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PREPARATION AND CHARACTERIZATION OF REPROCESSED COMPOSITES OF POLYPROPYLENE REINFORCED BY WOOD FIBERS: PHYSICAL AND MECHANICAL PROPERTIES

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Abstract. *This study aims to discuss the effects of the reprocessing on the performance of wood-plastic composites regarding their physical and mechanical properties. Composites composed of a polypropylene (PP) matrix reinforced by sawdust of Pinus elliottii compounded by a two-step extrusion process (wood fibers coated with PP/polymeric compatibilizer followed by the preparation of the composites) were prepared with 20 and 30 wt% of wood fibers, with and without the use of 10 wt% of a domestic compatibilizer maleated polypropylene (MAPP), produced by reactive extrusion. The composites were submitted up to six reprocessing cycles by extrusion in the same single-screw extruder used in the compounding step. To evaluate the changes in the mechanical properties due to the reprocessing cycles, tensile and flexural tests were carried out in specimens prepared by compression molding of both virgin and reprocessed composites after three and six cycles. The fractured surfaces of chosen tensile tested specimens were examined by Scanning Electron Microscopy (SEM). The influence of the reprocessing cycles were also discussed in terms of water absorption, viscosity, interfacial wood fiber/polymer adhesion, density and other characteristics such as color and odor for both virgin and reprocessed composites.*

Keywords: *WPC, Reprocessing, MAPP, Sawdust.*

1. INTRODUCTION

Any composites containing thermoplastic or thermosetting resin and wood may be called wood-plastic composites (Ashori *et al.*, 2013). In addition to these raw materials, composites may also contain additives as compatibilizing agents (for the purpose of generating adhesion at the interface between the natural fibers and the polymer matrix), flame retardants, lubricants, inorganic pigments, and even metal hydroxides to improve the thermal properties of these materials (Santos *et al*., 2015a; Arao *et al*., 2014; Calvimontes *et al*., 2014; Xu *et al*., 2015; Gwon *et al*., 2015). These materials, which were developed in the 1970s (with a thermoplastic matrix), can replace both plastics and wood, having the aesthetic appearance similar to the latter material (Lou *et al*., 2013).

Recycled polymers and biopolymers, as well as a wide variety of wood residues and by-products can be used as raw materials for these composites, and they can still be recycled (Teuber *et al.*, 2016). In this context, several works have studied the use of recycled products in the composition of composites of polymeric matrix and natural fibers, either from the point of view of the matrix with the use of recycled polymers (Sommerhuber *et al.*, 2015; Kazemi-Najafi *et al.*, 2013) or the natural fibers (Gozdecki *et al.*, 2015; Soucy *et al.*, 2014).

The present study proposes, then, that polypropylene matrix composites reinforced by sawdust and compatibilized with maleated polypropylene (MAPP), already produced and tested in laboratory, be reprocessed by the extrusion process up to six cycles, in order to verify the influence of this reprocessing on the properties of these materials.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Sawdust of *Pinus elliottii* was used as filler. The particles had the following size distribution: 27 wt% were greater than 40 mesh, 40 wt% had a size between 40 and 60 mesh, and 33 wt% of the particles were smaller than 60 mesh. For the polymeric matrix it was used polypropylene (PP) H-503 pellets, with a melt flow index of 3.5 g/10 min, obtained from Braskem (Brazil). For the compatibilizer MAPP, it was used dicumyl peroxide (DCP), donated by Avec Rubber, Brazil, and maleic anhydride (MAH) obtained from Carbomafra Company, Brazil.

2.2 Selection and preparation of the compatibilizer

The selected MAPP was previously developed in laboratory by Santos *et al.* (2015b), and it's composed of 98.5 wt% PP, 1.0 wt% of MAH and 0.5 wt% of DCP, and was selected because it resulted in the best mechanical properties in the final composites, according to that study. The MAPP was prepared by reactive extrusion and it was used in the compounding of the virgin composites (composites without reprocessing). The same equipment, a single-screw extruder (TECK TRIL, EMT 25) was used in the preparation of the compatibilizer, the composites and in their reprocessing.

