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TENSILE STRENGTH OF POLYESTER MATRIX COMPOSITES WITH INCORPORATION OF KAOLIN WASTE FROM THE AMAZON REGION

Alessandro José Gomes dos Santos
Federal University of Pará - UFPA, Department of Industrial Engineering, Abaetetuba, Brazil

Mauricio Maia Ribeiro
Federal Institute of Education, Science and Technology of Pará - IFPA, Ananindeua, Brazil

Jean da Silva Rodrigues
Federal Institute of Education, Science and Technology of Pará - IFPA, Materials Engineering Program, Belém, Brazil

Douglas Santos Silva
Military Institute of Engineering - IME, Materials Science Program, Rio de Janeiro, Brazil

Raí Felipe Pereira Junio
Military Institute of Engineering - IME, Materials Science Program, Rio de Janeiro, Brazil

Lucas de Mendonça Neuba
Military Institute of Engineering - IME, Materials Science Program, Rio de Janeiro, Brazil

Sergio Neves Monteiro
Military Institute of Engineering - IME, Materials Science Program, Rio de Janeiro, Brazil

Abstract: In recent years, growing interest in sustainable materials reflects the quest to reduce environmental impact. The strategy of reducing the proportion of polyester resin in composites stands out for its potential in saving resources and reducing carbon emissions, especially when using waste that would previously have been discarded. In this context, this study aims to evaluate how the incorporation of kaolin waste influences the tensile strength of polyester matrix composites. Five formulations were prepared, ranging from 0 to 40 wt.% kaolin waste, with a particle size of 50-100 mesh. The density of the kaolin waste and the composites were calculated. The composites were manufactured by the compression molding method, using a stainless-steel mold with dimensions of 300 x 160 x 2.5 mm. Through tensile tests, it was possible to identify that kaolin waste composites exhibit higher tensile strength, exceeding by 24.19%, 30.94%, 47.38% and 16.10% the tensile strength of the neat polyester for addition of kaolin waste 10, 20, 30 and 40 wt.%, respectively. The mechanical properties obtained were treated by analysis of variance (ANOVA) and Tukey test. Furthermore, a fractographic analysis was carried out using scanning electron microscopy (SEM) to analyze in detail the fracture surfaces of the tested samples and understand the failure mechanisms of each composite material.
Keywords: Polyester, composites, kaolin waste, tensile strength.

INTRODUCTION

In recent years, there has been a growing interest in the search for materials that not only meet design requirements, but are also sustainable [1]. Among the advances in this field, the promising strategy of combining polymers with waste has stood out, allowing the optimization of the use of materials. This occurs due to the reduction in demand for polymers, since there is a partial replacement of these by waste, as well as the use of materials that would have been discarded in the past [2-4].

The unsaturated polyester (UP), belonging to the third-largest class of thermoset molding resins, is a liquid polymer made of ethylene glycol and terephthalic acid [5]. This synthetic polymer having an ester functional group in its main chain, gathers interest from its low cost and easy processing [6]. These resins are widely used because of their excellent mechanical properties (such as strength and stiffness), versatility (in terms of processing), and resistance to chemicals. However, the mechanical properties of the polyester-polymer matrix are insuficient for many engineering applications [7].

Kaolin is a type of aluminosilicate mineral having a creamy white colour, primarily composed of the mineral kaolinite, which is a crystalline mineral composed of hydrated aluminium silicate and formed by the hydrothermal decomposition of granite rocks over millions of years. Kaolinite is a versatile material with a range of properties that make it useful in many applications. These properties include its non-swelling nature, low cation-exchange capacity, size, dimensional aspects, chemical structure, and element composition. Kaolin's non-swelling nature makes it a good choice for use in coatings and other materials that need to be dimensionally stable. Its low cation-exchange capacity means that it does not interact with other chemicals, which makes it a good choice for use in food-grade applications. Kaolin's size and dimensional aspects make it a good filler for coatings and other materials [8].

