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Original Article

Development and characterization of WPCs produced with high amount of wood residue

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ABSTRACT

In this work, high levels of wood residue were incorporated into high density polyethylene (HDPE). The composite processing was carried out in a twin screw extruder. Due to the large amount of solid charge, adaptation of the equipment was necessary. Wood polymer composites (WPCs) with 60, 65 and 70% wood residue were obtained. In order to use composites in applications such as profiles for application on floors, an analysis of the fatigue strength of the composites was performed, as well as the water absorption capacity. The effect of ultra violet (UV) radiation on the surface of the composites was also evaluated using simulation in a degradation chamber. There was a significant increase in the stiffness of the composites and a reduction in flexural strength. Due to the large amount of load, formation of agglomerates was observed, which reduced the impact resistance. In spite of the use of compatibilizers, it was observed a weak adhesion between the phases, which impaired in the number of cycles under fatigue. When subjected to UV-degradation, the materials exhibited a small reduction in tensile strength. In general, the results indicate that the developed WPCs, considering applications in floors, presented some suitable properties such as rigidity and flexural strength, however, the processing conditions must be adequate for greater interaction between the phases.

1. Introduction

Industrial and academic interests about the use of natural fiber from vegetal origin, known as natural lignocellulosic fibers (NLFs), as reinforcement for polymer matrix composites have grown exponentially since the beginning of the XXI century. Government environmental regulations motivated industries that employed inorganic fillers as reinforcements in polymer matrix composites to find environmentally friendly alternative materials. The use of renewable materials and the consolidation of circular economy are two important strategies towards a more sustainable future. In this scenario, NLFs appears as promising materials as they are capable of combining excellent mechanical properties and low den-

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sity with abundance, low cost of production and recyclability [1–5]. In addition, it has been reported the successful use of NLFs/polymer composites in a wide range of engineering applications such as building construction [6–9] and packing [10,11] as well as automobile industry [12–15] and even bullet proof applications [16–20]. Among innumerous types of NLFs, wood fiber stands up as promising candidate to be used as reinforcement for polymer matrix composites, especially for application in building materials [21]. Moreover, Liikanen et al. [22] showed that even waste from construction and demolition could be suitable for using as raw material for WPCs.

In spite of these interesting advantages, other inherent characteristics of WPCs could be considered as a challenge for a wider and more reliable use of it. Poor interfacial adhesion between the hydrophobic polymer matrix and the high hydrophilicity of the natural fiber reinforcement was addressed in several researches in the past years. In these researches, different chemical and physical treatments have been proposed to try to overcome such limitation [23–26]. Hünnekens et al. [23] studied the effect of plasma treatment on the surface properties of a polypropylene matrix WPC composite. The authors suggested that due the plasma treatment the adhesion between the two phases could be enhanced as the chemical compatibility is improved. Dimitriou et al. [24] investigated the effect of hydrogen peroxide for improving the adhesion in WPCs. In that work, it was showed that the use of an alkaline solution could increase the interfacial adhesion of this material in over 35% in comparison to an untreated composite. Nevertheless, other processing parameters as well as properties under certain conditions still lack of investigation and should be investigate properly. Which involves: the characteristics of the wood flour such as shape, size, distribution and volumetric concentration [27,28], as well as stability and durability of these WPCs that would directly impair in the application of these materials. In this context, the present work objective was, for the first time, to critically assess the evaluation on the properties of a high density polyethylene (HDPE) matrix reinforced with high amount, up to 70 vol%, of wood flour composite under fatigue, water absorption and mechanical properties after ultraviolet (UV) degradation.

2. Materials and methods

2.1. *WPCs production*

High-density polyethylene (HDPE) PE IA59, produced by Braskem, Brazil, with specific weight of 940 kg/m^3 was used as a matrix. The flow rate of the HDPE was determinate by the manufacturer as 0.73 g/min. Residue from the processing of Eucalyptus wood, botanically known as *Eucalyptus grandis*, with average particle size of 0.74 mm and density of 440 kg/m^3 was used as reinforcement. Other additives were also added to the composites to enhance the plasticity of the material and allows that higher contents of reinforcement be added to the composite formulation, as described in Table 1. The investigated composition of the WPCs produced in the present work was based on other researches as described elsewhere [29].

