Original Article

Development of unsaturated polyester/fiber glass composites with foundry sand

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Abstract

In this study, the effect of the use of foundry sand as a filler on the formation of different layer configurations in polyester/fiber glass composites was investigated. The composites were produced by hand lay-up lamination and their physical, mechanical and morphological properties were investigated. In the samples containing sand as the filler, the configuration of the layers affected the interface region of the phases, thus significantly affecting the mechanical properties of the samples. The morphological analysis of the composites revealed the presence of regions with moderate interaction between the phases (polyester / glass fiber and polyester / foundry sand), which can cause delamination of the layers and consequently the deterioration of the mechanical properties of the composites when compared to the composite without the sand.

Keywords: Foundry sand; Unsaturated polyester; Multilayer composite; Glass fiber.

1 Introduction

One of the sectors of the world economy that has been growing at an accelerated rate is the composites sector, and composites have gained immense attention for a wide range of applications such as in pipelines for transporting gas and oil and its derivative [1,2].

Glass fiber reinforced polymer (GFRP) composites much used in civil engineering applications due to present superior properties as conventional materials, such as thermomechanical properties, chemical resistance [2,3], high strength-to-weight ratio, lightness and corrosion resistance [4-6]. Depending on the type of resin used (polyester, epoxy, or others), pipes can work in different temperature ranges and in the most varied aggressive environments [7].

Polyester resins are frequently used in GFRPs and present chemical resistance, low cost and easy handling, in addition to combining high mechanical resistance to weight ratio [8] and low water absorption [9]. In addition, the combination of different types of fibers and loads allows the construction of pipes with a wide range of mechanical and structural properties [10]. The layering configuration and production method of these composites also have a significant effect on the properties of the product [6,7].

However, one of the main disadvantages of the tubes made up of composite materials is that they are expensive as compared to those made up of conventional materials such as metals and concrete. Thus, to meet the design requirements, such as rigidity and structural characteristics (internal diameter and thickness), the use of only polyester resin and fiberglass could make the product very expensive [1]. This problem can be solved by inserting fillers in the material.

Composite material tubes are typically produced with different layers and materials. The inner part of such tubes/pipes, especially those used for underground applications, is known as the core layer, which can be filled with a sand layer impregnated with resin. In composites with sandwich-type structures, also known as multilayer composites, thicker walls (thickness) are necessary to increase their apparent rigidity. The apparent stiffness of a tube represents its resistance to the deflection due to transverse loading. In this sense, the use of fillers is an efficient and inexpensive strategy to prepare composite pipes [11].

Melo et al. [1] reported the use of quartz sand as a filler in composites and evaluated their performance by experimental and numerical studies. Sand, which is commonly used in the production of composite pipes, is a naturally occurring granular material composed of finely divided rocks and mineral particles. The composition of sand strongly depends on the sources and local rocky conditions, but the

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most common constituent of sand is silica (SiO_2). It usually exists in sand in the form of quartz, which is the most widely used weather resistant and non-toxic mineral owing to its chemical inertness and considerable hardness [12].

The use of residues is a promising approach to control the composition of composites. Beycioğlu et al. [13] recently reported the use of fly ash as a filler in fiberglass-reinforced polyester composite tubes. The authors observed that fly ash could be an interesting substitute for sand as a filler in composites and that the evaluated formulations met the regulatory standards.

As an alternative to conventional sand, a waste that has always been the subject of many scientific investigations for reuse is the sand discarded from the smelting process [14,15]. In order to reduce the negative impacts of the generation of foundry sand, its adequate management by using it for the development of new engineering materials is imperative [15]. The chemically bonded sand, used in the metal smelting process, consists of 93–99% silica (SiO_2) [14,16] and approximately 1–3% of the chemical binder [14]. In this sense, the objective of this work is to evaluate the properties of glass fiber-reinforced polyester composites with different layers/contents of foundry sand as the filler.

