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Utilization of Pineapple Crown Fiber and Recycled Polypropylene for Production of Sustainable Composites

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Abstract: Nowadays, the production of consumer goods is based on the use of non-renewable raw materials, which in recent years has been performing as a problem for the environment. Considering the large number of available biofibers in nature, their use in the development of polymeric composites has inevitably emerged, it is also necessary to take into account the countless discarded plastics that still have the potential to be reused. In this work, fibers were extracted from pineapple crown residues and utilized to compose sustainable composites using recycled polypropylene from cups discarded in the trash as a matrix. However, it is known that for good performance, it is necessary to achieve a good chemical interaction between the fiber and the matrix. In order to improve this interaction, alkaline mercerization treatment was carried out on the surface of the fibers removing some components incompatible with the polymer. In this work, the effect of the mercerization treatment on the properties of the fibers was studied, as well as their interaction with the matrix. The effect of fiber concentration on the mechanical and thermal properties of composites was also evaluated. Levels of 5 and 7 wt% were used for both natural and mercerized fibers. A decrease in the number of degradation stages was observed through thermogravimetry analyses (from four in natural fiber to two in mercerized fibers), showing that the mercerization performed on the fibers was effective. An increase in the degree of crystallinity of mercerized fibers was also observed through the results of X-ray diffraction. Both techniques indicate that amorphous compounds, such as hemicellulose and lignin, were partially removed. Through the tensile test, it could be noted that all composites presented higher values of de elastic modulus than recycled polypropylene without added load; however, there were no differences in the elastic modulus between the different types of fibers and load levels. Therefore, it is interesting to use fibers as reinforcing agents in polymers;

however, the treatment did not increase the mechanical properties of the composites. In parallel, other factors, such as the dispersion of the components, must be taken into account to justify this result.

Keywords: Recycling; composites; polypropylene; biofibers; pineapple crown

1 Introduction

People live in a very worrying time when regarding the land overloading, that is when a broader set of resources is used than nature can renew. Consumer goods production is geared mainly towards the use of non-renewable materials, which in recent years has been a problem due to population growth and overconsumption [1,2].

Therefore, it is necessary to develop a worldwide awareness about the use and consumption of materials, as well as, to prioritize sustainable solutions [3]. The use of natural fibers for the production of polymer composites represents an alternative to the use of synthetic fibers. In addition to being low cost, they are renewable, abundant, and diversified due to the country's consolidated agriculture and decrease the amount of plastic used [4].

The pineapple crown has excellent potential for obtaining fiber, due to the high production of pineapple in the country and considering that pineapple is not fully utilized, these parts become an agro-industrial waste. The pineapple crown can be an alternative for use as reinforcement in polymeric composites since its use is still very limited mainly to the application in the textile and handicrafts production [5].

In addition, among the existing materials, synthetic polymers stand out when the subject is the environment since when they are improperly discarded, they are significantly contributing to increasing undesired pollution. After the useful life of these materials, most of them do not go through the recycling process and go directly to nature, taking a few centuries to decompose. Recycling them is an attractive and viable alternative to reduce this environmental impact. Also, it is a way to preserve non-renewable raw materials, save energy, reduce the costs of the final product, and generate jobs and income [6].

The interfacial region is responsible for adhesion between phases, being a fine line between the matrix and the reinforcement, which has unique properties. However, for fibers and the polymer matrix to work together effectively in a given product, there must be excellent adhesion between them. Nevertheless, it is well known that these are materials with low addition between them, i.e., incompatible. The incompatibility between fiber and polymer is due to their polarity difference since the fibers are hydrophilic and the polymers are hydrophobic, resulting in a weakening of the interfacial interaction of the composite and, consequently, a low-stress transfer capacity from the matrix to the fibers. Thus, it is necessary to use chemical or physical modifications on the fiber surface, which consist of the removal of some components from the fiber. Hemicellulose and lignin are responsible for the high water absorption of the fibers and are amorphous. The partial or total removal of these materials can increase the load compatibility with the matrix, which consequently will influence the mechanical properties of the obtained composite [7].

The main advantage of this work is the use of waste as raw material for obtaining composite materials since both the matrix and the reinforcement come from waste. Furthermore, from these, it can be demonstrated that it is possible to produce materials with added value, with remarkable properties, and still apply them in products. These sustainable materials are likely to show reduced properties compared to virgin materials. However, they can be applied to products that do not require as many mechanical requests, whereas the use of virgin materials can be focused on products that require greater performance. It can be a viable alternative to the large number of polymers that are discarded in the environment and also to the use of non-renewable materials.

Thus, fiber-reinforced polymer composites can be used to change properties and increase product performance as well as reduce costs and marketing strategy. Therefore, the main objective of this work was to use two residues, pineapple fiber and discarded PP, for the production of sustainable composites. Besides that, it will be carried out and evaluated one surface treatment on fibers. Finally, the effect of this fiber's treatment on the mechanical, thermal, and morphological properties of the composites will be evaluated.