A co-rotating twin screw extruder was used to process the materials and produce the WPCs. The processing parameters of this process are presented in Table 2.

Due to the high content of the wood flour residue, it was necessary to adapt the equipment to the mixing process by removing the matrix, as showed in Fig.1. This product was further process in a knife mill and dried in an oven at 100 °C for 4 h to reduce the moisture content. The final shape of the WPCs produced was obtained by injection molding in accordance with ISO 527 [30] and ISO 178 [31] standards, in a Romi model Primax injector. Injection molding parameter are presented in Table 3.

2.2. *Mechanical properties*

Mechanical properties and impact resistance of the produced WPCs were measured at quasi-static strain rates as well as low strain rates using an Izod pendulum. Tensile and flexural tests were performed, in accordance with aforementioned ISO 527 [30] and ISO 178 [31] standards, in model DL 2000 Emic equipment with a dimension of the specimen 4 mm thick and 10 mm wide, using 5 mm/min and 2 mm/min, respectively. The flexural tests were performed in a three-points configuration system. Data was treated using the Tesc software. Izod impact resistance test was performed according to ISO

Table 1 – Materials used in the WPC production and nomenclature of the conditions.

Materials	Weight %			Characteristic
PE IA 59	19%	13%	8%	
Wood residue	60%	65%	70%	
Orevac CA 167*	6%	6,5%	7%	Flow index 0.3 g/min; Tm = 180 °C
Calcium carbonate	10%	10%	10%	D = 325 mesh
Struktol	5%	5%	5%	Dropping point = 67–77 °C
Composite nomenclature	WPC60	WPC65	WPC70	

Table 2 – Processing parameters.

Screw diameter	Screw speed	Mixing zones	Heating profile
30 mm	120 rpm	45° and 90°	Z1 = 155 °C; Z2 = 155 °C; Z3 = 160 °C; Z4 –Z7 = 170 °C; Z8 – Z10 = 175 °C; Z11 = 32 °C.
L/D ratio	Feeding speed		
40	9 rpm		

Table 3 – Injection parameters.

Injection pressure	Injection speed	Cooling time	Heating profile
1250 bar	180 cm^3/s	20 s	Z1 = 200 °C; Z2 = 200 °C; Z3 = 190 °C; Z4 = 180 °C

Fig. 1 – Co-rotating twin screw extruder adapted for high amounts of wood residue.

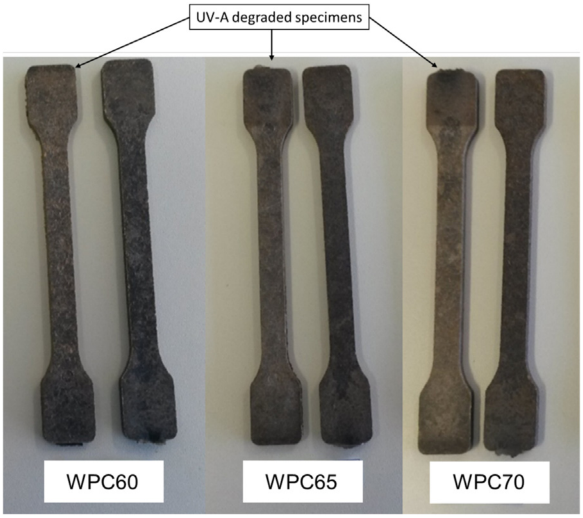

Fig. 2 – Comparison of WCPs specimens exposed to UV-A radiation source and non-exposed.

180 standard [32]. The impact specimen with4 × 10 × 80 mm^3 were evaluated in a model CEAST 9050 Instron equipped with 2.7 J Izod hammer. For all the mechanical tests five specimens were used to guarantee statistical validation of the measured results.

Fatigue tests were conducted, as described in ASTM D7791 standard [33], in a model Electron Plus 3000, Instron fatigue machine with 10kN load cell, at 25 °C. Frequency of 1Hz and maximum loading set as 25% of the maximum load obtained in the tensile test were the parameters used in this evaluation.