Brazil and the U.S. have some of the largest reserves of kaolin in the planet. In Brazil, the state of Pará (Amazon region) stands out for having one of the most important international kaolin deposits for the paper industry [9]. During the processing steps for purifying raw kaolin, waste is produced and stored in settling ponds which occupy large area. This situation presents a problem due to the impact on the environment [10].

The process of processing kaolin intended for filling and covering paper generates two types of waste: a compound of coarse particles (mainly quartz in the form of sand), which are replenished at the mining site itself, and another which is stored in voluminous sedimentation lagoons [10]. Research with this waste demonstrates great viability for it to be used as a raw material in various industrial segments [10,11].

Polymer composites with reinforced fillers have gained popularity because the presence of fillers changes the structure, mechanical qualities and physical characteristics of this polymer composite. Kaolinite, the primary component of kaolin, is a white, soft, and non-plastic clay that acts as an important filler in this polymer field. It produces a smooth surface finish, reduces shrinkage and cracking during curing, improves impact strength, and enhances resistance to the effects of weathering and chemical action for polymers [12].

According to research done by Al-asade et al. [13], adding kaolin to an UP resin at a 3–9% (which is considered a relatively low percent) improves the mechanical properties of the UP resin. This demonstrates that kaolin is a sufficiently good binder, and its application as a particle reinforcing material resulted in an improvement in the mechanical characteristics of UP at relatively low kaolin percentages [13-16].

In composite studies, bentonite nanoparticles with maleic anhydride added to polymers at levels of up to 5% mass provided an increase in tensile strength and modulus of elasticity of up to 35%, as demonstrated by Venkatesan et al. [17] and Rao and Sankar [18].

Furthermore, the theory of circular economy is developed from the industrial ecosystem, which aims to gather the thoughts from different scientific grounds that represent the collective characteristics of reduce, reuse and recycle, concept of cleaner production, environmental efficiency, green manufacturing approach, capitalization and the idea of minimum discharge of effluents [19–22]. The concept of circular economy came into existence to enhance the existing manufacturing and utilization model of "takemake-dispose", which is intimidating the sustainability of society [23]. Now it is required to reuse various industrial wastes resources in construction sector by developing valuable sustainable products [24–26].

In view of this, the present work evaluates the tensile strength of polyester matrix composites with incorporation of kaolin waste from the amazon region, contributing to the search for new materials that are more sustainable alternatives, with economic viability and appropriate technology.

MATERIALS AND METHODS

MATERIALS

Polyester Resin

The polymer matrix used was the medium viscosity unsaturated terephthalate-based polyester resin (Arazyn AZ 1.0 #34) and the methyl-ethyl-ketone peroxide (MEK) catalyst, PERMEC D-45, supplied by Ara Química SA (São Paulo, Brazil). The polyester resin was mixed with 1.0 wt.% hardener.

Kaolin waste

The kaolin waste used in the study was provided by Imerys-Caulim Ltda. (Barcarena, Pará, Brazil).

EXPERIMENTAL PROCEDURES

Processing of kaolin waste composites

The composites were manufactured by the compression molding method, using a stainless-steel mold with dimensions of 300 x 160 x 2.5 mm. The kaolin waste was dried in an oven at 100°C for 24 hours. After drying, the kaolin was sieved to obtain a particle size of 50-100 mesh, kaolin used in present work, as indicated in Figure 1.

Then, the kaolin powder was mixed with polyester resin by mechanical stirring, remaining under low-speed agitation for 10 minutes, during which the homogeneity of the mixture was observed. The kaolin was added to the resin in four different proportions: 10, 20, 30 and 40 wt.%. After homogenization, 1.0 wt.% of MEK catalyst was added to the mixture. Next, the mixture was poured into the steel mold, the gel time (material in the curing process) was determined in the range of 15 to 20 minutes. Immediately afterwards, the metallic mold was closed and pressed in a hydraulic press (model MPH-15, MARCON brand, Brazil) with a load of 2.5 kN for 20 minutes. The plates were removed from the metal molds and left at room temperature for the total curing process, which lasted 24 hours. After the total curing process, the composite plates were cut on a circular bench saw, to produce the test specimens in accordance with the standard test. Table 1 shows the configurations produced and tested.

