CHARACTERIZATION PROPOSAL FOR PCL MEMBRANES WITH NANOPARTICLES FOR A FOG WATER HARVESTING DEVICE

Materials Characterization

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INTRODUCTION

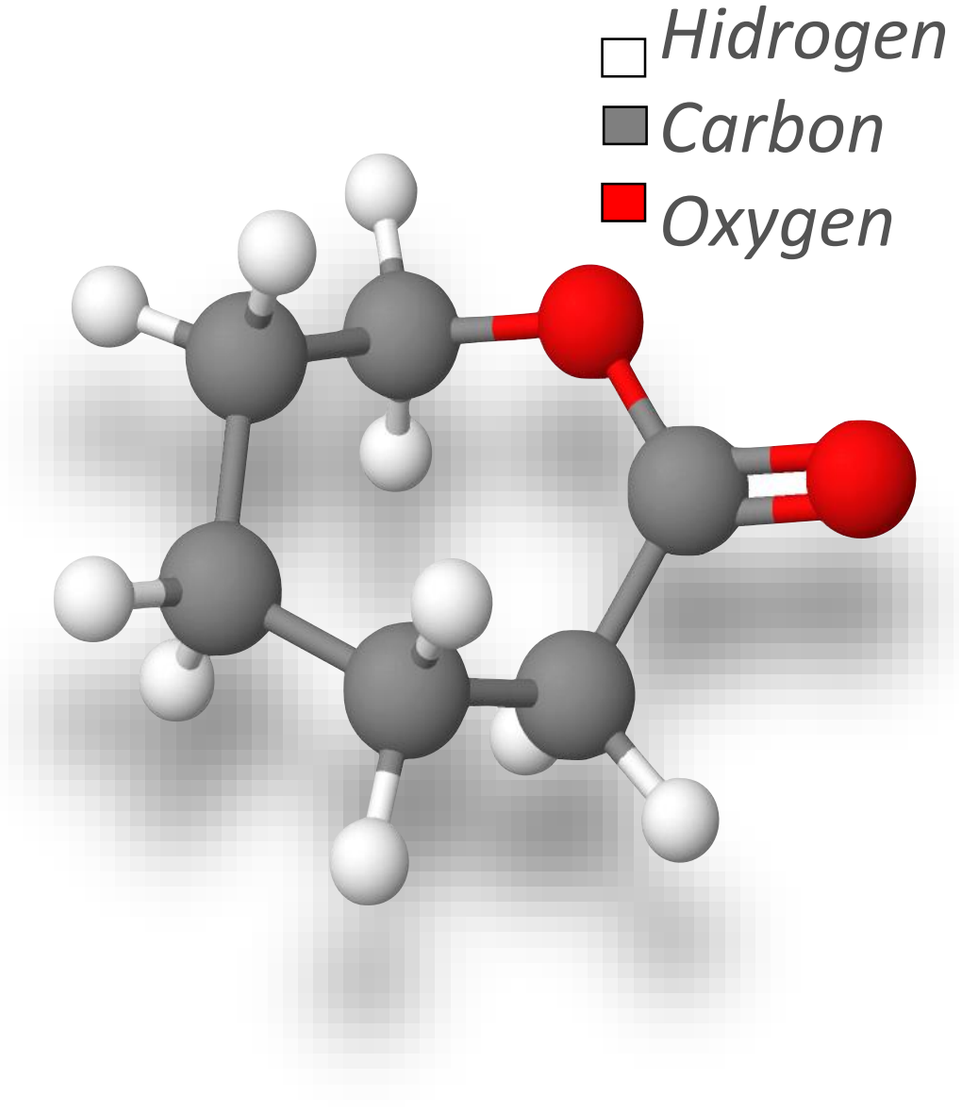
Polymer materials require an extensive set of characterizations to being able to fully understand their physicochemical properties.

This characterization proposal centers around light-matter interaction based and thermal characterization methods in order to understand the morphology, the structure and the thermal behavior of the polymer as well as the interaction with the nanoparticles that are used as dopants.

Poly-Caprolactone membrane (source:http://www.genoss.com/product/osteoguide.php)

INTRODUCTION

The idea to use dopants is to enhance the physicochemical properties of the material as a whole and the characterization methods will allow us to understand better this interaction as well as the changes produced trough the use of solvents and nanoparticles for the fabrication of a PCL membrane to be later applied to a base material to enhance fog water harvesting.



The attractive advantages of PCL are: its approval by the Food and Drug Administration (FDA) for use in humans, its biodegradability, good processability, ease of melt processing due to its high thermal stability and its relatively low cost [1].

Poly-Caprolactone molecule (source: molview.org)

THERMOGRAVIMETRIC ANALYSIS (TGA)

Justification: *Provides information regarding the degradation temperature of the material and its behavior along the time (as temperature raises).*
This parameter is crucial in understanding how the material behaves with temperature and is necessary when working with polymers, since they need a solvent and temperature for generating a solution.

Parameters:

