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Preparation and characterization of composites from plastic waste and sugar cane fiber

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Abstract: This study presents the preparation and characterization of composite materials using Plastic Waste from Hydrapulper (PWH) from paper industries extruded with sugar cane fiber residues from ethanol industries. The factorial design showed that composite material with 40% of sugar cane fiber, pressed with 5 ton was the optimized condition. The main findings attested that the composite is resistant up to 250 °C and its hardness is increased compared to the raw PWH. The material presented woodsy aspect although water absorption has increased. So, this study offers a good alternative for the use of plastic waste generated as a by-product of recycled paper industry as well as a destination to the sugar cane bagasse.

Keywords: cane bagasse fiber, composite material, hidrapulper equipment, plastic waste, alternative material for wood.

1. Introduction

The multiple application possibilities of plastic materials, stimulated by the diversity of its properties and characteristics have been conducted to its frequent use in several applications, including civil engineering. The high cost of metals and cement for the production of reinforced concrete and the lack of wood allied to the large amount of plastic material available for recycling increased the use of thermoplastic composites. The use of materials which are able to combine basic requirements, such as the conservation of natural resources and the environment preservation, can be essential for the future of the planet. Considering these problems, a challenge is the production of environmental friendly materials or perform the correct destination and reutilization of plastic materials^[1,2]. The transformation process of plastic waste in composites materials

is a viable option to stimulate practices that prioritizes recycling and development of new materials through the utilization of environmental liabilities.

A polymeric composite is formed by two phases: a continuous one, that is a thermoplastic base matrix and the dispersive phase, which are reinforcing fillers of organic or inorganic fibers [3]. The incorporation of fiber have been widely investigated and used as reinforcement in polymer matrices such as sponge gourd fiber^[4], sisal^[5], curauá^[5], sugar beet pulp^[6] and sugarcane bagasse fiber^[7]. Acacia bark residues was used as reinforcing filler in polypropylene composite can produce higher impact properties and higher degradation temperature^[8]. Taffick et al.^[8] attributed this behavior to the of tiny and short fibers due distributed more homogeneously of composite. Researchers done by Martins et al.^[9] and Rzatki and Barra^[10] proved the effectiveness of the natural amorphous silicate short fibers as reinforcing agents increasing the tensile strength of reinforcement matrix of the epoxy and poly (butylene terephthalate), respectively. The final product will be a polymeric and natural fiber composite as an alternative or replacement to wood and its derivatives, reducing deforestation and pollution.

The residues from the Hydrapulper machinery represent high destination costs both financially and environmentally. The technological equipment, Hydrapulper, is used at the separation of polymer residues, plant fibers and additives coming from containers of the recycled paper industry. This machinery is similar to a giant blender with a pond in cylinder form. The procedure is performed adding water to the paper that will be recycled and by a physical agitation process, the paper fibers are detached forming a cellulose pulp. These fibers are washed and purified, being used for the production of paper in the form of cardboard boxes, napkins, and other products. In a sieve located below the spinning rotor, the impurities in the process, such as various plastics, non disaggregated fibers and small quantities of metals that are retained originating the Plastic Waste from Hydrapulper (PWH), which is a very difficult waste to be recycled and is commonly sent to specific landfills causing concern with the disposal and vast environmental problems.

On the other hand, the abundance of natural fibers such as sisal, coconut, jute, ramie bast, eucalyptus pulp, banana, hemp, flax, pineapple leaf, bamboo, palm, cotton, waste of mate-tea and sugarcane fiber among others and the possibility of using these fibers as reinforcement in composites stimulate the studies of different products combining these raw materials^[11-15]. Each of these natural fibers has a wide range of mechanical and physical properties governing its wider applications. They are renewable, biodegradable, non-toxic, non-abrasive, they have low density with specific strength and low cost. Moreover, they are available worldwide been characterized as “environmentally-friendly” materials^[16-18].

Sugarcane bagasse is the by-product obtained after sucrose extraction from the sugar cane plant or ethanol fuel plant. It is especially interesting

because it is considered the main residue from Brazilian agro industry, which generates around 270 kg of bagasse per ton of cane ground with an annual accumulation of 132 million tons. They are used in industrial furnace and for second generation ethanol, organic fertilizing, animal feed and cellulose and paper production. Sugarcane fibers are composed of cellulose, hemicelluloses and lignin as well as small amounts of mineral, wax, and other compounds^[19,20].

