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MICROSTRUCTURAL CHARACTERIZATION OF SOAPSTONE-REINFORCED POLYETHYLENE COMPOSITES AND CORRELATIONS WITH THE MACROSCOPIC MECHANICAL BEHAVIOR

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Abstract. *An experimental study was carried out to assess the use of soapstone residue as reinforcement for polymeric composites. Soapstone waste particles were incorporated into a high-density polyethylene (HDPE) matrix in three weight fractions: 10, 20 and 30 wt%. The aim of this study is to perform a microstructural characterization of these composites and to understand its relation to the macroscopic mechanical behavior observed in tensile tests. Automated image analysis was used to determine size and shape parameters of the filler. X-ray microtomography was used to quantify the filler volume fraction, void content, and the microstructural homogeneity of the composites. The analysis revealed that the soapstone particles have a strong tendency of forming clusters in the HDPE matrix and the amount of particle clustering increases with the amount of reinforcement. For higher filler loadings, the volume fraction of clusters in the sample was up to 6.03%. However, statistical analysis revealed that filler distribution was homogeneous throughout the length of the samples. The data from the tensile tests was compared to a macro-mechanical model from the literature. The results revealed that, despite the particle clustering, there was good interfacial adhesion, which resulted in higher strength of the composites in comparison to the polymer matrix.*

Keywords: *x-ray microtomography, automated image analysis, soapstone, polyethylene, particulate composites.*

1. INTRODUCTION

Nowadays, one of the main challenges in materials science is to counterbalance the need for environmental preservation with the global industry demands. Due to their non-biodegradable alkaline nature and their highly wasteful exploitation and production processes, stone waste is generally considered as a highly polluting waste, that can represent a health hazard and an environmental issue (Sahu *et al.*, 2020). Accordingly, there is a growing scientific interest in using such waste materials as alternative fillers for polymeric materials (Girge *et al.*, 2021). Fillers are often incorporated into polymers in order to reduce costs and/or improve required properties for specific applications. Recent studies reveal a growing tendency of substituting commercial standard fillers for industrial or mineral waste particulates, which can bring both economic and environmental advantages. For instance, by incorporating waste glass powder into a recycled high-density polyethylene matrix (RHDPE), Sadik *et al.* (2021) obtained a fully recyclable composite material with improved mechanical properties and high thermal stability, that can be used as cost-effective automobile dashboard material. With the addition of a maleic anhydride compatibilizer, the RHDPE/glass waste composites presented an increase of up to 29.26% in tensile strength and up to 126.8% in elastic modulus in comparison to the neat material (Sadik *et al.*, 2021). In another study, Sahu *et al.* (2020) showed that stone waste is a promising material for improving the properties of polymeric green composites. Stone waste powder was incorporated into an epoxy matrix, obtaining a moisture resistant, electrical insulating composite material with high mechanical strength that can be used for electrical insulating and advanced construction materials applications (Sahu *et al.*, 2020).

Soapstone is a talc-rich, metamorphic rock, used for ornamental and construction purposes, which exploration can produce a considerable amount of waste (Huhta and Kärki, 2018; Rodrigues and Lima, 2012). Worldwide, Brazil is one of the ten largest talc and soapstone producers, corresponding to approximately 10% of the global production (Bolen,

2019). Locally, the major production occurs in the Ouro Preto region of Minas Gerais state, where there are several soapstone quarries that exploit this rock as dimension stone. During the exploitation of the soapstone, only a small portion of the rock is destined to the ceramic industry, the majority (70%) is composed of small, low-quality blocks which are destined to artisan workshops (Rodrigues and Lima, 2012). The global recovery of soapstone exploited in quarries is only 40 wt%, so approximately 60 wt% of the rock is discarded as residue (Rodrigues and Lima, 2012). In addition, the artisan workshops produce a large quantity of soapstone powder (approximately 10%-15% rock recovery), which is disposed careless, often causing environmental problems (Rodrigues and Lima, 2012).

