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Novel green composite material manufactured by extrusion process from recycled polypropylene matrix reinforced with eucalyptus fibres and granite powder

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Abstract. The development of sustainable composites materials, from recycled polymeric materials and waste from the wood industry and stone processing, allows reducing the volume of these by-products, minimizing impacts on health and the environment. Nowadays, Polypropylene (PP) is the most recycled polymer in industry, while the furniture industry has increasingly used timber felled from sustainable forest plantations as a eucalypt. The powder tailing from the ornamental stone extraction and processing industry is commonly disposed of in the environment without previous treatment. Thus, the technological option for the development of composite materials presents itself as a sustainable alternative for processing and manufacturing industries, enabling the development of new materials with special technical features. The results showed that powder granite particles may be incorporated into the polypropylene matrix associated with short eucalyptus fibres forming green hybrid composites with potential application in structural engineering, such as transport and civil construction industries.

Keywords: eucalyptus fibres; granite particles; green material; recycled polypropylene; sustainable composites

1. Introduction

According to Vini and Daneshmand (2019), the attempt to adapt circular economy concepts to economic growth necessarily involves the efficient use of resources. This implies the use of fewer

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resources in the production of goods and services, namely waste reuse, and material recycling (Sari *et al.* 2020).

In recent years, the increased production of both manufacturing residues and post-consumption waste has represented a serious problem to the environment. As polymeric materials come from a non-renewable source origin, can be considered as one of the main polluting agents, but it is widely used due its low cost, versatility in applications, and excellent mechanical performance (Elmushyakhi 2021).

These materials have slow spontaneous degradation and, when burned, produce different degrees of toxicity, representing serious environmental damage. As a sustainable alternative, recycling has been the solution to the increased cycle-life of these materials. Thus, factors such as the use of virgin raw materials reduction, the quality of recycled products, low production costs, and the production of new economic-commercial activities have been the reason for searching for novel development (Lopez *et al.* 2020, Vini and Daneshmand 2022). So, some authors affirmed that because of these environmental impacts the society has encouraged the development of new environmentally friendly polymer composite materials, minimizing both the use and dependence on petroleum source products (Sari *et al.* 2020). On the other hand, the incorporation of natural fibre reinforcements, nucleating agents, and organic pigments, have been used to improve mechanical performance to the recycled polymer composites allowing potential use in engineering (Nourbakhsh and Ashori 2009).

According to Zaaba and Ismail (2019), a wide interest in the use of natural waste as reinforcement in composites has increased in recent years which allows the waste reduction, since the acquisition until the disposal involve higher costs, such as planning, execution, and monitoring in the process of transport and stockpiling.

In this context, the production of solid waste in the wood industry generates large amounts of tailings because they are a continuous process, which requires large spaces to lodge sawdust and offer potential risks of fires (Maziero *et al.* 2019).

In Brazil, a large portion of these residual volumes are from eucalyptus wood (IBÁ 2021). In addition, the tailings extraction industry and processing of ornamental stones also cause substantial environmental damage. It can be attributed to wrong methods of disposal without previous treatment which would cause an imminent soil contamination risk and groundwater (Zainuddin *et al.* 2019).

The dry abrasive sludge resulting from the processing of granitic stones, which is constituted by a fine powder that causes straight damage to public health due to the high content of silicates (Medina *et al.* 2017).

Thus, the granite choice waste as a component in new composites materials provides a sustainable option which is still not very approached in the literature. Most research on the use of granite waste is limited to the use of the sludge from cutting/polishing processes or the crushed granite itself as aggregates in the production of concrete and ceramics (Shamsabadi *et al.* 2018, Gonzalez-Triviño *et al.* 2019). A proposal to minimize the environmental impact of these wastes is to add them into polymeric matrices, such as polypropylene and polyethylene, to improve the mechanical properties of composites (Yu *et al.* 2016, Zdiri *et al.* 2018).

