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

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Abstract: The industrial process plays a vital role in the manufacturing of various products, but it also faces challenges such as that arising from waste generation. Transforming these byproducts into value-added resources is a growing goal to promote economic and environmental sustainability. In this context, this study aims to evaluate how the incorporation of red mud waste influences the tensile strength of polyester matrix composites. Five formulations were prepared, ranging from 0 to 40 wt.% red mud waste, with a particle size of 50-100 mesh. The density of the red mud 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 10, 20, 30 and 40 wt.% red mud waste/ polyester composites exhibit higher tensile strength, exceeding by 42.75%, 53.83%, 31.76% and 22.54% the tensile strength of the neat polyester, 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, red mud waste, tensile strength.

INTRODUCTION

In recent times, there is a growing interest in using industrial wastes as reinforcing additives/filler materials for polymers. These wastes like inorganic fillers can easily be integrated with thermoplastic or thermosets matrices to improve their thermal, mechanical and tribological characteristics. In addition, these waste filler materials have shown a significant improvement in thermal and electrical conductivity [1].

The unsaturated polyester (UP), belonging to the third-largest class of thermoset molding resins, is a liquid polymer resin made of ethylene glycol and terephthalic acid [2]. This synthetic polymer resin having an ester functional group in its main chain, gathers interest from its low cost and easy processing [3]. UP is widely used because of its excellent mechanical properties (such as strength and stiffness), versatility (in terms of processing), and resistance to chemicals. However, the mechanical properties of the plain polyester are insuficient for many engineering applications [4].

Red mud (RM) is a waste discarded during the aluminium production from bauxite [5–8]. It is mainly a multicomponent material and consists of both major and minor elements. RM is basically a mixture composed of a variety of minerals and has wide range of combinations of Fe_2O_3 (20–60%), Al_2O_3(10–30%), SiO_2 (2–20%), Na_2O (2–10%), CaO (2–8%), TiO_2 (2–10%) [9–14].

In Brazil, the bauxite ore refinement to produce alumina occurs mainly the state of Pará, in the Amazon region. Although alumina production is extremely important for the region from the social and economic points of view, it is associated with a high environmental impact since each ton of produced alumina approximately generates between 1 and 1.5 tons of bauxite residues [15,16].

RM waste is the major part of these residues, is generated in large amounts and is highly alkaline (pH >11). The inadequate storage of this residue can cause serious environmental damage if it leaches to groundwater or surface bodies of water [17,18]. In addition, the management of this residue represents an additional cost of approximately 4–12 US$ per ton of RM, as storage space that must be built for the proper management and maintenance

of this material [16,19]. Approximately 160 million tons of RM is generated annually worldwide, and the amount stored already exceeds 2.5 billion tons [20]. This large amount of RM has been considered as an obstacle for the production of alumina due to the lack of storage space and potential danger of environmental pollution [21].

Only a minor fraction of the generated RM is being utilized for the manufacture of construction materials such as glasses, cement, ceramics, concrete blocks, and bricks [22-25]. The RM is also used as a filling material in post mining activity, road construction material, ingredient in plastic manufacturing industry [26,27], and pollution control [28]. Several researchers have reported on the utilization of RM as hybrid composites [29], geopolymer materials [30], neutron shielding polyester composite [31], functionalized biochar [32] and persulfate oxidation [33].

In view of this, the present work evaluates the tensile strength of UP matrix composites with incorporation of different contents of RM waste (bauxite residue) 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 UP was mixed with 1.0 wt.% hardener.

RM waste

The RM waste used in the study was provided by Hydro Alunorte Ltda. (Barcarena, Pará, Brazil).

EXPERIMENTAL PROCEDURES

Processing of RM 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 RM waste was dried in an oven at 100°C for 24 hours. After drying, the RM was sieved to obtain a particle size of 50-100 mesh, as illustrated in Figure 1.

