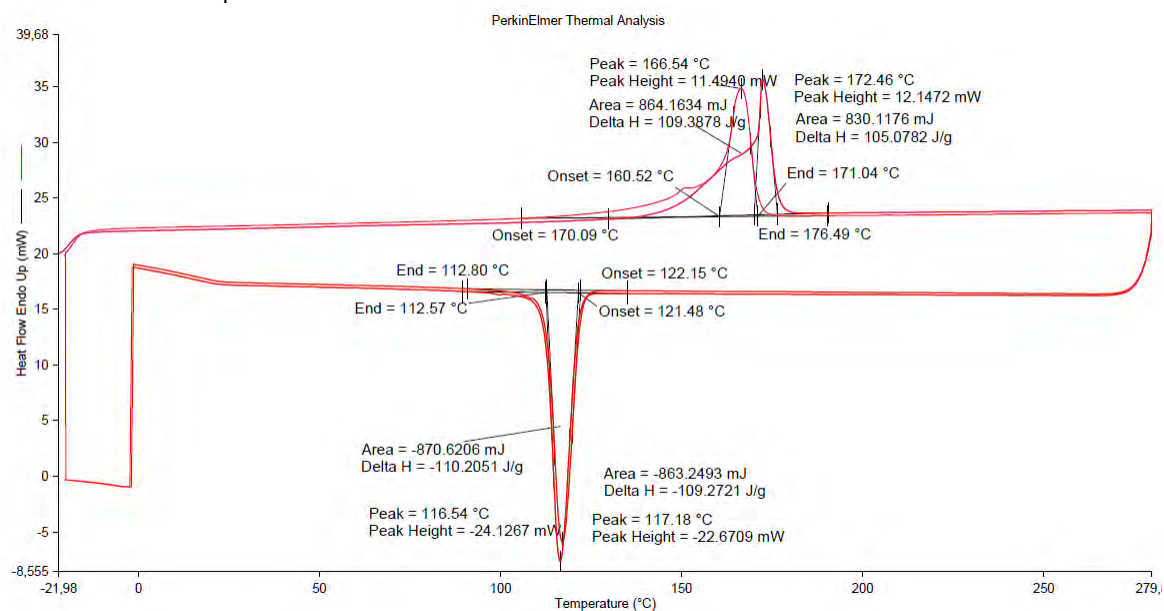
ANNEX I

Thermograms obtained with program 1

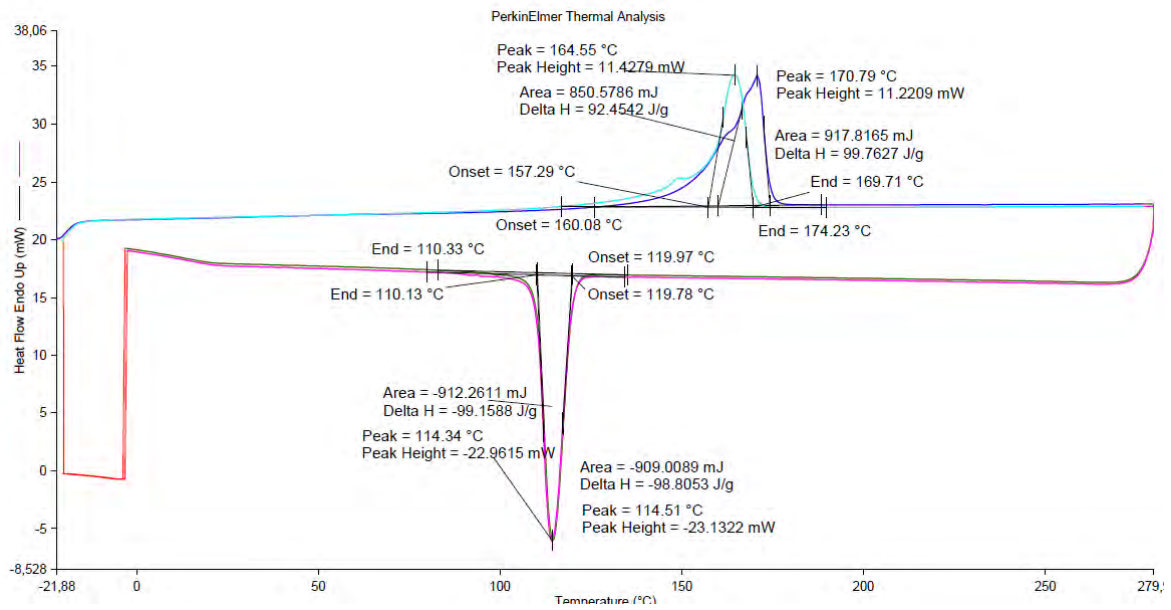