The morphological aspects of fractured surface of these tests were analyzed by scanning electron microscopy in model JSM 6510 LV Jeol equipment. In order to make the samples conductive, carbon sputtering coated the analyzed surface on a Denton Vacuum equipped with auxiliary carbon yarn accessory.

2.3. *Water absorption*

The water absorption of the WPCs was carried out following the ISO 62 standard [34]. Initially, the samples were dried at 50 °C for 24 h in an air oven. The specimens were weighed on a GEHAKA brand scale (accuracy of 0.001 g), model AG 200 and immersed in beakers with distilled water. At predetermined time intervals, the specimens were removed from water, weighed, and put back in the bath. This procedure was repeated until saturation on the measured weight was reached. The water absorption calculation was performed using Eq. (1).

$$\text{Water absorption}\,(\%) = \frac{m_2 - m_1}{m_1} \times 100\% \qquad (1)$$

Where m_1 is the mass of the sample before immersion and m_2 is the mass after immersion in water.

2.4. *Ultraviolet (UV) degradation*

The WCPs specimens were exposed under UV-A radiation source, wavelength 340 nm, as shown in Fig. 2. The accelerated weathering equipment displayed temperature control, timers and atmosphere humid. Cycles with duration of 4 h in temperatures of 60 and 50 °C were performed for 30 days. Atmosphere humidity led to a combination of photochemical and thermal degradation. The degraded WPCs specimens were mechanically characterized according to ISO 527 [30] standard in model DL 2000 Emic equipment. A qualitative analysis of the surface was performed using a Zeiss Scope A1 optical microscope, equipped with Axiocam ERc5s Zeiss camera.

3. Results and discussion

Fig. 3 (a)–(c) exhibits the specific mechanical properties for all WPCs conditions investigated in the present work.

It can be observed that the tensile strength increases with the increase in the amount of wood flour reinforcement, Fig. 3 (a) black curve. This increment in the tensile strength may be associated with the main loads' distribution being transferred from the matrix to the reinforcement [35]. The tensile strength observed by the WPC70 condition is in the same order as high-density polyethylene (HDPE), ~25 MPa. If one considers the specific tensile strength, the lower density of the wood flour in comparison with HDPE, 440 and 940 kg/m^3 respectively, would have a major impact in the specific mechanical properties of the composites. Analyzing the flexural strength,