Then, the RM powder was mixed with UP resin by mechanical stirring, remaining under low-speed agitation for 10 min, during which the homogeneity of the mixture was observed. The RM was added into 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 min. 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 (RT) for the total curing process, which lasted 24 h. 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 RM waste

The determination of the density of RM followed the guidelines of NBR NM 52/2009 [34], 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

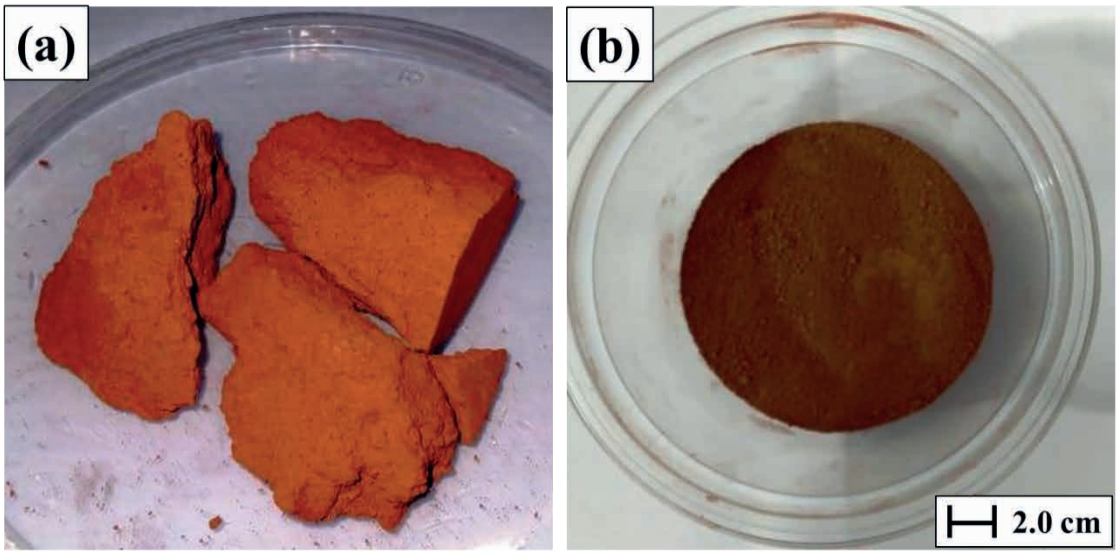

Figure 1: (a) RM waste as obtained, (b) RM waste particle size of 50-100 mesh.

Configuration code	Samples description
NP	Neat Polyester
PRMW10	Polyester/RM waste – 10 wt.%
PRMW20	Polyester/RM waste – 20 wt.%
PRMW30	Polyester/RM waste – 30 wt.%
PRMW40	Polyester/RM waste – 40 wt.%

Table 1: Specimens configuration.

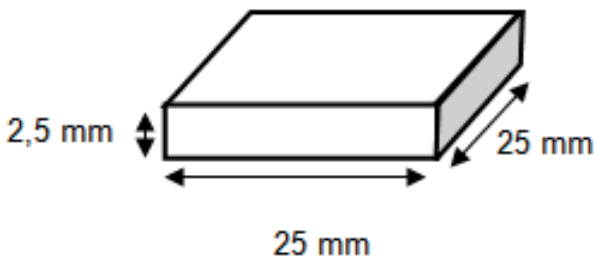

Figure 2: Dimensions of specimens for density tests.

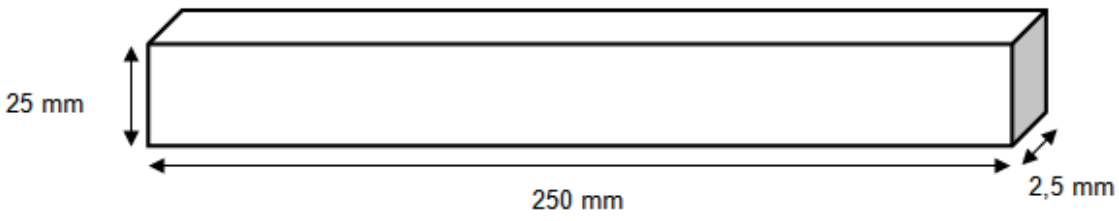

Figure 3: Dimensions of specimens for tensile tests.

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/cm^3. 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/cm^3).

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 [35] standard with dimensions shown schematically in Figure 3.