Density determination of kaolin waste

The determination of the density of kaolin waste followed the guidelines of NBR NM 52/2009 [27], where the method used was pycnometric using distilled water and three samples of approximately 1.0 g of particulates.

Density determination of composites

Density were carried out in eight specimens with 25x25 mm for each condition were tested, totaling 40 specimens, at 23 °C according to the Archimedean principle with dimensions shown schematically in Figure 2.

The cured composite was then weighed in air and then again weighed in a liquid with a known density. The calculated density from measured values was reported in g/cm³. The physical property of density is calculated from Equation (1).

$$D = \frac{(M_s \times \rho_L)}{(M_U - M_I)} (g/cm^3) \quad (1)$$

Where, $\mathbf{M_U}$ is the wet mass (g), $\mathbf{M_S}$ is the dry mass (g), $\mathbf{M_I}$ is the immersed mass (g) and ρ_L is the specific mass of water (g/cm³).

Tensile tests

The tensile tests were carried out in eight specimens for each condition were tested, totaling 40 specimens, at 23 °C according to the ASTM D3039 [28] standard with dimensions shown schematically in Figure 3.

The tests were conducted using na KRATOS model IKCL3 universal testing machine with a 5 kN load cell. The crosshead speed was set as 2 mm/min for all tensile tests.

Statistical analysis

Analysis of variance (ANOVA) was used, through the F test, to verify if there were significant differences between the means of the mechanical properties. To compare the means of treatment, Tukey's Significant Difference test was applied (posttest), if necessary. The significance level adopted was (α) of 5%, with the null hypothesis (H_0) being equivalence between means; in which for P-value smaller than α, reject if H_0. Statistical analysis was conducted entirely using the R CORE TEAM Environment [29], using the RSTUDIO TEAM [30] integrated development environment, and supported by additional packages.

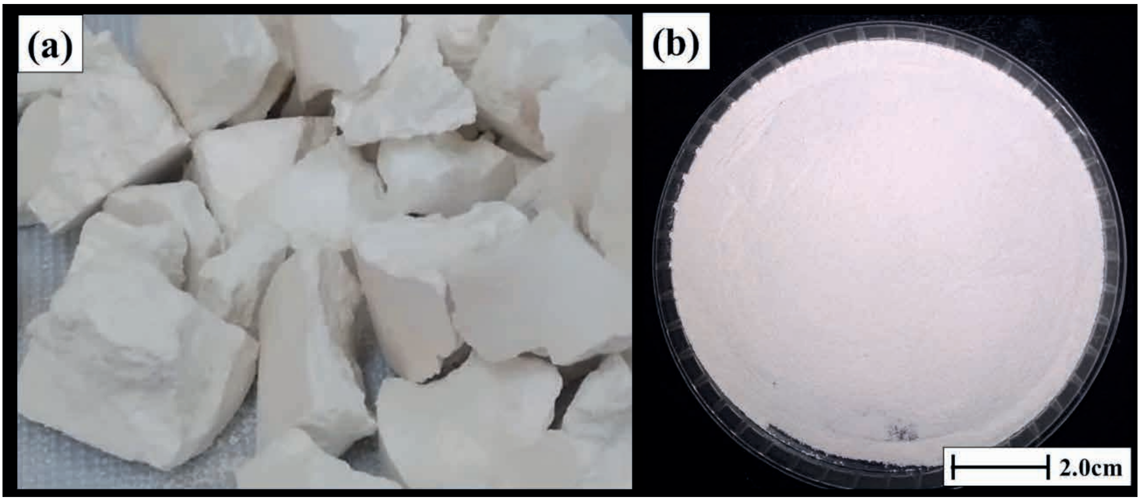

Figure 1: (a) Kaolin waste as obtained, (b) kaolin waste particle size of 50-100 mesh.