2 Materials and Methods

2.1 Fibers Obtaining

Pineapple crown leaves obtained from a local market (Pelotas-RS, Brazil) were thinned, washed, and oven-dried at 65°C for 48 h. After drying, which aims to remove moisture, the leaves were ground in a Marconi knife mill, model MA 340. Then, the fibers (denoted as natural fibers) were sieved using the 32-mesh sieve in order to obtain a uniform grain size.

2.2 Fibers Modification

The chemical treatment of the surfaces of the fibers, known as a mercerization process, was made by an alkaline solution of sodium hydroxide (NaOH), aiming to improve the adhesion between fibers and matrices. For this, 20 g of fibers were used for each 200 mL of NaOH solution, with 10 wt% concentration. The fibers were kept into this solution for 1 h at 80°C under mechanical agitation. Then, they were recovered by filtration and washed distilled water until the final solution reached pH 7, removing any NaOH residue. For last, the mercerized fibers were oven-dried at 65°C for 16 h. Fig. 1 presents a flowchart of the methodology used for the mercerized fibers obtention [8].

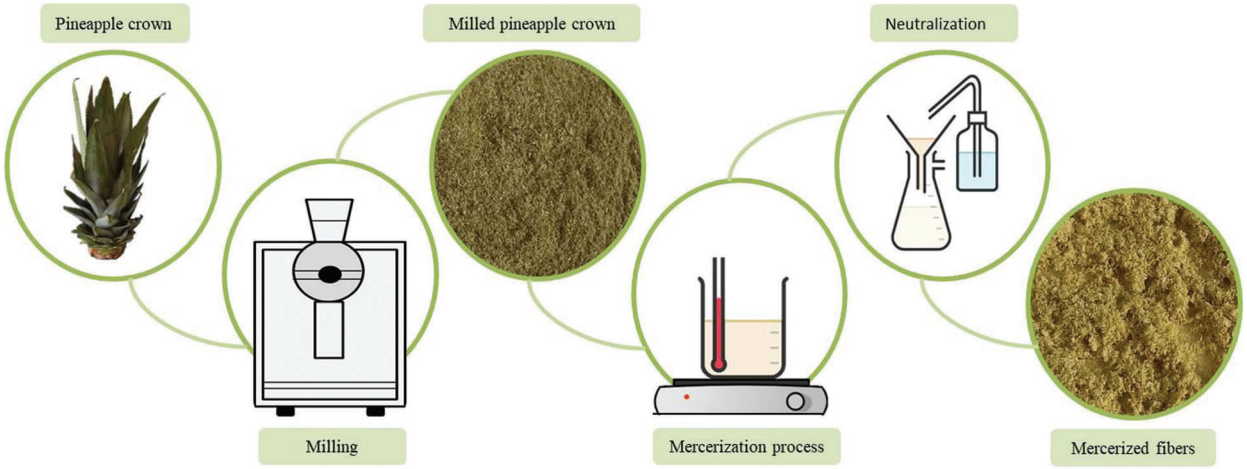

Figure 1: Flowchart of the mercerization process

2.3 Polypropylene Recycling

In order to use polypropylene (PP) as a polymeric matrix, PP plastic cups found in the streets of downtown Pelotas's city were collected. The cups were sanitized, dried at room temperature, and cut into smaller pieces. These smaller pieces were ground in a knife mill to ensure a grain size close to that of the fibers, thereby facilitating the mixing process in the extruder.

2.4 Composites Processing

The composites were obtained by melt blending processing to promote good homogenization. The mixture was made in a single screw extruder Eco Solutions brand, and a temperature profile of 190/204/

221°C was used in zones 1, 2, and 3, respectively. The fibers concentration was fixed in 5 and 7 wt% concerning the total mass of PP.

After the extrusion, processed material was pelleted and oven-dried at 60°C for 24 h. Injection molding was performed using a mini bench injector, AX Plastics brand, model AXINJET, with an injection pressure of 0.5 bar. The following injection parameters were used: injection temperature 210°C, mold temperature 80°C and an injection pressure of 0.6 MPa. The specimens were obtained following dimensions specified by ASTM D-638 for the evaluation of mechanical properties.

2.5 Characterization

The natural and mercerized fibers were characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Thermogravimetric Analysis (TGA). For FTIR analysis, a Shimadzu Prestige-21 spectrometer was used in a range from 400 to 4000 cm^{-1}. The XRD analyses were performed on a Shimadzu diffractometer, model XRD-600, with a scanning speed of 0.5°/min, for 2θ values between 10° and 90°. The thermal stability of natural and mercerized fibers was evaluated by TGA in equipment from TA Instruments, model Q50, using a heating rate of 20 °C/min, from room temperature to 800°C under a nitrogen atmosphere.