- Sample 1



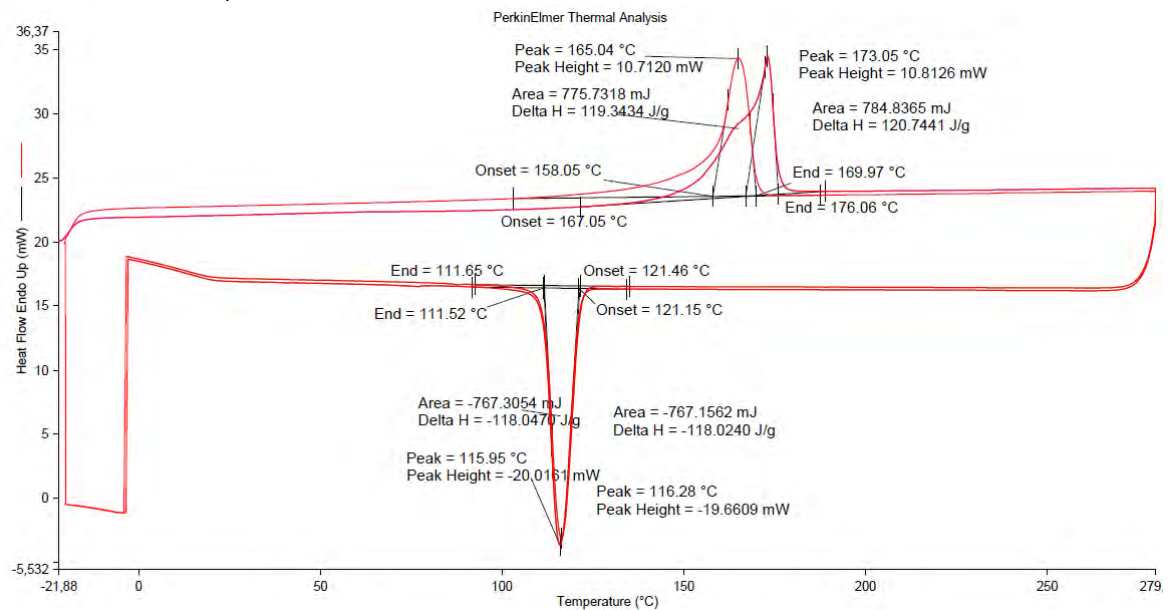
- Sample 2



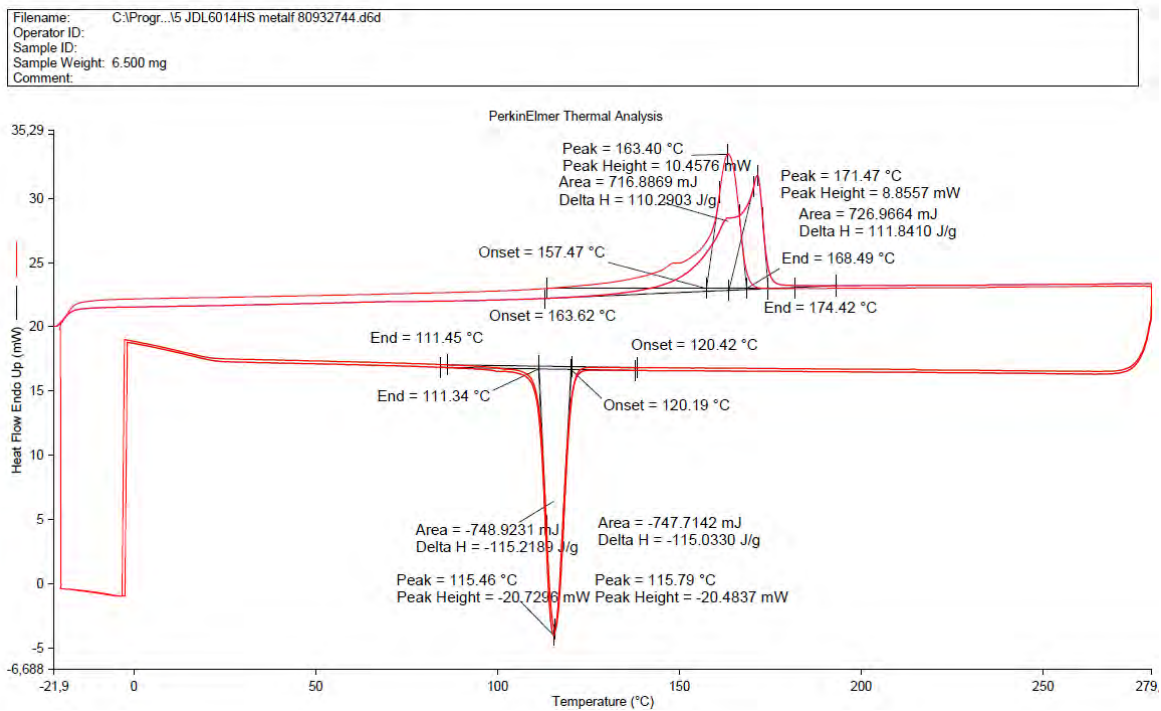
- Sample 3