2.3 Preparation and reprocessing of the composites

Virgin composites were prepared with two different sawdust contents (20 and 30 wt%) with the addition of 10 wt% of MAPP. It was also prepared virgin composites, with the same sawdust content, but without the addition of the compatibilizer. The preparation of the composites was carried out through a two-step extrusion process. The first step consisted in the wood fiber coating (processing, by extrusion, a compound consisting of all the sawdust previously dried in an oven at 60^{o}C for 24 h, the MAPP and a part of the polypropylene). In the second step, the coated fiber was pelletized, and reprocessed along with the rest of the polypropylene. The four different virgin composites obtained were reprocessed through extrusion process up to six times. For the virgin composites, and the composites submitted to three and six extrusion cycles, it was prepared specimens by compression molding. Fig. 1 shows the stages of the composites preparation and the reprocessing method. Table 1 presents the prepared composites for this study and their names.

Table 1. Virgin and reprocessed composites.

Composites prepared without MAPP			Composites with 10 wt% of MAPP		
Sawdust content	Number of reprocessing cycles	Name	Sawdust content	Number of reprocessing cycles	Name
20%	0 (virgin)	B1a	20%	0 (virgin)	D1a
	3	B1d		3	D1d
	6	B1g		6	D1g
30%	0 (virgin)	B2a	30%	0 (virgin)	D2a
	3	B2d		3	D2d
	6	B2g		6	D2g

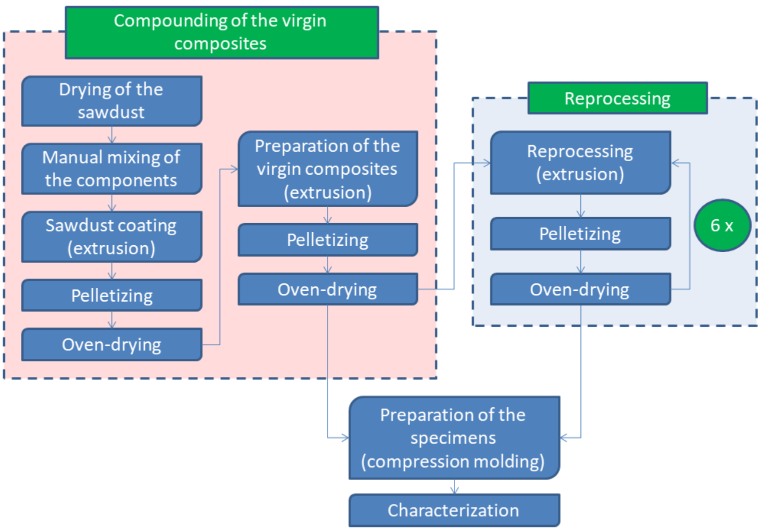

Figure 1. Preparation and reprocessing of the composites

2.4 Characterization

The mechanical properties of virgin composites, and the composites reprocessed three and six times were evaluated through:

Tensile tests (ASTM D638 – 14). The equipment used was an Instron tester, model 5567, with a load cell of 5 kN. The test speed was 5 mm/min.

Flexural tests (ASTM D790 – 10). The three point bending tests were performed using an EMIC universal tenting machine (model DL – 2000), with a load cell of 20 kN. The distance between the supports was 80 mm, and the test speed was 15 mm/min.

The physical properties of the same composites tested for the mechanical tests were evaluated through:

Water absorption (ASTM D570 – 98). The samples, previously dried at an oven for 24h at 50°C, were tested by the procedure of water immersion for 24 hours.