2 Materials and methods

2.1 Materials

Orthophthalic unsaturated polyester resin with 30% styrene was purchased from Redelease Produtos Para Industrias Ltda. The MEKP catalyst based on methyl-ethyl-ketone (Butanox-M50) was used in the proportion of 1 wt.%, as indicated by the supplier. Glass fibers with a weight of 200 g/m^2 were used as the bidirectional fabric and fiberglass with a weight of 300 g/m^2 was used as the multidirectional non-woven blanket. The glass fibers were supplied by Texiglass Indústria e Comércio Têxtil. According to the supplier, the glass fibers used are compatible with the unsaturated polyester resin. The foundry sand residue used in this study was supplied by WEG Motor, located in the city of Joinville (Brazil), in the powder form and was used without any further treatment. The sand comes from the production of cast iron foundry and contains resins and organic binders. The particulate classification of the foundry sand was carried out by sieving it with Tyler series sieves. Figure 1 presents the optical microscopy (OM) of fiberglass bidirectional fabric and fiberglass multidirectional non-woven blanket used in this work.

3 Methods

3.1 Sample preparation

The preparation of the composites was carried out by manual lamination (hand lay-up) according to the configurations and mass fractions shown in Figure 2. A silicone mold with the dimensions of 200 mm × 150 mm was used for the production of the samples.

The thickness of the composites varied depending to the number of layers used for the lamination.

After their layer lamination, the composites were placed in a thermal oven at 70 °C for 30 min and were subsequently maintained in an environment with a controlled temperature of 23 °C for 72 h.

3.2 Characterization

The densities of the samples were measured according to ASTM D792-00 [17] standard. The samples were weighed on an analytical balance, dipped in ethanol, and weighed again. Each test value was calculated as the average of at least seven independent measurements. Density calculations were performed according to Equation 1:

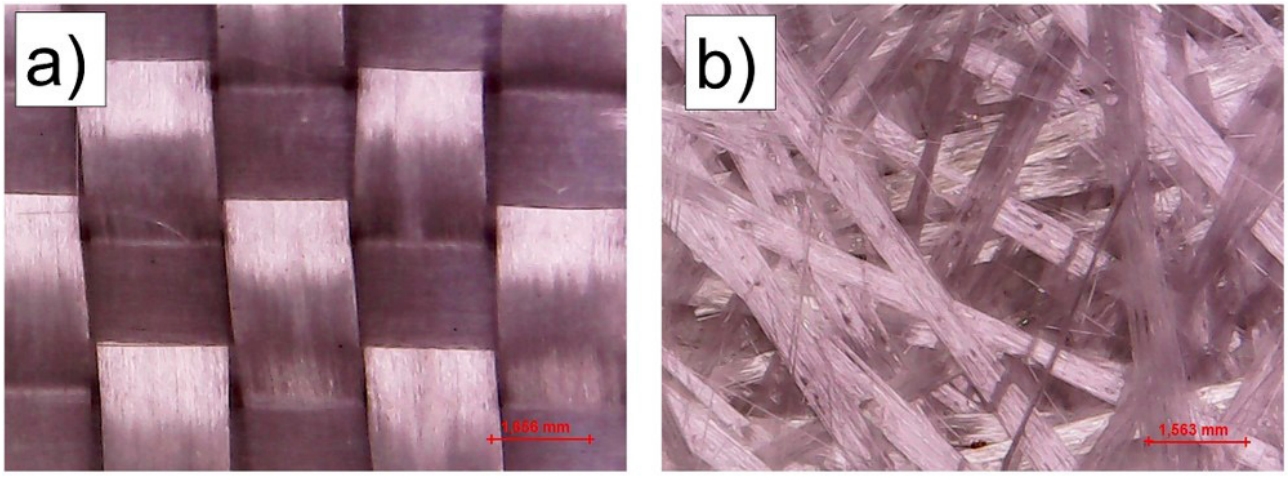

Figure 1. OM images of the surfaces of the (a) bidirectional fiberglass fabric and (b) multidirectional fiberglass blanket.