Product developed from the use of the waste not only guarantees a lower environmental impact and consequently higher preservation of the resources, but also favors the economic and social development of the producer region. In this sense, several materials have been used as reinforcement in composites materials, which can highlight the natural fibres, such as wood, coconut, cotton, jute, sisal, curaua, kenaf, bamboo, among others. Similarly, the use of mineral

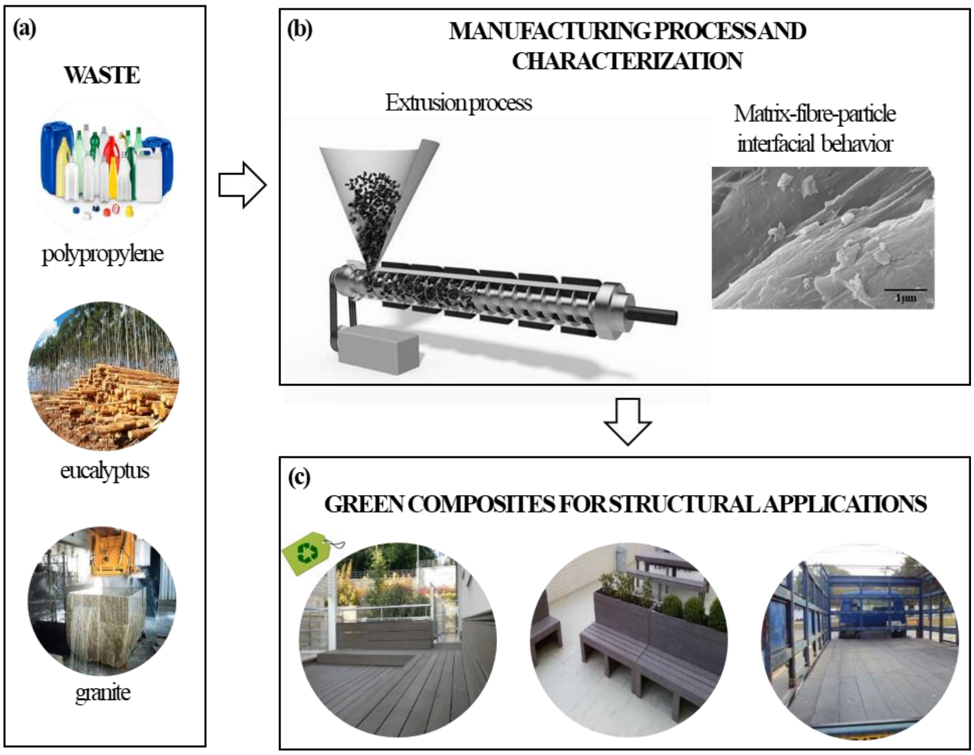

Fig. 1 Hybrid green composites with eucalyptus fibres and granite powder: (a) use of waste to produce composite materials, (b) manufacturing process by extrusion and interfacial adhesion analysis, and (c) structural applications of the new green composite

particles, such as clays, talc, calcite, silica, barite, have been studied by some authors (Ragunathan *et al.* 2017, Ramos *et al.* 2020).

According to Battegazzore *et al.* (2019), many studies show that particle-reinforced composites have lower performance than fibre-reinforced composites. Since that particle reinforced composite has poor performance in tension, some authors have used the bonding of FRP plate to reinforce it (Vini and Daneshmand 2020). As one of the main uses of natural fibres in terms of the reinforcement of plastics and rubbers stand out increasing quality and commercial application (Castro *et al.* 2020). Accordingly, Vieira *et al.* (2016) the development of hybrid composites has gained prominence in the industrial scenario because the adequate mixture of the particles and fibres allows combining different properties into polymeric matrices resulting in materials with better mechanical and physical properties. On the other hand, according to Silva *et al.* (2013) the addition of silica microparticles into hybrid composites was characterized by a statistically noticeable contribution to the porosity and the water absorption.

Currently, polyethylene is the most attractive thermoplastic in making the natural fiber reinforced composites which are mainly used as the exterior building components. Based on these remarks, the main purpose of this study is to investigate the mechanical behaviour of a new hybrid composite, which uses a recycled polypropylene matrix reinforced with short eucalyptus fibres and fine granite particles (powder).

An unprecedented evaluation on the effect of granite particulates in improving the properties of

Table 1 Experimental conditions tested

Experimental condition	Fibre content (% m/m)	Particle content (% m/m)
rPP	-	-
rPP-10F	10	-
rPP-20F	20	-
rPP-10F-10G	10	10
rPP-20F-20G	20	20

short fibres-reinforced polymers produced by extrusion and injection-molded plastic process was made. Its performance was analyzed by tensile, flexural and impact tests. Also, the interfacial adhesion and the fracture mode analysis of the composite specimens was made by optical microscopy (OM) and scanning electron microscopy (SEM). Fig. 1 shows a schematic figure for the production of this green composite and its applications.