Density tests (ASTM D2395 – 14). The experimental densities of the composites, the PP and the MAPP were determined through water immersion. The composites experimental densities were compared with their theoretical densities, to determine the real amount of sawdust that was incorporated in the composites. The theoretical density was obtained through Eq. (1), already used by Santos *et al*. (2015b):

$$\frac{1}{\rho_C} = \frac{W_{PP}}{\rho_{PP}} + \frac{W_{PPMA}}{\rho_{PPMA}} + \frac{W_S}{\rho_S} \tag{1}$$

Where:

ρ_C: theoretical density of the composite (g/cm^3);
W_{PP}: weight fraction of PP (%);
ρ_{PP}: experimental density of the polypropylene (g/cm^3);
W_{PPMA}: weight fraction of MAPP (%);
ρ_{PPMA}: experimental density of the MAPP (g/cm^3);
W_S: weight fraction of sawdust (%);
ρ_S: theoretical density of the sawdust (g/cm^3).

For Klyosov (2007), the extrusion process results in the compression of the cellulosic fibers, and therefore the sawdust density in the composites exhibits values between 1.3 and 1.5 g/cm^3, which is in agreement with the value presented by Sommerhuber *et al.* (2016). The theoretical densities of the virgin composites were then calculated for the two values mentioned (1.3 and 1.5 g / cm^3).

Other characterization techniques were used to study the effects of the reprocessing cycles:

Microstructure and other characteristics. The fractured surfaces of the tensile tested specimens which had the best results for the tensile strength were examined by scanning electron microscopy (SEM), to evaluate the fiber/polymer interfacial adhesion and the dispersion of the wood fibers in the polymeric matrix. The influence of the reprocessing cycles in the composites was also evaluated through the change of their characteristics such as color, odor and the viscosity during processing.

3. RESULTS AND DISCUSSION

3.1 Surface characteristics of the composites

Figure 2 shows the surfaces of the test specimens of both virgin and three and six times reprocessed composites, with 20 and 30 wt% of sawdust, respectively. It can be seen that, with the successive extrusion cycles, the color of the composites became darker. That was expected, once that a higher number of reprocessing cycles implies in a longer exposure time of the sawdust to the temperatures involved in the extrusion process, thus causing the thermal degradation of the sawdust. This same effect was observed by Soccalingame et al. (2015) who, when studying the reprocessing of WPC with a polypropylene matrix reinforced by wood flour, attributed this effect not only to the degradation of hemicellulose, but also the migration of lignin, which due to the high temperatures involved in the process, may melt and migrate from the wood particles to the matrix, which also justifies the darker color of the composites in this work.

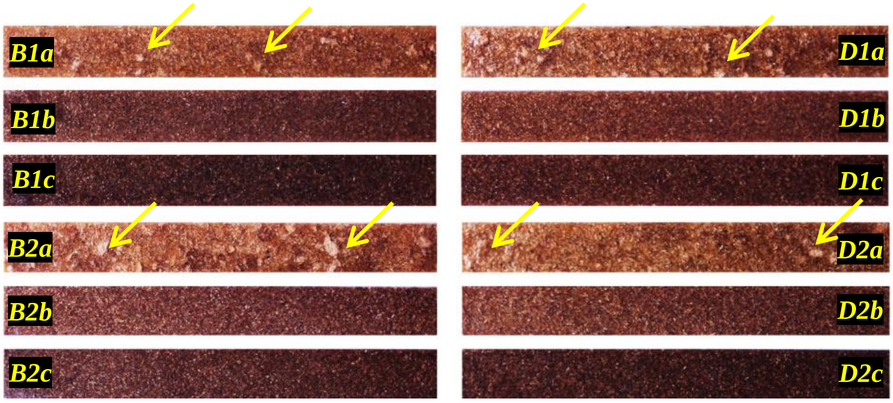

Figure 2. Surface of the test specimens for both virgin and reprocessed composites