In previous works, the authors have proposed the use of soapstone waste powder as filler for high-density polyethylene (HDPE) composites (de Sousa *et al.*, 2019, 2021). Soapstone waste obtained with craftsman from Ouro Preto, Brazil was characterized and used as filler. Talc was the only mineral identified in the waste and the tensile test results revealed that the mechanical properties of HDPE were improved with the addition of up to 30 wt% of soapstone particles. In the literature, there is a large number of studies that report gain in mechanical strength and stiffness by incorporating commercial talc particles into polyolefins. In a study of talc-filled polypropylene (PP) composites, Lapcik Jr. *et al.* (2008) reported an enhancement of up to 25% in the polymer's tensile strength. Within the frame of pressure pipe applications, Mehrjerdi *et al.* (2013) reported that the addition of talc particles in a HDPE matrix resulted in higher impact resistance, thermal conductivity, thermal diffusivity, and specific density. However, only a marginal improvement was observed in tensile strength (approximately 7.5%). A subsequent study showed that the addition of a titanate coupling agent to the talc particles promoted a better filler-matrix adhesion, that lead to an increase in tensile strength of up to 18% and of up to 82% in stiffness, in comparison to the virgin polymer (Mehrjerdi *et al.*, 2014).

The macroscopic behavior of a particulate reinforced composite is intrinsically dependent of the microstructure formed during processing. In regard to mechanical behavior, two very relevant factors are the particle distribution and the interfacial adhesion between particles and matrix. Particle distribution is often not uniform in these type of composites, instead, the particles are clustered in some local regions, forming agglomerates throughout the material. Agglomeration of the reinforcement phase can have a negative effect on the practical behavior, in fact, particle clustering has been shown to have deleterious effects on tensile strength, flow stress, ductility and fatigue performance (Dastgerdia *et al.*, 2018).

One particularly powerful characterization method is X-ray microtomography. This non-destructive technique is based on computational reconstruction of the three-dimensional (3D) structure of the sample from a number of two dimensional X-ray images taken from different directions, which allows a qualitative assessment of spatial relationships (Dastgerdia *et al.*, 2018). Furthermore, perhaps the most compelling aspect of tomographic data is the digital nature of the 3D images, that makes possible, through image processing techniques, to extract a wide array of quantitative measurements of the internal structure, such as phase volume fractions and spatial distributions (Landis and Keane, 2010).

The aim of the present study is to perform a microstructural characterization of HDPE/soapstone waste composites and to understand its relation to the macroscopic mechanical behavior observed in tensile tests. Automated image analysis was used to determine size and shape parameters of the filler. X-ray microtomography was used to quantify the filler volume fraction, void content, and the microstructural homogeneity of the composites. The analysis of the mechanical behavior was done by fitting the microstructural parameters and tensile tests data into a macro-mechanical model from the literature, which allowed the quantification of the interfacial bonding.

2. THEORETICAL BACKGROUND

2.1 Microstructural parameters

In particulate polymer matrix composites (PMMCs), particles with random size distributions and shape irregularities, are incorporated into a polymer matrix. Due to processing conditions and the randomness of the fillers, it is difficult to obtain a uniform and homogeneous distribution of the particles along the composite (Dastgerdia *et al.*, 2018; Karimi and Milani, 2019). Additionally there may be a tendency for particle clustering, where individual reinforcement particles form clusters (agglomerates) whose mechanical properties differ from those of individual particles and matrix (Dastgerdia *et al.*, 2018).

The amount of clustering in the composite's microstructure can be expressed by dividing the total volume of particles v_p into two parts: $v_p = v_p^c + v_p^i$, where v_p^c is the volume of clustered particles and v_p^i is the volume of individual particles. Accordingly, the fraction of particles in clusters can be defined as:

$$\zeta = \frac{v_p^c}{v_p} \tag{1}$$

which is the volume ratio of the particles in the clusters over the total particles (Dastgerdia *et al.*, 2018). In addition, the amount of clustering can be described by the volume fraction of clusters as:

$$\gamma = \frac{v_p^c}{v} \tag{2}$$

where v is the total volume of the sample (Dastgerdia *et al.*, 2018).

The present work proposes an approach to analyze the variability of reinforcement distribution in the composite by dividing the sample into equal subsets and measuring the amount of filler in each subset. Considering the volume of all the particles in each subset as v_p^{sub}, two parameters can be defined: the fraction of particles in the subset, calculated as:

$$\xi_p^{sub} = \frac{v_p^{sub}}{v_p} \tag{3}$$

and the local volume fraction of particles, defined as:

$$\Phi_p^{sub} = \frac{n \, v_p^{sub}}{v} \tag{4}$$

where n is the number of subsets. Comparisons between the local volume fractions of particles (Φ_p^{sub}) and the total volume fraction of particles (Φ_p) can give insights to the uniformity of the filler distribution in the composite.