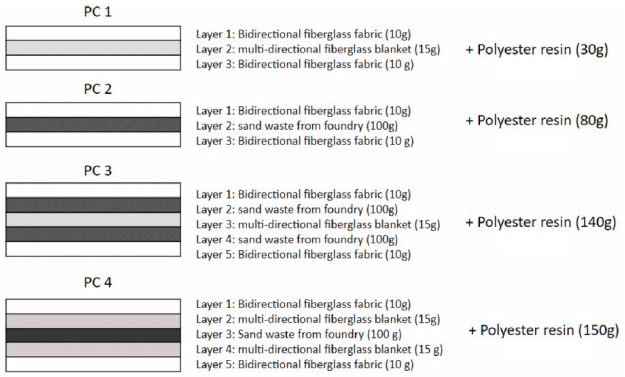

Figure 2. Identification and composition of the layers used for preparing the composites.

$$\rho = \frac{(a\,x\,b)}{(a-c)} \tag{1}$$

Where ρ is the density (g/cm^3) of the sample, *a* is the mass of the sample (g), *b* is the density of ethanol (g/cm^3), and *c* is the mass of the sample immersed in ethanol.

The void contents of the samples were determined according to ASTM D2734-16 [18] standard.

The void content can be obtained by the relative difference between theoretical and measured composite density. The theoretical densities of the composites were calculated using Equation 2:

$$\rho_T = (M.\rho_m) + (G.\rho_g) + (F.\rho_f) \tag{2}$$

Where ρ_T is the theoretical density of the composite (g/cm^3); M = mass percentage of the polymer matrix in the composite; ρ_m is the density of the polyester matrix (g/cm^3), which was equal to 1.17 g/cm^3; G is the mass percentage of the fiberglass in the composite; ρ_g is the density of the fiberglass (g/cm^3) = 2.55 g/cm^3; F is the mass percentage of the foundry sand residue in the composite and ρ_f = density of discarded foundry sand (g/cm^3). The void contents of the composites were determined from the difference between their experimental and theoretical densities, according to Equation 3:

$$V = \left(\frac{\rho_T - \rho_E}{\rho_T}\right) x100 \tag{3}$$

Where V is the void content, ρ_T is the theoretical density of the composite (g/cm^3) and, and ρ_E is the experimental density of the composite (g/cm^3).

The water absorption test of the composites was carried out according to ASTM D570-98 [19] standard. The composites were first dried in an oven for 4 h at 80 ºC. Water absorption tests were conducted by immersing the samples in a deionized water bath at 23 °C for different time durations (1, 2, 5, 24, 48 and 72 h). After immersion of the test period, the specimens were taken out from the water and all surface water was removed with a clean dry cloth and re-weighed. Five replicate measurements were made for each sample. The water absorption of the composites was assessed using Equation 4.

$$WA(\%) = \frac{m_f - m_i}{m_i} x100 \tag{4}$$

Where *WA* is the percentage of water absorption, *mi* is the mass of the specimen before immersion (g), and *mf* is the mass of the specimen after the immersion (g).

The tensile strength tests of the samples were carried out according to ASTM D3039/D3039M–17 [20] using an Emic universal testing machine (model DL10000), with 2 mm.min^{-1} crosshead speed. From this test, the stress of the samples was determined and the tensile force per width (*Fr*) is presented in Equation 5.

$$F_r = \frac{Q}{b} \tag{5}$$

Where *Fr* = tensile force (kN/m), *Q* = load applied to the specimen at the moment of rupture (kN), and *b* is the specimen width (m).

The flexural strength test of the composites was carried out on an Emic universal testing machine, model DL10000 (load cell: 200 kg$_f$) at a test speed of 1.8 mm/min in accordance with ASTM D7264M-07 [21].

The morphological characterization of the composites was carried out using scanning electron microscopy (SEM) (Carl Zeiss LS-10) at an accelerating voltage of 10 kV. All the samples were covered with a thin layer of gold. The composite samples were ruptured during their tensile test. The optical microscopy (OM) analysis of the composites was carried out on an Olympus BX41M-LED microscope.