According to Santos (2011), the fibers, because of their hydrophilic nature, have a tendency to agglomerate, and in the virgin composites it is possible to notice the presence of these sawdust aggregates for all cases, making the surface of these materials quite heterogeneous. Some of these aggregates are indicated, in Fig. 2, by yellow arrows, in order to facilitate their observation. The presence of these aggregates is a strong indication of poor dispersion of the wood fibers in the polymeric matrix. In the composites submitted to three and six cycles of reprocessing, these aggregates are no longer observed, and the surface of the specimens becomes very homogeneous, indicating that the reprocessing cycles resulted, in principle, in a better dispersion of the fibers in the matrix. This better dispersion of the sawdust fibers in the polymer matrix, throughout the cycles of reprocessing, was already expected, having been discussed by several authors (Soccalingame *et al*., 2015; Beg and Pickering, 2008; Shahi *et al*., 2012).

Throughout the reprocessing cycles, for all of the composites, a reduction in the viscosity was observed, which was evident after the third reprocessing cycle. This observation was made based on the flow characteristics of the material when leaving the extruder, which started to form thinner filaments, such as those observed for MAPP, which had a higher melt flow index then the virgin composites. The melt flow index of the MAPP used in this work was determined by Trombetta (2010) as being 27.8 g/10 min. Moreover, for the final cycles of reprocessing (fifth and sixth cycles), a burnt wood odor could be felt during the processing of the composites, evidencing the thermal degradation of the wood fibers.

3.2 SEM micrographs

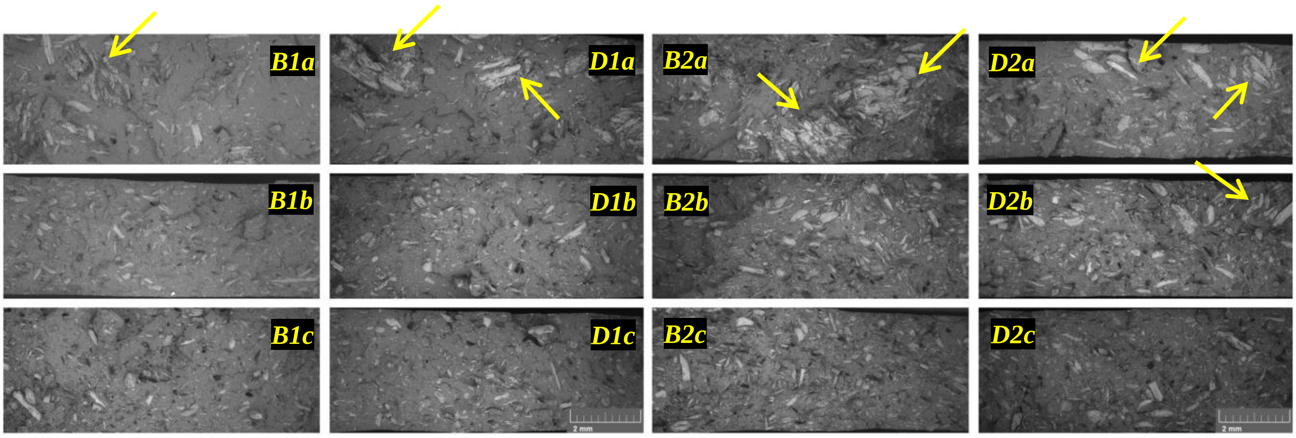

Figure 3. Surface of the tensile tested specimens for both virgin and reprocessed composites which had the best results for the tensile strength

The images in in Fig. 3 were made by backscattered electrons, and with this technique, wood fibers (light gray regions) can be easily observed dispersed in the continuous polymer matrix (dark gray). The black regions, inside the samples surface, constitute voids that were formed by the pulled out wood particles from the matrix during the test.

These images allow the observation of both the sawdust aggregates formed mainly in the virgin composites (indicated with the yellow arrows) and the evolution of the dispersion of the wood fibers in the polymeric matrix, which has become more uniform as composites were reprocessed.