The objective of this study was to prepare and characterize composite materials using PWH from paper industries extruded with sugar cane fiber (SCF) residues from ethanol industries. In Specific (a) a factorial design has been conducted to optimize the proportions and compaction pressure in the composite preparation and (b) characterization tests consisting of physical, chemical and mechanical tests, thermal stability and morphology of the composites materials.

2. Materials and Methods

2.1 Raw materials and composite

The PWH was obtained from a paper recycling factory from Guarapuava, PR, Brazil. The material showed moisture content ~50% and was first centrifuged and then dried in an oven at 100 °C. Clamps, clips, wires, nails and other metallic residues were magnetically separated by a device adapted in the entrance of a Wiley knife mill. In the sequence the plastic waste was bonded for 5 minutes in a thermo-kinetic mixer. Finally the material was cut into small particles using the same mill with a 30 mesh sieve (Figure 1 ac).

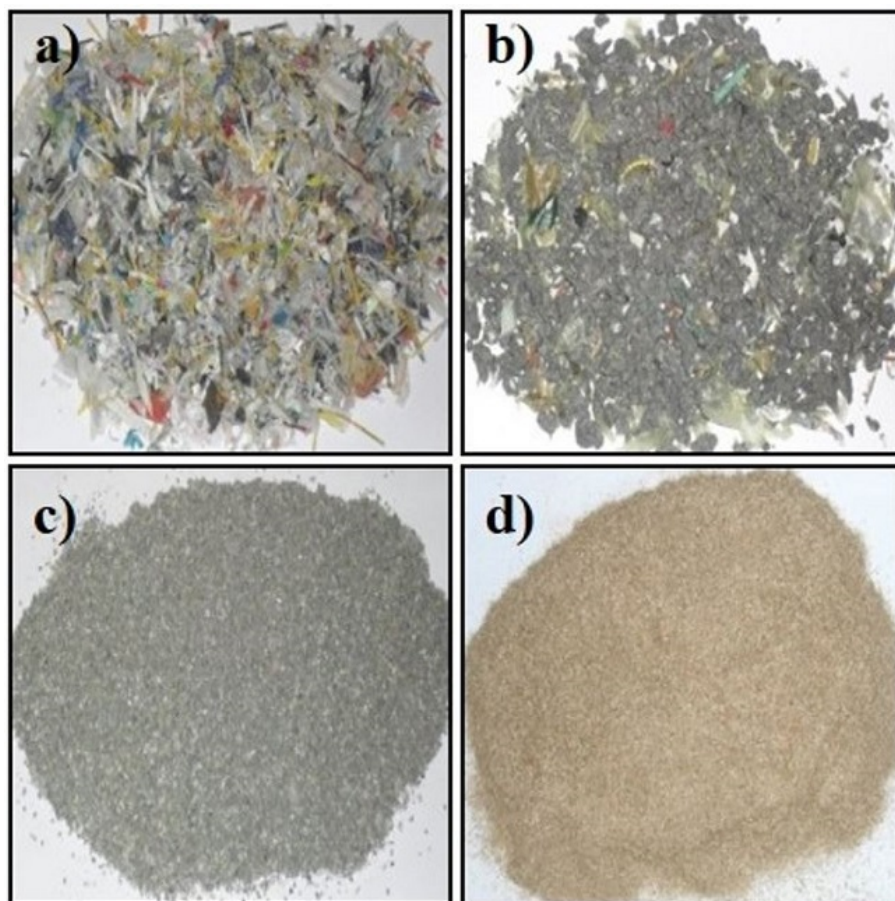


Figure 1.

(a) PWH after first milling and removal of metals. (b) PWH after agglutination process (c) PWH after micronization. (d) Particles of Sugar cane fiber.

Sugar cane fibers were collected from a sugar and ethanol plant in São Pedro do Ivaí, PR, Brazil. The material was dried in an oven at 100 °C and milled in Wiley knife mill with 30 mesh sieve (Figure 1 d).

2.2 Preparation of the composites

The PWH and sugar cane particles were mixed and homogenized at 120 rpm for 5 minutes at room temperature using a thermo-kinetic mixer. The proportions of fiber and polymer contents were performed according to the factorial design that is presented in section 2.4.