4 Results and discussion

Figure 3 shows the SEM image of the foundry sand used in this study and Figure 4 its granulometric analysis. The particles were heterogeneously distributed in terms of the size and shape. The foundry sand particles had a diameter of 100–400 µm and had an irregular granular shape (some were rounded, while the others were sub-angular). It can be observed from Figure 3b, indicated by arrows, that the sand particles have a rough micro texture, which may be due to the presence of binder residues used in the casting activity, such as phenolic resin. It was also observed that the sand particles had a rough microtexture.

Table 1 lists the densities, void contents, resin content in the composites and thicknesses of the laminated composites prepared in this study. PC3 presented the highest density of 1.68 g/cm^3 among all the samples investigated in this study, as it has two layers of foundry sand. The PC2 and PC4, with only one layer of foundry sand, showed similar densities of 1.59 and 1.58 g/cm^3, respectively. The increase in the

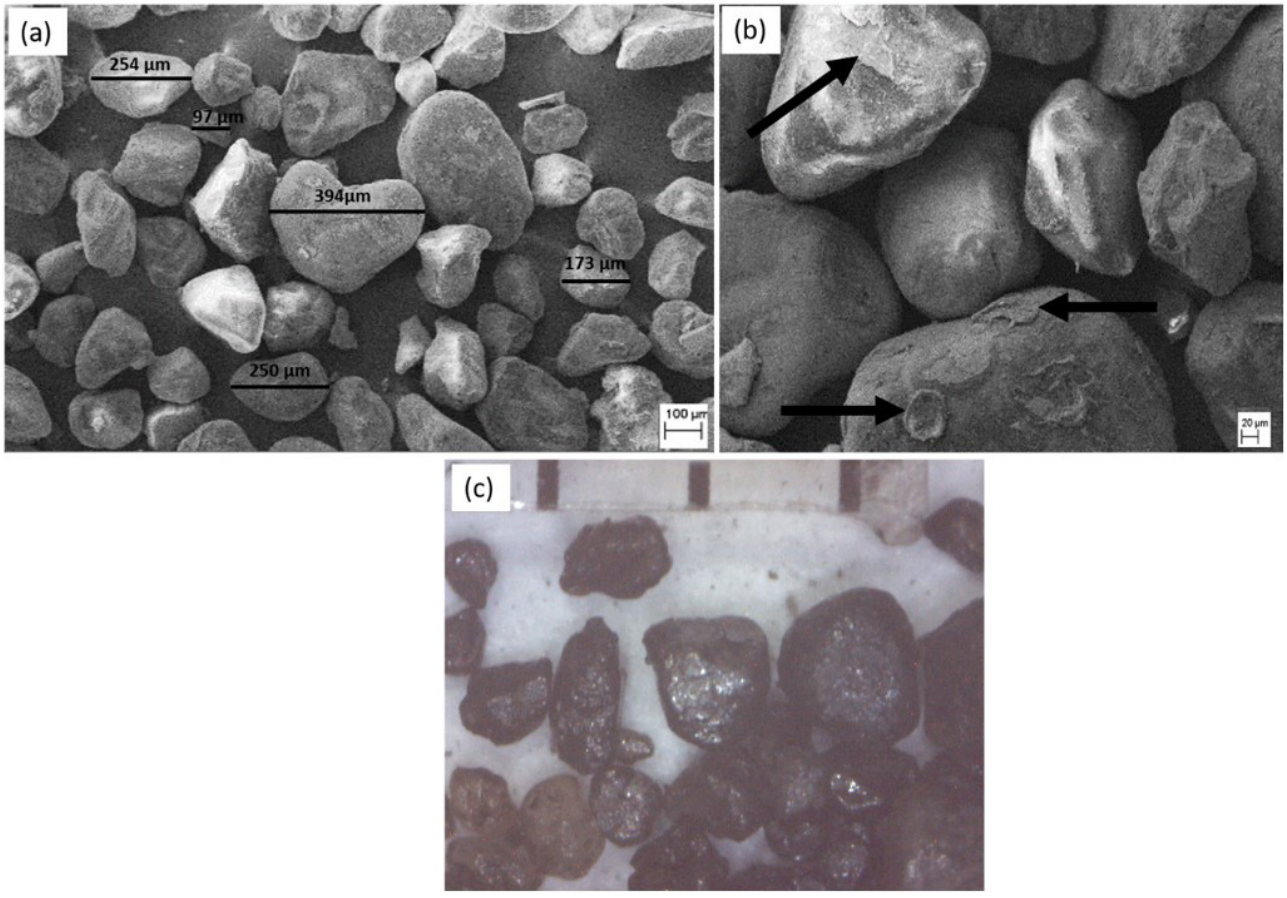

Figure 3. Micrographs obtained by the (a), (b) SEM and (c) OM of the sand discarded from the foundry process.

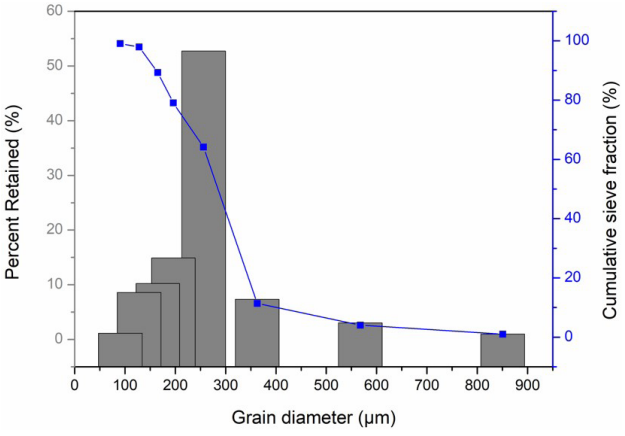

Figure 4. Granulometric analysis of foundry sand.

density of the composites was proportional to the increase in the foundry sand content, since the density of the foundry sand was higher than that of the pure orthophthalic polyester resin. Due to the fact that the density of the foundry sand used in this study was 2.65 g/cm^3, which is higher than that of the polyester/fiberglass composites.

The real and theoretical densities of the samples were used to determine their void contents. It was found

