

Recycled PET Nanofibers Produced by Electrospinning Technique

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ABSTRACT

Increasing concerns for the preservation of environmental and sustainability of resources have been draw and the use of recycled polyethylene terephthalate (PET) is one of alternatives to non-renewable resources in material technology.

The main purpose of the work is to produce by recycled PET a nanofiber by the electrospinning technique. The work consists of polymeric solution preparation to be electrospun, followed by definition of parameters of electrospinning process and finally the nanofiber production. Afterwards, characterization of PET nonwoven nanofiber that includes Thermal analysis, Thermogravimetric analysis, Scanning Electron Microscope (SEM) and contact angle measurement. The results show that the conditions of electrospinning process adopted were suitable to obtain a nonwoven fiber, with filaments in nanoscale. Moreover, the fiber, even with the processing, the PET polymer retained its properties without showing signs of degradation, thus creating a new alternative to the use of the material, with high functional value.

Keywords: Polyethylene terephthalate, PET, Nanofibers, Electrospinning.

1 INTRODUCTION

The present work aims to contribute to the sustainability of the 3rs (reduce, reuse and recycle), practical actions that establish a more harmonious relationship between consumers and environment, through the proposal of development of nanofibers, which are produced from recycled PET, providing new material in polymer platform, able to equate, or even replace, previously used materials for different applications with the advantage of differential cost, integrating efficiency and advanced technology.

The universe of science and technology, in addition to being in constant evolution, has been revolutionized from the discovery of nano-scale materials, by being able to introduce features that support to optimize properties not

previously provided by the same materials as micrometric scale^[1].

Nanofibers are a new class of materials with special properties. Within this class, are the materials with dimensions varying from filaments less than 100 nm to 1000 nm. The nanofibers are of great interest in studies, due to its unique structure and properties due to this structure^[2]. There are different methods used in the production of Nanofibers, such as, centrifugal spinning, molten material blowing, bi-component wiring, phase separation, synthesis of mould and electrospinning technique^[3]. The technique selected for this study was that of electrospinning technique, chosen held due to instrumental simplicity the process required and low cost necessary for the development and implementation in the process of production of nanofibers. The formation of nanofibers by electrospinning technique is in principle controlled by an extensive group of parameters, which are unique for each polymer and electrospinning technique system chosen. Thanks to this wide range of parameters when set, that can produce nanofibers with pore diameter, guidance and controlled, based also on control of environmental conditions, especially humidity, contributing to the stability of these nanofibers.^[1,2,5]

In a historic survey, performed by Zanin et al.^[4], the technique of electrospinning technique has been developed for over 70 years. The electrospinning technique is a technique applied in the process to obtain solid fibers of micrometric to the nano scale, from a polymer material in the molten state or in solution. The system that make up the process of electrospinning technique should work in environment with controlled atmosphere and is basically made up of a pump, a syringe, a sink, two electrodes and a source of tension, as shown in Figure 1. This technique uses an electrical charge to turn the polymer solution in filaments.^[3,4,5]

2 METHODS

The nanofibers was based on flakes of recycled PET. The steps of production of nanofibers and

characterization are presented in the flowchart shown in Figure 1.

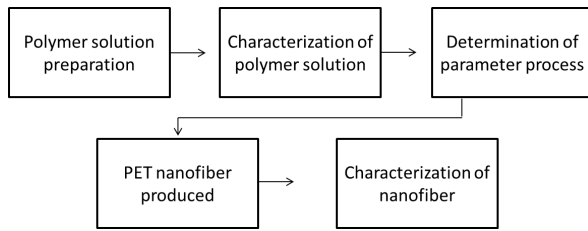


Figure 1: Flowchart of PET nanofiber production.

2.1 Solution preparation

The PET was added to the jacketed reactor and then the solvent Hexafluoro-2-propanol (HFIP) was poured. The solution was maintained by constant stirring for a period of 2 hours with a controlled temperature of 20° C. The solution obtained was then discharged from the reactor and stored in an appropriate bottle. The full route of the solubilization and details of the process is presented in the table 1.

Table 1: Conditions of process used to produce nanofiber.

PET	5 (wt%)
Solvent	HFIP
Proportion	95 (wt%)
Preparation of the solution	jacketed reactor (20 °C, 2h)
Cylinder Rotation	30 rpm
Feed flowing	3 mL/h
Distance (cm)	10 cm
Tension (kV)	25 kV
Temperature (°C)	25 ± 3
Moisture (%)	50 ± 3
Time (h)	1h

2.2 Characterization of the solution

In order to study the process and find the best condition to electrospun the polymer solution and achieve process (batch-to-batch) with repeatability, the solution was analyzed as the conductivity and the viscosity.