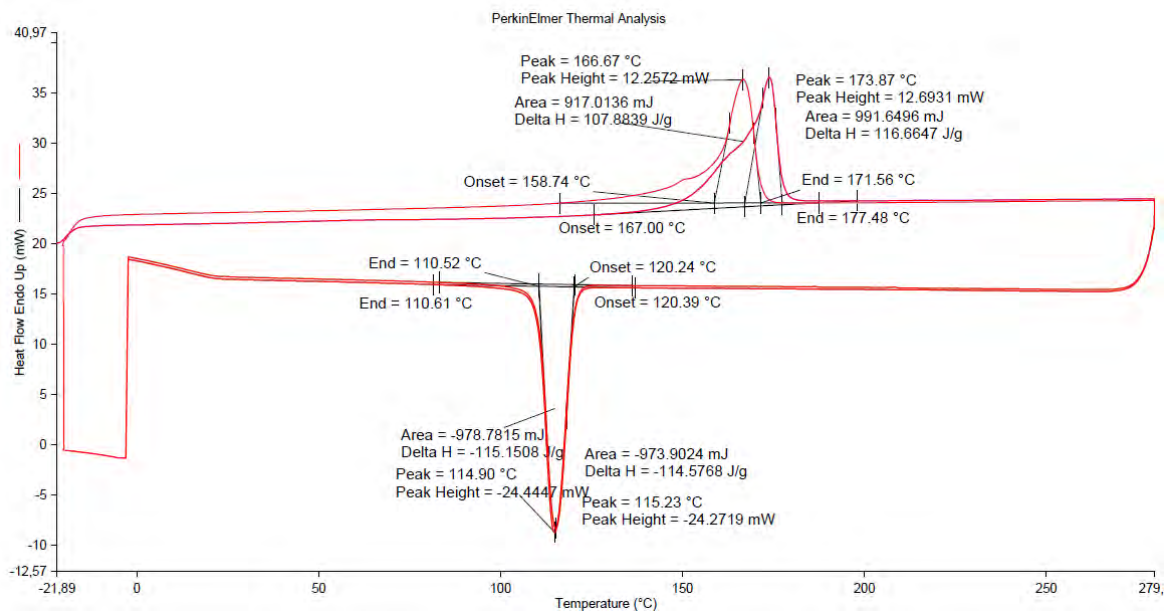
- Sample 4



- Sample 5



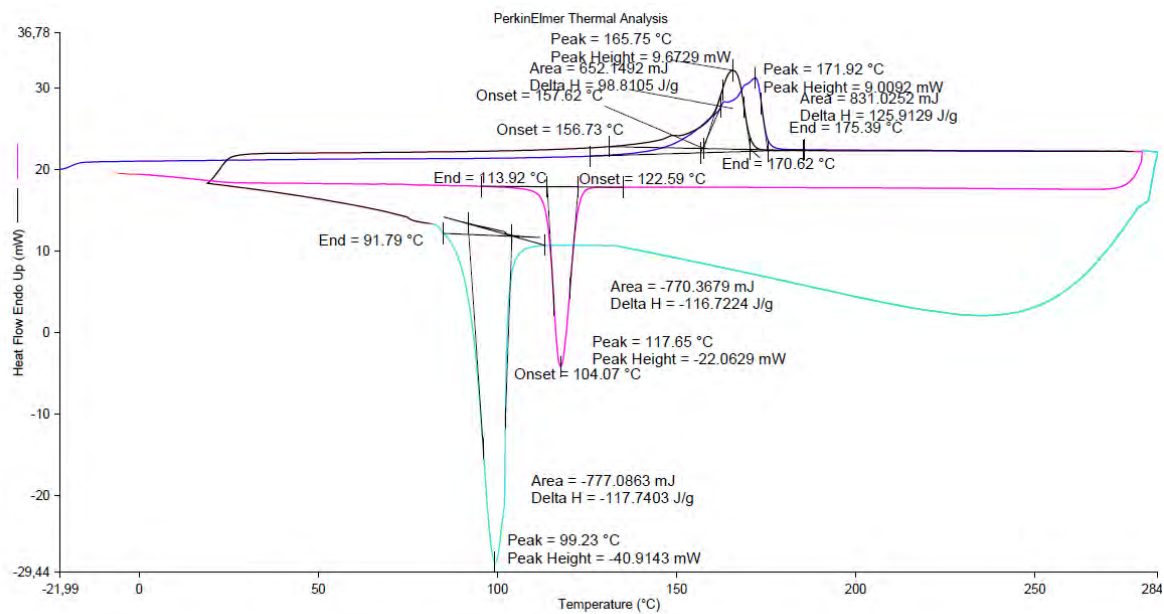
- Sample 6