The tests were conducted at RT 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 [36], using the RSTUDIO TEAM [37] integrated development environment, and supported by additional packages.

The value of the minimum significant difference (m.s.d.) was found through Equation (2).

$$m.s.d. = q.\sqrt{\frac{QMR}{n}} \quad (2)$$

Where, **q** is the total student amplitude (tabulated value), which is a function of the degree of freedom of the residue and number of treatments, **QMR** is the mean square of residue within the group and **n** is the number of repetitions of each treatment within the group.

Scanning Electron Microscopy (SEM)

Morphological analysis of the surface of the RM 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 10 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 RM WASTE

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

The SEM micrographs indicates that RM is made of small particles ranging in size from a few to hundreds of microns. The varied forms of red dirt include spherical and flaky particles [38]. It is observed that there is a heterogeneous and irregular granulometric distribution, with the presence of fine particles that tend to agglomerate [39]. These agglomerated particulates tend to form relatively larger

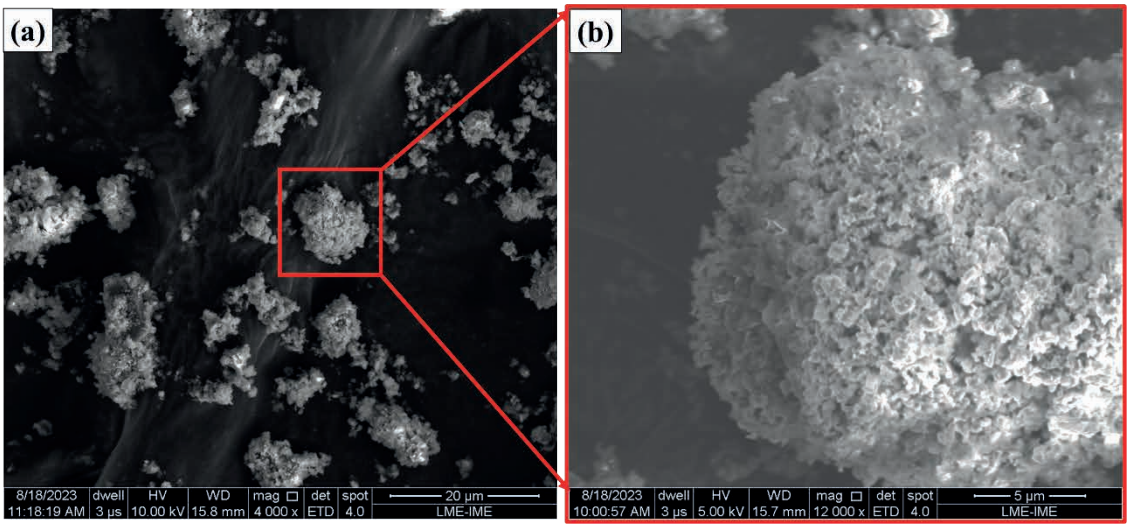

Figure 4: SEM micrographs of the RM waste. (a) 4000x and (b) 12000x.

Nomenclature	Density (g/cm^3)
NP	1.20 (± 0.09)
PRMW10	1.27 (± 0.07)
PRMW20	1.36 (± 0.09)
PRMW30	1.47 (± 0.08)
PRMW40	1.57 (± 0.08)

Table 2: Results of the density test of polyester composites incorporated with RM 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)
PRMW10	32.99 (± 5.05)	0.0280 (± 0.0026)	1.264 (± 0.111)
PRMW20	35.55 (± 2.49)	0.0331 (± 0.0050)	1.344 (± 0.092)
PRMW30	30.45 (± 3.91)	0.0312 (± 0.0020)	1.104 (± 0.209)
PRMW40	28.32 (± 2.94)	0.0273 (± 0.0027)	1.273 (± 0.208)

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

aggregates [40,41]. RM's porous character and large specific surface area lead to its adsorption capacity and interfacial adhesion behaviors.