2. Materials and methods

2.1 Materials

The matrix used is recycled polypropylene (rPP), prepared from a post-consumer blend in granules, pigmented with 0.3% m/m carbon black (CB, ρ: 1.70-1.90 g cm^{-3}, from Evonik Industries AG), melt flow index of 13.097±0.028 g/10 min (230ºC/2.16 kg), and density of 0.971 g cm^{-3}.

Eucalyptus short fibres and granite particles are used as reinforcement. Eucalyptus fibres measuring between a maximum of 1190 μm (16 mesh) to minus 53 μm (270 mesh) were used. It has a quantity of 70.2±0.2% holocellulose and 26.3±0.6% lignin, which is without chemical treatment, and they are a density of 0.12±0.01 g cm^{-3} (aspect ratio 55). Granite particle sizes are from 841 µm (20 mesh) to minus 53 μm, without chemical treatment, with a specific mass of 2.74±0.12 g cm^{-3}, moisture content of 1.0±0.5%, 67.25%, 12.30%, and 9.32% of SiO_2, Al_2O_3, and Fe_2O_3 in composition, respectively.

The factors studied in this work such as eucalyptus fibre percentage (10% and 20%) and granite powder concentration (10% and 20%), are shown in Table 1. Thus, the effect of incorporating to the rPP matrix, both the short natural fibres reforested wood as micro-particles originating from the processing of decorative stone can be evaluated.

2.2 Composite manufacturing

To produce the composite material processing using an extruder machine. In this process, the polymer is in powder, granules, or pellets, fed continuously from the hopper into the extruder barrel. The heated barrel melts the polymer joining the reinforcer while transporting the composite material through the nozzle. After this, the final material is pressured on to fill an injection die.

Thus, the composites are processed on a SEIBT ES35 L/D 30 single screw extruder, using a screw speed of 90 rpm, feed rate of 2.0 kg h^{-1} and mixing time in the chamber of 180 seconds. Fig. 2 shows the schematic diagram of the single screw extruder, the thread profile, and the temperatures used in the five heating zones.

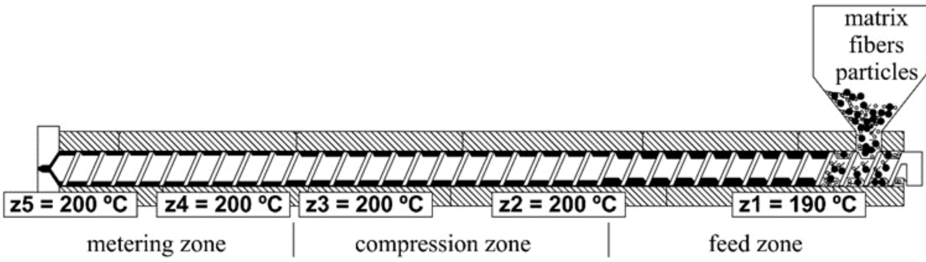

Fig. 2 Schematic diagram of the extruder, thread profile, and extrusion temperatures used

Table 2 Results of ANOVA and Tukey Pairwise Comparisons for the mean values of the mechanical properties, considering the fibre content effect ("EF" effect)

Experimental condition		Tensile tests		Flexural tests		Impact tests
		Tensile strength	Tensile modulus	Flexural strength	Flexural modulus	Impact strength
rPP		A	C	A	C	B
rPP-10F-10G		C	A	B	B	A
rPP-20F-20G		B	B	C	A	C
"EF" effect	*p*-value	0.00	0.00	0.00	0.00	0.00
	R^2 (adj) (%)	94.02	94.52	97.00	96.15	97.27

The specimens were produced from the extruded using standard granules dried in an oven at 100.0±5.0ºC for 5 hours. The injection molding machine was a Pavan Zanetti model HXF 220, with ratio Length and Diameter of 22.2, the feed temperature profile for the injection nozzle of 170 (z5), 180 (z4), 190 (z3), 200 (z2), and 210 (z1) ºC, the injection pressure used to differents zones was 45 (z5), 45 (z4), 50 (z3), 50 (z2), and 50 (z1) bar, respectively.

2.3 Mechanical characterization

Tensile and three-point bending tests were performed on a Lloyd LS 5 universal testing machine. Tensile tests (DIN EN ISO 527-2:2012) were performed with a test speed of 5 mm min^{-1} and a distance between grips of 115 mm, using a 5 kN load cell. The flexure tests follow DIN EN ISO 178:2019-08, using a load cell of 2.5 kN and a strain rate of 1.70 mm min-1. In the Izod impact tests (notched samples), an Instron® CEAST 9050 machine was used, with a 5.5 J hammer and a speed of 3.46 m s^{-1}.