Table 1. Physical properties of foundry sand and GFRP with and without the addition of foundry sand

Sample	Density (g.cm^{-3})	Void fraction (%)	Resin content in the composite (%)	Laminate thickness (mm)
Sand	2.65 ± 0.01	--	--	--
PC 1	1.50 ± 0.14	21.6	46	1.1 ± 0.1
PC 2	1.58 ± 0.10	22.8	40	2.1 ± 0.2
PC 3	1.68 ± 0.17	19.5	37	4.8 ± 0.2
PC 4	1.59 ± 0.13	16.0	50	4.0 ± 0.1

that the void content of the composites decreased with an increase in their thickness. PC3 and PC4 showed low void contents of 19.5 and 16.0%, respectively. PC4 showed lower void content than PC3 because of its larger polyester resin content, and hence greater adhesion among the sand particulates. Nayak and Satapathy [22] when evaluating the incorporation of marble powder in unsaturated isophthalic polyester resin observed similar behavior.

The PC1 sample without the foundry sand showed a relatively large void content of 21.6%, which indicates that the process commonly used, via hand lay-up, is susceptible

to constructive errors due to the presence of voids in the composite. As expected and observed, during the composite lamination, the greatest formation of voids occurs in the layer with the multidirectional fiberglass blanket due to the resin's difficulty in filling the voids in the blanket. PC2 composite with a layer of sand between the two layers of fiberglass in the form of a bidirectional fabric, showed the highest void content of 22.8% among all the samples. Voids can be formed during the manufacturing process for several reasons as the production method involving manual lamination is inaccurate and prone to failure, high viscosity of the used resin, humidity, different types of fibers and fillers and chemical incompatibility between the components [23].