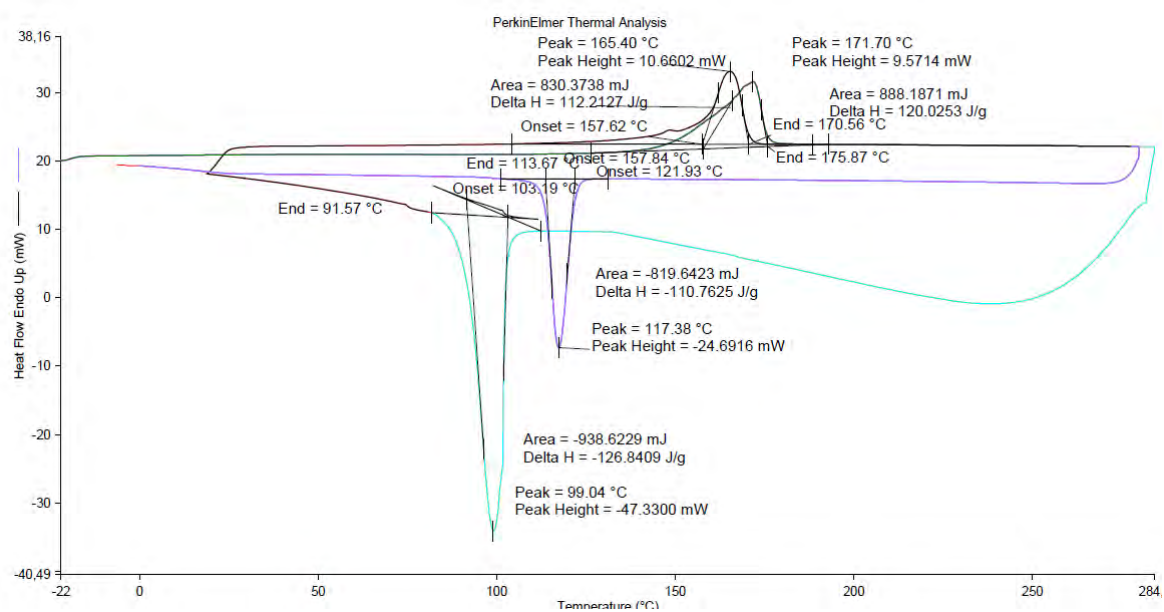
ANNEX II

Thermograms obtained with program 2

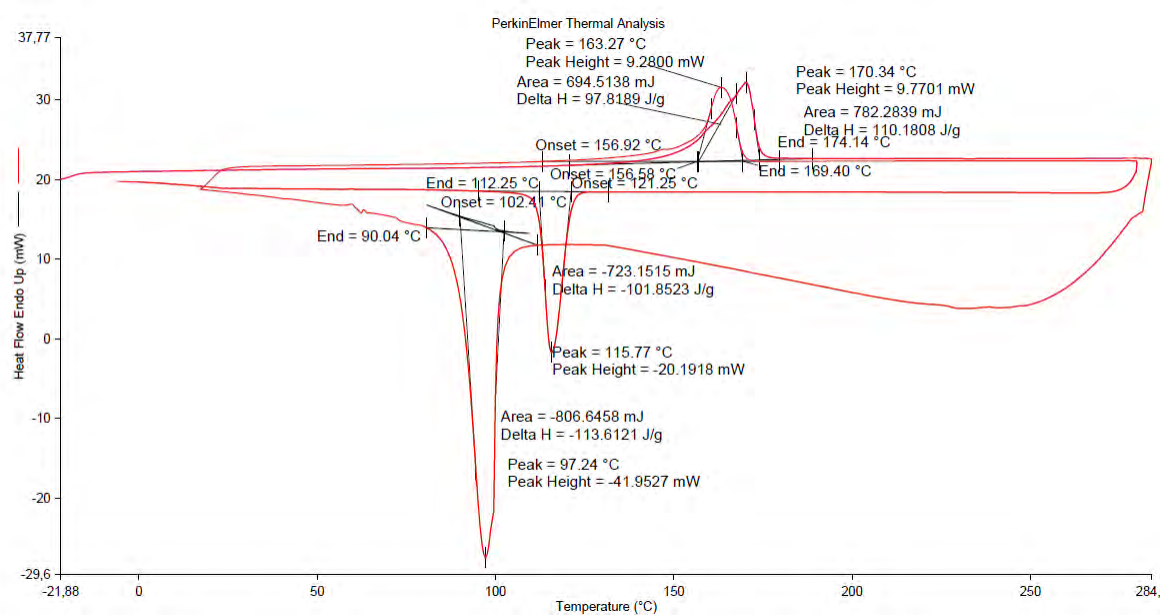