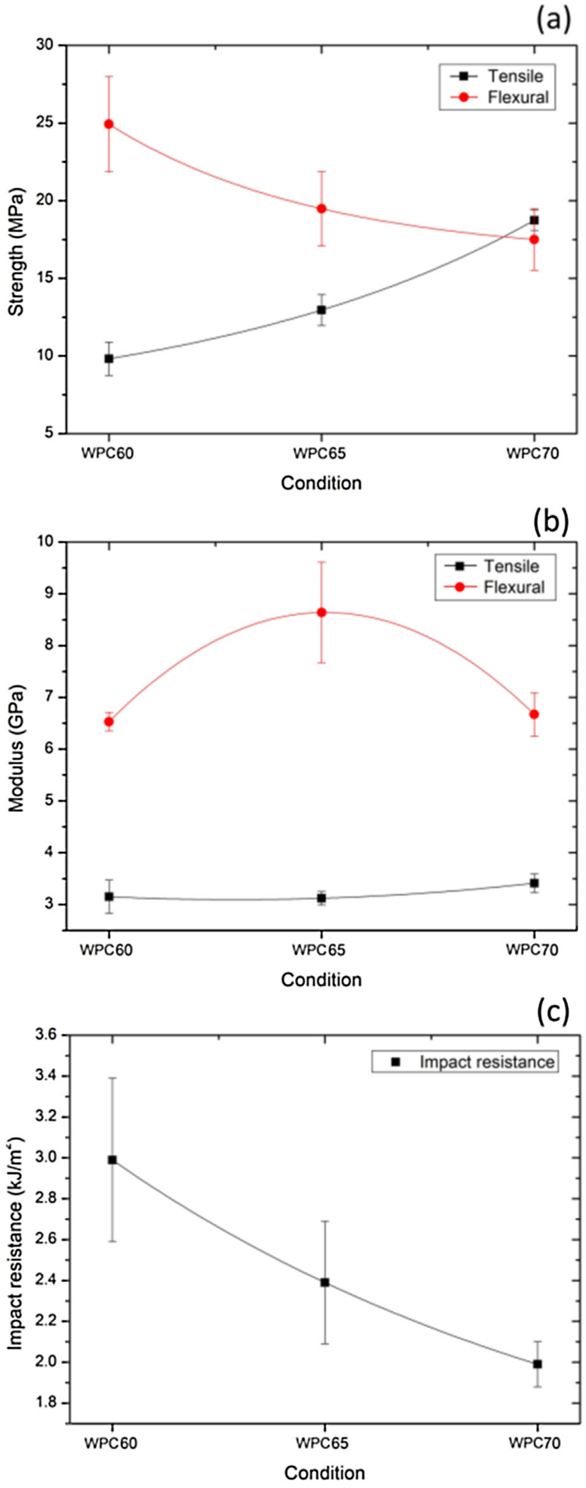

Fig. 3 – Mechanical behavior of the investigated conditions (a) tensile and flexural strength, (b) elastic modulus under tension and flexion and (c) impact resistance.

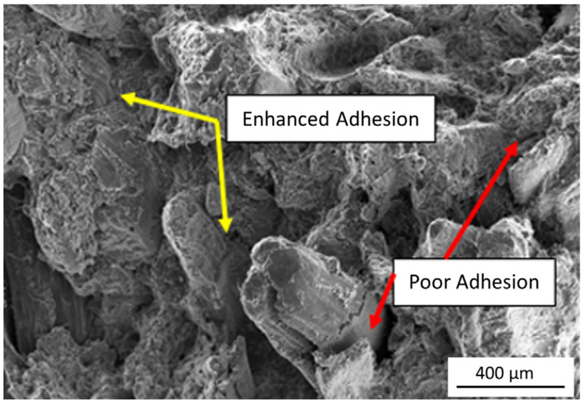

Fig. 4 – Interaction between the reinforcement and the matrix.

Fig. 3 (a) red curve, it is possible to verify an inverse trend as higher amounts of reinforcement exhibited lower values of flexural strength. The applied mechanical forces can result in a heterogeneous strain distribution, implying the existence of localized stresses, which results in a decrease in the maximum force supported by the material [35]. Fig. 3 (b) shows the results obtained for Youngś modulus under tension and flexion. An increase in these properties is observed as the amount of reinforcement increases which is associated with a great increase in the stiffness of the composite. A considerable increase in the modulus of elasticity, when adding the fiber, was observed in other studies, varying its maximum values also depending on the type of fiber used [36]. The increase in the elastic modulus is the result of a good distribution of the fiber in the matrix as well as its orientation, interaction and adhesion between the phases [37]. An example of that can be observed in Fig. 4, which displays the microscopic aspect of the fractured surface of the WPC70 condition under flexion. In this figure, one may observe two opposite situations: a significant interaction between the phases and a weak adhesion.

Regarding the impact resistance, Fig. 3 (c), one should verify the influence of wood reinforcement content on it. Despite the increased stiffness that is associated with the wood reinforcement addition, there is inherent brittleness in such composites. Impact resistance is a complex combination of strength, stiffness and toughness. A factor that could contribute to the decrease of the impact resistance, due to high concentrations of reinforcement, is the agglomeration of particles as shown in Fig. 5. Agglomerates can act as stress concentrators, providing the formation of cracks which reduces the energy absorbed under impact.

Table 4 presents the parameters and number of cycles under fatigue for all investigated conditions. It is important to notice that only the 25% of maximum load was considered in this fatigue test.