Recycled PP and composites were characterized by TGA, Differential Scanning Calorimetry (DSC), tensile strength, and Scanning Electron Microscopy (SEM). TGA analyses were performed on Shimadzu equipment, model TGA-50, using a heating rate of 20 °C/min, from room temperature to 800°C, under the nitrogen atmosphere. DSC was performed in a Shimadzu model DSC-60 to determine the thermal transitions of the composites and PP. The used conditions were: heating ramp from 30°C to 200°C, using a heating rate of 10 °C/min and isotherm of 3 min for the first heating, with subsequent cooling. The second heating was performed under the same conditions as before, once the thermal processing history of the material was removed. Tensile strength tests of composite materials were performed on an EMIC universal testing machine, model DL30000N with a load cell of 300 kN, at room temperature. Morphological analyses were performed in a Jeol SEM, model JSM-6610LV.

The nomenclature assigned to each composite can be seen in Tab. 1.

Table 1: Identification assigned to each sample used in this work

Sample	Identification
Recycled PP	Recycled PP
5 wt% natural fiber-reinforced composite	5 wt%NF
7 wt% natural fiber-reinforced composite	7 wt%NF
5 wt% mercerized fiber composite	5 wt%MF
7 wt% mercerized fiber composite	7 wt%NF

3 Results and Discussion

3.1 Characterization of the Reinforcements

3.1.1 FTIR

Fig. 2 shows the infrared spectra of natural and mercerized pineapple crown fibers. Regarding the modification, the bands at 2921 cm^{-1} and 2847 cm^{-1} are observed, and they are associated with the aliphatic C-H stretch of the methyl and methylene groups. The band found at 3350 cm^{-1} shows a higher absorption, and it is more defined in mercerized fibers, which indicates an increase of O-H groups. It occurs due to the breakdown of lignin and hemicellulose bonds, as a result of the alkaline treatment; thus,

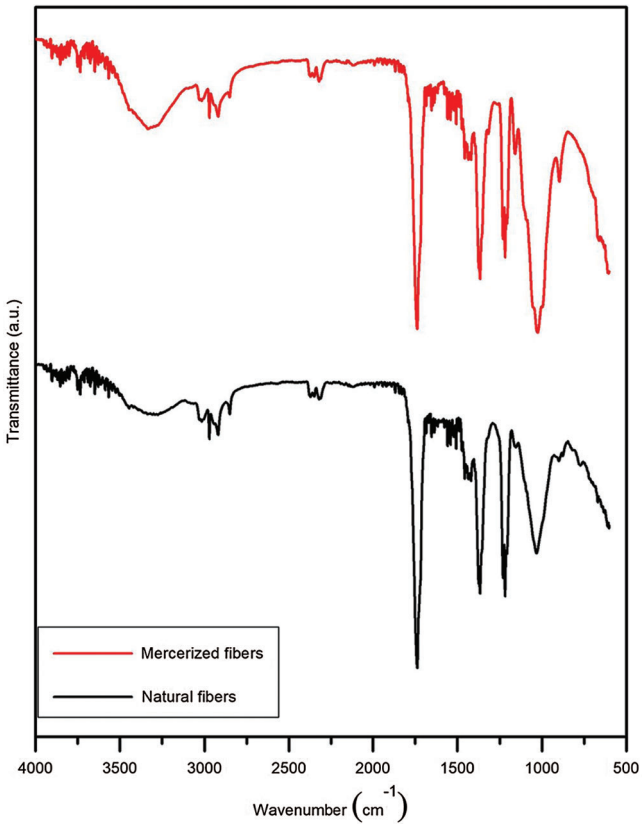

Figure 2: FTIR spectra of natural and mercerized fibers

this removal serves to expose the O-H groups and to create a more reactive surface. Stretch modification bands are observed for the carbonyl groups present in hemicellulose and lignin. The band at 1158 cm^{-1} refers to the asymmetric deformation of the methyl group [9]. The band at 1030 cm^{-1} is related to the C-O stretch bonds and C-H vibrations of cellulose; the increasing spectral intensity corresponds to the treatment performed, indicating greater exposure of cellulose due to the removal of other constituents [10]. The band at 897 cm^{-1} is attributed to the deformation of CH in cellulose, and the one at 667 cm^{-1} to the angular deformation outside the plane of the C-OH bond. The bands at 3500 cm^{-1} and 760 cm^{-1} are attributed to cellulose. Due to the high intensity of these bands, it is suggested that due to the treatment, cellulose was efficiently separated from other components [11]. These results indicate the removal of lignocellulosic components, which can be proven with the collaboration of other characterizations.

3.1.2 XRD

Fibers were also characterized by XRD, and the results are shown in Fig. 3. The mercerization treatment expands the fibers and partially removes some compounds, which provides better packaging of the cellulose chains, increasing its crystallinity. It can be noted that for modified fibers, all the peaks have become more defined. The graphics present two peaks at 16.35° and 22.35°, which are characteristic peaks related to vegetal fibers and a peak at 34.49°, which corresponds to the alignment and direction of the fibers [12]. It is noticeable that mercerized fibers have sharper and more defined peaks, indicating increased fiber crystallinity, which suggests that amorphous compounds such as hemicellulose and lignin have been removed [13,14].