For the processing of PWH and the formulated composites, a laboratory single-screw extruder with 45 mm cannon by 300 mm in length and a screw with 25 mm of diameter by 350 mm in length was used, Tornoeste brand (model ET 001). A rotation process of 100 rpm and four heating temperatures were employed, respectively: 160 ± 5 °C (power zone), 175 ± 5 °C (compression zone), 190 ± 5 °C (dosage) and 200 ± 5 °C on the coupling head.

It was possible to check the viscosity and fluidity of plastic waste and the polymeric composites, proposed by the factorial design, by

the pressure of extruded paste, evaluated on the pressure gauge of the extruder.

After the extrusion process, the composites were molded for the mechanical testes according to ISO and ASTM standards. The samples were pressed with 10 or 5 tons for 90 s, according to the factorial design (section 2.4). Additives or coupling agents were not used in the molding process.

2.3 Thermal analysis

Thermal analyses have been performed to analyze the thermal behavior of the raw PWH, the raw sugarcane fiber and the composite material. Tests were done with a equipment SDT Q600 from TA Instruments. Thermogravimetry (TG) and differential scanning calorimetry (DSC) have been applied to determine the weight gain, the mass loss rate and the degradation process. Five milligrams of each samples have been analyzed at the temperature range from 20 °C to 1000 °C at a heating rate of 10 °C.min⁻¹ in a nitrogen flow of 100 mL.min⁻¹.

2.4 Factorial design

A 2² factorial design was performed with the purpose to optimize the production conditions of the polymeric composite^[21], considering fiber proportions and compaction pressure. Initially, one sample of each of the following charge content (sugar cane fiber) has been prepared: 20%, 30%, 40% and 50%. The factorial design was performed considering 30% and 40% because with 50% the extrusion process is very hard and the material has low hardness. With 20% of fiber content the material loses its wood appearance. The compaction factor was evaluated to analyze if differences in the pressure eliminates some empty spaces in the interfacial region of the materials which interfere in moisture and mechanical hardness of the samples. The studied values were 10 and 5 ton. Table 1 present the factorial design experiment. Five samples of each condition were prepared and evaluated for tensile strength, impact, hardness, moisture modifications and composite density.

Table 1.
Factorial design description.

Essay	Factor 1 (proportion)	Factor 2 (compaction)
1	70% PWH/30% bagasse fiber (-)	5 ton (-)
2	60% PWH/40% bagasse fiber (+)	5 ton (-)
3	70% PWH/30% bagasse fiber (-)	10 ton (+)
4	60% PWH/40% bagasse fiber (+)	10 ton (+)

2.5 Physical and mechanical tests

Water absorption was carried out according to ASTM D-1037 with 24 h immersion time in a thermostatic bath at 20°C. The body tests were weighed before and after the immersions. Water absorption was determined using Equation 1 :

$$\% \text{ moisture} = \frac{\text{wet sample mass} - \text{dry sample mass}}{\text{dry sample mass}} \times 100 \quad (1)$$

The density of the composites and of the raw PWH was carried out with a Multipycnometer from Quantachrome Instruments using He gas. The measurements were performed in triplicate.

The mechanical properties of the raw PWH and of the composites were evaluated through tensile strength at break (ASTM D-638) in an EMIC DL 10000 machine, flexural strength (ASTM D-790) in an LLOYD LR10K, Impact strength (Izod) with V notch (ASTM D-256) at room temperature in a RESIL impactor 5R, compression strength (ISO 604) in a EMIC DL 30000 and hardness shore (ABNT 7456) with a WOLTEST GSD 702D. All mechanical properties were determined using at least 5 samples for each test.

2.6 Morphological analysis

Analysis of fractured surfaces from raw PWH and from polymeric compounds obtained from Izod Impact test were performed by SEM using a TM300 Hitachi microscope. The samples were fixed in a carbon tape prior to analysis.

The PWH and sugar cane particles were mixed and homogenized at 120 rpm for 5 minutes at room temperature using a thermo-kinetic mixer. The proportions of fiber and polymer contents were performed according to the factorial design that is presented in section 2.4.

For the processing of PWH and the formulated composites, a laboratory single-screw extruder with 45 mm cannon by 300 mm in length and a screw with 25 mm of diameter by 350 mm in length was used, Tornoeste brand (model ET 001). A rotation process of 100 rpm and four heating temperatures were employed, respectively: 160 ± 5 °C (power zone), 175 ± 5 °C (compression zone), 190 ± 5 °C (dosage) and 200 ± 5 °C on the coupling head.