Ten samples of each condition were tested at 23.0±2.0ºC and a relative humidity of 65.0±5.0%, considering two replicates of five samples for each experimental condition.

Analysis of variance (ANOVA) were used to verify if the differences between the sample means are statistically significant, evaluating the effect of the insertion of eucalyptus fibres in the recycled polypropylene matrix and the effect of the insertion of the mixture of eucalyptus fibres with granite powder in the polypropylene matrix, to produce the hybrid composites.

2.4 Morphological characterization

The adhesion between the matrix-fibre-particle interfaces and also the dispersion of the fibres

Table 3 Results of ANOVA and Tukey Pairwise Comparisons for the mean values of the mechanical properties, considering the effect of fibre content and granite powder content (“EF+GP” effect)

Experimental condition		Tensile tests		Flexural tests		Impact tests
		Tensile strength	Tensile modulus	Flexural strength	Flexural modulus	Impact strength
rPP		A	C	A	C	B
rPP-10F-10G		C	A	A	B	A
rPP-20F-20G		B	B	B	A	B
“EF+GP”	*p*-value	0.00	0.00	0.00	0.00	0.00
effect	R^2 (adj) (%)	96.21	94.00	94.61	92.72	81.76

and particles in the matrix was observed by the fracture surface of the samples from the tensile tests. A Bioptika B500 Series optical microscope and a ZEISS® EVO MA10 scanning electron microscope, respectively, were used to obtain the surface images.

3. Results and discussion

The analyze of variance (ANOVA) was used to verify the influence of the fibres (“EF” factor) and the particles as reinforcement (“EF+GP” factor) on the response variable, as a support to the conclusions found in the descriptive statistics, verifying if the means of each experimental condition are different. A p-value lower or equal to 0.05 indicates a significant effect provided by the main factors, or the presence of the interaction of factors on the response variables, considering a confidence interval of 95%. The assumptions of a normal probability of residuals and the homogeneity of variances were checked and confirmed. Values of adjusted R^2 greater than 90%, show that the models explain a considerable variation of data.

The results of ANOVA for the mean values of the mechanical properties (flexural, tensile and impact tests) for each experimental condition are shown in Tables 2 and 3. The “EF factor” and “EF+GP factor” factors were significant on all response variables, presenting *p*-values of 0.00.

Tables 2 and 3 shows the results of the analysis of variance (ANOVA) obtained in the tensile, flexural and impact tests. The adjusted R^2 values values were satisfactory and show a good model explanation. A *p*-value less than 0.05 allows the statistical inference that the sample means are different. Considering only the insertion of eucalyptus fibres (“EF” effect) in the recycled polypropylene matrix (rPP), it is observed that there is a significant change in the mean values of tensile strength and tensile modulus. Moreover, the Tukey test showed a particular grouping for each condition in these tensile properties, indicating different average values between the rPP, the composites with eucalyptus fibres in 10% m/m (rPP-10F) and in 20% m/m (rPP-20F). The ANOVA results also indicated significant changes in the sample means of the tensile tests using the granite powder waste and eucalyptus fibres as reinforcement ("EF+GP" effect), compared to pure rPP. Tukey test also denoted different groupings for the conditions with pure rPP, composites with eucalyptus fibres at 10% m/m, granite powder at 10% m/m (rPP-10F-10G) and using 20% m/m for each waste (rPP-20F-20G).

In the flexural tests, there was a significant difference in the mean values of flexural strength of the conditions. Analyzing the effect of the insertion of the fibres in 10% m/m and 20% m/m in the rPP matrix, different groupings were verified in the Tukey tests for all conditions. The addition of