- Sample 1



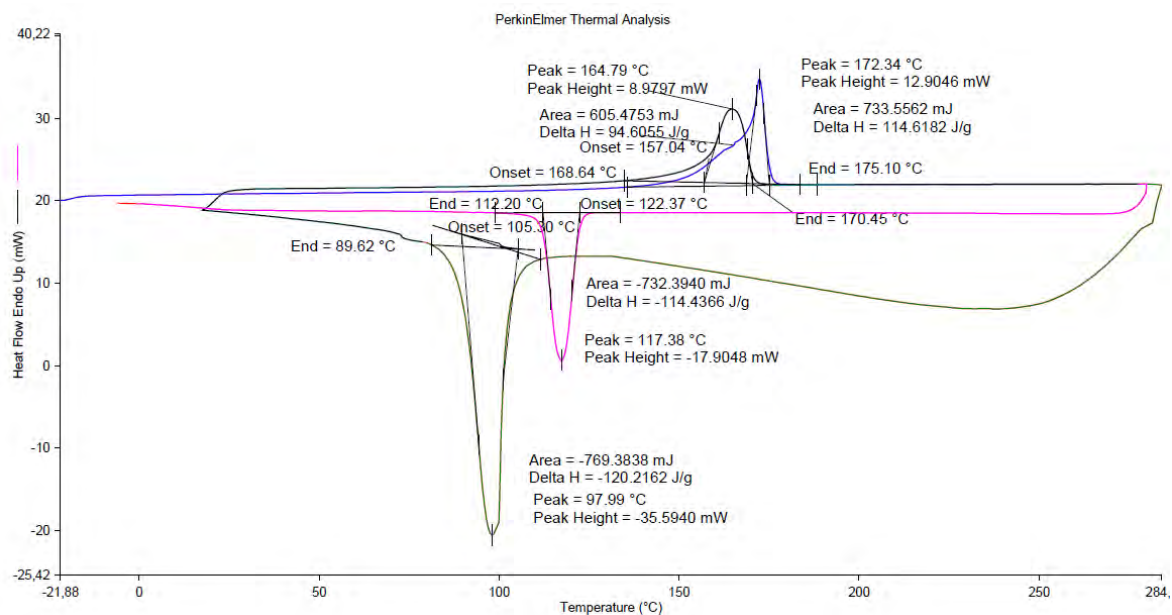
- Sample 2



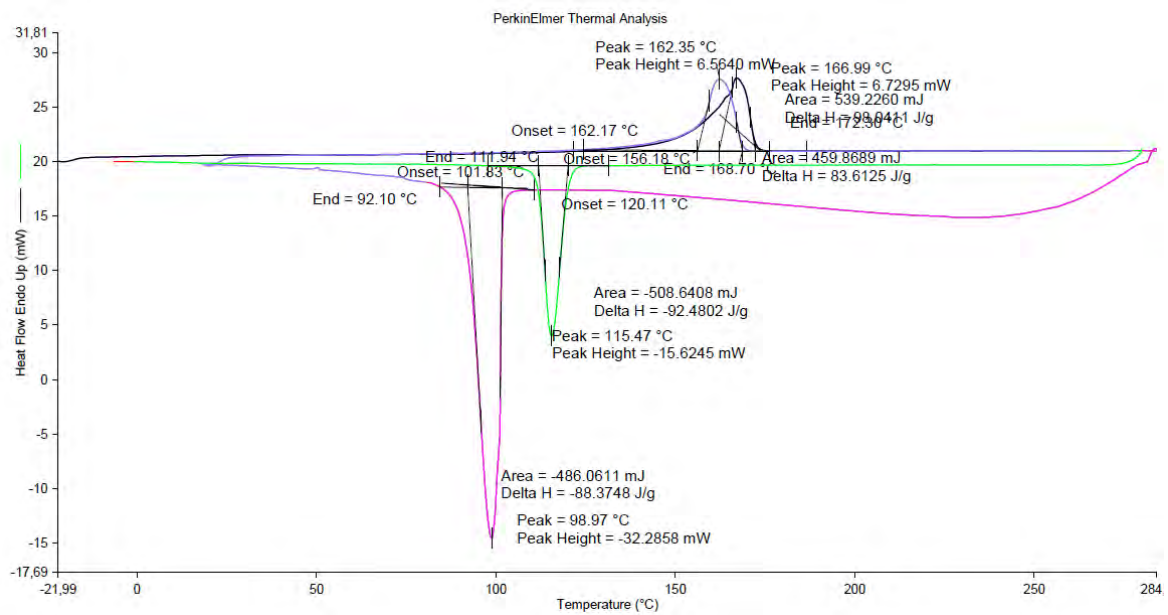
- Sample 3