It was expected that, by subjecting the materials to reprocessing cycles, the dispersion of the fibers in the matrix would be improved. This better dispersion was probably due to two reasons. The first, and more direct, concerns to the fact that the reprocessed composite is subjected to a greater number of mixing cycles, and therefore more shear stresses, which leads to a better distribution of the wood particles in the matrix, making the structure of the material more homogeneous (Shahi *et al*., 2012).

The second reason is the reduction in the viscosity of the matrix (which was observed during the reprocessing of the materials), allowing better dispersion and wettability of the wood particles. This reduction in viscosity is caused by the subsequent cycles of reprocessing, which also involve a greater exposure of the material to heating cycles, which combined with the aforementioned shear stresses present in the extrusion process, leads to chain scissions in the polymeric matrix. This phenomenon was also observed in other studies (Petchwattana *et al*. 2012; Paukszta *et al*., 2015; Dickson *et al*., 2014). Therefore, the best distribution of the sawdust in the polypropylene matrix could easily be observed after six cycles of reprocessing, where, for none of the composites studied, the presence of the wood particles aggregates was verified.

Figure 4 (a) and (b) show, respectively, examples for the virgin and three times reprocessed composites of an aggregate made of wood particles, and an apparently loose wood fiber, which is an example of bad adhesion in the wood/fiber interface. Figure 4 (c) shows the structure of a composite subjected to six reprocessing cycles, where the matrix with the highest melt flow index is expected (or the lower viscosity, due to the chain scissions that occurred because of the successive extrusion cycles). It is possible to observe that the matrix covered the surface of the fiber, resulting in a greater interaction between these two phases. It is also possible to note that, with the fracture of the specimen in the tensile test, the matrix deformed around the fiber, being possible to affirm that, in this case, there was a better interfacial adhesion.

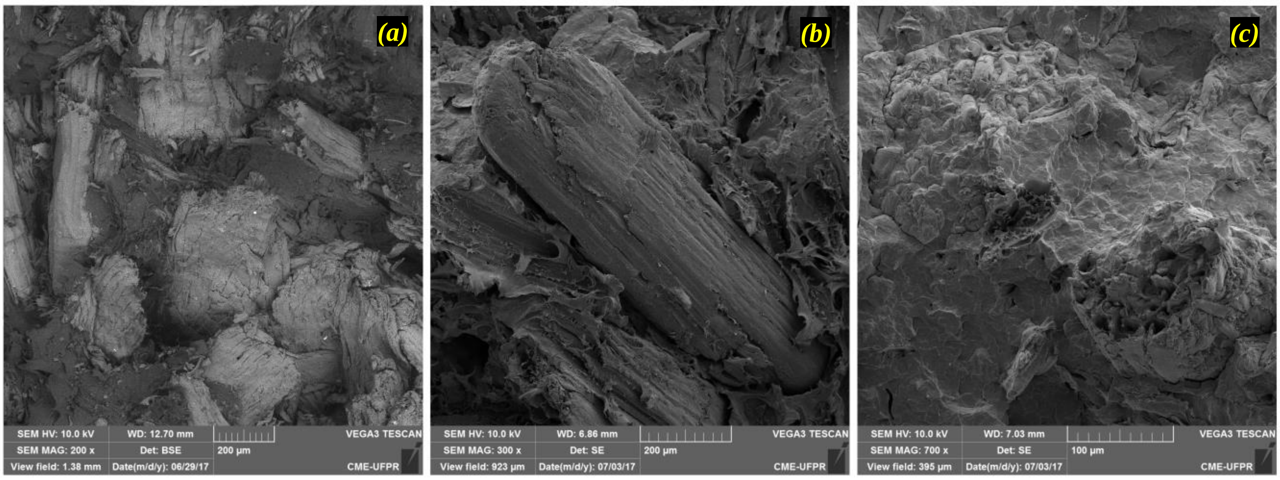

Figure 4. Surface of the tensile tested specimens, showing the following composites: (a) B2a, D2d (b) and (c) D2g.