It was possible to check the viscosity and fluidity of plastic waste and the polymeric composites, proposed by the factorial design, by the pressure of extruded paste, evaluated on the pressure gauge of the extruder.

After the extrusion process, the composites were molded for the mechanical testes according to ISO and ASTM standards. The samples were pressed with 10 or 5 tons for 90 s, according to the factorial design (section 2.4). Additives or coupling agents were not used in the molding process.

3. Results and Discussions

3.1 Thermal analysis

Figure 2 presents the thermograms for raw fiber, raw PWH and composites with 30% and 40% of fiber charge. It was observed that all these materials keep stable up to 250 °C eliminating retained moisture. It demonstrates that these raw materials are apt for extrusion, injection and thermoforming process, since 200 ± 5 °C was the maximum temperature applied in the composites preparation.

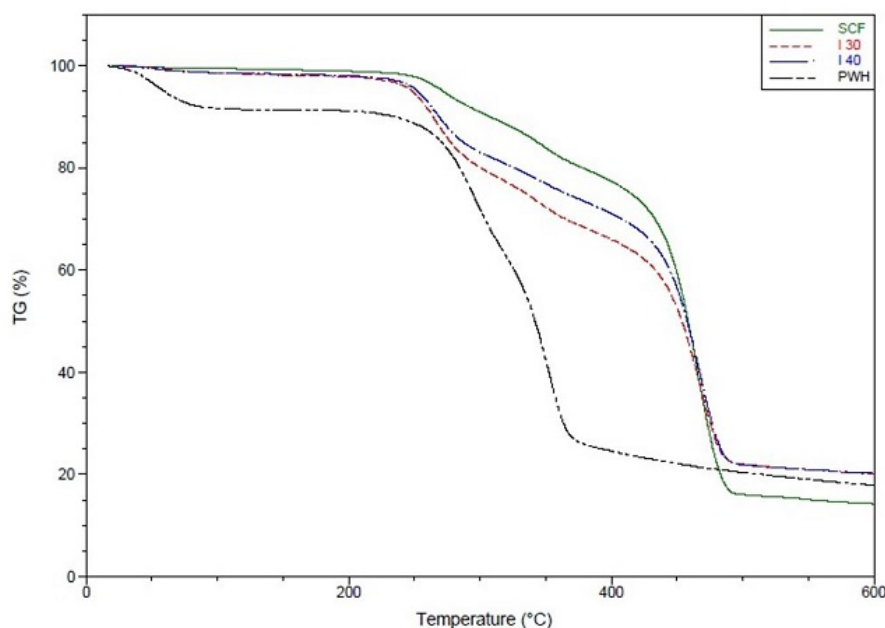


Figure 2.

Thermograms (TG) curves for sugar cane fiber (SCF), and polymeric composites with 30% (I 30) and 40% (I 40) fiber charge, and pure plastic waste from hydrapulper (PWH).

The curves for raw PWH show mass loss and small degradation in the 250 °C to 450 °C interval. In the end of the composition process, the residual mass is smaller than in the polymeric composites.

Between 250 °C and 450 °C the composite with 40% fiber loses less mass and degrades more slowly than the sample with 30% fiber. It is in agreement with the literature which shows that natural fiber compounds have higher heat resistance and consequently high resistance to decomposition. So, cellulose and lignin contribute to the thermal resistance of the polymeric composite with higher fiber charge ^[22]. For temperatures over 450 °C the mass loss is equivalent.

Endothermic crystalline transitions are evidenced by DSC curves, Figure 3 . For temperatures below 120 °C second order transitions are present, demonstrating the presence in smaller quantities of amorphous polymers (PS, ABS, PVC). Over 200 °C the discontinuities occur due to the presence of semi-crystalline high fusion polymers (PET, PA 6/6.6, PC)^[23,24]. The main events are polyolefin fusion (polyethylene and polypropylene) at 125.6 °C and 160.8 °C, besides the onset thermal degradation at 266.5 °C and offset at 465.9 °C, demonstrated in heat absorption peaks. As for sugar cane bagasse fibers, an endothermic peak was observed at 66.6 °C related to the moisture loss. The peaks at 300.3 °C (endothermic) and 355.3 °C (exothermic) represent hemicelluloses and cellulose degradation, respectively. Lignin degradation occurs at 395 °C as shown by the exothermic peak^[25,26].