3.2.1 Viscosity

Punctual polymer solution viscosity was analyzed by a shear rate generated at 150 rpm. The determination was conducted on a rotational Viscometer with concentric cylinder geometry, Brookfield brand, model DV-III Ultra, with controlled temperature and using a spindle for 34 small center. To obtain a more reliable measure, the reading of the viscosity was made immediately after starting the

analysis, due to the fact that the measuring system was used open, which favours the evaporation of solvent, thereby changing the concentration and viscosity of the solution. The ambient temperature was approximately 20° C.

3.2.2 Conductivity

The conductivity of the polymer solution is a parameter to be considered, bearing in mind that the smaller the conductivity, the greater the difficulty to form filaments.^[1] The solution then was taken to the conductivity meter MC model 226 of the brand Mettler Toledo. The temperature of the solution at the time of analysis was 25° C, and the environment had a relative humidity of 76%. The calibration of the equipment with the electrode in air and then immersed the electrode in polymer solution. The analysis was performed in triplicate.

2.3 Electrospinning technique

The process of electrospinning occurred inside a hood, with controlled room temperature of 23° C ± 3, and relative humidity of 50% ± 3. The electrospinning set up used is shown in Figure 2.

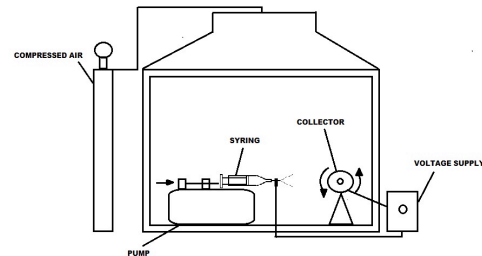


Figure 2 : Set up of Electrospinning process.

2.4 Characterization of nanofiber

3.4.1 Morphology observation

A sample of nanofiber generated was segregated for verification of their morphology, in order to evaluate the surface and the nanofiber diameter. The sample was then affixed to a support, known as stub, with the aid of copper double-sided tape and covered with gold. After the preparation, the sample was taken to the scanning electron microscope with Field emission SEM-FEG FEI Quanta 3D model brand, which images were obtained with the magnification of 20000 times.

3.4.2 Contact angle

The contact angle measurement was performed aiming to identify whether the setting of the PET nanofibers could affect the PET contact angle. According to the literature^[6], the value of the contact angle of the PET is 70 ± 5. The analysis took place in the laboratory of

Electrochemistry and Nanostructured Materials, (LEMN), located on the premises of the Federal University of ABC (UFABC), using the goniometer equipment Ac stabilizer, brand Summit, provided with a still camera pixelink brand. For the test, the sample is placed on a glass slide and deposited gently using an automatic pipette a drop of deionized water with volumes of 5 μ l. Moisture from the lab at the time of the test was 58% and the temperature was 22.5° C. The analysis was performed using the Visiodrop software.

3.4.3 Termal analysis

To evaluate the thermal profile of the samples of recycled PET flakes and PET Nanofibers, the both samples were submitted to the gravimetric analysis. The equipment used was the Analyzer Mettler Toledo, TGA/DSC model 1 Stare System. The samples were then submitted to an an inert atmosphere and the temperature range range of 25 to 800° C, under gas flow to 50 ml/min and a heating rate of 10° C/min, with the use of the software STARe.

3 RESULTS

3.1 Charactezation of the solution

The results obtained for the conductivity and viscosity of the solution will serve as a sample history to facilitate the reproduction of the same. The table 2 shows the results obtained for the conductivity of polymer solution.

Table 2: Conductivity measurement of PET solution before electrospun.

Readings	Conductivity (μ S /cm)
1	0.36
2	0.37
3	0.35
Avarege	0.36

The table 3 shows the results obtained through the analysis of viscosity. These results are of great importance, due to this parameter has a great influence on the profile and diameters of nanofibers.

Table 3: results obtained in Rotational Viscometer for recycled PET solution.

Sample	PET Solution
Viscosity (Cp)	13.4
Torque (%)	54.0
Shear Stress (D/cm ²)	22.8
Shear Rate (1/s)	42.0

The values obtained for the viscosity test are related to pre-established conditions, remembering that, through this technique cannot validate the value of intrinsic viscosity of

the sample, and the results represent the behavior of the sample under conditions of temperature and rotational speed of the spindle. The choice of spindle was held based on the percentage of torque, whereas to obtain reliable results these values must meet between 10% and 100%.