3.3 Density of the composites

The results shown in Table 2 indicate that the values obtained experimentally for the density of the virgin composites are very close to the theoretical ones, calculated by Eq. (1). This fact indicates that the amount of sawdust previously determined, was effectively incorporated into the composites in all cases, and also that the specimens prepared by hot compression did not present significant amounts of pores or voids, which were not observed in the fractured specimens in the mechanical tests. The presence of these defects, as well as of the sawdust content below that stipulated for the material, would imply in the lower experimental density of the composite when compared to the theoretical values. It is also possible to observe that the experimental density of the polypropylene is in accordance with the manufacturer's value of 0.905 g/cm^3 as shown in Table 2. This result demonstrates that the methodology used to determine the experimental density of the composites is coherent.

Since wood is the component with the highest density, it can be seen from Table 2 that the addition of sawdust to polypropylene resulted in an increase of the material density. Thus, it is possible to expect that, the larger the quantity of wood, the greater the density of the composite, which is confirmed by the graph presented in Fig. 5 (a), which shows the experimental densities of the studied composites. From the graph, it is clear that the composites with higher sawdust contents presented higher densities, which is in agreement with other studies (Sommerhuber *et al*. 2016; Soleimani *et al*., 2008; Santos *et al*., 2015b).

Table 2. Experimental and theoretical densities.

COMPOSITE / MATERIAL	THEORETICAL DENSITY (g/cm^3)	EXPERIMENTAL DENSITY (g/cm^3)
PP	0,905$^{(1)}$	0,91 ± 0,03
MAPP	-	0,88 ± 0,02
B1a	0,98 ± 0,01	0,96 ± 0,01
B1d	-	0,98 ± 0,02
B1g	-	0,93 ± 0,03
B2a	1,02 ± 0,02	1,02 ± 0,04
B2d	-	1,06 ± 0,03
B2g	-	1,04 ± 0,03
D1a	0,97 ± 0,01	0,96 ± 0,01
D1d	-	0,94 ± 0,01
D1g	-	0,96 ± 0,01
D2a	1,01 ± 0,02	1,01 ± 0,03
D2d	-	1,01 ± 0,01
D2g	-	1,00 ± 0,03

$^{(1)}$value given in the polymer data sheet provided by the manufacturer.

The changes in the density of the composites when reprocessed were quite small, when comparing the same wood contents. Tabkhpaz Sarabi et al. (2014) also found no significant difference in the densities of virgin and reprocessed WPC. For the authors, this was expected since the matrices, which had different flow rates, had very similar densities, and the wood contents were the same. These statements can certainly be applied to the present study, where only one type of matrix was used, and the most relevant density variation was in function of the wood content.

3.4 Water absorption

In this study, the composites, besides being reprocessed, were prepared with different sawdust contents, with and without the addition of MAPP. So the results can be evaluated under three different points of view: the influence of the sawdust content, the influence of the presence of the compatibilizer and finally, the influence of reprocessing cycles.

Figure 5 (b) shows the results for water absorption for the virgin and reprocessed composites. In all cases, materials containing higher sawdust content absorbed a greater amount of water. Since PP is a hydrophobic material, the water absorption in the composites occurs due to the presence of the sawdust. This is explained by the fact that wood is a very hydrophilic material. So, a higher sawdust content (which means more lignocellulosic material and, therefore, more free –OH groups, responsible for attracting the water molecules, will be in contact with water), usually implies in a greater amount of water absorbed by the composite (Santos *et al.*, 2015b; Gozdecki *et al.*, 2015).

The effect of adding the MAPP compatibilizer can be verified by observing the amount of water absorbed for the composites in the virgin state. Comparing the same wood contents, it is possible to observe that the reduction was, on average, of more than 40% and 95% for the composites with 20 and 30 wt% of sawdust, respectively. This result is probably due to the improved dispersion and wettability of the fiber by the polymeric matrix, making the wood less exposed to the water (Santos *et al.*, 2015b; Yeh *et al*, 2013).