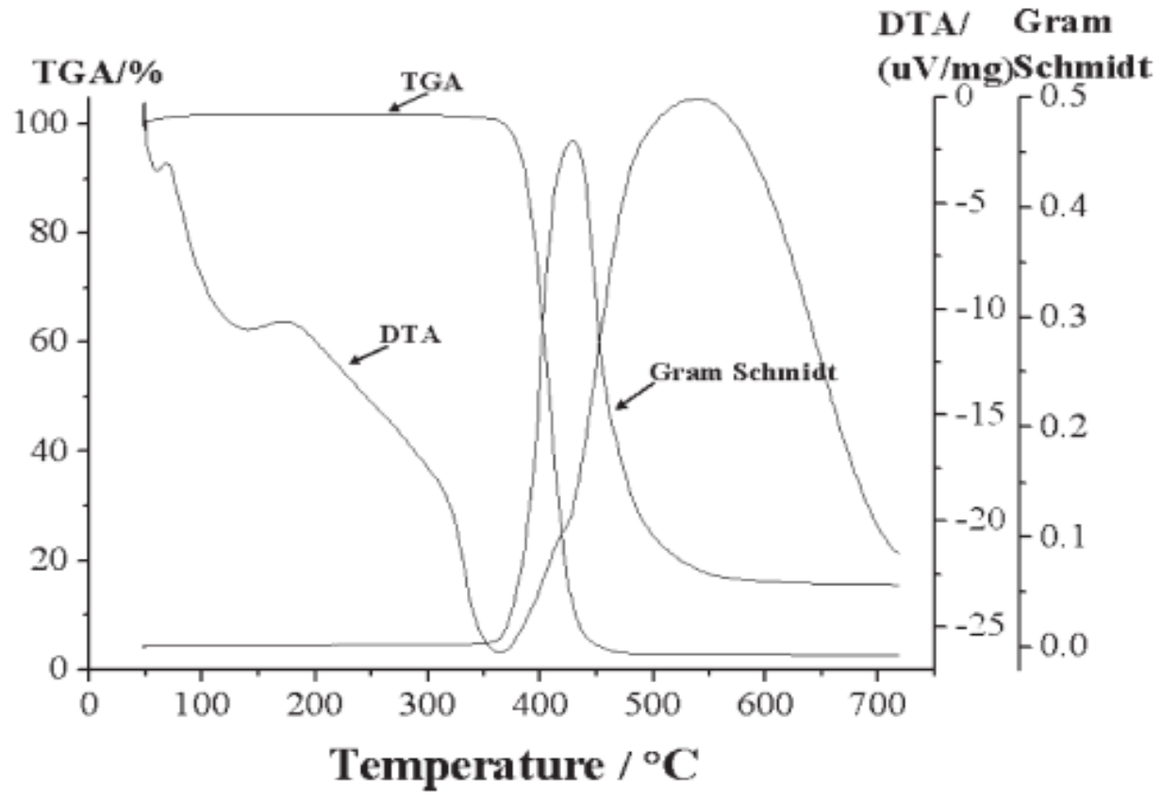
Perkin Elmer TGA

Around 5mg of sample

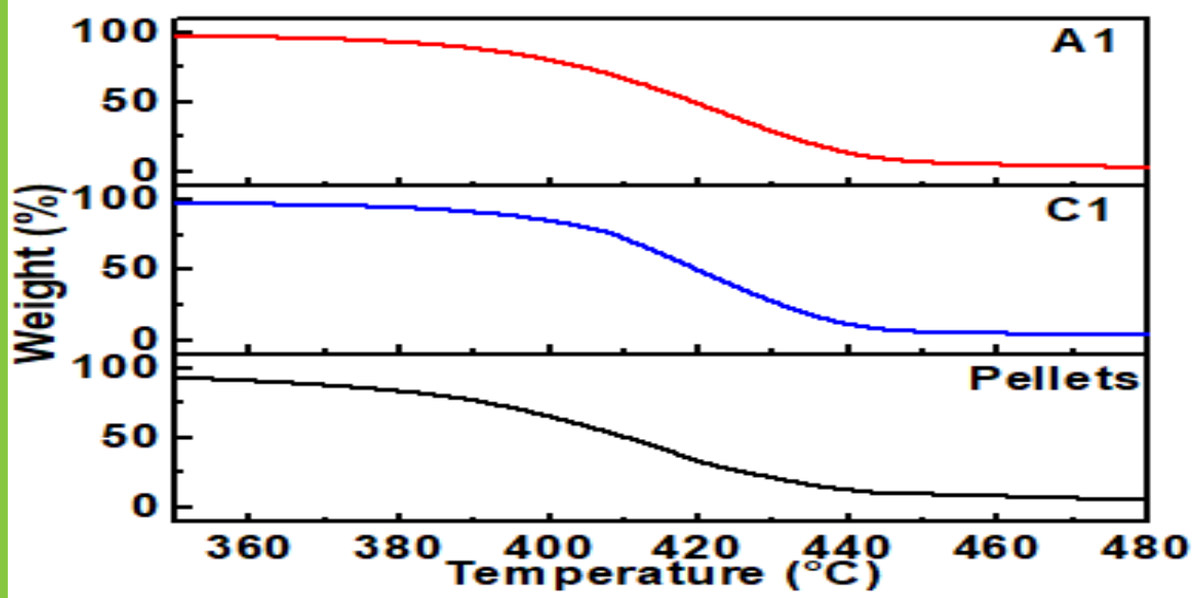
From 25°C to 500°C

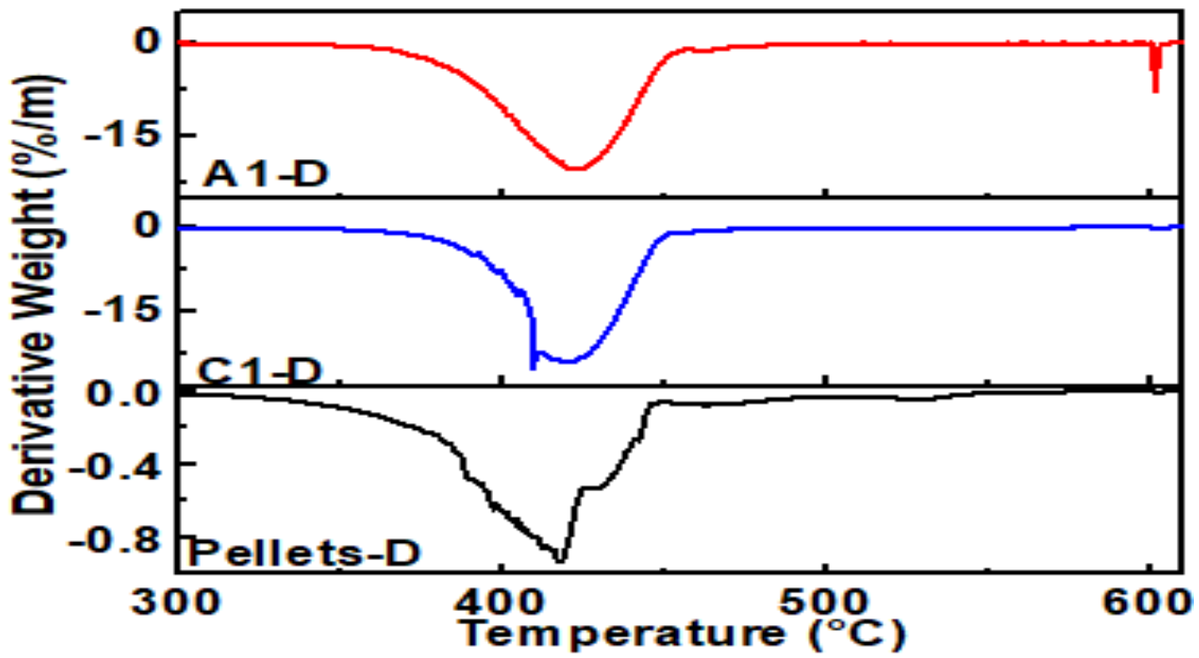
Run in a Nitrogen Atmosphere

EXPECTED RESULTS



Degradation curve for PCL taken from [3]





DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Justification: *It is useful to indicate how the virgin material must be handled and manipulated when it is in pellets form to dilute it and generate a polymeric solution. The information of the thermal behavior of the material and the crystal phases that form is shown in this technique. Different shapes in the endotherms indicate differences in the structure of the polymer.*

This information is important in order to validate if the polymer will be able to function at the application temperature.

Parameters:

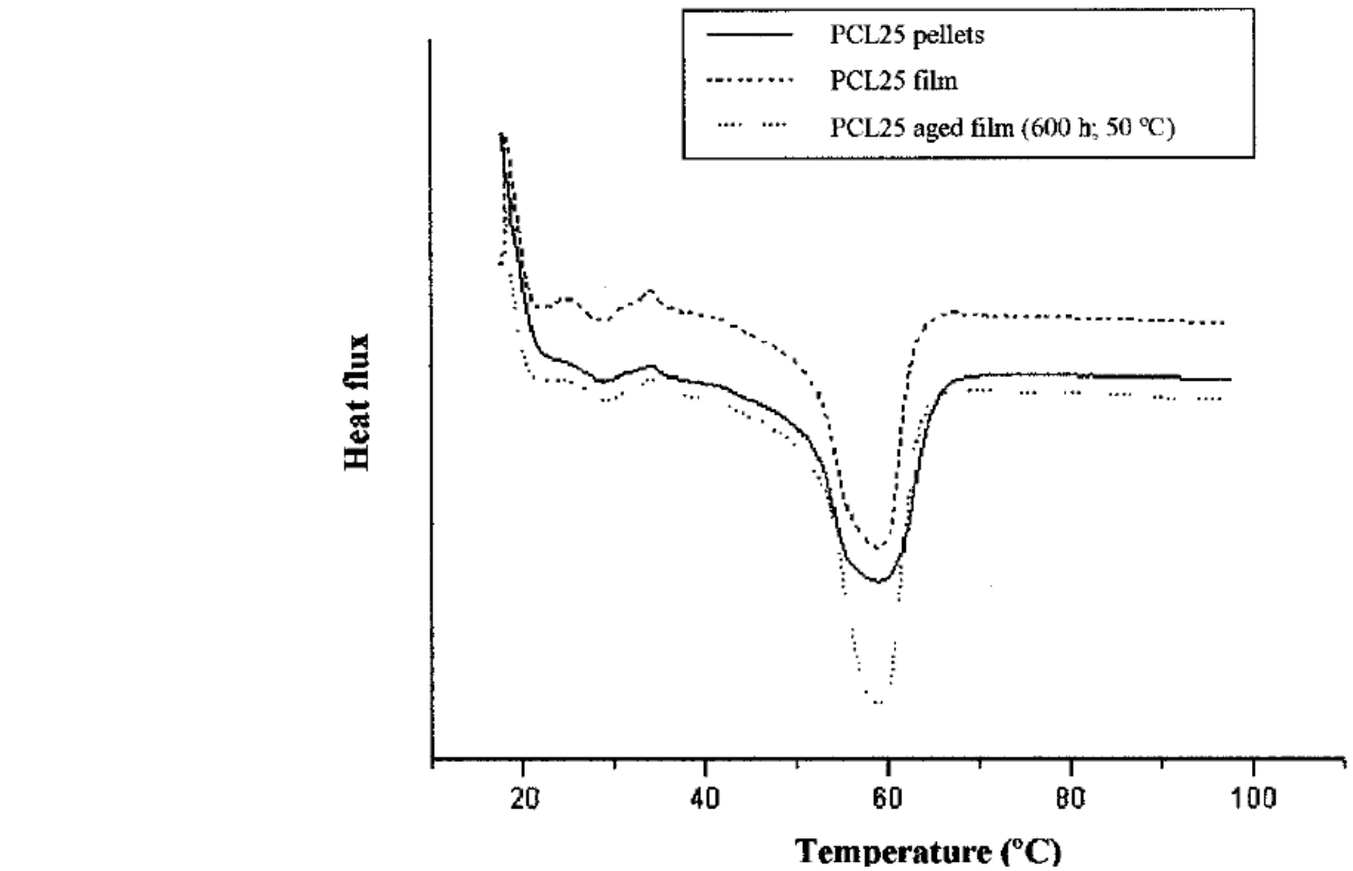
Perkin Elmer DSC 8000

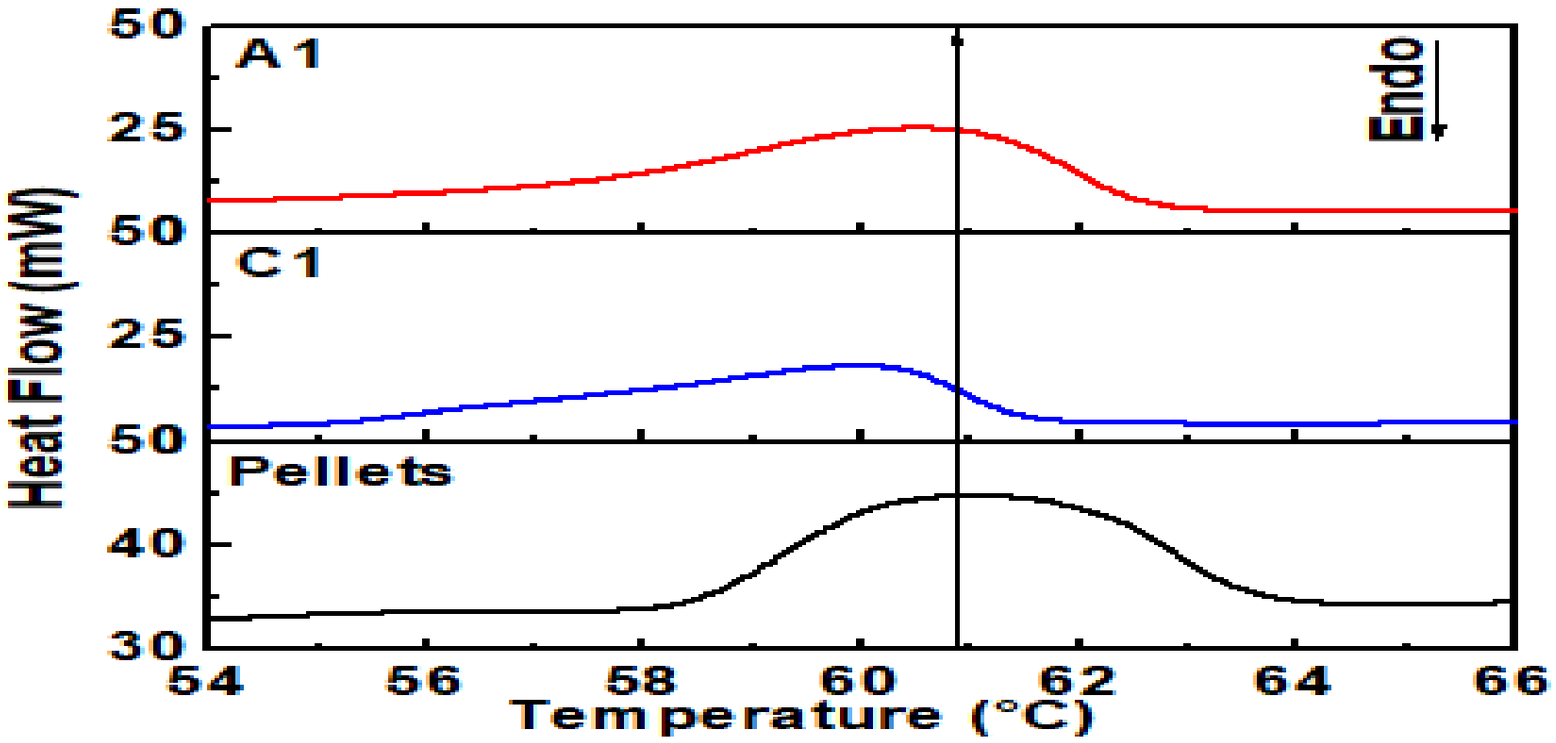
Around 5mg of sample

From 25°C to 270°C

Run in a Nitrogen Atmosphere

EXPECTED RESULTS





DSC result of PCL pellets that shows melting point around 60 °C taken from [4]

X-RAY DIFFRACTION (XRD)

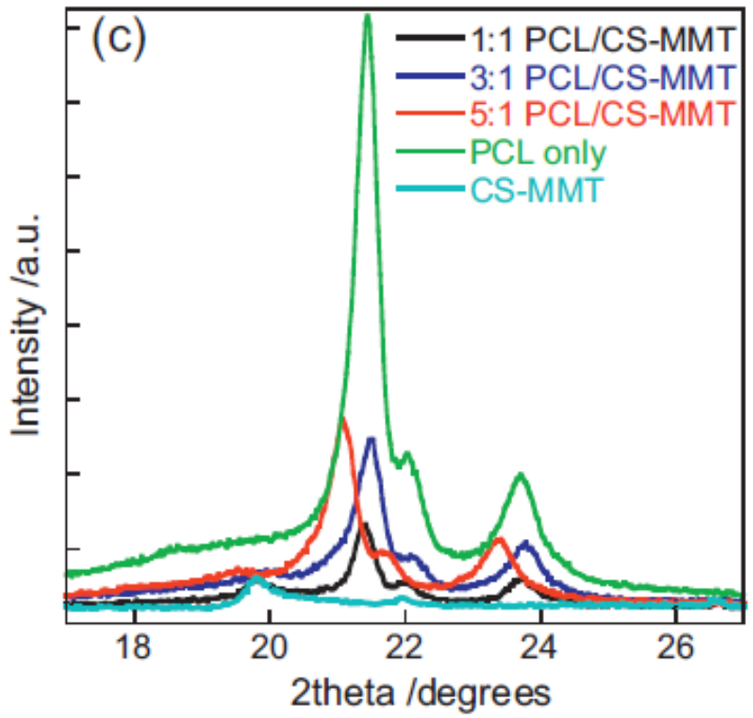
Justification: *It determines the diffraction angles that the material shows, allowing the confirmation that the material we obtained is what we desired. Also, differences regarding solvents usage can be shown trough this method.*

Parameters:

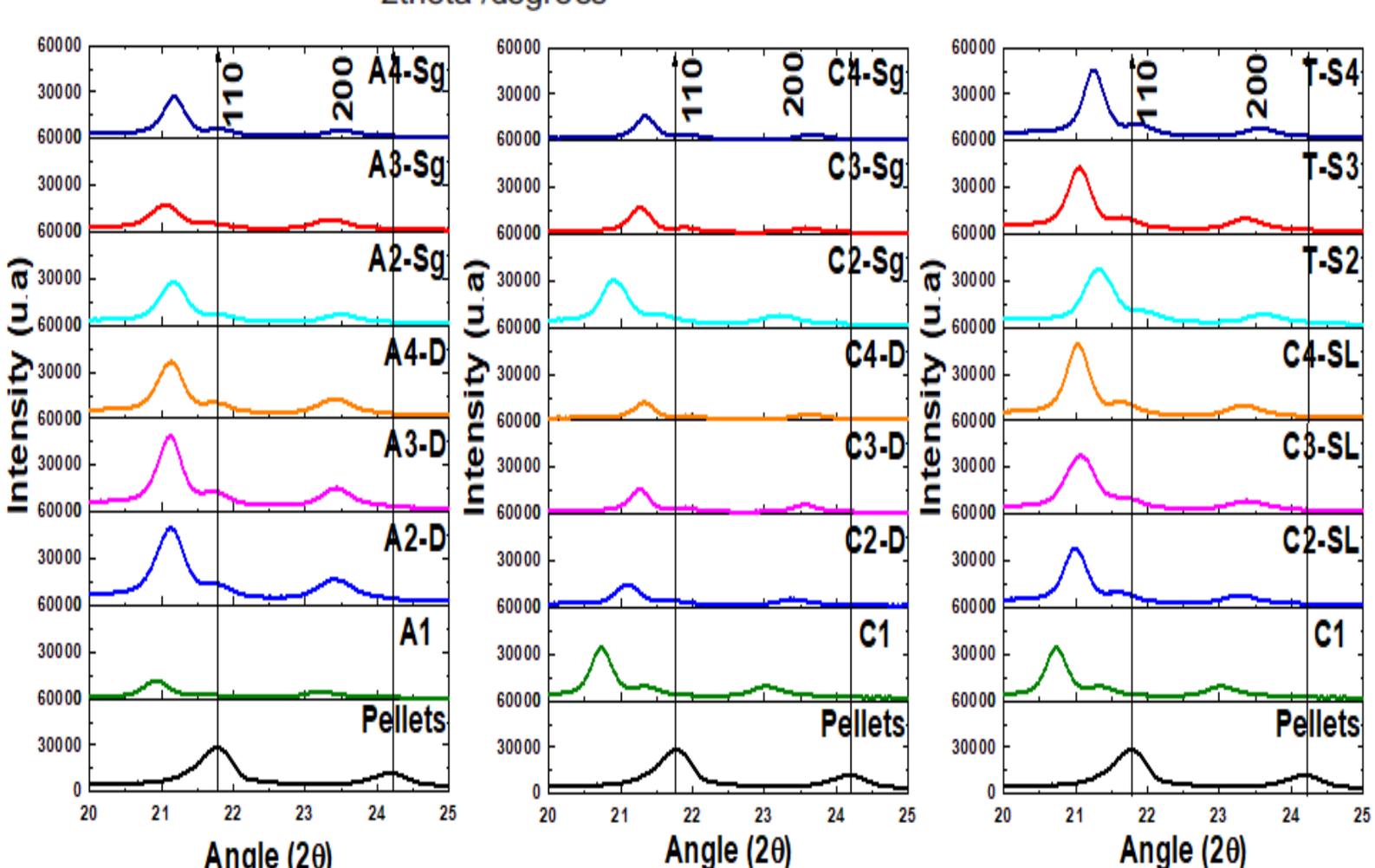
PANalytical EMPYREAN

2Θ=6-80°

EXPECTED RESULTS



Green curve shows typical PCL diffraction planes taken from [5].



SCANNING ELECTRON MICROSCOPE (SEM)

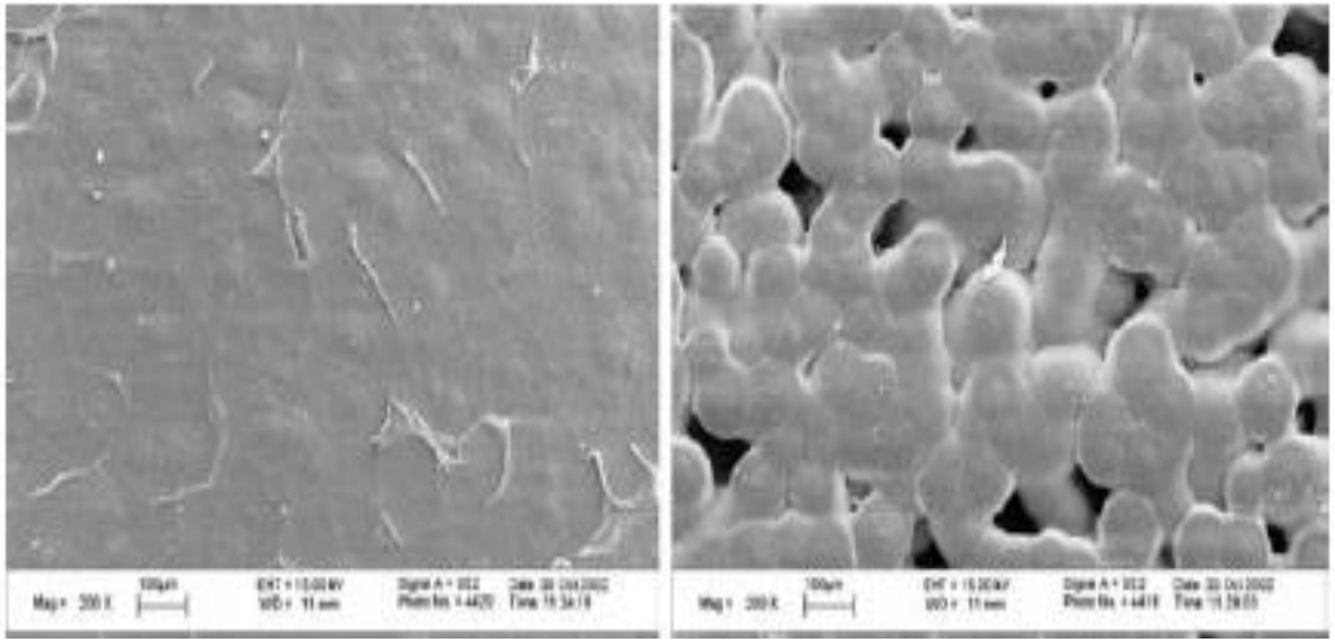
Justification: ***T****he morphology of the surface of the sample can be observed. This shows patterns, defects, porosities and structures of the surface. Finally, the dispersion of the nanoparticles and their sizes can also be observed with these techniques. These parameters are important because, normally, good dispersion is better for the performance of the material, as well as less porosities (in some cases, as it is in mine).*

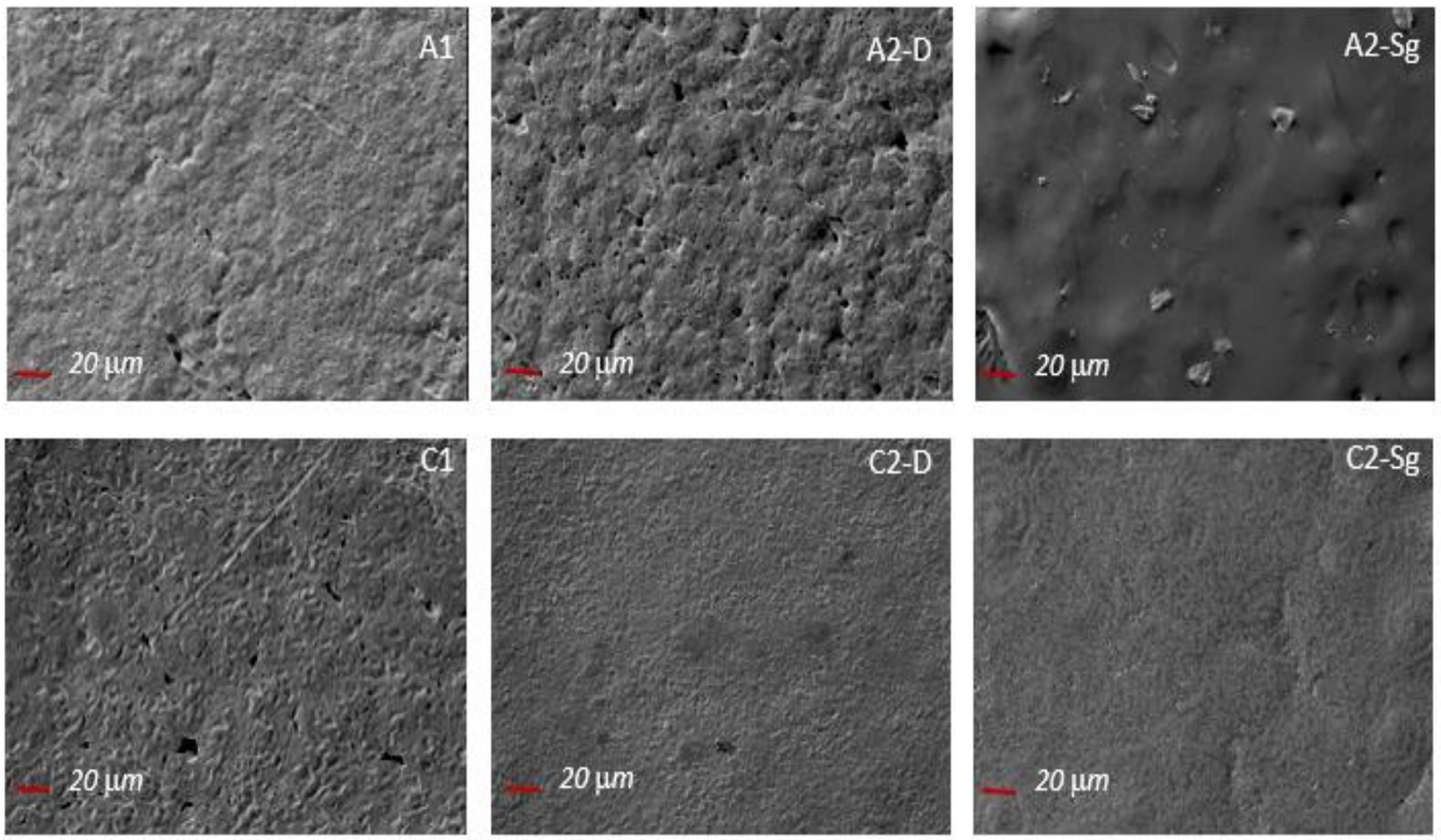
Energy Dispersive X-Ray Spectroscopy (EDS) can be performed in order to ensure that a composite has been formed and the presence of the desired nanoparticles in the material.