Volatile components or even contamination can form voids by vaporization during the curing cycle of the composite [24-26]. Consequently, these voids can crack nucleating elements in the composite microstructure, and care must be taken both in terms of their quantity (indicated by the void volume) and shape. Spherical voids are almost always present inside the layers, while elongated voids are located at the interface between the composite layers, which can cause intralaminary defects [27-30]. The presence of voids also significantly deteriorates the mechanical and physical properties of composites [31]. The most common cause of voids is the inability of the matrix to displace all the air that is carried by the fibers as it passes through the impregnation of the matrix [32].

As seen in Table 1, there was a significant variation in the polymer content and in the thickness of the composites produced. PC1 and PC2 have respectively 46 and 40% of resin. PC3 has 37% resin in its composition and PC4 has 50%. The thickness of the composites depended on the number of layers used in the lamination process. The thickness of the PC3 and PC4 samples, which consisted of more sand and glass fiber layers, was two times larger than that of the other samples. Depending on the application, for example a tube, the thickness of the composite is of great importance, since there are several standards that predict relationships between the diameter and thickness of the tube.

Figure 5 shows the water absorption results of the composites. Water absorption can deteriorate the properties of a composite, and hence is a very important characteristic determining its final application [24,25]. In composites for packaging, civil construction, and waste water treatment, water absorption is a very important factor, because it can alter the physical and mechanical properties of these materials, affecting the structure of the matrix and fiber-interface [32].

The samples were monitored for 72 h. It was found that after 5 h of immersion, the samples showed a significant increase in water absorption. After 72 h, the PC3 composite showed the highest water absorption content (8.6%), followed by the PC1 sample (6.3%). PC4 samples showed lower water absorption contents with 4.4% after 72 h. Water absorption is strongly associated with the resin content in the composites composition. It is observed that the higher the resin content, the lower the water absorption in the samples. The water absorption of composite materials depends on many factors, such as the temperature, fiber volume fraction, void content, reinforcement orientation, fiber nature (permeable or impermeable), geometry of the exposed surfaces, and diffusivity and surface protection [33]. The main mechanism of moisture penetration in composites is diffusion [34]. This mechanism involves the direct diffusion of water into the matrix and, to a lesser extent, into the fibers. The other common mechanisms are capillarity and transport by microcracks [35]. According to Georgiopoulos et al. [36] the strong intermolecular resistance of the fiber/matrix connection decreases the water absorption rate of the material. For better adhesion between the fibers and matrix, the water absorption rate must be reduced because there are fewer gaps in the interfacial region [37-39]. The addition of the foundry sand in the composites facilitated the penetration of water into them. This water was then deposited at the polymer/filler interface. Therefore, an increase in the load content of the composites increased the number of voids filled with water. Thus, the PC3 composite with two foundry sand layers interspersed between three layers of the polyester fiber showed higher void content than PC4, which consisted of only one layer of the foundry sand.

Table 2 lists the tensile strengths of the composites with and without the foundry sand layers. The tensile strength of the composites decreased with the addition of the foundry sand.

According to Wong et al. [10] the incorporation of sand into composites decreases their flow stress, ductility, and

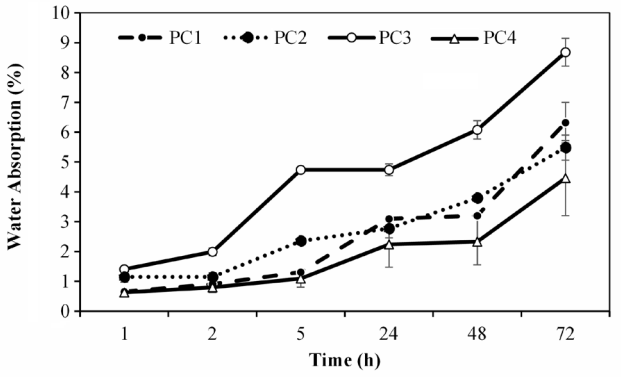

Figure 5. Water absorption of the composites with and without foundry sand.