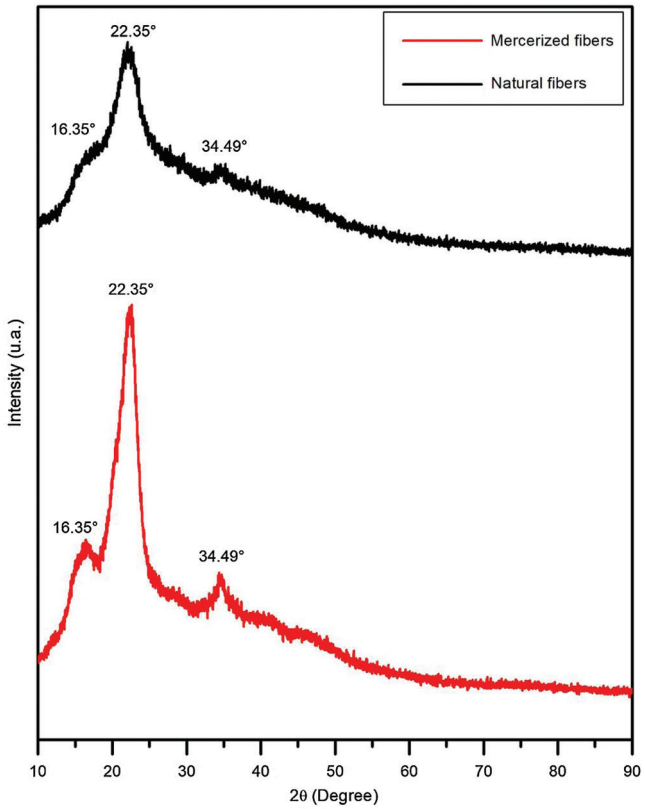

Figure 3: XRD diffractograms of natural and mercerized fibers

3.1.3 TGA

TGA was performed to evaluate the influence of mercerization treatment with sodium hydroxide on the thermal properties of fibers. The weight loss curves as a function of temperature and the derivative thermogravimetry (DTG) of natural and mercerized fibers can be observed in Figs. 4 and 5, respectively.

In both types of fibers, it is possible to identify an initial weight loss stage between 50°C and 100°C, corresponding to the elimination of water from fibers. Looking at the mass loss and DTG curves, it is possible to observe that mercerized fibers have two main weight loss stages. The first one, as already mentioned, is characterized by water loss (around 8 wt%). The second stage is related to fiber degradation at approximately 350°C when the elimination of cellulose occurs (mass loss of around 75 wt%) [15,16].

Natural fiber presents a higher weight loss process than mercerized fibers, once mercerization removes soluble compounds from fibers. It is possible to highlight four stages of mass loss for natural fiber. The first stage corresponds to water loss (6 wt%) as already mentioned; the second stage from 200 to 230°C is related to thermal depolymerization of hemicellulose with a mass loss of around 10 wt%. Between 230°C and 340°C, a weight loss of 27 wt% refers to lignin degradation. The fourth and final stage at 350°C corresponds to the decomposition of cellulose (weight loss of around 33 wt%) [17,18].

These results show that mercerization treatment promotes changes in the degradation process of the fibers, generating an increase in the thermal stability of pineapple crown fibers. The results of the thermal analysis indicate that the mercerized and natural fibers are suitable material to reinforce the polymeric matrix.