Sample preparation:

A gold coating with sputtering technique is required.

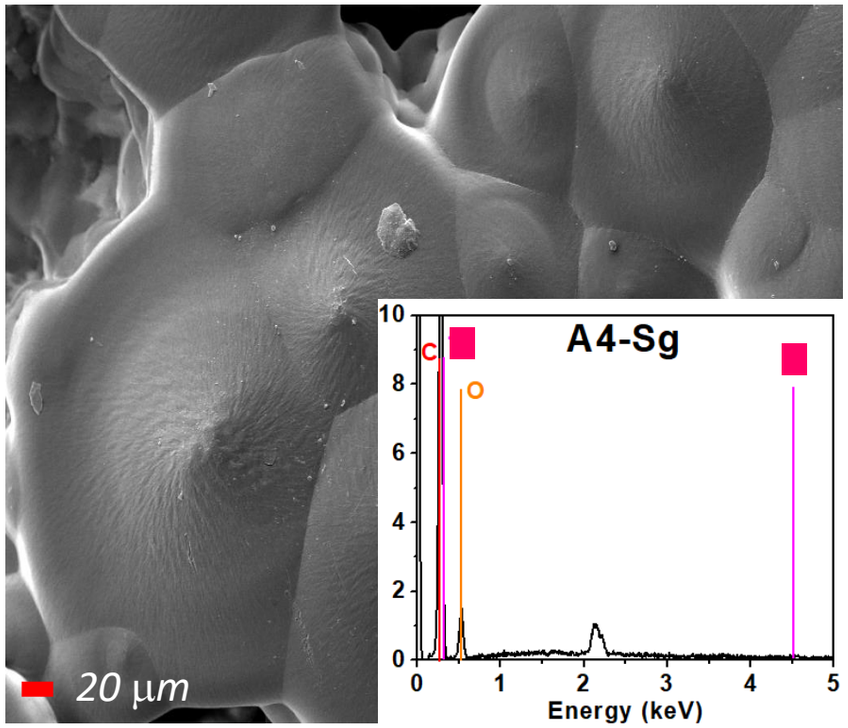
EXPECTED RESULTS

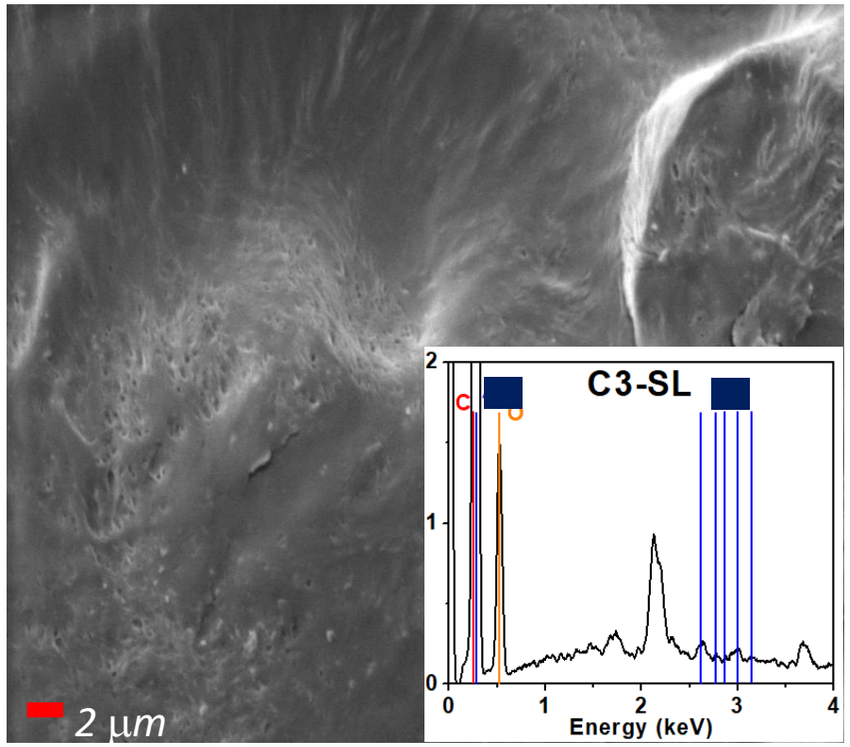


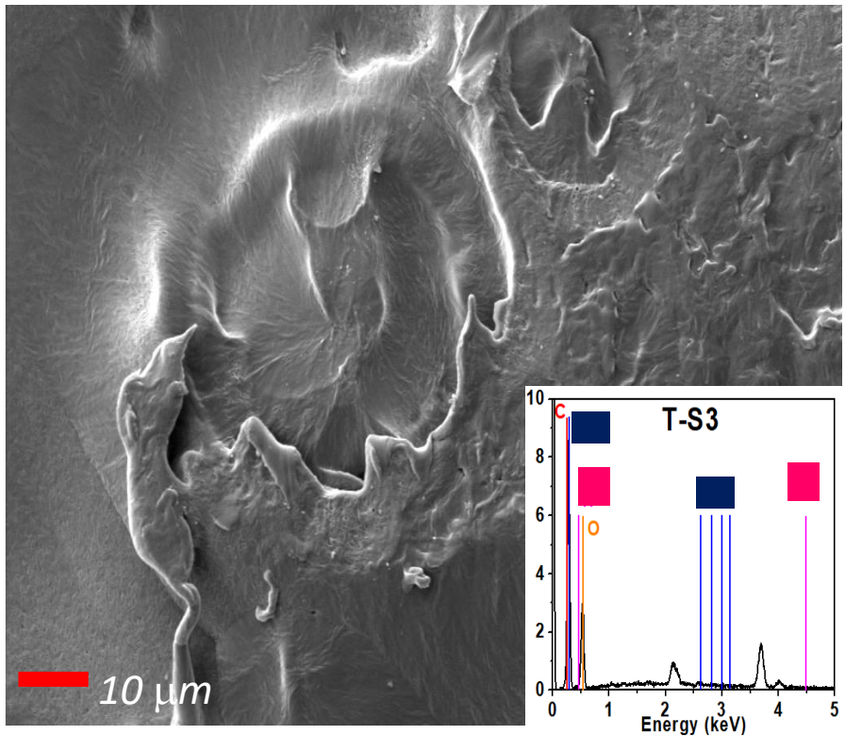


PCL film morphology observed through SEM taken from [6].

EXPECTED RESULTS







DYNAMIC LIGHT SCATTERING (DLS – ZETASIZER)

Justification:

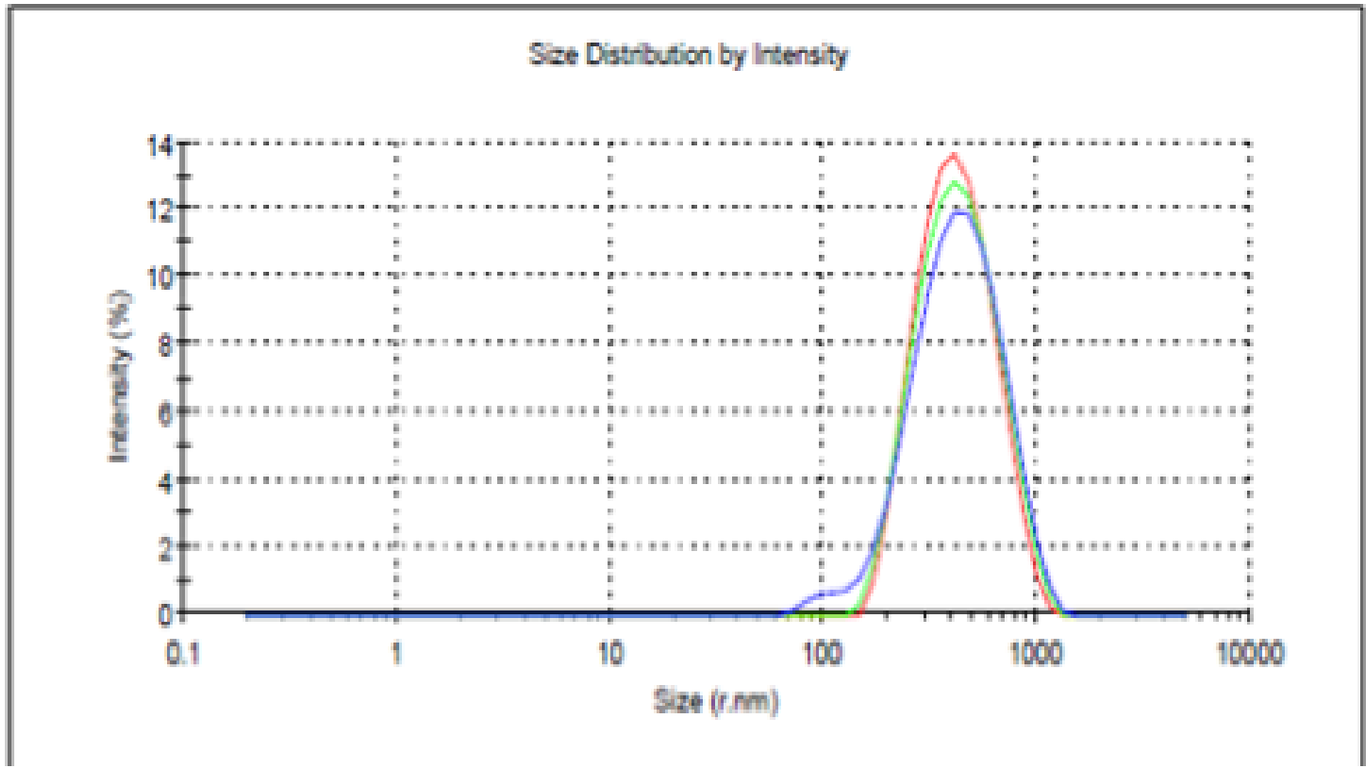
Acts as a complementary technique of the microscopy, since it allows us to know the dispersion of the medium size of the nanoparticles. The statistical distribution that this method presents allows us to confirm that we are working with the correct size of particles we desire.

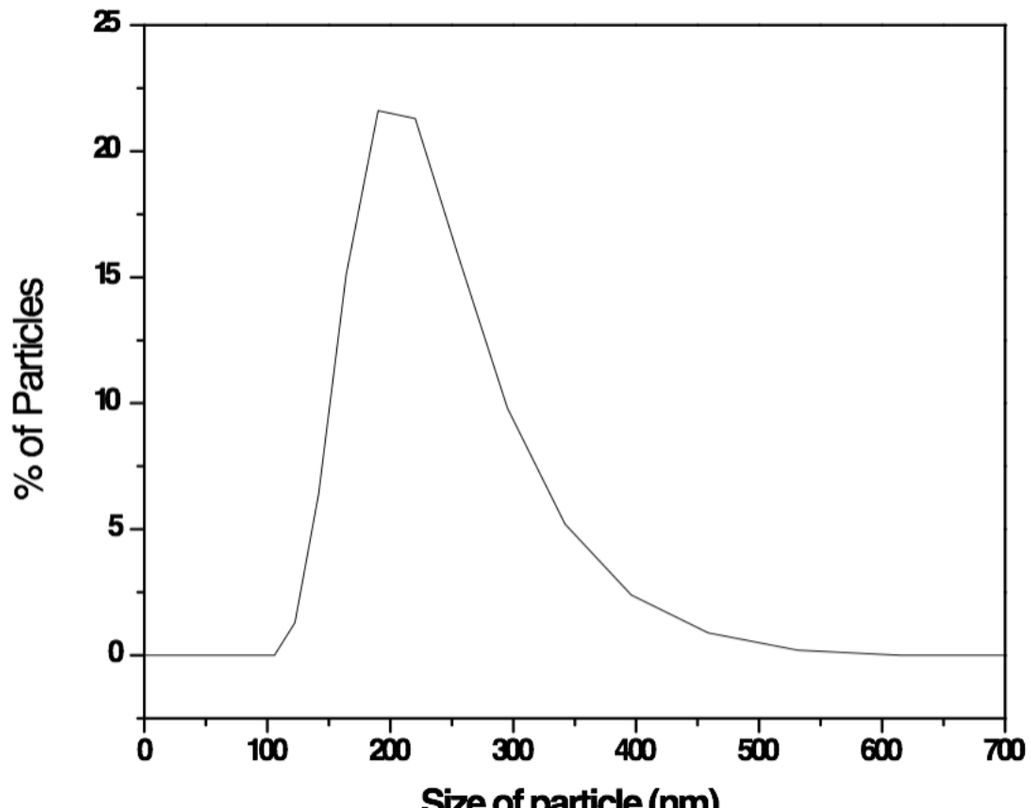
Sample preparation:

Nanoparticles diluted in distilled water

An ultrasonic bath is required for dispersion.

EXPECTED RESULTS





Size distribution of nanoparticles obtained via DLS which shows bigger particles than expected and suggest agglomeration and cluster formation taken from [8]

TRANSMISSION ELECTRON MICROSCOPE (TEM)

Justification:

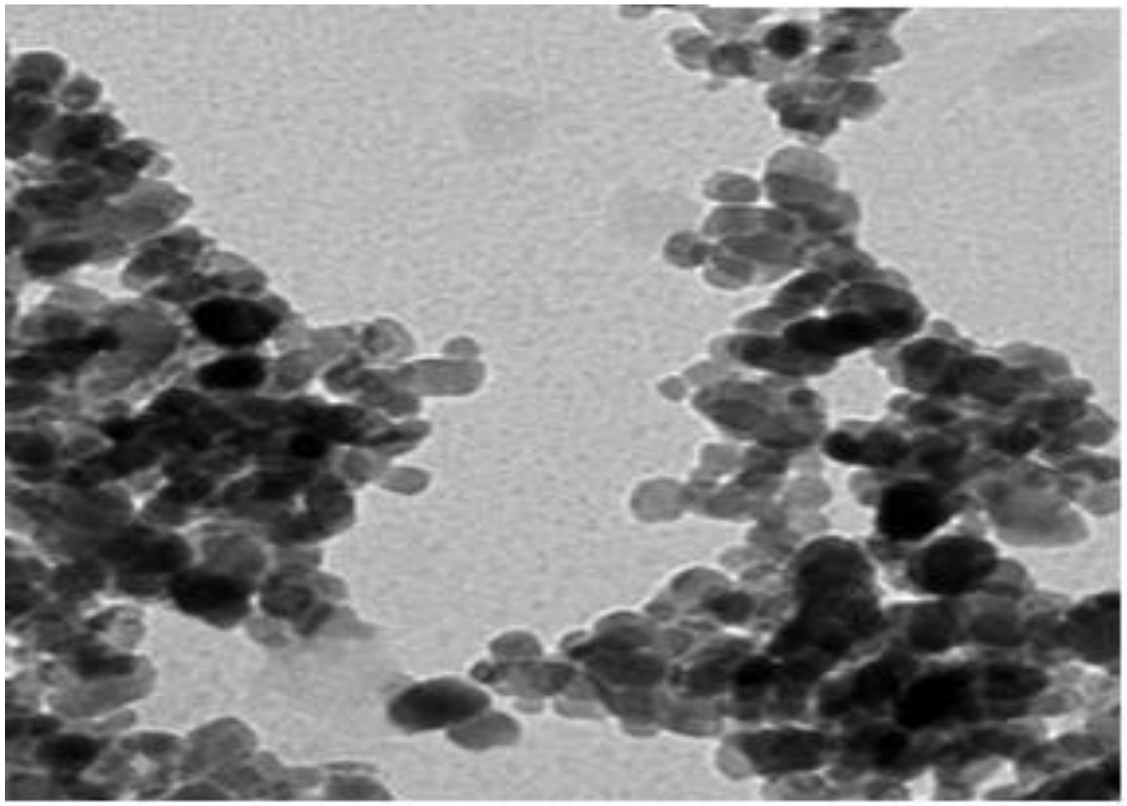
This technique allows us to see trough the nanoparticles. In case that SEM shows big clusters or aglomerations, with TEM it is possible to see the real composition of these aglomerations and the size of the nanoparticles. Nanoparticles presence is important because of the surface-to-area ratio that is increased.