One should verify that the increment in the amount of wood reinforcement led to an abrupt decrease in the number of cycles prior to failure. According to Fotouh et al. [38] failure of particle-reinforced composites due to cyclic stress generally occurs due to the accumulation of internal damage. Damages are initiated by the formation and growth of microcracks or voids, clustering, coalescence, formation and growth of early fissures until the dispersion of cracks and ultimately sample failure [39]. The propagation of the failure depends on the consolidation of the dispersed phase as well as on the interface generated with the matrix [40]. In this case, due to the large amount of wood residue reinforcement, the transfer

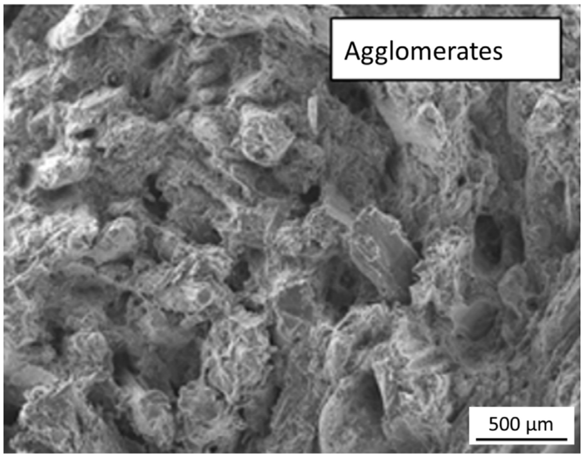

Fig. 5 – Wood reinforcement agglomerates observed in the fractured surface of the impact resistance WPC65 specimen.

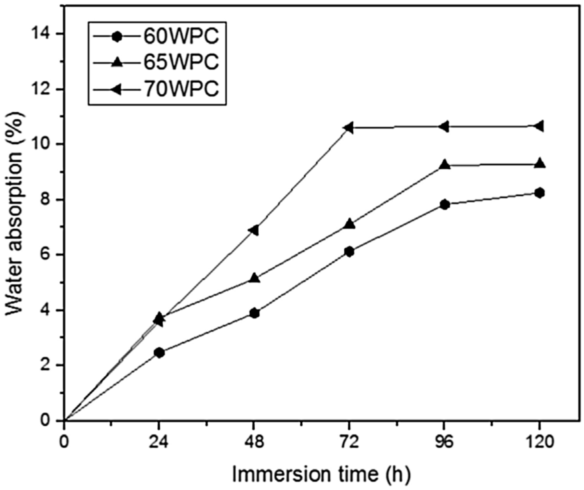

Fig. 7 – Water absorption of the investigated conditions.

of the mechanical load between the phases is compromised, which may justify the results obtained. Fig. 6 presents the morphological analysis of samples after testing. It is possible to observe the pullout of the fibers which suggests the weak interaction between the phases and, consequently, the capability in transferring the mechanical load from the matrix to the reinforcement.

As expected the water absorption increased with the amount of wood flour reinforcement. Fig. 7 shows the result of this investigation.

The results indicate that after 4 days the WPC60 and WPC65 reached saturation weight. As for the WPC70 due to the higher amount of wood flour reinforcement there is a greater water absorption reaching saturation in just 3 days. This behavior is due to the characteristics of NLFs, which are rich in free hydroxyl (OH) groups of cellulose. Water encounters these groups and forms hydrogen bonding which justify the water affinity of this material [41,42].

Both microstructural aspect and mechanical assessment of UV-degraded samples were analyzed. Fig. 8 displays the microstructure from an interface region on WPC60. One may notice the difference between the degraded and the non-degraded region. The region subjected to UV radiation becomes whiter. This behavior is associated with the photo oxidation process resulting in samples discoloration, brittle surfaces, superficial hardening and cracks [43].

Fig. 9 compares the tensile strength of the WPC composites degraded and non-degraded. One can see that the UV-degradation did not significantly impair in the tensile properties of the composite. Which suggests that the UV photo degradation process is restrict to the surface of the material

Table 4 – Fatigue resistance.