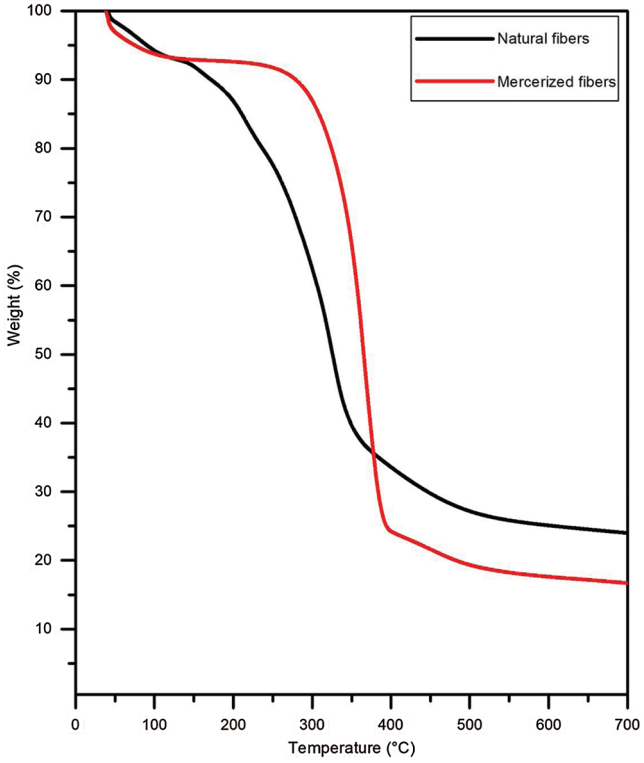

Figure 4: TGA curves of natural and mercerized fibers

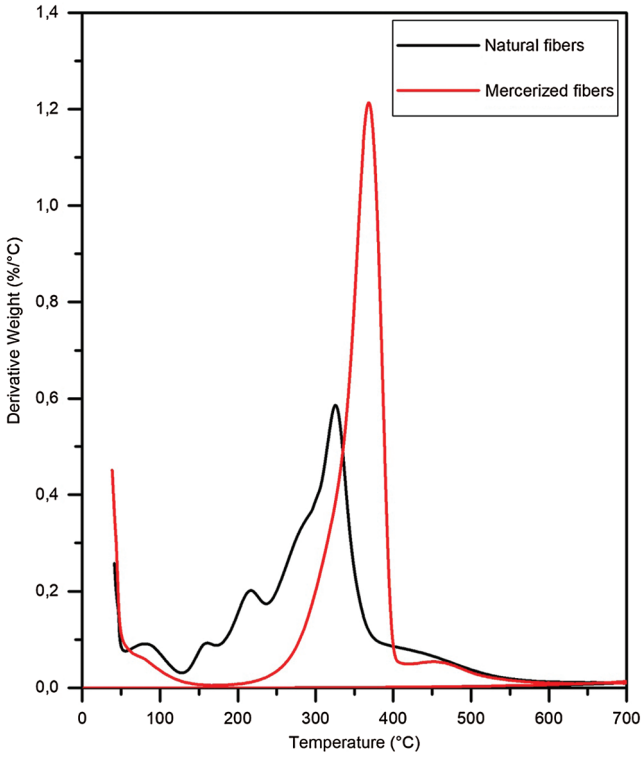

Figure 5: DTG curves of natural and mercerized fibers

3.2 Characterization of the Composites

3.2.1 TGA

TGA was performed to evaluate the thermal stability of recycled PP and polymeric composites with different contents of natural and mercerized fibers. The TGA and DTG curves are shown in Figs. 6 and 7, respectively.

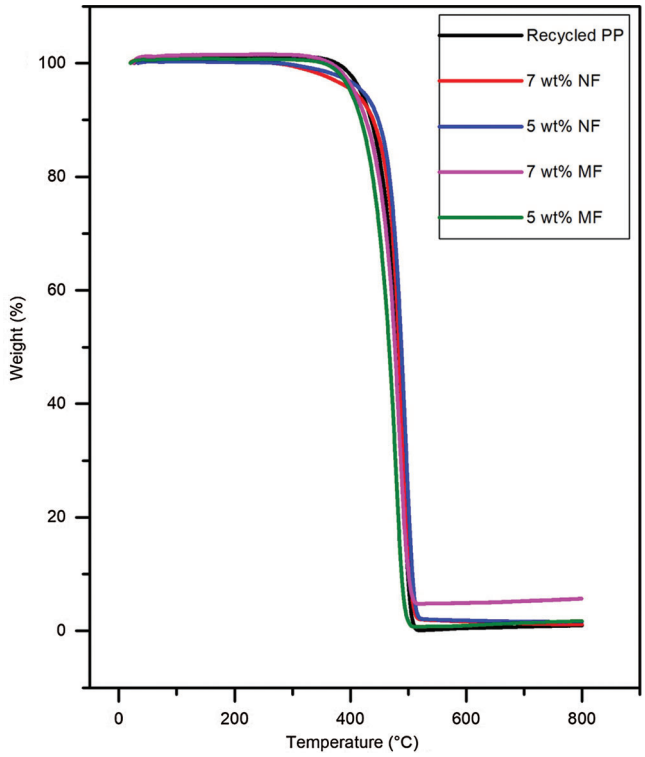

Figure 6: TGA curves of recycled PP and the composites reinforced with natural (5 wt%NF and 7 wt%NF) and mercerized (5 wt%MF and 7 wt%MF) fibers

The composites reinforced with natural fiber have an initial decomposition temperature close to the recycled PP, presenting a single degradation stage, with the poor influence of fiber on the thermal resistance to degradation. It can be noted that the composites reinforced with mercerized fibers started to degrade at lower temperatures than the other ones. The composites that showed higher thermal stability were the composites reinforced with natural fibers, where the 5 wt%NF sample had a maximum degradation peak at 450°C. A hypothesis for this fact that the treated materials have less thermal stability is that treated fiber composites present better interfacial interaction due to reactions that occur on fiber surface with sodium hydroxide. Thus, it promotes a higher interaction in the degradation process of the two phases, i.e., the degradation of one component can accelerate the degradation of another one [19,20].

The results of pure recycled PP were similar to all composites; that is, there were no major changes in thermal stability when the fibers were added. One reason for this is that the dispersion of particles is also an important factor that contributes to the improvement of thermal stability and may have influenced the result of these materials [21].