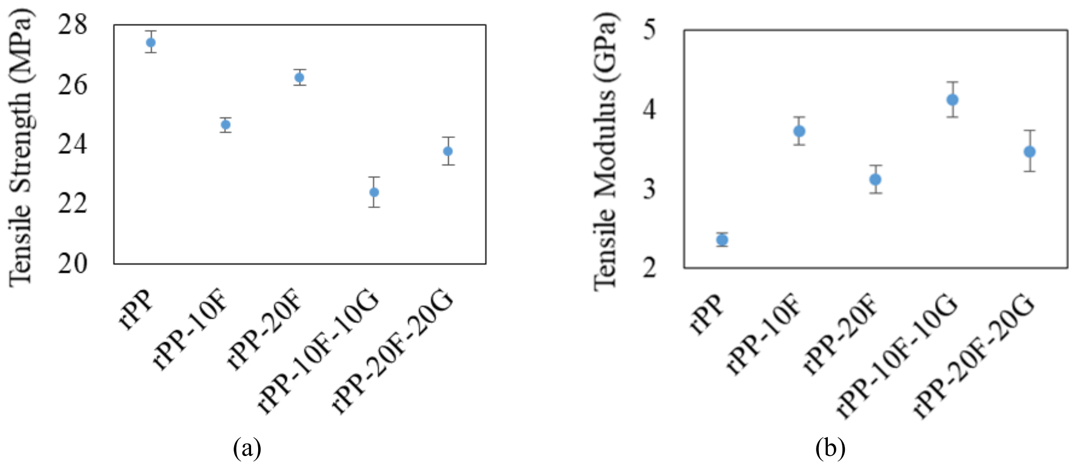

Fig. 3 Tensile properties of the experimental conditions: (a) tensile strength and (b) tensile modulus

granite powder along with the fibres, only the condition with 20% m/m concentration for each residue (rPP-20F-20G) presented a significantly different average value. The flexural modulus was also statistically different for all experimental conditions, with different groupings for each sample mean, evidencing the influence of the matrix-reinforcement interface on the stiffness of the composites. The experimental conditions also showed significant differences in impact strength, with sample means in different groupings by Tukey test.

Fig. 3 shows the results for tensile strength and tensile modulus obtained in the tensile tests of the experimental conditions. In all conditions, the matrix-reinforcement interaction reduced the tensile strength, compared to the pure rPP matrix. The agglomeration of the short fibres and particles acts as stress concentrators, causing the material to rupture with a lower strain. Moreover, the weak matrix-reinforcement interaction caused by the lack of wettability of the fibres determines the macroscopic behaviour and decreases the mechanical strength of the composites. According to Caicedo *et al.* (2018), the formation of aggregates is a disadvantage in the use of mineral particulate materials in composites, and therefore a decrease in the particle size is indicated to obtain a greater surface area.

Fig. 3(b) shows a significant increase in tensile modulus to the composites with 10% m/m of fibres (rPP-10F and rPP-10F-10G), which occurs due to the loading transference between the polymer matrix and the dispersed phases. Also, it can be affirmed that the increase in tensile modulus caused by the incorporation of particulate and fibrous wastes occurs due to the greater limitation to polymer deformation. Other authors have also seen this increase in the tensile modulus during the addition of vegetable fibres and mineral particles in recycled polypropylene matrix when compared to recycled polypropylene (Ibrahim *et al.* 2017, Srivabut *et al.* 2018).

Fig. 4 shows typical stress-strain curves of the experimental conditions evaluated. About the behaviour of the elongation and rupture, for all conditions, there was a ductility decrease of the matrix when the residue (10% to 20% m/m) were added. It can be explained because the fibres and particles have higher stiffness and lower deformation than the pure polymeric matrix. This behaviour is motivated by the restriction to the mobility of the polymer chains of the matrix, making the composite stiffer. According to Zdiri *et al.* (2018) when a mineral filler is added this behaviour is more evident. In addition, during the cure of the composites, the formation of voids