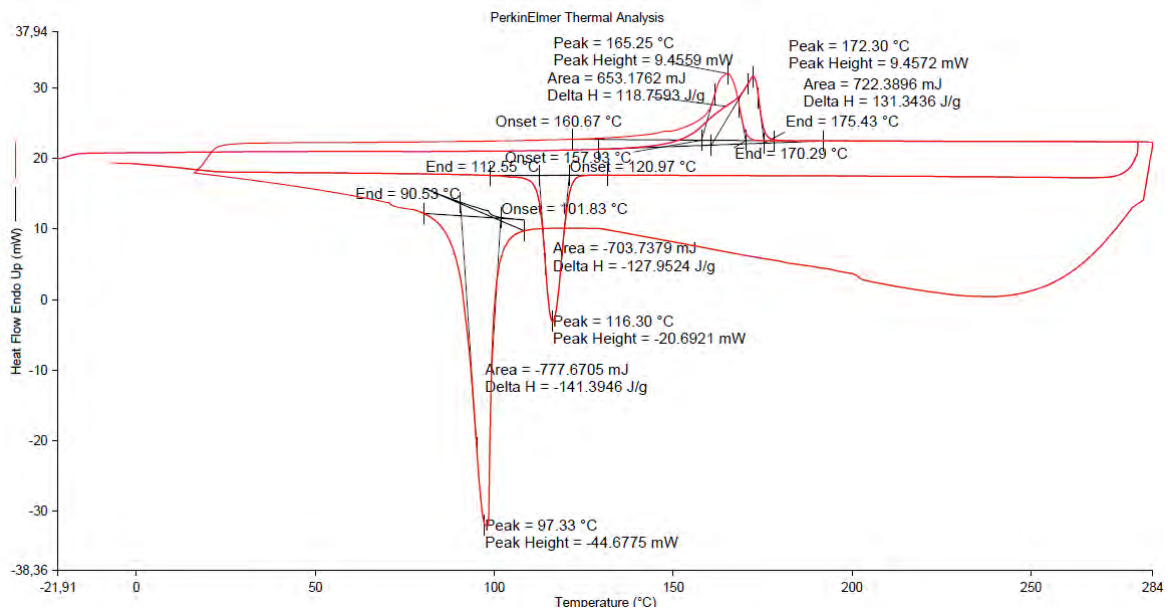
- Sample 4



- Sample 5



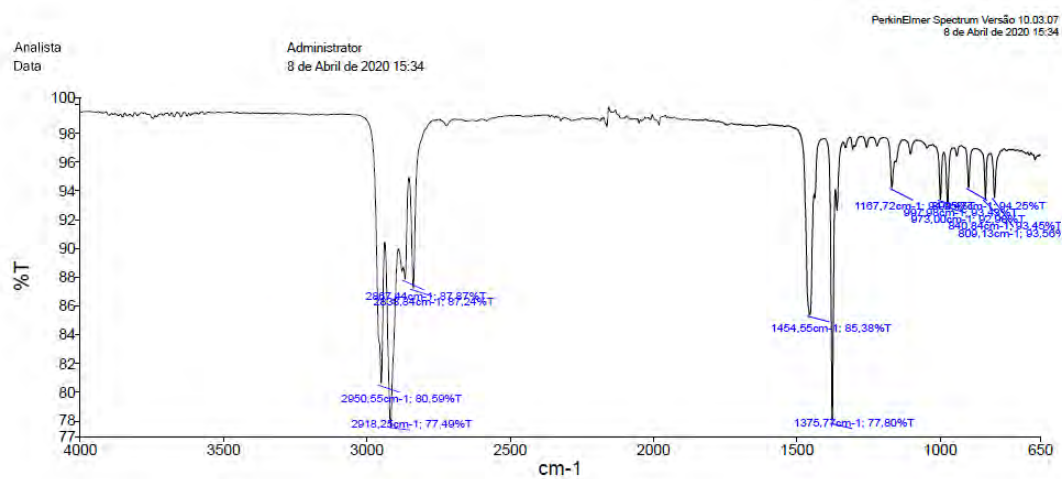
- Sample 6