Configuration code	Samples description
NP	Neat Polyester
PKW10	Polyester/Kaolin waste – 10%
PKW20	Polyester/Kaolin waste – 20%
PKW30	Polyester/Kaolin waste – 30%
PKW40	Polyester/Kaolin waste – 40%

Table 1: Specimens configuration.

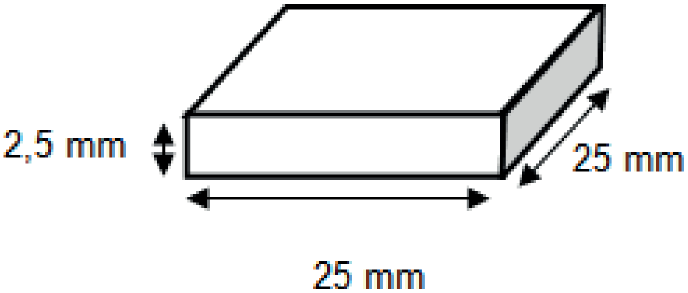

Figure 2: Dimensions of specimens for density tests.

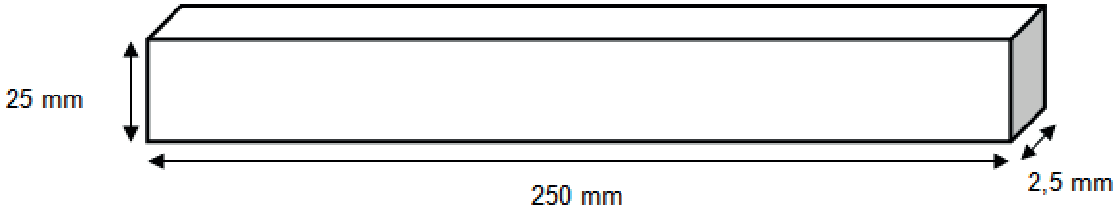

Figure 3: Dimensions of specimens for tensile tests.

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Scanning Electron Microscopy (SEM)

Morphological analysis of the surface of the kaolin waste was performed by scanning electron microscopy (SEM) using a Quanta FEG 250 Fei model microscope equipped with na Everhart-Thornley secondary electron detector, operating at a 5 kV acclimation voltage.

The fracture surfaces of the composites were investigated using a Hitachi scanning electron microscope, model TM3000. The electron beam voltage was set to 20 kV. The samples were coated with a thin gold layer using a cathodic sputter model ACE600 (Leica, Germany) for 30 min.

RESULTS AND DISCUSSION

MORPHOLOGY OF KAOLIN WASTE

SEM micrographs of the kaolin waste are presented in Figure 4.

Figure 4 is presented that the material, is primarily composed of many clustered particles of pseudo-hexagonal morphology, characteristic of kaolinite, which can be called poorly selected "coarse particles" because they are industrial waste [31,32].

Density test results

The results of the density of composites are presented in Table 2.

It is important to note that the kaolin waste has a specific mass value of 1.96 g/cm³, which is slightly higher in relation to the polyester matrix of 1.26 g/cm³ and with the insertion of the waste in the matrix, there is a behavior of gradual growth of the density of the composites.

Tensile test results

The results of tensile strength, Young's modulus and total strain of composites are presented in Table 3.

Results of Table 3 show that kaolin waste composites exhibit higher tensile strength, exceeding by 24.19%, 30.94%, 47.38% and 16.10% the tensile strength of the neat polyester for addition of kaolin waste 10, 20, 30 and 40 wt.%, respectively. For the total strain in all cases, the neat polyester matrix shows higher results, where for 10, 20, 30 and 40 wt.% of kaolin waste, the matrix was superior by 11.39%, 11.71%, 10.76% and 21.52%, respectively. Consequently, under this condition, the Young's modulus for the 10, 20, 30 and 40 wt.% kaolin waste composites exhibit the highest values, exceeding by 24.35%, 24.89%, 27.81% and 24.57% the stiffness of the neat polyester.