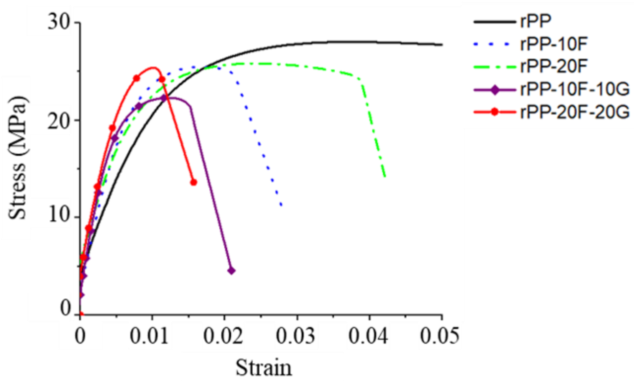

Fig. 4 Tensile stress-strain curves

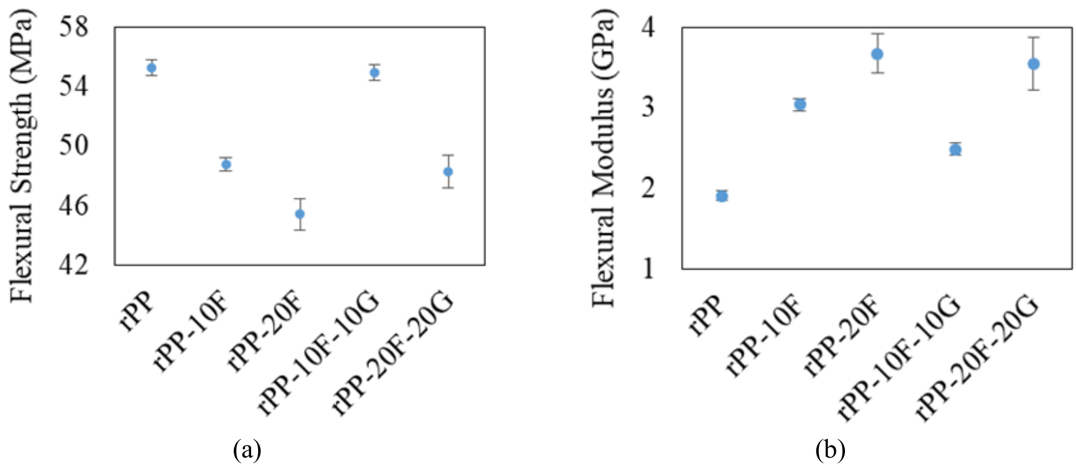

Fig. 5 Flexural properties of the experimental conditions: (a) tensile modulus and (b) flexural modulus

occurs, which act as stress concentrators, decreasing the real cross-section of the specimens and leading to crack nucleation and propagation, as well as material fracture.

The results of the flexural tests (Fig. 5), show a slight reduction in flexural strength with the addition of waste, especially for higher concentrations of waste (rPP-10F, rPP-20F and rPP-20F-20G). Flexural loads impose a particular state of stress on the samples, with compressive stresses on the contact surface with the central displacement pin, and tensile stresses on the bottom surface of the sample (Castro *et al.* 2021). Thus, the deformation capacity can be reduced by the presence of the reinforcements into the composite, as the carbon black and high contents of fillers, as well as the failures of the interface reinforce-matrix adhesion.

The flexural modulus was notably higher for the composites compared to the pure rPP matrix, showing the efficiency of the residues in the load transfer with the matrix.

It is important to mention that the stiffening load reduces the yield stress in the ductile rPP matrix, increasing the modulus of elasticity in all the waste-reinforced composites. This significant increase in stiffness with the addition of eucalyptus fibres and granite particles, indicated by the mean values of flexural modulus, and also tensile modulus, is in agreement with other authors results (Islam *et al.* 2016, Srivabut *et al.* 2018).

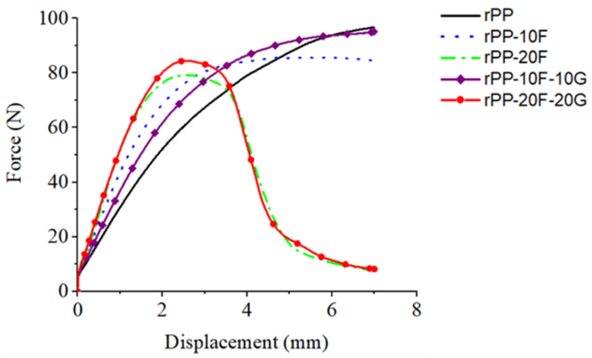

Fig. 6 Flexural tests: typical force-displacement curves

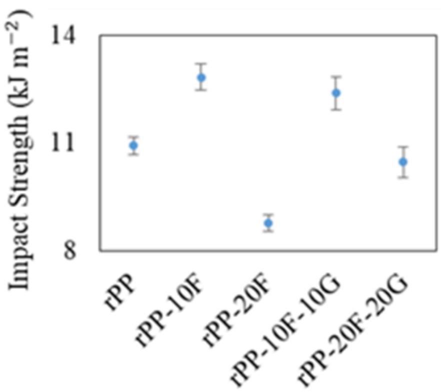

Fig. 7 Impact strength of the experimental conditions

Fig. 6 shows force comportment by displacement of the pure rPP matrix and the reinforced composites with waste. A higher slope in the initial stage of the curves can be observed to the composites with 20% m/m in fibres (rPP-20F) and of the hybrid composites with fibres and granite powder (rPP-20F-20G), proving the high flexural stiffness of these composites. Moreover, higher energy absorption is verified by the composites with lower mass concentrations (rPP-10F and rPP-10F-10G), showing a ductility similar to that presented by the rPP matrix.