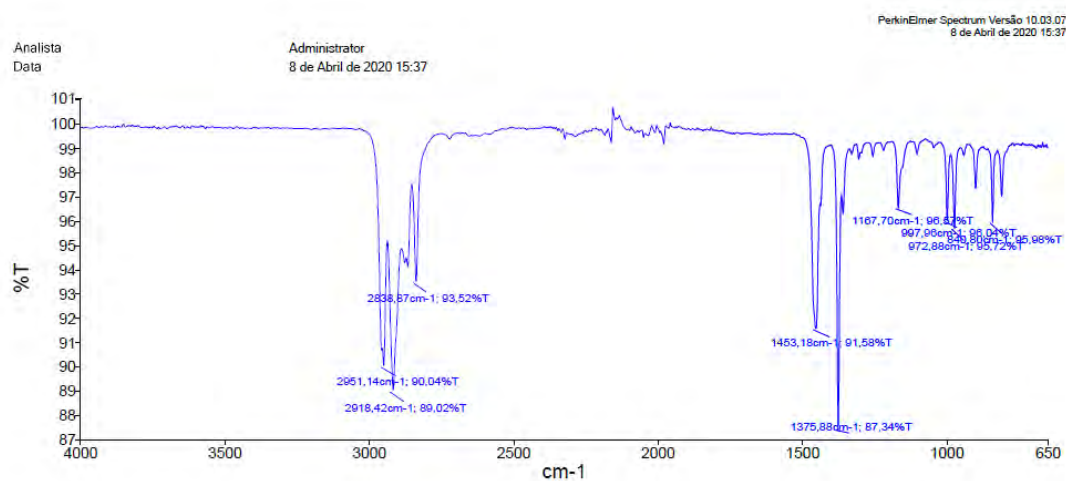
ANNEX III

Spectrum

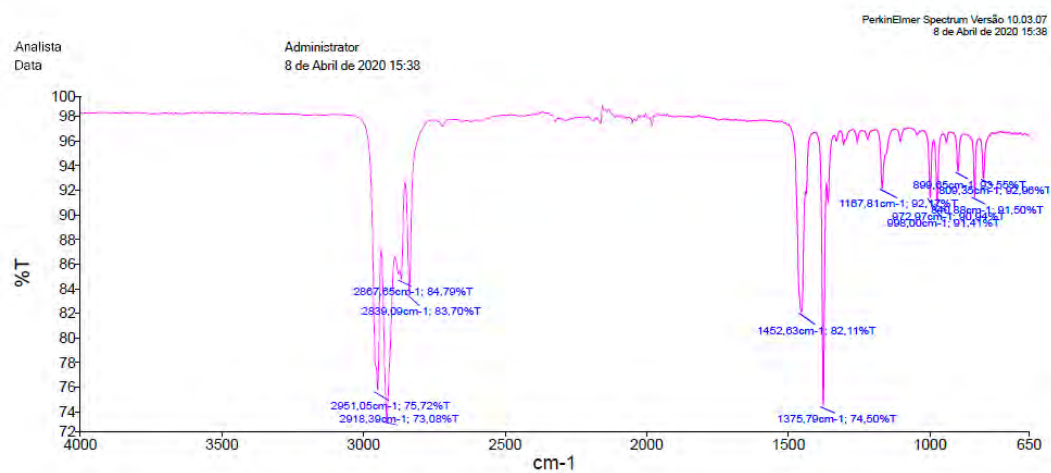
- Sample 1



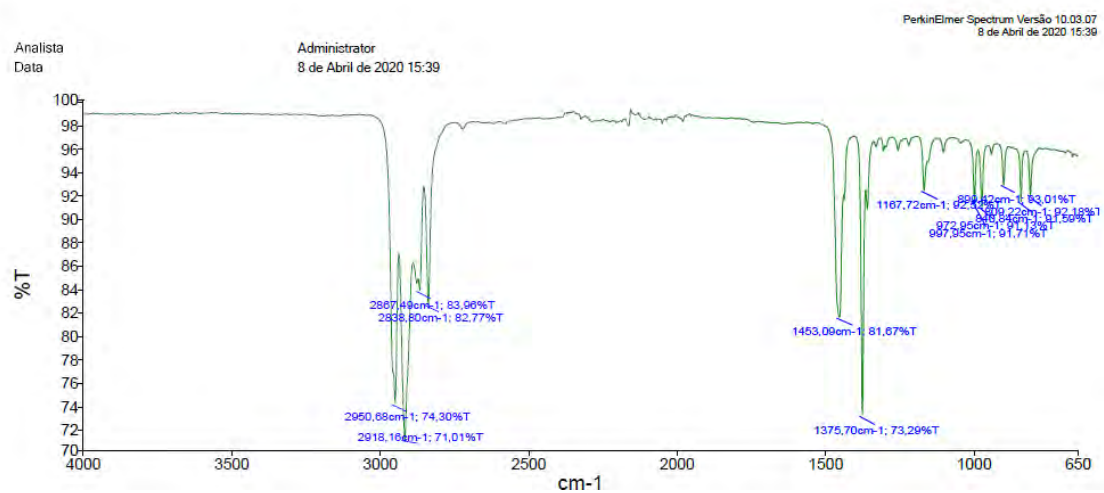