Abd et al. [6] reported that the incorporation of kaolin into an UP composite led to an increase in the material's termal properties, a decrease in its heat conductivity, a reduction in its glass transition temperature, degree of crystallinity, and melting point. Henceforth enhancing the manufacturing process and reducing the cost of the formulation process. Cobalt actuate and Methylene ethyl ketone peroxide (MEKP) were used to condense an UP matrix that was then filled with varying amounts of kaolin (5%, 10%, 15% and 20%).

Additionally, compared to other kaolin loadings, it has been reported that polyester with a 20 % gm of kaolin content exhibits better mechanical and thermal properties. This is due to the physical interaction between molecules chain by Vander walls bonds between matrix molecules and filler as indicative in UV spectrum absorption. The work was done with the intention of improving the thermal, electrical and mechanical properties of the polyester composite as well as providing smooth surfaces, dimensional stability, resistance to chemical attack, reduced shrinkage and cracking characteristics.

The mixture of polymer matrix (UP resin), hardener (Methyl Ethyl Ketone peroxide), accelerator (Cobalt naphthenate), and kaolin as reinforcement material was

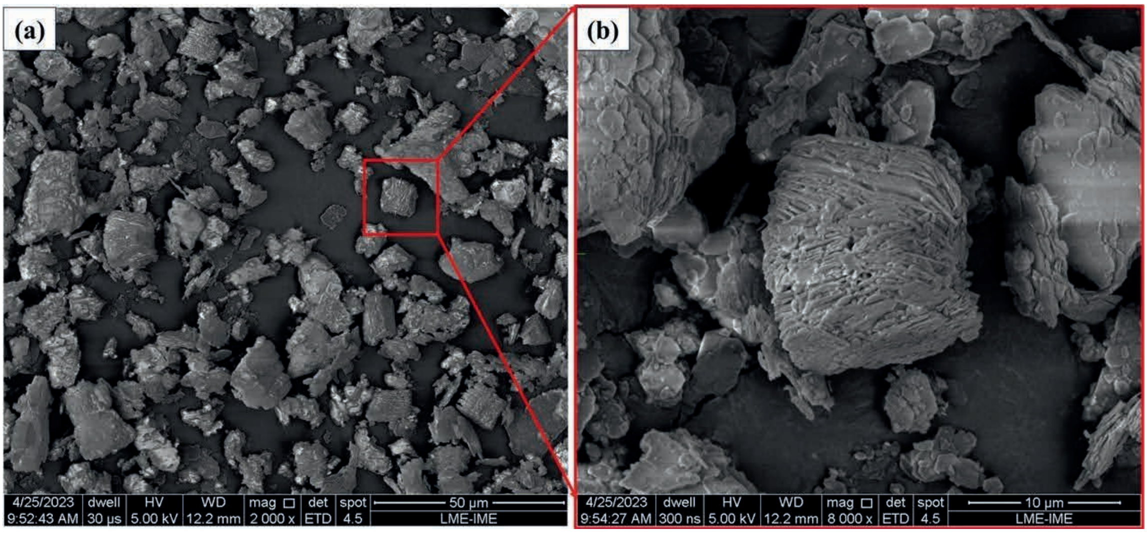

Figure 4: SEM micrographs of the kaolin waste. (a) 2000x and (b) 8000x.

Nomenclature	Density (g/cm³)
NP	1.26 (± 0.08)
PKW10	1.44 (± 0.04)
PKW20	1.65 (± 0.06)
PKW30	1.87 (± 0.03)
PKW40	2.09 (± 0.09)

Table 2: Results of the density test of polyester composites incorporated with kaolin waste.