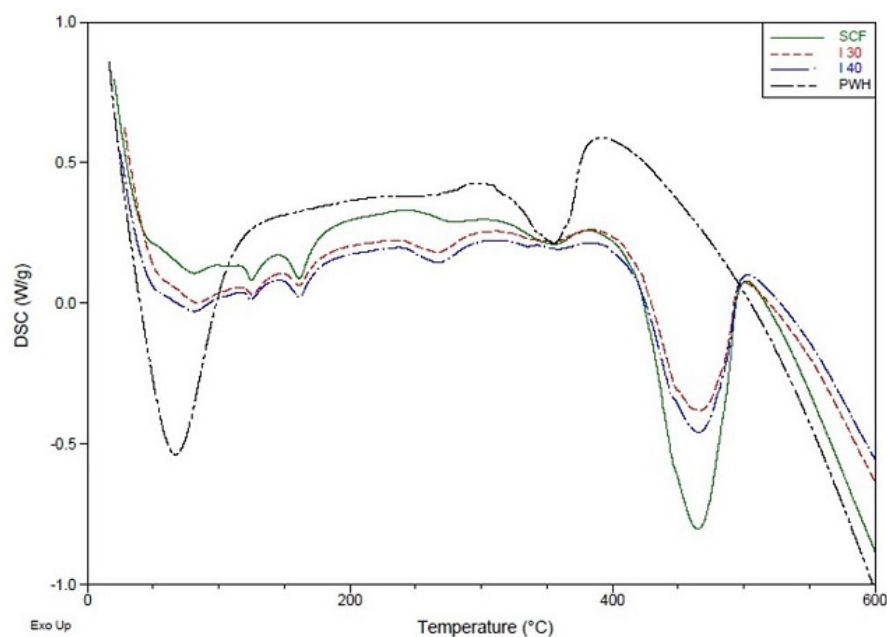


Figure 3.

Differential Scanning Calorimetry (DSC) curves for sugar cane fiber (SCF), polymeric composites with 30% (I 30) and 40% (I 40) fiber charge, and pure plastic waste from hydropulper (PWH).

3.2 Physical and mechanical properties

Incorporated as polymeric matrix in composites, the purpose is to use PWH to replace raw polymers like polyolefin that are the most used thermoplastics. Table 2 presents the results of physical and mechanical properties of the raw PWH and also compares it with the main polymers commonly used to prepare composites.

Table 2.

Mechanical and physical properties of PWH compared to virgin and recycled polymers.

Polymeric Matrix	Hardness Shore D (0 to 100)	Flexural strength	Tensile strength	Izod impact strength (J/m)	Moisture absorption (%)	Density (g cm ⁻³)
PWH	41.4 ± 0.7	18.6 ± 0.3	9.6 ± 0.4	73.9 ± 2.0	0.95 ± 0.06	1.00 ± 0.06
PEHD	62.0 ± 0.79 ^[27]	18.99 ± 0.45 ^[27]	20.73 ± 0.44 ^[27]	-----	< 0.01 ^[28,29]	0.94-0.98 ^[28,29]
Recycled	-----	≅ 20.5 ^[2]	≅ 21.0 ^[2]	≅ 46.5 ^[2]	-----	-----
	64.0 ^[30]	24.87 ^[30]	25.66 ^[30]	33.9 ^[30]	-----	-----
PEHD	62.9 ± 0.55 ^[27]	-----	24.53 ± 0.12 ^[27]	-----	-----	-----
Virgin	65.0 ^[30]	24.87 ^[28]	24.73 ^[30]	38.34 ^[30]	-----	-----
	-----	19.76 ± 0.39 ^[31]	18.9 ± 0.2 ^[31]	-----	-----	-----
PP	68.2 ± 0.57 ^[27]	41.74 ± 0.49 ^[27]	28.96 ± 0.67 ^[27]	-----	-----	-----
Recycled	-----	-----	22.78 ± 1.14 ^[32]	26.25 ± 4.5 ^[32]	-----	-----
	-----	18.2 ^[33]	16.1 ^[33]	-----	-----	-----
PP	71.0 ± 0.71 ^[27]	43.48 ± 0.51 ^[27]	30.72 ± 0.45 ^[27]	-----	< 0.03 ^[34,35]	0.90-0.91 ^[29,35]
Virgin	-----	-----	37.36 ± 1.87 ^[33]	25.05 ± 1.6 ^[33]	-----	-----
	-----	55.74 ^[36]	34.11 ^[36]	5.68 ^[36]	-----	-----
	-----	-----	23.4 ± 1.3 ^[37]	8.5 ^[37]	-----	-----