Fig. 7 shows the results for the impact strength in notched specimens. When the composites reinforced with fibres and hybrids (fibres and granite powder) are analyzed, it presents a decrease in the impact strength with the increase of waste from 10% to 20% m/m. This could be associated with a possible agglomeration of particles, this evidence possible problems of dispersion of the reinforcements in the composites, which can negatively affect the load transfer and the propagation of cracks in the samples.

Figs. 8 and 9 show the particle dispersion (white circles), confirming the main feature of the manufacturing process (injection), which allows a homogenous distribution of the powdery material and high fineness (31.95%), and the good fibre-particle adhesion in the matrix, respectively. So, it can be affirmed that good dispersion of particles will be responsible for a high impact strength compared to pure rPP, as verified in the statistical analyses. A Bioptika B500 Series optical microscope and a ZEISS® EVO MA10 scanning electron microscope were used to obtain the images of the surface respectively.

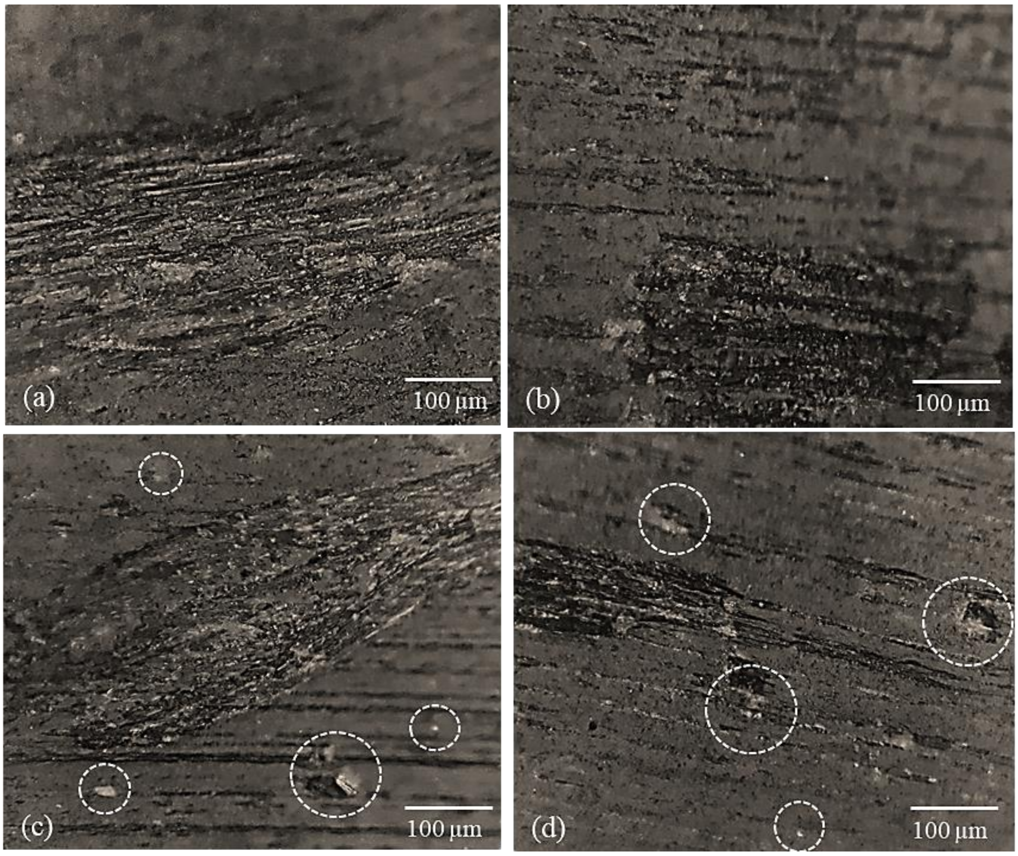

Fig. 8 Dispersion of fibres and particles in the matrix at 40x magnification: (a) rPP-10F, (b) rPP-20F, (c) rPP-10F-10G, and (d) rPP-20F-20G

The SEM images of Fig. 9 allow verifying that when the reinforcements were added to the composite material, a change of mechanical behaviour was caused. Thus, the results obtained in the tensile tests in composites with reinforcement only of short eucalyptus fibres (10% and 20%) present more ductile fracture behaviour in the specimen fracture zone (Figs. 9(a) and 9(b)). On the other hand, when particulate reinforcement (powder) is added the behaviour becomes clearly brittle (Figs. 9(c) and 9(d)). Finally, it can be affirmed that the higher the percentage of particles, the clearer is this brittle behaviour.