Nomenclature	Tensile Strength (MPa)	Total Strain (mm/mm)	Young's Modulus (GPa)
NP	23.11 (± 4.04)	0.0316 (± 0.0037)	0.924 (± 0.096)
PKW10	28.70 (± 5.78)	0.0280 (± 0.0030)	1.149 (± 0.126)
PKW20	30.26 (± 6.54)	0.0279 (± 0.0087)	1.154 (± 0.091)
PKW30	34.06 (± 3.09)	0.0282 (± 0.0016)	1.181 (± 0.043)
PKW40	26.83 (± 2.49)	0.0248 (± 0.0021)	1.151 (± 0.012)

Tabel 3: Results of the tensile test of polyester composites incorporated with kaolin waste.

used for compression tests, flexural tests, pendulum Charpy test and Brinell hardness test with suitable testing techniques to draw out conclusion. It was observed that when there was a low percentage of kaolin, the compression strength increased due to a high degree of adhesion between the polymer molecules and the kaolin particles, which indicates that kaolin acts as a binding material [33]. On the other hand, when there was a high percentage of kaolin, the compression strength decreased due to the kaolin particles extending the distances between the polymer molecules, which means that they broke the intermolecular forces that were holding these molecules together [34].

However, unlike compression strength, the flexural modulus and flexural strength were found to be increasing with the increase in kaolin percent, due to high degree of adhesion between the kaolin and polymer while in high percent of kaolin the properties decreased due to lowering of the cross-link density of the polymer. It was also noticed that the particles worked as stress concentrators when there was a high percentage of kaolin present [34].

As per the results of Brinell hardness test, it was reported that the Brinell Hardness decreased with low percent of kaolin. But then an inversion in the behavior was observed at high percent, confirming the fact that at low percent, kaolin acts as binder material while in high percent it reduces the cross-linking density for UP. Also, it was found that the impact strength dropped with the increase in kaolin content and this is due to the kaolin particles acting as a crack initiator, but at high contente the particles act as crack stoppers so that the impact strength increases with high percent of kaolin.

It is well-known that the filler type and amount used in defining a polyester-filler composite greatly affect the material's mechanical properties [34]. It was reported that the compressive strength values increase as the concentration of untreated kaolin increases. This trend continued until a maximum was reached at a styrenated polyester/kaolin ratio of 60:40 wt%. After reaching this point, a slight decrease in the compressive strength value was also observed. In this case of kaolin filler, the degree of adhesion between the kaolin and polymer may be the cause of the observed direct link between mechanical characteristics and kaolin percentage. With that it is obvious that, when it comes to achieving a product with high mechanical strength, the kind of initiator and the concentration of the accelerator that are used in the cross-linking reaction tend to play a vital role [35].

Dalila Laouchedi et al. [36] investigated the impact of locally produced clay (Algeria), its rate and size on mechanical and physical properties of the composite material. The study comprised of two parts with the first part studying kaolin-epoxy resin based composite materials, using size fractions varying from 2% to 20%; while the second one examining the influence of composite with metakaolin (calcined kaolin) to study its particle size, structure and charge rate on the properties of material. The properties of clay fillers modified epoxy resin were observed to be different from that of neat epoxy resin, reducing the material's cost. Simultaneously, authors also stated that, it is necessary to use chemically treated clay fillers, in order to achieve better interaction and dispersion among the filler and resin. According to the author, fillers improved mechanical properties of the material by increasing its rigidity. At 18% kaolin content, the rigidity was maximum with a value of 2.4 GPa and the elasticity modulus increased by more than 325% as compared to unfilled resin. It was also noted that elasticity modulus increased proportionally with loading rate whereas, rigidity was proportional to filler rate. Moreover, for both the employed fillers, it was observed that lower fraction yields better results.

Statistical analysis

Statistical analysis of mechanical properties is presented in Table 4.