Regarding the effect of the reprocessing cycles, the results shown in Fig. 5 (b) indicates that, for all cases, the amount of water absorbed was reduced more drastically for the reprocessed composites three times in relation to the virgin composites. When comparing the three and six times reprocessed composites, the vulnerability to water was practically maintained, but there was a slight tendency to reduce these values, from the third to the sixth cycle. Two facts, observed in the SEM images, that occurred during the reprocessing cycles may explain in part the reduction in the amount of the absorbed water by the reprocessed composites: the best dispersion of the fibers in the polymeric matrix and the reduction in fiber size, which combined, make the wood particles less exposed to the water.

An interesting fact is that, when comparing the composites with the same wood contents and reprocessed for the same times, the amount of absorbed water is very similar for the reprocessed composites prepared in the presence of MAPP and without the addition of this compatibilizer. This is due to the fact that the reprocessing cycles ends up homogenizing the microstructure of the material, reducing the exposure of the wood to the water, which has already been discussed. Thus, considering only the factors that affect water absorption, the verification of the effects of the presence of the MAPP, becomes restricted to the virgin composites.

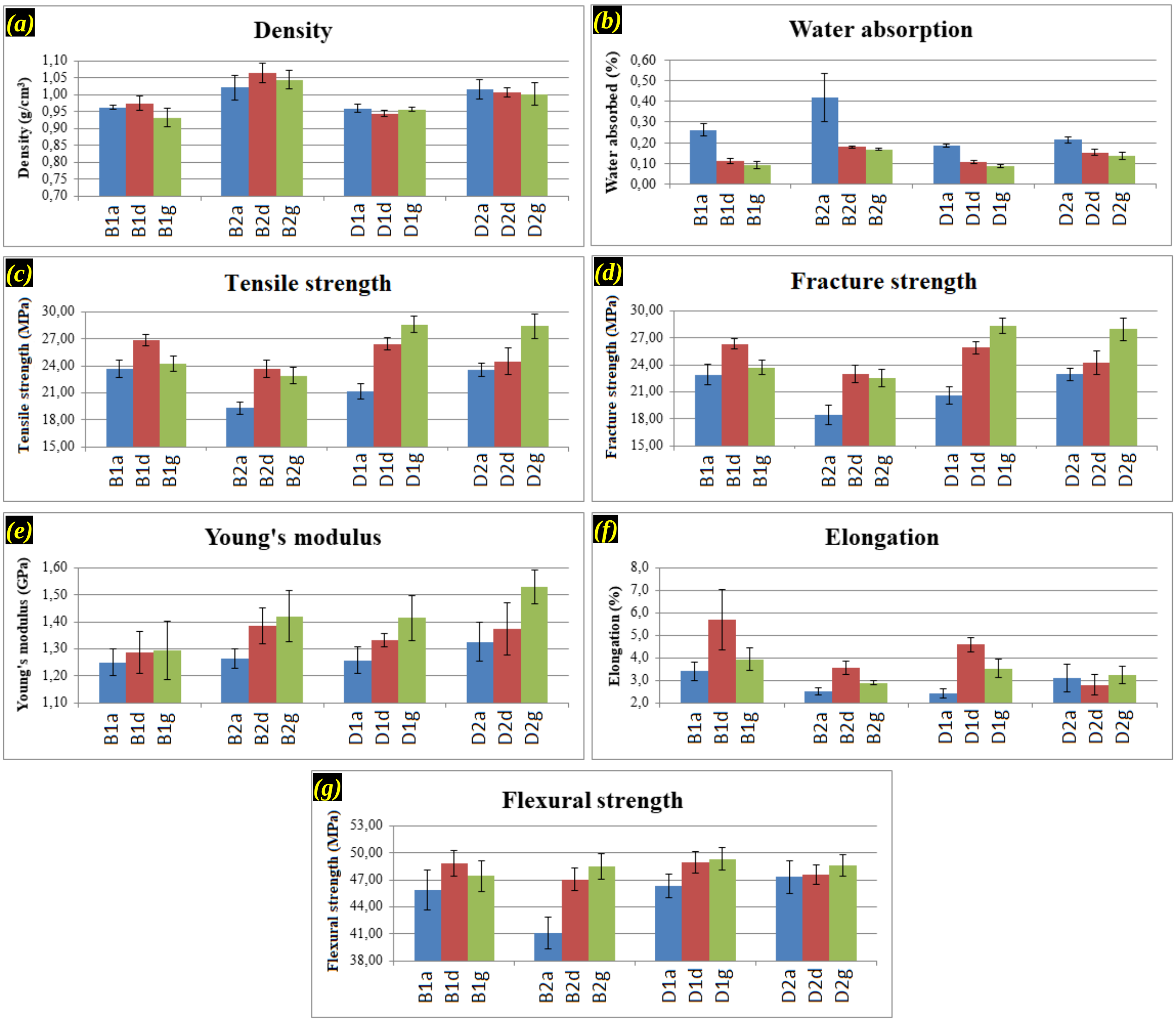

Figure 5. Mechanical properties for the virgin and reprocessed composites

3.5 Mechanical properties

Tensile ad flexural tests were performed on the virgin and reprocessed three and six times composites in order to evaluate the mechanical properties of these materials. Through tensile tests, tensile strength (Fig. 5 (c)) and fracture strength (Fig. 5 (d)) were calculated, as well as the Young's modulus (Fig. 5 (e)) and elongation (Fig. 5 (f)). By the flexural tests, the flexural strength (Fig. 5 (g)) was verified.

The first point to be discussed is that the dispersion of the wood fibers in the polymer matrix obtained for the virgin composites proved to be quite precarious, which was made clear by the observation of the sawdust aggregates in the photographs of the surfaces of specimens and in the images obtained by SEM. This poor dispersion was clear when the composites were reprocessed, since an expressive improvement was observed in terms of the mechanical properties, especially in the tensile tests. The images obtained by SEM allowed concluding that a better dispersion was obtained with the reprocessing of the composites.