5. Conclusions

This study investigated a new environmentally friendly hybrid composite using eucalyptus fibres and granite powder reinforcing recycled polypropylene. The main conclusions of this study are:

- The hybrid composites reinforced with eucalyptus fibres and granite powder showed good processability and good consistency in the investigated samples, validating the manufacturing process by extrusion and injection molding.

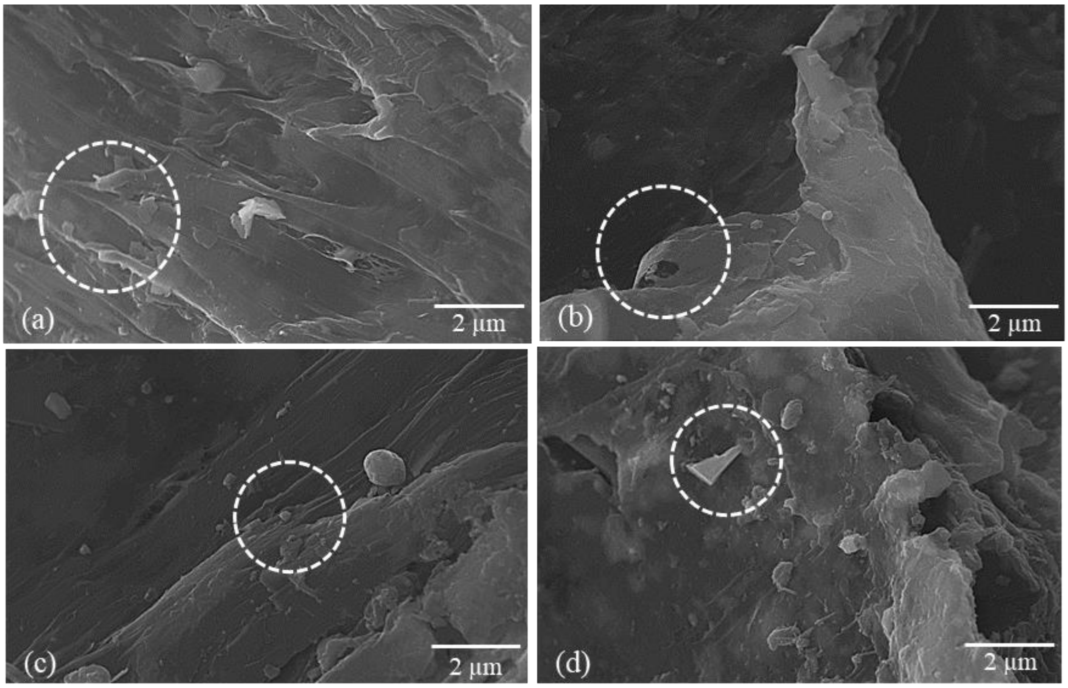

Fig. 9 Matrix-fibre-particle interface of the different composites tested (a) rPP-10F, (b) rPP-20F, (c) rPP-10F-10G, and (d) rPP-20F-20G. Magnification 2000x (30 kV)

- The composites with 10% and 20% m/m eucalyptus fibres showed higher tensile modulus (158% and 132%) than the pure rPP matrix. With the incorporation of granite powder residue in 10% and 20% m/m, this increase was even higher, of 175% and 147%.
- The addition of granite powder to composites reinforced with eucalyptus fibres increased the flexural strength by 113% and 106% for concentrations of 10% and 20% in waste, respectively.
- The composites with eucalyptus fibres showed 159% and 192% increase in flexural modulus with the incorporation of 10% and 20% m/m fibres in the rPP matrix. The hybrid composites also reached increments in the flexural modulus compared to the pure rPP matrix, being 130% with incorporation of 10% m/m and 186%, with 20% m/m of granite powder.
- Lower concentrations of waste (10% m/m) achieved higher averages of Izod impact strength in the composites, showing possible problems of dispersion of the reinforcements and failures in interfacial adhesion in the composites.
- The morphological analysis showed a ductile fracture in tensile of composites reinforced with eucalyptus fibres, and a brittle fracture with the addition of granite dust particles, evidencing the interaction between the matrix and the waste in the hybrid composite.
- The green hybrid composites showed promising mechanical behaviour, enabling applications in structural engineering, such as in civil and transportation industries.

Acknowledgments

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