The PWH absorbs higher water quantity when compared to polyethylene and polypropylene because in this plastic waste there is an incorporation of small quantities of cellulose residues in the plastic surface. The presence of these fibers and other impurities increases water retention. The PWH is denser than the polyolefin due to smaller proportion of thermoplastics, thermosets and elastomers which enlarge the final density.

Shore D hardness and tensile strength presents smaller values in the PWH compared to the other polymers. For flexural strength the values were similar to virgin or recycled PEAD. As mentioned before, the PWH is composed by a mix of thermoplastics, thermosets and elastomers that are melted to form the polymeric matrix. But the polymers that have high melting temperatures are incorporated to the composite as particulate charge generating micrometric empty spaces between the particulate and the polymeric matrix. This fact restricts the mechanical properties that require higher interface adhesion of the materials as noted in the tensile strength test.

In the Izod impact test is possible to verify that the PWH has greater resistance compared to the polyolefin. So the composite could be employed in applications that require high impact resistance.

Factorial design results and the comparison among the raw PWH and the composites are presented in Table 3. When shore hardness is analyzed, it is possible to verify that with 5 ton the results are equivalent in both proportions, but with 10 ton and 40% fiber the shore hardness decreased. For tensile strength all the results are equivalent considering the standard deviation with a slightly decrease for higher pressure quantities. The same equivalence was obtained for traction test. In the impact test, the samples with higher fiber proportion have significant higher Izod impact strength, reaching around 58 J.m^{-1} . The resistance to compression was slightly better for 5 ton compaction. The moisture physical test shows that increasing the fiber content, it increases the moisture. And also at 10 ton the moisture is higher than in 5 ton. Density of the material remains constant independently on the proportion and compaction value.

Table 3.

Factorial Design results for the polymeric composites compared to the mechanical and physical properties of raw PWH. Values and standard deviation for 5 different samples.

Property	Raw PWH	Composite 1	Composite 2	Composite 3	Composite 4
		70% PWH/30% SCF 5 ton	60% PWH/40% SCF 5 ton	70% PWH/30% SCF 10 ton	60% PWH/40% SCF 10 ton
Hardness (Shore D) (0-100 scale)	41.4 ± 0.7	51.9 ± 2.8	52.2 ± 1.6	51.5 ± 1.3	48.1 ± 1.3
Flexural strenght (MPa)	18.6 ± 0.3	16.9 ± 0.6	18.1 ± 0.4	15.6 ± 0.7	14.3 ± 0.5
Tensile resistance (MPa)	9.6 ± 0.4	7.5 ± 0.3	7.6 ± 0.7	7.5 ± 0.4	7.9 ± 0.8
Impact resistance (Izod) (J/m)	73.9 ± 2.0	45.6 ± 1.5	57.5 ± 4.0	50.5 ± 4.7	58.0 ± 2.7
Compression strength (MPa)	27.7 ± 0.9	11.9 ± 0.8	13.9 ± 0.5	8.9 ± 1.2	10.3 ± 1.8
Moisture retention (%)	0.90 ± 0.06	1.8 ± 0.1	2.1 ± 0.1	1.9 ± 0.1	2.4 ± 0.2
Density (g cm ⁻³)	1.00 ± 0.06	1.22 ± 0.01	1.19 ± 0.01	1.20 ± 0.01	1.18 ± 0.01

Comparing the composite material with raw PHW it is possible to verify that the reinforcement with sugar cane fiber, although present visually good appearance, decreases the mechanical resistance and increases moisture and density in the samples. Inserting the fiber content in the PWH makes it harder than the composite. Analyzing the factorial design results the application of 10 ton seems to be inefficient. We suppose that with higher compaction pressure the natural fibers shifts from the interface region decreasing the interaction with the polymeric matrix. With the pressure excess, cracks were formed in the sample decreasing the mechanical properties of the final composite.

Considering the four possibilities of the factorial design, the tests with higher fiber proportion and lower compaction pressure demonstrated the best result. Table 4 present a comparison with the best result of our study with other similar studies in the literature.