In relation to the MAPP, its presence, regarding to the mechanical properties, was better observed for the virgin composites, however, only for composites prepared with 30 wt% of sawdust. These results indicate that, for higher wood contents, the addition of the compatibilizer is fundamental for the material, since both the tensile and flexural strength obtained for the composites prepared with MAPP, were much higher when compared to the composites without the compatibilizer.

However, the presence of MAPP is not noticeable for the three times reprocessed composites, since the tensile and flexural strengths present very similar values when compared to the composites prepared with and without the addition of the compatibilizer, for the same sawdust contents. It demonstrates the great dependence of these properties on the dispersion of the wood particles in the polymeric matrix, and also that reprocessing was fundamental for a good dispersion to be obtained, allowing the wood to act as a mechanical reinforcement to the composite, which can also be observed for the Young's modulus, where higher values were obtained for the reprocessed composites.

One negative effect that was expected when reprocessing the composites was the excessive reduction of the molecular weight of the matrix. If at first, such a reduction in viscosity allowed a better dispersion of the wood particles in the matrix (resulting in better mechanical properties), it was expected that, after a certain point, it would be detrimental to the properties of the materials. In fact, the six times reprocessed composites presented, in general, smaller values of elongation, which was attributed to the reduction in the molecular weight of PP, however, the tensile and flexural strength, as well as the Young's modulus, apparently were not impaired by this phenomenon.

4. CONCLUSIONS

It is possible to affirm that this work demonstrated that for the adopted process conditions (single-screw extruder for compounding), the reprocessing of the composites was not only feasible, but also fundamental in terms of obtaining better properties for the composites. However, it is important to emphasize that the final characteristics of the composites will depend largely on the equipment used in the compounding and molding stages of the composites. However, since these steps generally involve heating, cooling, and shear stresses, the results obtained here may be useful to other researchers.

5. ACKNOWLEDGEMENTS

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