Table 2. Mechanical properties of tensile strength of GFRPs with and without the addition of foundry sand

Sample	Strain (%)	Tensile strength (MPa)	Tensile modulus (MPa)	Fr (kN/m)
PC 1	10.1 ± 0.2	287.0 ± 9.7	6673 ± 120	319 ± 25
PC 2	6.0 ± 1.0	68.1 ± 1.1	3329 ± 380	135 ± 20
PC 3	5.7 ± 1.2	37.1 ± 5.1	2877 ± 133	163 ± 42
PC 4	7.5 ± 0.5	77.6 ± 12.8	3208 ± 341	295 ± 32

the absorbed energy for fracture resistance. The efficiency of a reinforcement material depends on its particle size. The larger the reinforcement particle size, the lower is its efficiency. As the size of the foundry sand particles used from diameter of 100-400 μm, that is, they were relatively large, a low filler reinforcement efficiency was expected. The presence of multiple phases, such as layers of fiberglass and foundry sand, as shown in Figure 2, also contribute to the deterioration of properties, as the interphase region creates weak points in the composite.

In polymeric systems, the introduction of particulate materials generally does not lead to a substantial improvement in the properties of the polymer as compared to the introduction of fibrous materials. This is because in the case of particulate reinforcement materials, stress is not effectively transferred from the matrix to the reinforcement particles because of their small surface area. Furthermore, these particles can also act as nucleating agents for the formation of cracks, which reduce the mechanical strength of the composites.

By comparing the mechanical properties of the PC2 and PC3 with one and two foundry sand layers, respectively, it can be observed that an increase in the sand content resulted in decreased strain capacity of the composites and the deterioration of their mechanical properties. Elongation appears to have a strong correlation with the amount of polymer used in the manufacture of composites. The PC3 sample, which had the lowest resin content, had the lowest deformation capacity. The decrease of the mechanical properties of the composites with the sand, in comparison with the sample PC1, can also be attributed to the poor adhesion between the fiberglass fabric and sand layers, which resulted in debarking and delamination. This phenomenon is shown in Figure 6, which shows the SEM images of the fracture regions of the composites after the tensile strength test.

From Figure 6, it is possible to observe that the main fracture mechanism is phase delamination, more pronounced in the PC2 and PC3, as indicated by the arrows. In the PC4 sample, no significant phase delamination was observed.

The PC4 sample showed improved mechanical properties as compared to the PC3 because of the higher polymer content and good contact between the sand layer and the fiberglass blanket, which was rougher than the bidirectional fiberglass fabric and allowed better anchoring of the sand particles in the central layer. This effect was also shown by the tensile force (Fr) of the composites. As observed in Figure 1, that shows the OM images of the surfaces of both the glass fibers used in this study. The blanket with multidirectional glass fibers shows rougher texture than the bidirectional glass fiber fabric. This greater roughness improves the physical adhesion of the phases, favoring the mechanical response of the PC4 sample in relation to the other samples reinforced with foundry sand.

The tensile test of a specimen expresses its mechanical response as a function of its area. Moreover, the thickness of a specimen is directly related to its stress response. For the preparation of tubes, this thickness can be adapted according to the design requirements. Thus, the analysis of the load applied over the width of the specimen, without considering its thickness provides promising results with the insertion of foundry sand in composites. The PC1 (without sand particulates) and PC4 samples showed similar Fr, while the PC2 and PC3 samples showed lower Fr than the PC1 and PC3 samples. This result corroborates the hypothesis that the blanket layer with multidirectional fibers was more effective for interacting with the sand layer than the bidirectional fiberglass blanket.

As the resin content of the PC4 was higher than those of the PC2 and PC3 samples, it can be stated that the properties of the composites were also affected by their resin to filler mass ratios.