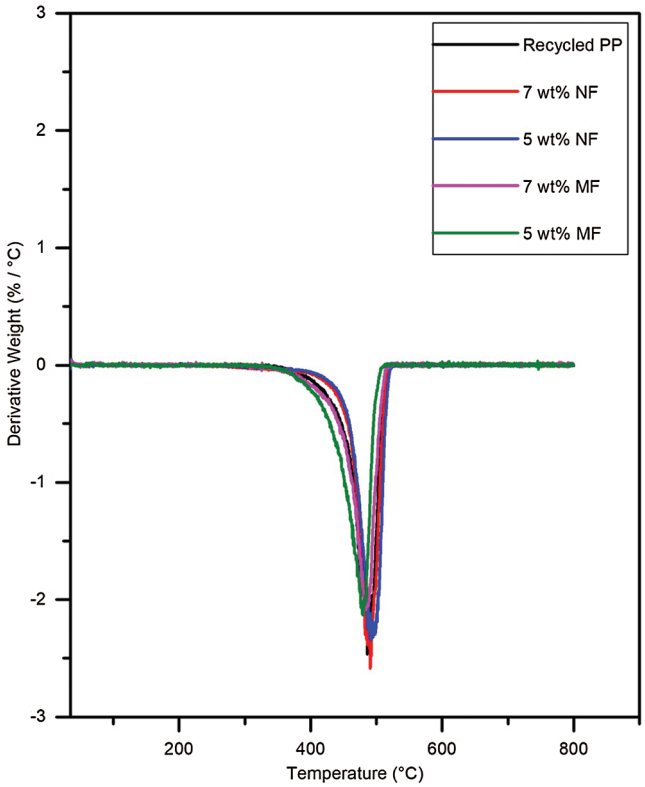

Figure 7: DTG curves of recycled PP and the composites reinforced with natural (5 wt%NF and 7 wt%NF) and mercerized (5 wt%MF and 7 wt%MF) fibers

3.2.2 DSC

Figs. 8 and 9 show the cooling and second heating thermograms, respectively, for the recycled PP and the composites. It can be noted that the melting temperature (Tm) of recycled PP is slightly different when compared to the composites (see Tab. 2); thus, the presence of reinforcements did not change the Tm of composites. A single endothermic peak close to 165°C is observed for the composites, corresponding to the Tm of recycled PP [22]. The same behavior is observed for the crystallization temperatures (Tc), without significant differences.

The values of degree of crystallinity (Xc) of recycled PP and the composites were calculated using the relationship between experimental melt heat (ΔHf) and melt heat of a 100% crystalline PP ($\Delta H_{f100\%}$), given by Eq. (1):

$$X_c = \frac{\Delta H_f}{\Delta H_{f100\%}} \times \frac{1}{W_{PP}} \times 100\% \quad (1)$$

where $\Delta H_{f100\%}$ = 165 J/g, ΔH_f is the enthalpy of crystalline melting during second heating, and W_{PP} is the mass fraction of PP in the composites.

The values of degree of crystallinity (Xc) obtained using the DSC results show that there was an increase in its parameter for composites with natural fiber (5 wt%NF and 7 wt%NF) when compared to recycled PP. This fact can be explained because the nucleation capacity of the fibers accelerated the crystallization process of PP [22]. Moreover, it was also observed that the mercerized fibers (5 wt%MF and 7 wt%MF) suffered a reduction in enthalpy of fusion and presented a lower degree of crystallinity related to the recycled PP. This

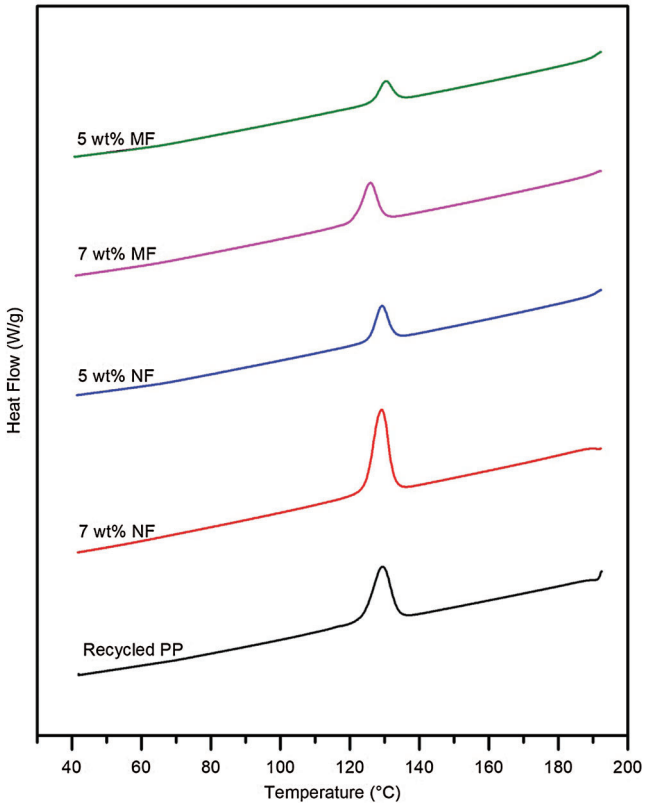

Figure 8: DSC thermograms obtained under the cooling ramp for recycled PP and composites with natural (5 wt%NF and 7 wt%NF) and mercerized (7 wt%MF and 5 wt%MF) fibers

fact indicates that the addition of modified fiber allows the crystallization process to occur at lower temperatures, with smaller lamellae due to fiber-matrix interaction [23,24].