Sample preparation:

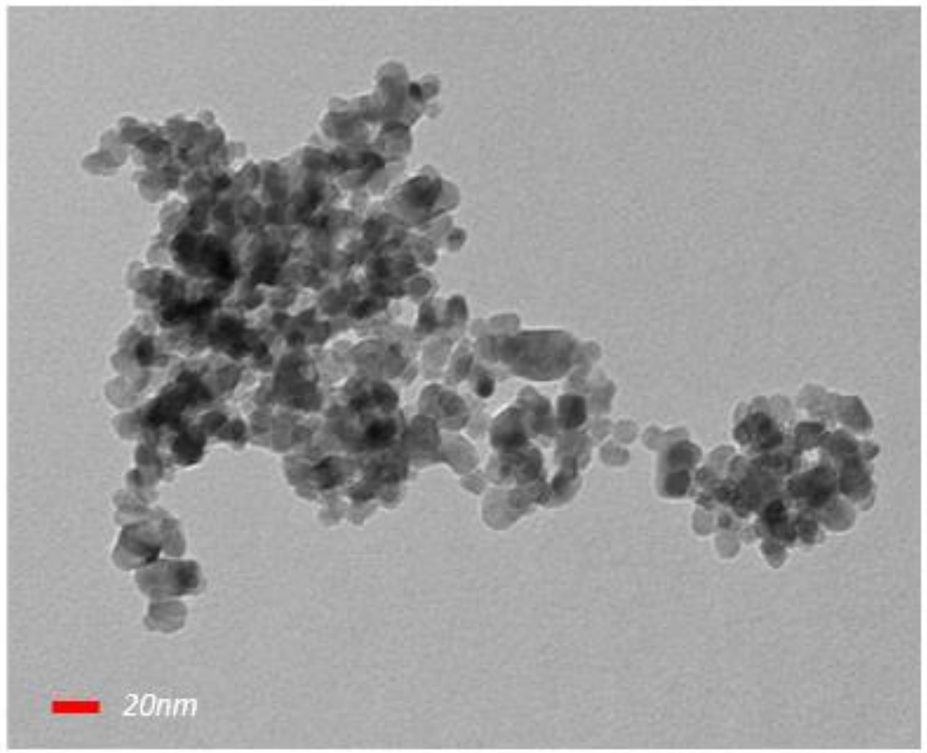
The nanoparticles are diluted in alcohol

Dispersion in ultrasonic bath is required

EXPECTED RESULTS



Observation of nanoparticles using TEM microscopy taken from [7]



ULTRAVIOLET-VISIBLE SPECTROSCOPY (UV-VIS)

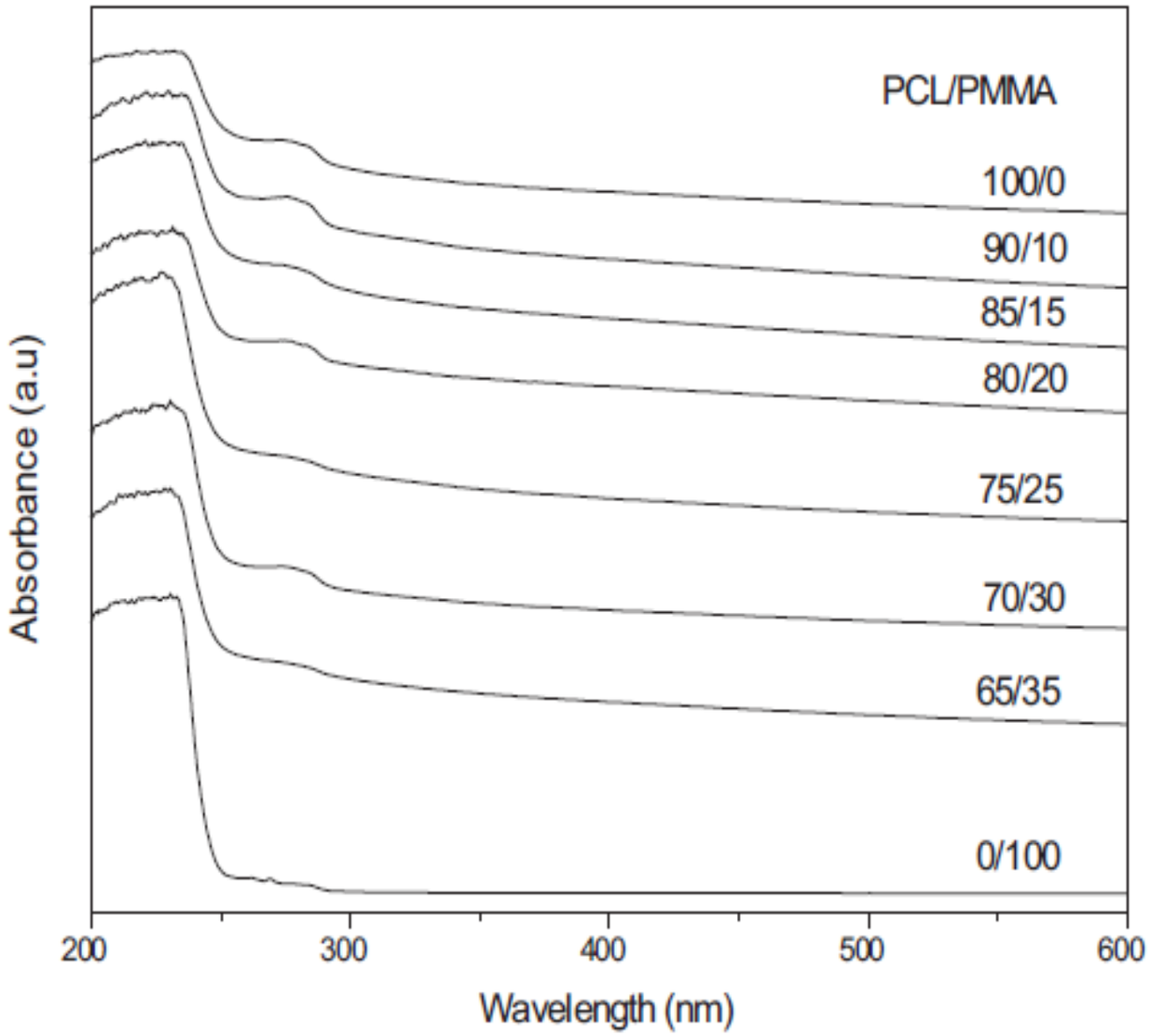
Justification:

Since the application of my material interacts with the sun rays and has photoactivable dopants, this characterization is crucial to know how the molecule behaves and excites along different wavelengths and times. The wavelength is important since it stablishes a parameter for the dopant and its activity in order to increase the efficiency of the photoactivation.

Parameters:

200nm to 400nm

EXPECTED RESULTS



UV-Vis spectra of a PCL membrane taken from [9]

FOURIER TRANSFORMED INFRARED (FT-IR)

Justification:

With this method, we can observe the vibrations of the bonds involved inside the material (also known as the blueprint of the material). These interactions are related to the behavior of the material in the application and in the environment, as well as the other characterizations performed previously. Some shifting in the vibrational bands can be observed, thus confirming the effect of the dopants and changes induced to the material.

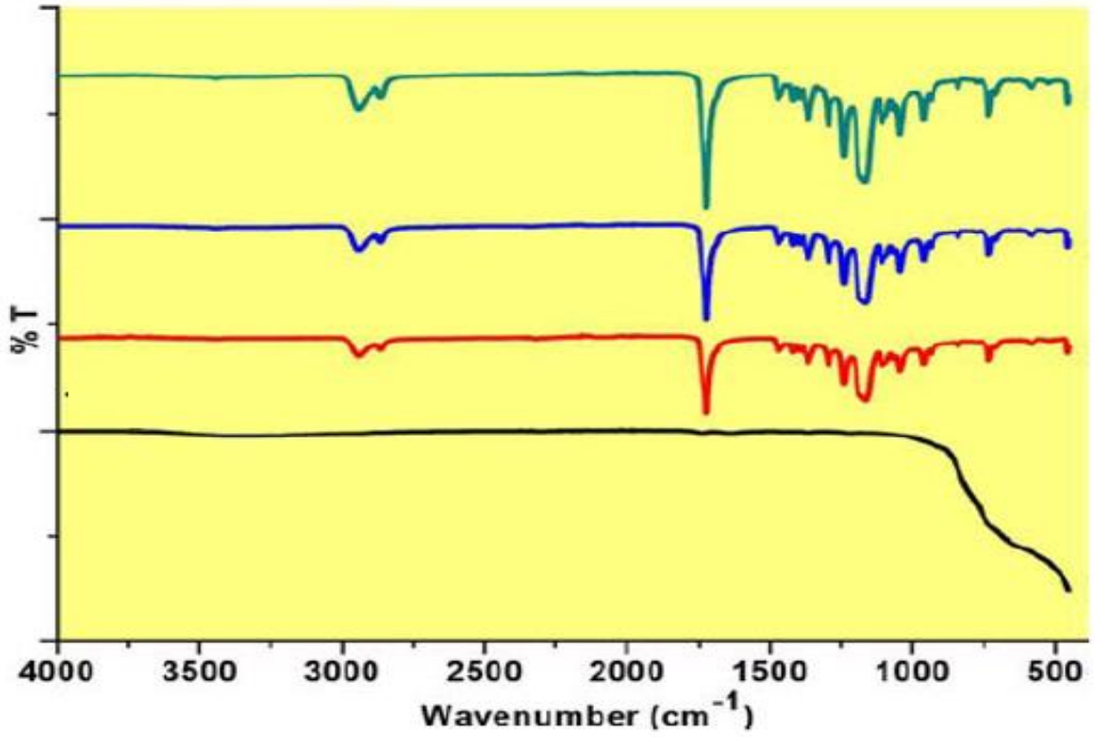
Parameters:

PerkinElmer FT-IR/ FIR Spectrometer Frontier

4000-400 cm^{-1}

16 scans

EXPECTED RESULTS



FT-IR result from PCL doped films and their comparison, taken from [10].

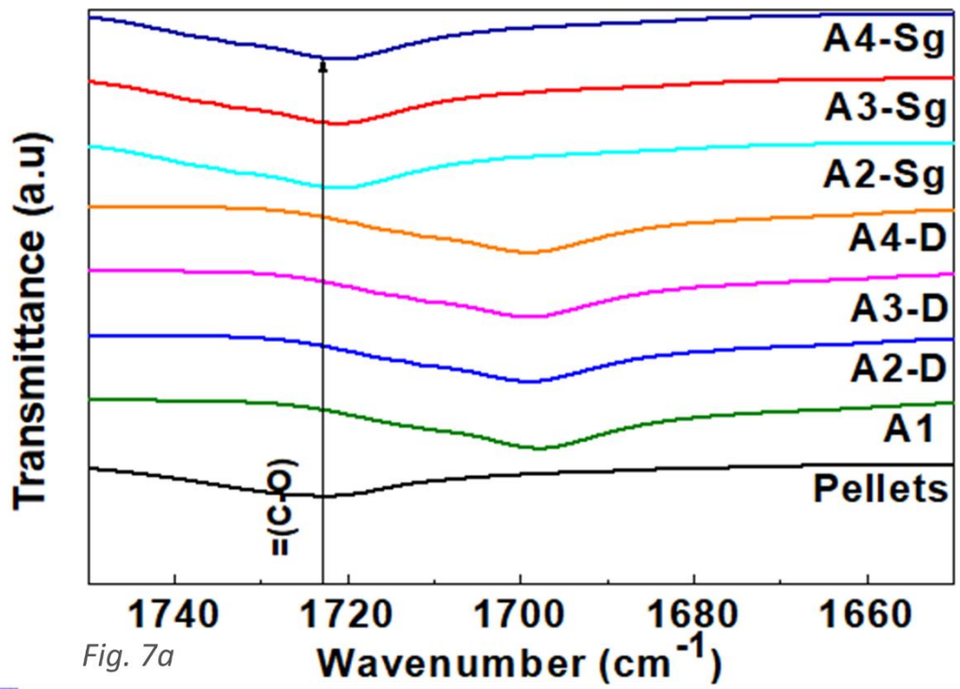
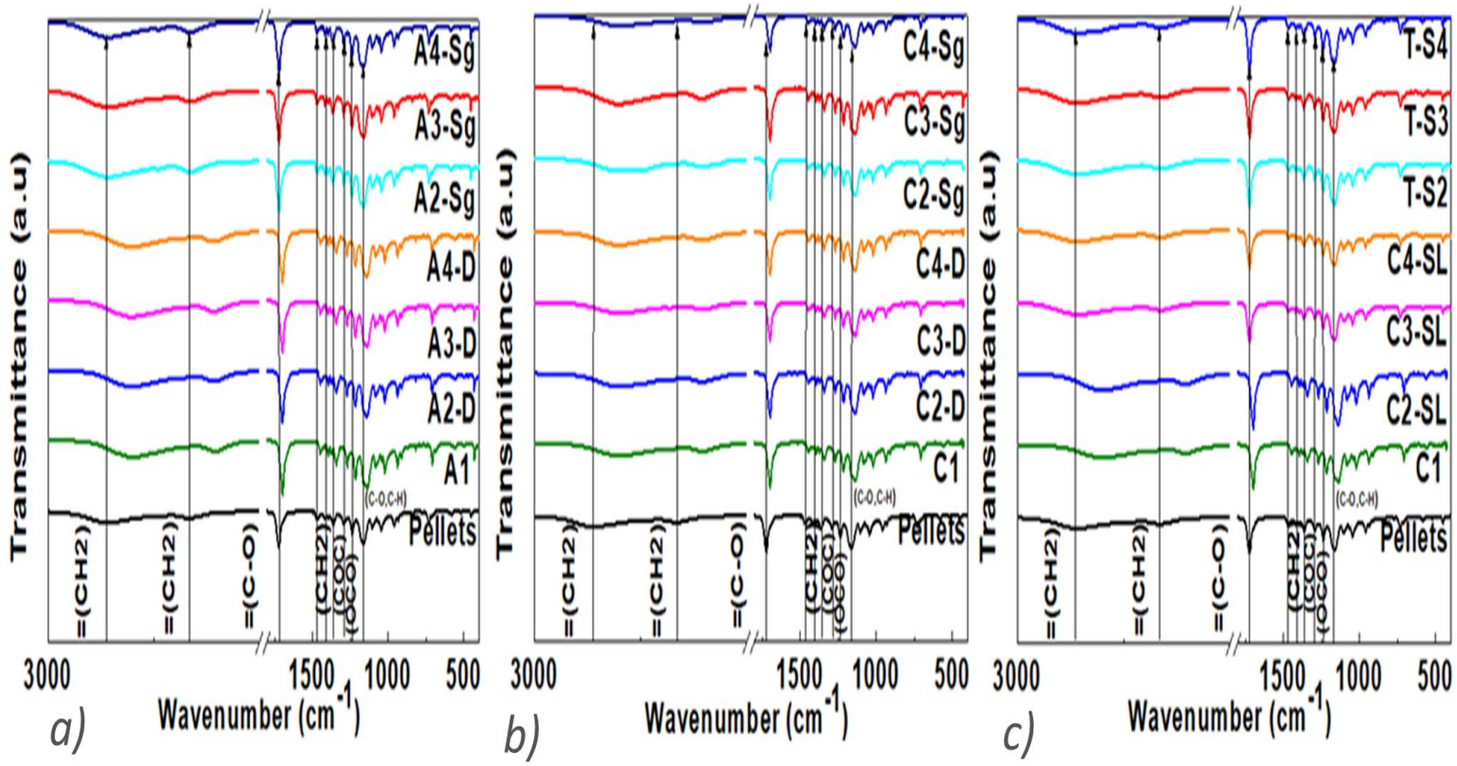


Fig. 7a



CORRELATIONS

- *TGA with DSC: a relationship between the thermal degradation of the material and its behavior as temperature increases with the phase change endotherms provided by DSC*

- *DSC with XRD: relationship of the percentage of crystallinity of the material, provided by the endotherm´s heat capacity´s value and the diffractogram.*

- *XRD and SEM: validate the phases present in the material and the material present in the diffractogram and compare them with what is shown in the images.*

CORRELATIONS

- *SEM/TEM with Zetasizer: a validation and relationship may be stablished to see the size of the particles and their distribution in the surface of the material.*

- *UV-VIS with FT-IR: modification of the molecule can be observed trough UV-Vis (mineralization of the particle) and the vibrations generated with this modification can be observed in FT-IR thus allowing a comparison.*

- *FT-IR with XRD: The characteristic angles observed in the diffractogram may be related with the vibration of the carboxyl groups characteristic of the polymer I´m working with and then the bondings found in the FT-IR diagram with the phases of the material observed in the diffractogram.*

CONCLUSION

The presented characterizations are needed in order to understand the material´s behavior and its interaction both with the solvent and the nanoparticles added.

Further characterization methods must be performed in order to fulfill all the necessities the project has which include: Contact angle, HPLC, Mechanical test (uni-axial and bi-axial) and Roughness measurement (Alicona).

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