- Sample 2



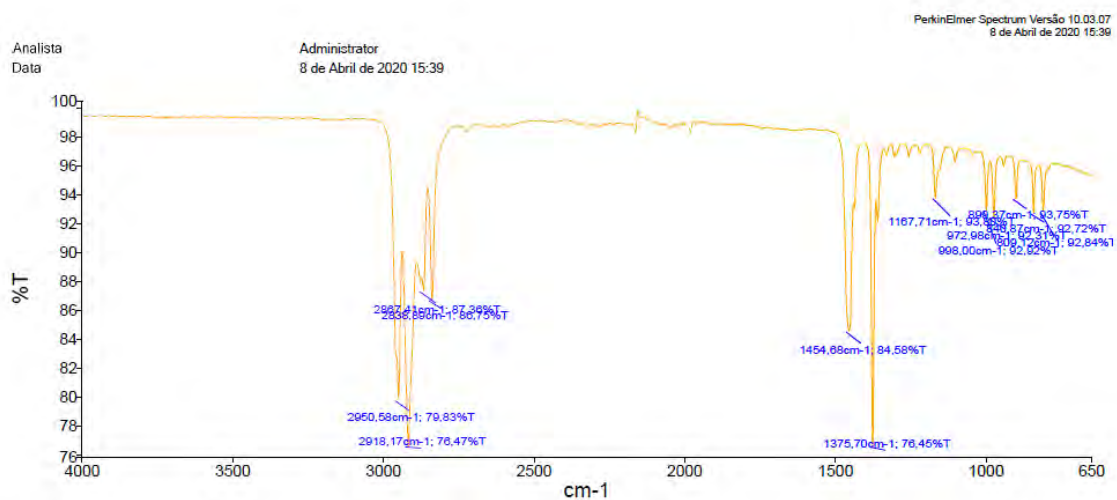
- Sample 3



- Sample 4



- Sample 5



- Sample 6