3.2.3 Tensile Strength

Tab. 3 presents the results obtained for the tensile strength test of recycled PP and composites with different reinforcement contents. It is possible to observe that the tensile strength did not present significant changes among the composites. This property is directly related to the average fiber length, which can be affected during processing [25].

It can be observed that all composites presented higher values of elastic modulus than the recycled PP. Besides, this effect was more pronounced for the 7 wt%MF sample. These results confirm the increased stiffness of the matrix with the addition of reinforcement [26,27].

3.2.4 SEM

SEM images of the composites reinforced with 7 wt% of natural and mercerized fibers were taken from the fractured surface of each specimen after the tensile strength tests. The images are presented in Fig. 10.

From some of the images, it can be observed typical features of a brittle type fracture, which means a relatively flat fracture surface. This type of fracture occurs when there is no significant plastic deformation and rapid crack propagation, causing the material to rupture rapidly [28].

SEM images indicate that the composite with 7 wt% of natural pineapple crown fiber (Figs. 10a and 10b) has poor dispersion and poor fiber adhesion in the PP matrix. On the other hand, for composites reinforced

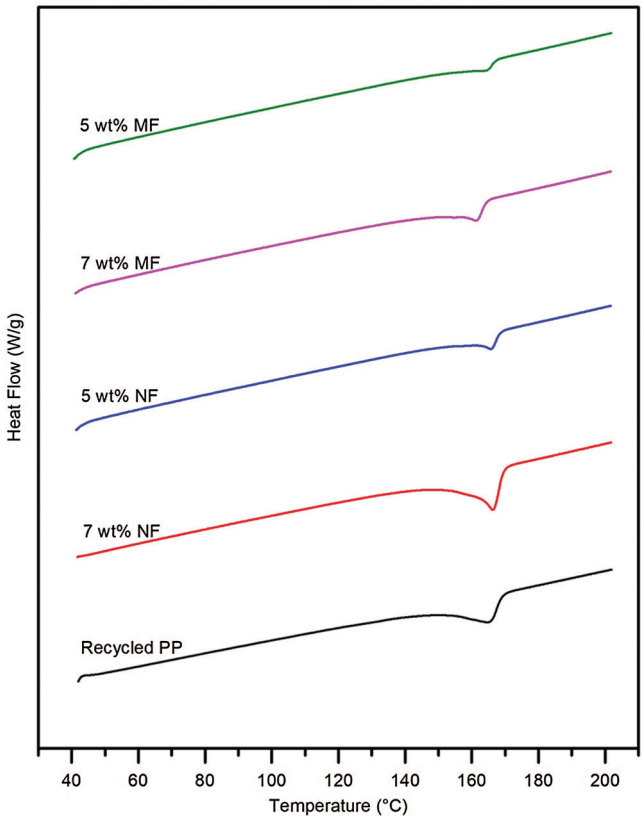

Figure 9: DSC thermograms obtained under the second heating ramp for recycled PP and composites with natural (5 wt%NF and 7 wt%NF) and mercerized (7 wt%MF and 5 wt%MF) fibers

Table 2: Data obtained from DSC analyses from cooling and second heating ramps

	T_m (°C)	ΔH_f (J/g)	ΔH_c (J/g)	T_c (°C)	X_c (%)
Recycled PP	165.7	63.6	73.0	129.4	38.5
7 wt%NF	166.4	99.6	113.3	129.2	64.2
5 wt%NF	165.9	74.4	125.6	129.4	47.3
7 wt%MF	161.9	52.2	93.9	125.9	33.2
5 wt%MF	165.4	41.4	77.2	130.5	26.3

Table 3: Tensile strength and Young's modulus of recycled PP and the composites

Sample	Tensile strength (MPa) (± Standard deviation)	Young's modulus (MPa) (± Standard deviation)
Recycled PP	31.8 ± 3.6	535.2 ± 18.7
5 wt%NF	30.7 ± 0.7	704.5 ± 53.8
7 wt%NF	30.0 ± 2.0	672.3 ± 136.0
5 wt%MF	31.8 ± 0.5	708.9 ± 38.1
7 wt%MF	30.9 ± 1.5	730.7 ± 94.9