Table 4.

Comparison of the optimized condition reached in this study with literature results.

Conditions/references	Tensile strength (MPa)	Flexural strength (MPa)	Izod impact strength (J/m)	Compression strength (MPa)
60% PWH/40% sugar cane fiber (our study)	7.6 ± 0.7	18.1 ± 0.4	57.5 ± 4.0	13.9 ± 0.5
1-80% Epoxi resin/15% FBC/5% glass fiber ^[23]	4.69	3.96 ± 0.16	24.01 ± 0.28	9.60
2-80% raw PEAD/20% sisal fiber ^[38]	22.71 ± 0.10	35.27 ± 1.02	65.5 ± 2.37	-
2-80% raw PP/20% sisal fiber ^[38]	28.62 ± 0.13	49.74 ± 1.39	34.60 ± 2.56	-
3-50% recycled PEAD/50% wheat straw ^[39]	8.11 ± 1.44	13.16 ± 1.64	47.37 ± 6.42	-
3-50% recycled PP/50% wheat straw ^[39]	6.42 ± 1.11	17.76 ± 1.06	11.06 ± 1.50	-
4-90% recycled PEAD reciclado/10% banana fiber ^[40]	20.4 ± 0.3	-	45.2 ± 1.9	-
5-90% recycled PEAD/10% sisal fiber ^[2]	21.2	25.3	62.0	-
6-50% PSAI/50% pineapple leaf fiber ^[41]	22.64 ± 1.25	31.66 ± 3.25	24.39 ± 1.34	-

3.3 Scanning electron microscopy (SEM)

An image of the fractured surface from the Izod impact sample of raw PWH is presented in Figure 4. It can be noted that there are components that were not melted and kept in the samples as charged particles. The Figure 4 b highlights the empty spaces between melted and not melted polymers (white points). This indicates the necessity of some kind of reinforcement charge or some coupling agent to increase the interfacial adhesion^[42,43].

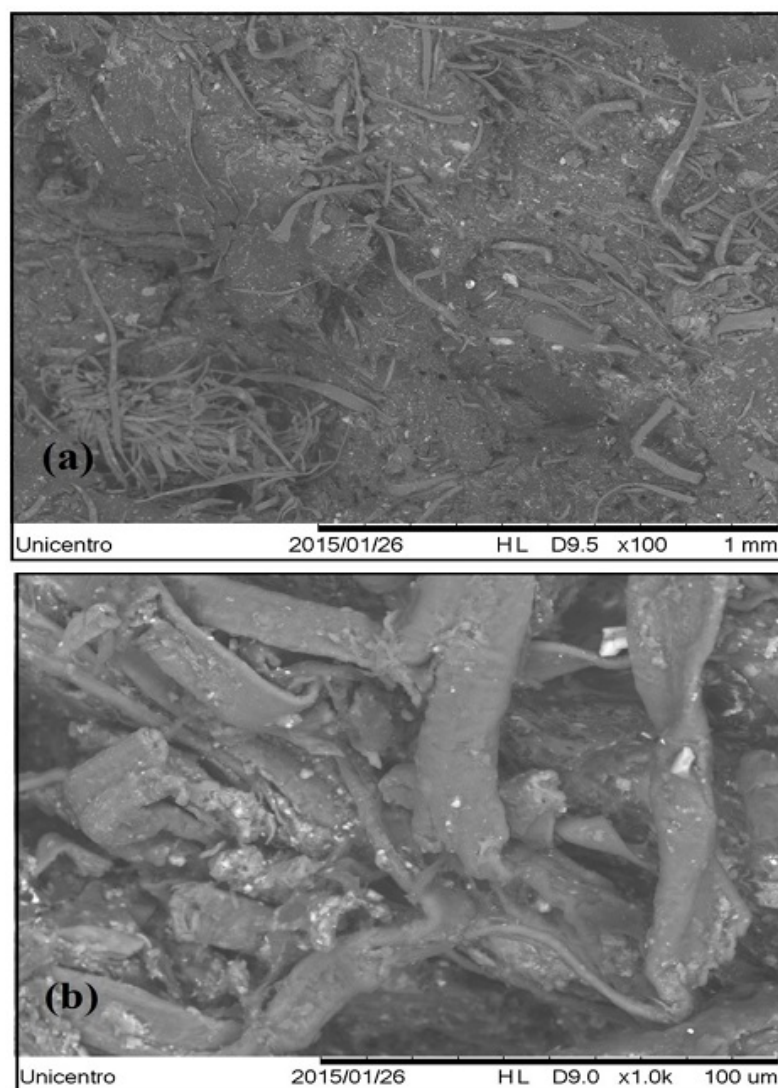


Figure 4
Scanning electron microscopy of the (a) fracture of raw plastic waste from hydropulper and (b) higher magnification with empty spaces.