DENSITY TEST RESULTS

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

It is important to note that the RM waste has a specific mass value of 2.11 g/cm³, which is higher in relation to the UP matrix of 1.20 g/cm³ and with the insertion of the RM 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 RM waste composites exhibit higher tensile strength, exceeding by 42.75%, 53.83%, 31.76% and 22.54% the tensile strength of the neat polyester for addition of 10, 20, 30 and 40 wt.% RM, respectively. For the total strain in all cases, the neat polyester matrix shows higher results, where for 10, 20, 30 and 40 wt.% RM, the matrix was superior by 11.39%, 4.75%, 1.27% and 13.61%, respectively. Consequently, under this condition, the Young's modulus for the 10, 20, 30 and 40 wt.% RM waste composites exhibit the highest values, exceeding by 36.80%, 45.45%, 19.48% and 37.77% the stiffness of the neat polyester.

Rodrigues et al. [42] evaluated the tensile mechanical properties of polyester matrix composites loaded with an industrial residue of RM, with a mass fraction of 20%. The results obtained in the tensile test of the polyester/RM composites were calculated by the extensometer and the Digital Image Correlation (DIC) technique. The samples showed a tensile strength limit of 14.973 MPa, the average value obtained for the modulus of elasticity was 0.6621 GPa and for the failure strain 0.0393 mm/mm, calculated from the results of the strain gauge. By means of DIC, it was obtained for the modulus, 0.6766 GPa and for the strain 0.0387 mm/mm, presenting errors in the DIC versus Extensometer ratio, of +2.20% and -1.61%, respectively.

Prabu et al. [43] filled and modified polyester and epoxy resins with RM and studied their mechanical properties. They found that the tensile strength and impact strength of RM–polyester resin/epoxy resin composites showed downward trends, but the hardness value of the composites increased.

Akinci et al. [44] prepared isotactic polypropylene polymer matrix composites using RM as the filler. The mechanical property analysis showed that with increasing RM content, the tensile strength and flexural strength exhibited a linear downwards trend. However, the hardness of the composites increased with increasing RM content.

Zhang et al. [45] prepared RM/polypropylene (PP) composites by using twin-screw extruders and other equipment. Analysis of their mechanical properties showed that when the RM content was 15%, the tensile strength of the composite reached its maximum value. However, with increasing RM content, the impact property of the composites decreased. Thus, RM/PP composites exhibited better mechanical properties at relatively low RM contents.

In the recent studies it has been observed that RM serves as a good filler material in polymer composite. As it is stable in nature due to absence of toxic elements, it hardly affects the tensile strength, physical–mechanical properties and hardness of these composite. The compatibility of polyester resin is also good enough with RM particles. Further it has been found that using RM minimizes the wear rate of composites as well. Instead of normal particles, spherodized particles of RM are used for filling in polymer composites

[46].

On comparison with banana fiber composites, the impact and flexure strengths of RM filled sisal polyester composites are greater. Alongside sisal and banana fiber, the addition of RM enhances flexural strength. RM filled polymer composites are ideal for higher resistance and load carrying capacity requirements due to elevated flexural strength [47].

STATISTICAL ANALYSIS

Statistical analysis of mechanical properties is presented in Table 4.

In Table 4, for maximum strength, one can see that the $F_{calculated}$ (7.83) is higher than the $F_{critical}$ value (2.87). In the same way, for Young's modulus, the $F_{calculated}$ (6.04) 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 RM 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 $F_{calculated}$ (2.70) 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 7.18 and m.s.d for Young's modulus was calculated as 0.290. Thus, it might be seen that the inclusion of RM in polyester resin was essential to cause changes in mechanical properties. The PRMW10, PRMW20, PRMW30 and PRMW40 composites have indeed the highest tensile strength and Young's modulus, which effectively stiffened the material. Similar behavior was observed by Santos et al. [48] verifying the influence of the incorporation of kaolin waste on the tensile strength of polyester matrix composites.

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 wt.% RM waste (PRMW10) and it is clear that cracks are spreading. Figure 5(b) shows the fracture surface of the composite incorporated with 20 wt.% RM waste (PRMW20), there is also the presence of cracks and agglomerates. The direct addition of RM to the polyester matrix leads to a decline in some mechanical properties. This is because the high surface activity of RM causes it to agglomerate in the polyester matrix, resulting in uneven distribution and poor interfacial bonding properties, which ultimately leads to a decline in the mechanical properties.