In Table 4, for maximum strength, one can see that the $F_{calculated}$ (3.81) is higher than the $F_{critical}$ value (2.87). In the same way, for Young's modulus, the $F_{calculated}$ (7.86) is higher than the $F_{critical}$ value (2.87). Thus, the hypothesis that the averages of the properties presented are equals, with a confidence level of 95%, is rejected. Thus, the hypothesis that the averages of the properties presented are equals, with a confidence level of 95%, is rejected. Because of the ANOVA results, the Tukey test was necessary in order to investigate if an increase in the amount of kaolin was more effective in causing significant changes in the mechanical properties in this composite.

Table 5 shows the results of Tukey test. For total strain, the $F_{calculated}$ (1.34) is lower than the $F_{critical}$ value (2.87), because of the ANOVA result, the Tukey test wasn't necessary.

The minimum significant difference (m.s.d) is a value that can discriminate which treatment shows difference in its average values. Once the difference between the average values of groups, compared two by two, is higher than the m.s.d value, this pair is considered to be different. The m.s.d for maximum strength was calculated as 8.79 and m.s.d for Young's modulus was calculated as 0.159. Thus, it might be seen that the inclusion of kaolin waste in polyester resin was essential to cause changes in mechanical properties. The PKW10, PKW20, PKW30 and PKW40 composites have indeed the highest tensile strength and Young's modulus, which effectively stiffened the material.

Microstructural analysis

The SEM micrographs of the fractured surfaces of the studied composites after the tensile test are presented in Figure 5.

Figure 5(a) shows the fracture surface of the composite incorporated with 10% kaolin waste (PKW10) and 5(b) shows the fracture surface of the composite incorporated with 40% kaolin waste (PKW40), where it can be seen in both images the presence of cracks in the matrix and sedimented points and some defects caused by the difficulty of homogenization, impregnation and compaction at the matrix/reinforcement interface, which has repercussions on the decrease in tensile strength of the composites incorporated with kaolin waste in the mass fraction of 40%, as shown in the values obtained.

CONCLUSIONS

Regarding physical properties, the density results showed an increasing trend as there was an increase in waste in the matrix. Kaolin waste composites exhibit higher tensile strength, exceeding by 24.19%, 30.94%, 47.38% and 16.10% the tensile strength of the neat polyester for addition of kaolin waste 10, 20, 30 and 40 wt.%, respectively. Statistical analysis of the data reinforced the validity of the results obtained and highlighted the viability of kaolin waste composites as a sustainable solution. SEM micrographs of the fracture surfaces shown sedimented points, caused by the difficulty of homogenization, impregnation and compaction at the matrix/reinforcement interface.

In general, the research carried out presented viable results, considering the incorporation of an industrial waste as mineral filler in composite materials, it is important to highlight that the reuse of kaolin waste contributes to reducing the environmental impacts generated in its storage process and

Maximum Strength (MPa)						
Source	**Sum of Squares**	**Degrees of Freedom**	**Mean of Squares**	**F (Calculated)**	**F Critical**	**P-*value***
Between the groups	329.65	4	82.41	3.81	2.87	0.018
Inside the group	432.43	20	21.62			
Total	762.08	24				
Total Strain (mm/mm)						
Source	**Sum of Squares**	**Degrees of Freedom**	**Mean of Squares**	**F (Calculated)**	**F Critical**	**P-*value***
Between the groups	0.0001	4	2.83×10^{-5}	1.34	2.87	0.291
Inside the group	0.0004	20	2.12×10^{-5}			
Total	0.0005	24				
Young's Modulus (GPa)						
Source	**Sum of Squares**	**Degrees of Freedom**	**Mean of Squares**	**F (Calculated)**	**F Critical**	**P-*value***
Between the groups	0.224	4	0.056	7.86	2.87	5.61×10^{-4}
Inside the group	0.142	20	0.007			
Total	0.366	24				

Table 4: Analysis of variance for polyester composites incorporated with kaolin waste.