Thakur et al. have shown that it is possible to perform a surface modification, for example, of natural polymers using silane coupling agent^[44] or graft copolymerization^[45], this change in morphology can cause changes in the properties of the polymers.

Figure 5 a and b present the micrographs for the composites with 30% and 40% of natural fibers. Compared to the micrograph of the raw PWH is noted that the reinforcement charge incorporation filled the porous to obtain a more homogeneous material. Figure 5 c, with amplification power of 1000 times in the sample with 40% fiber it still presents several “white dots” related to the polymers not melted, it attests that sugar cane fiber charge was not able to cause an efficient adhesion. However, the final product has interesting properties and applications.

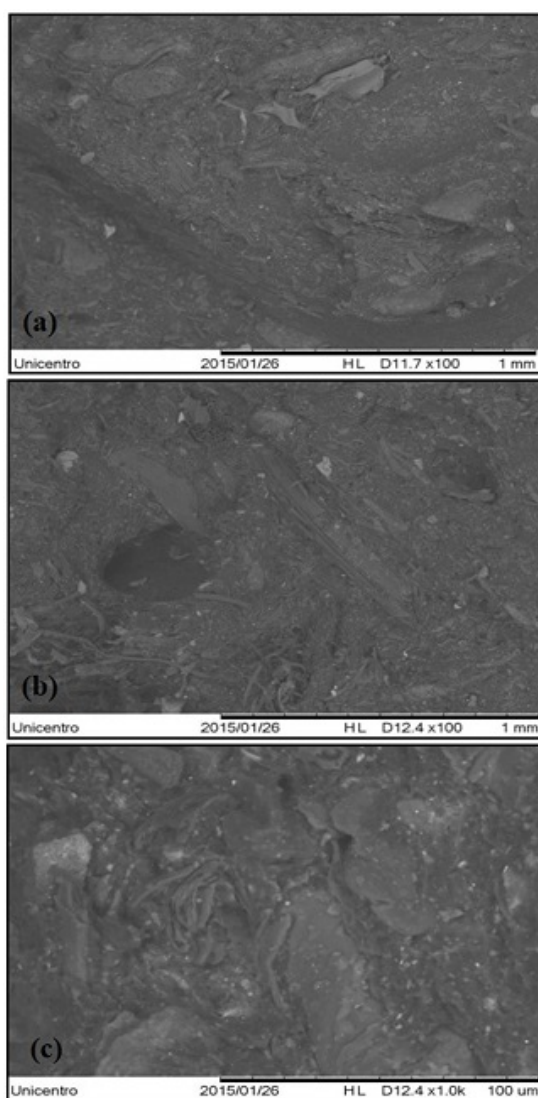


Figure 5.

Scanning electron microscopy of the fracture of composites (a) 30% sugar cane fiber, 100x; (b) 40% sugar cane fiber, 100x; (c) 40% sugar cane fiber, 1000x.

4. Conclusions

Thermal analysis attested that the composite is resistant up to 250 °C, so it is not degraded in extrusion, injection and thermoset process. The PWH is composed in its majority by polyolefin (PEAD, PP, PEBD) as concluded by DSC analysis.

Compared to the raw PWH, the addition of sugar cane as reinforcement fiber increased the hardness of the material. The other physical and mechanical properties were not improved. Higher fiber proportion in the composite presented positive effects, mainly in the compression and impact tests, although water absorption increased. It was observed that the material presented a woody aspect losing its plastic appearance. So, the composite material with 40% of sugar cane fiber, pressed with 5 ton just after extrusion was the optimized condition reached in this study. Higher pressure values caused a decrease in resistance and an increase in moisture.

In the segment of recycled paper and cellulose this study contributes to give a profitable destination to the plastic waste generated as a by-product of recycled paper industry. The polymeric composite produced presents a material with strong environmental appeal as an alternative to wood and some derivatives to be used in buildings and furniture industries.

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