3.2 Nanofiber

At the end of the process of electrospinning technique obtained a nonwoven fiber mat white, as seen in figure 3.



Figure 3 : Photograph of nonwove nanofiber based on recycled PET.

3.3 Charactezation of the Nanofiber

In Figure 4, it is possible to observe with 20000 times of magnification the recycled PET filaments. The filaments have a tendency to rectilinear guidance, no direction and standardized with dispersion of diameters size.

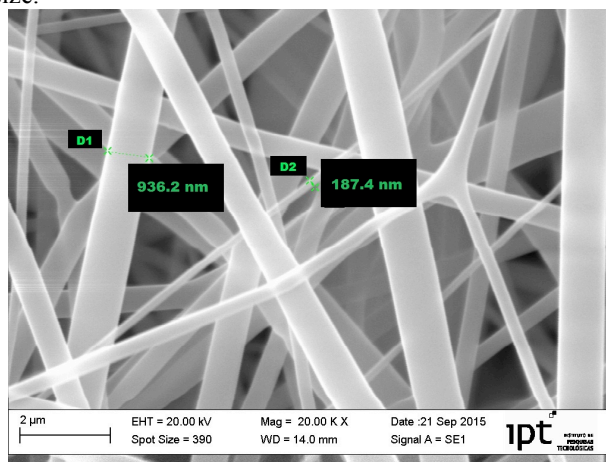


Figure 4: Scanning electron microscopy of nanofiber sample of recycled PET (20000 x).

As highlighted in the Figure 4, the fiber diameter vary, but within the range of nanometers. Measuring the diameter of two filaments that apparently represent the

extremes of larger and smaller diameter, we have the dimensions of 187.4 nm and 936.2 nm, respectively.

The results of static contact angle for the PET nanofiber were collected immediately after deposit of the drop of water on its surface, and each minute for six minutes. The average result is presented in table 4.

Table 4: Result of contact angle.

Liquid	Substrate	Average result of contact angle
H ₂ O	PET nanofiber	75.6°

The Figure 5 shows the images of the surface of the nanofiber without the drop (a), with drop in time zero (b) and (c) drop after five and ten minutes (d), respectively.

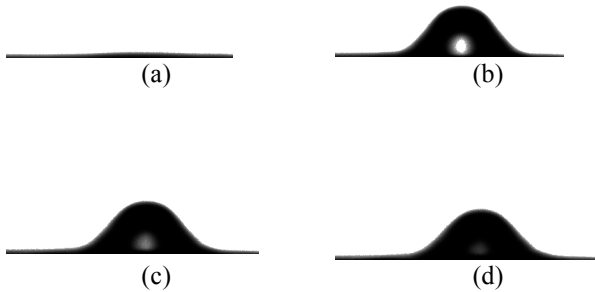


Figure 5: Images obtained through the sessile drop test.

It is observed that over time the drop tends to conform, wetting the surface, showing so a character of wettability intermediary, noting that hydrophilic materials are considered with static contact angle below 50° and hydrophobic materials with contact angle greater than 90°.

For thermal analysis, the thermogravimetric analysis, show that the degradation profile for recycled PET and recycled PET nanofiber are similar for both (figure 6), indicating that the polymer processing has not changed the thermal properties of PET.

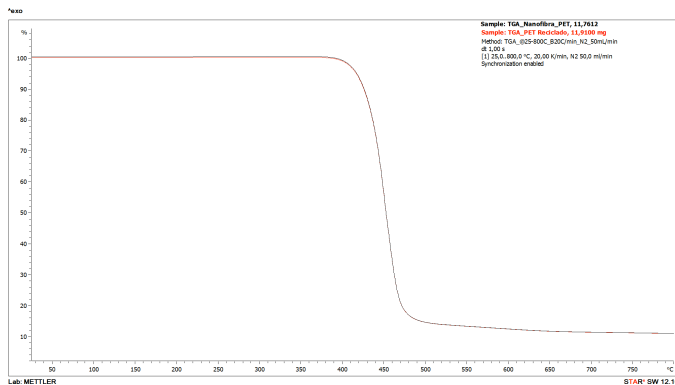


Figure 6: Curve Superpositions of thermogravimetric analysis of recycled PET and recycled PET nanofiber.

4 CONCLUSION

Conducting an overview of the results obtained, it can be concluded that not only it is possible to obtain nanofiber based on recycled PET via electrospinning process, but also conclude that it is possible to obtain an alternative for processing recycled PET. Moreover, the fiber, even with the processing, the PET polymer retained its properties without showing signs of degradation, thus creating a new alternative to the use of the material, with high functional value.

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