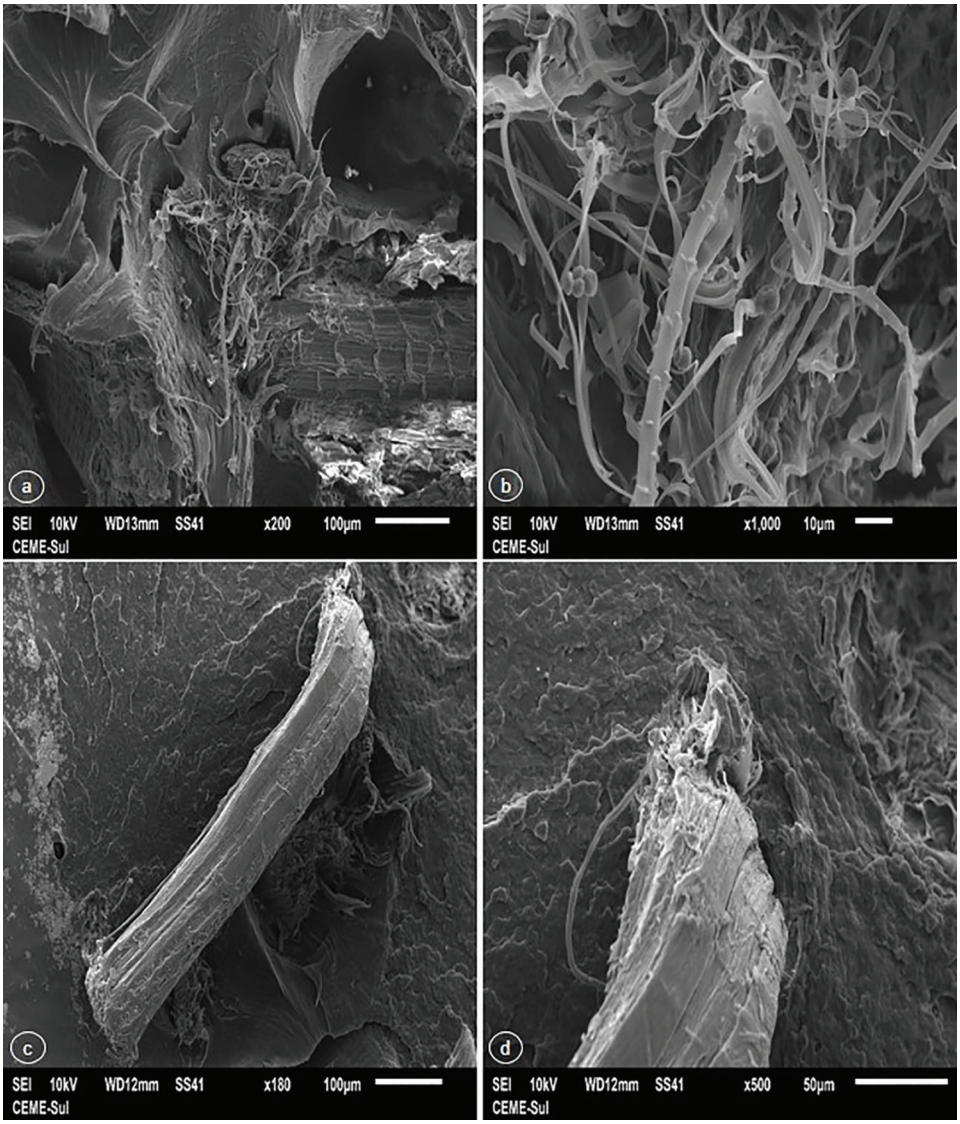

Figure 10: SEM images of fractured surface of the composites: (a) 7 wt%NF (200×), (b) 7 wt%NF (1000×), (c) 7 wt%MF (180×), and (d) 7 wt%MF (500×)

with mercerized pineapple crown fiber, it is possible to note a change in adherence (Figs. 10c and 10d), indicating a better interaction between modified fiber and PP [29,30]. However, the poor dispersion of both fibers influenced the tensile strength and stiffness of the composites and did not make a significant difference in the mechanical properties [31,32].

4 Conclusions

According to FTIR, XRD, and TGA results, the fiber mercerization treatment was effective, indicating that there was the removal of compounds such as hemicellulose and lignin, in which mercerized fibers presented higher thermal stability and crystallinity. Through the characterization of the composites, it can be concluded that composites with natural fibers presented higher thermal stability than composites with mercerized fibers. Besides, they presented a lower degree of crystallinity in relation to composites with natural fibers. Regarding the mechanical evaluation, the values of Young's modulus for the composites

were higher than the recycled PP. However, there were no significant changes between the different composites, nor was there any significant difference in the maximum stress values. Analyzing the SEM images, it can be noticed a better adhesion between the mercerized fibers and the polymer matrix; however, the degree of dispersion was not sufficient, influencing the mechanical properties.

Therefore, it can be concluded that among the evaluated materials, the natural pineapple crown fiber showed the capacity to be used as a reinforcing agent in polymeric matrices. Besides, recycled polypropylene showed great potential to be applied to products, highlighting the importance of recycling and reuse of materials. The use together of these residual materials is essential to emphasize conscious consumption and to solidify a new culture of waste reuse. Therefore, considering the properties studied, the composites in question have a low cost because they are based on waste materials, adding value to these raw materials, and also because no treatments are required.

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Conflicts of Interest: The authors declare that they have no conflicts of interest to report regarding the present study.

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