The distribution of RM particles in resin composites determines the structure and properties of the composites [49]. Combined fillers formed by RM and other materials also create different synergistic effects in the resin matrix. The distribution of particles in the composite material has an important influence on the properties of the composite materials such as mechanical properties, thermal stability and wear properties [50].

CONCLUSIONS

Regarding physical properties, the density results showed an increasing trend as there was an increase in waste in the matrix. RM

Maximum Strength (MPa)						
Source	**Sum of Squares**	**Degrees of Freedom**	**Mean of Squares**	**F (Calculated)**	**F Critical**	**P-*value***
Between the groups	451.42	4	112.85	7.83	2.87	5.72×10^{-4}
Inside the group	288.15	20	14.41			
Total	739.57	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	$3{,}11 \times 10^{-5}$	2.70	2.87	6.02×10^{-2}
Inside the group	0.0002	20	$1{,}15 \times 10^{-5}$			
Total	0.0004	24				
Young's Modulus (GPa)						
Source	**Sum of Squares**	**Degrees of Freedom**	**Mean of Squares**	**F (Calculated)**	**F Critical**	**P-*value***
Between the groups	0.569	4	0.142	6.04	2.87	2.34×10^{-3}
Inside the group	0.470	20	0.024			
Total	1.039	24				

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

Maximum Strength => m.s.d. = 7.18					
	NP	PRMW10	PRMW20	PRMW30	PRMW40
NP	0.00	**9.89**	**12.44**	**7.35**	5.21
PRMW10	**9.89**	0.00	2.55	2.54	4.68
PRMW20	**12.44**	2.55	0.00	5.09	**7.23**
PRMW30	**7.35**	2.54	5.09	0.00	2.14
PRMW40	5.21	4.68	**7.23**	2.14	0.00
Young's Modulus => m.s.d. = 0.290					
	NP	PRMW10	PRMW20	PRMW30	PRMW40
NP	0.000	**0.340**	**0.420**	0.180	**0.349**
PRMW10	**0.340**	0.000	0.080	0.159	0.010
PRMW20	**0.420**	0.080	0.000	0.239	0.070
PRMW30	0.180	0.159	0.239	0.000	0.169
PRMW40	**0.349**	0.010	0.070	0.169	0.000

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

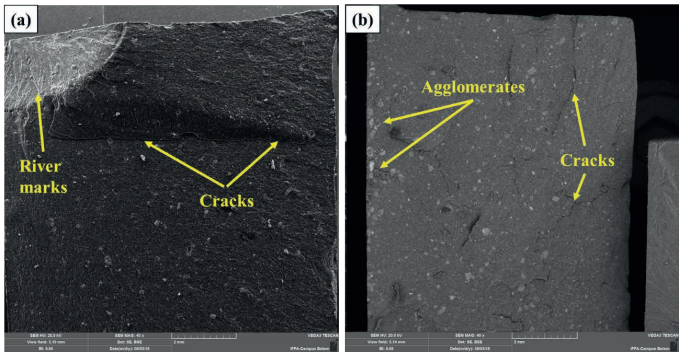

Figure 5: SEM Micrographs of the fracture surfaces of the composites after tensile testing: (a) sample PRMW10 (40x); (b) sample PRMW20 (40x).

waste composites exhibit higher tensile strength, exceeding by 42.75%, 53.83%, 31.76% and 22.54% the tensile strength of the neat polyester for addition of 10, 20, 30 and 40 wt.% RM, respectively. Statistical analysis of the data reinforced the validity of the results obtained and highlighted the viability of RM waste composites as a sustainable solution. SEM micrographs of the fracture surfaces revealed cracks and agglomerates.

In view of this, RM can be used as a conventional filler in resin composites to greatly reduce production costs. More important, the application of RM in plastic products can reduce waste residue and carbon dioxide emissions from the metallurgical industry, which not only protects the ecosystem but also promotes the sustainable development of the building, metallurgy and plastics industries.

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