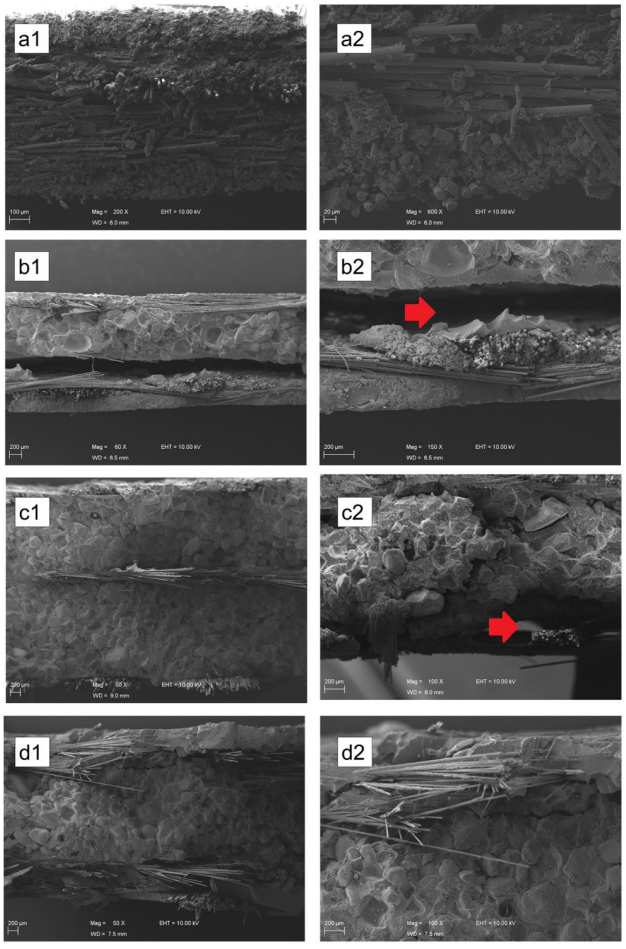

Figure 6. SEM micrographs, in the fracture region, after the tensile strength test of the sample (a) PC1; (b) PC2; (c) PC3 and (d) PC4.

Table 3. Mechanical properties of flexural strength of GFRPs with and without the addition of foundry sand

Sample	Maximum force (N)	Flexural strength (MPa)	Flexural modulus (GPa)
PC 1	8.75 ± 0.9	335.8 ± 21.2	9.38 ± 0.1
PC 2	23.5 ± 4.8	270.4 ± 81.4	6.56 ± 0.2
PC 3	80.1 ± 4.8	181.7 ± 20.1	5.06 ± 0.1
PC 4	100.0 ± 6.2	234.9 ± 10.1	6.94 ± 0.1

Table 3 lists the flexural strengths of the composites with and without the foundry sand. Similar to the tensile strength, the stress of the composites decreased with the addition of the foundry sand. However, the maximum strength of the composites increased with the insertion of the foundry sand. For example, in PC3 with a resin consumption 4.6 times higher than PC1, the maximum force required for breaking was greater than 9.1 times. As compared to PC2, PC3 showed a significant increase in the strength because of its large thickness and the configuration with two sand layers, which contributed to its high stiffness. The PC4 sample showed better maximum strength response than the PC3 sample despite its lower thickness owing to the better compatibility of its constituent phases and higher polymer content.

5 Conclusions

The results obtained in this study demonstrated the feasibility of incorporating foundry sand residue into multilayer composites. Although the incorporation of sand resulted in the deterioration of some mechanical properties of the composites, the manipulation of the configuration and mass fractions of the resin, fiberglass, and sand layers, allowed the production of composites suitable for piping applications. In addition, the incorporation of the sand increased the stiffness and thickness of the composites. The incorporation of one layer of foundry sand did not significantly influence the water absorption, when compared to the PC1 sample, however with the addition of two layers of foundry sand (PC3), a greater water absorption was observed. The polymer content also had a great influence on the water absorption properties of the composites, and the higher the polymer content, the lower the water absorption. The PC4 composite showed the best performance because of the good contact between the sand layer and the multidirectional fiberglass blanket, which was rougher than the bidirectional fiberglass fabric (used in PC2 and PC3) and allowed better anchoring and adhesion of the sand layer.

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