Maximum Strength => m.s.d. = 8.79					
	NP	PKW10	PKW20	PKW30	PKW40
NP	0.00	5.59	7.15	**10.96**	3.72
PKW10	5.59	0.00	1.56	5.37	1.87
PKW20	7.15	1.56	0.00	3.81	3.43
PKW30	**10.96**	5.37	3.81	0.00	7.24
PKW40	3.72	1.87	3.43	7.24	0.00
Young's Modulus => m.s.d. = 0.159					
	NP	PKW10	PKW20	PKW30	PKW40
NP	0.000	**0.225**	**0.230**	**0.257**	**0.227**
PKW10	**0.225**	0.000	0.005	0.033	0.002
PKW20	**0.230**	0.005	0.000	0.028	0.003
PKW30	**0.257**	0.033	0.028	0.000	0.030
PKW40	**0.227**	0.002	0.003	0.030	0.000

Table 5: Results obtained for differences between the average values after applying the Tukey test.

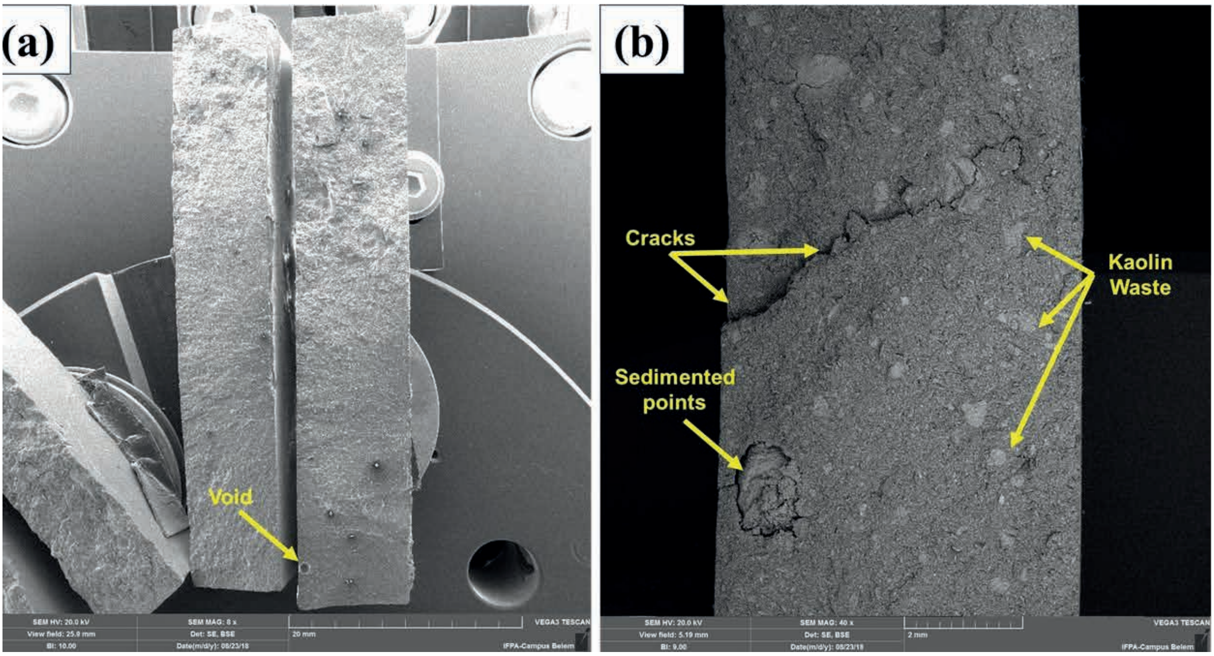

Figure 5: SEM Micrographs of the fracture surfaces of the composites after tensile testing: (a) sample PKW10 (8x); (b) sample PKW40 (40x).

also reduces the cost of the final product, considering that part of the polymer volume is being replaced by a material of lesser value.

Depending upon specific application and desired properties of the polymer composite, kaolin can be used both as a filler or a reinforcement material to improve its critical application-process problems.

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