Fatigue parameter	Condition		
	WPC60	WPC65	WPC70
Maximum load (N)	95.3	117.5	188.0
Minimum load (N)	9.5	11.8	18.8
Mean load (N)	52.4	68.3	103.3
Amplitude (N)	42.9	55.9	84.5
Number of cycles	329,894	298,457	156,808

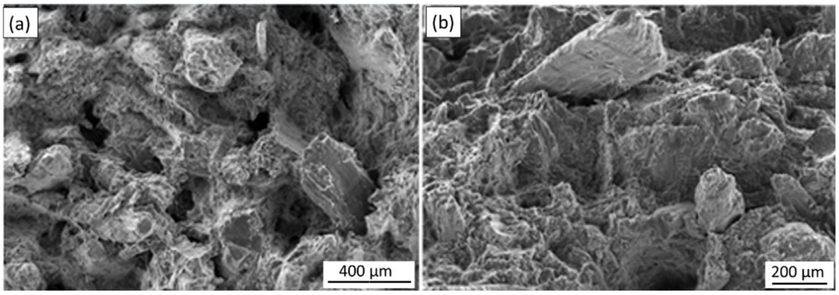

Fig. 6 – Failure surface of WPC composites displaying: (a) Weak interaction between reinforcement and matrix in WPC65 and (b) pull out effect of WPC70.

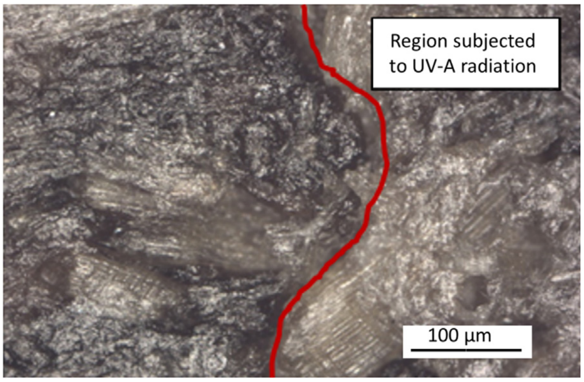

Fig. 8 – Microscopical interface between reinforcement and matrix subjected to UV radiation in the WPC60 composite. Magnification of 50×.

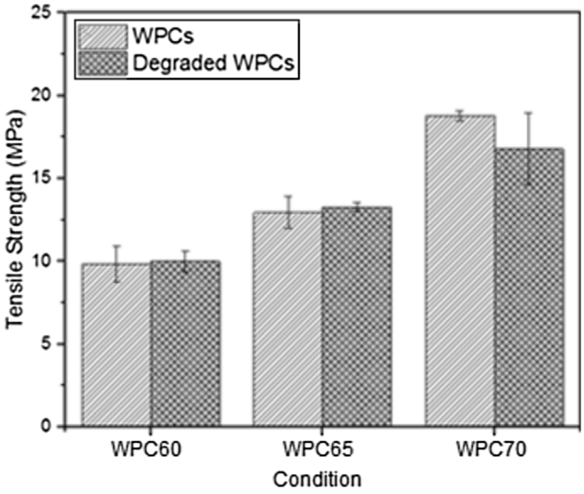

Fig. 9 – Comparison of tensile strength of the specimens before and after UV-A degradation.

and therefore does not impair in the mechanical properties of the material [44–46].

4. Conclusions

WPCs composites with high concentrations of wood flour residue, up to 70 vol%, and additives were processed in a twin screw extruder.

- It was observed an increase in the tensile properties with the amount of wood flour reinforcement. However, an inverse trend was observed for the flexural strength of the material. These relations were associated with the capability of the matrix to successfully transfer the loads to the reinforcement. While the formation of agglomerates could be considered as main responsible for the decrease in some properties.
- Fatigue resistance is strongly influenced by the amount of reinforcement. It is observed that the addition of wood residue significantly decreases the number of material cycles under the conditions evaluated due to the compromised transfer of the mechanical load between the phases by the large amount of load used.
- The presence of wood residue significantly increases the water absorption capacity of the WPCs. On the other hand, the UV-degradation exposure time to which the specimens were submitted was not capable of significantly affects the mechanical properties of the composites.

Conflicts of interest

The authors declare no conflicts of interest.

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