2.2 Macro-mechanical model

The strength of a composite material is intrinsically dependent on the load transfer between both phases, which it is dependent of filler-matrix adhesion. The degree of the interfacial bonding is usually divided into poor adhesion, good adhesion and some adhesion. In the case of poor adhesion, the interface will act as an inherent flaw in the microstructure and a lower bound response is observed (Ahmed and Jones, 1990). In the case of good filler-matrix adhesion, the filler has an reinforcing effect on the matrix properties, thus an upper bound response is observed (Liang and Li, 1998). In the literature there is a variety of equations that model either lower or upper bound responses (Ahmed and Jones, 1990). A more general approach, however, was proposed by Turcsányi *et al.* (1988), to model the yield strength of particulates composites, according to the following semi-empiric equation:

$$\sigma_{yc} = \frac{1 - \Phi_p}{1 + A\Phi_p} \, \sigma_{ym} \, exp(B\Phi_p) \tag{5}$$

where σ_{yc} and σ_{ym} are the yield strength of composite and matrix, respectively, and *A* is a shape factor, related to the spatial distribution of the filler. The generality of the equation is given by the parameter *B*, which is an adimensional parameter directly related to the particle-matrix adhesion, that can be used to provide a quantitative characterization of the interfacial adhesion. In the case that $B = 0$, there is no adhesion and, consequently, there is no load transfer in the interface. For values of $B \geq 3$ there is some matrix-filler adhesion and a reinforcement effect (Turcsányi *et al.*, 1988).

3. EXPERIMENTAL

3.1 Materials

High density polyethylene (HDPE) supplied by BRASKEM, Brazil, was used as matrix. The material was received as pellets with the specification IA59U3, which means that this HDPE has a narrow molecular weight distribution and is commercially used for injection molding. The HDPE used has a melt flow index (ASTM D1238 - for testing melt flow rates of thermoplastics by extrusion plastometer) of 0.73 g/min, density (ASTM D792 - for testing density and specific gravity of plastics by displacement) of 0.960 g/cm^3 and softening temperature (ASTM D1525 - for testing Vicat softening temperature of plastics) of 129 °C. The soapstone waste was obtained with craftsmen in Ouro Preto - Minas Gerais, Brazil. The waste consists of a orange fine powder and it was used without any kind of processing.

3.2 Preparation of composites

The composites were produced in a mini parallel double-screw extruder (DSM Xplore 5cc), in which the inlet, homogenization and exit temperatures were set at 180 °C , 190 °C and 200 °C, respectively. The rotation used for the mixture was 160 rpm. The homogenization time was about three minutes in the extruder, under constant flux of N$_2$. After the extrusion, the tensile specimens were injected using a mini injector (DSM Xplore 5,5cc), with the injection nozzle at 200 °C and the mold at 80 °C. The specimen fabricated were type V as per ASTM D638 (standard test method for measuring the tensile properties of plastics). Three different soapstone waste concentrations were used: 10%, 20% and 30% by weight, which are referred to as 90/10, 80/20 and 70/30 composites, respectively.

3.3 Automated Image Analysis

Shape and size parameters of the soapstone particles were determined by automated image analysis with a Malvern Morphology 4, which has an integrated dry powder dispersion unit and can determine particle sizes in the range from 0.5 to 1300 μm.

3.4 X-ray microtomography and image analysis

One specimen from each composite fraction was analyzed with a Zeiss micro CT (Xradia Versa 510), with 3.5 μm pixel size. The X-ray tube was used without filter, with acceleration voltage and power set to 80 kV and 7 W, respectively. 1601 projection images over 360°of rotation were acquired with a total of 4 s exposure time per angular position.

The image obtained in micro CT, was submitted to an image processing and analysis following the steps of: pre-processing (through the application of the Non Local Means filter, according to Buades *et al.* (2005)); segmentation of the particles by the threshold method; and extraction of attributes (volume and feret diameters). The digital image processing was performed with Dragonfly software. Typically, the spatial resolution refers to the minimum separation at which a feature pair can be resolved by an imaging system. Due to this fact, it is important to note that image resolution and particle distinction are limited to twice the pixel size (2 x 3.5 μm), according to the recommendation of Zeiss (2013).

4. RESULTS AND DISCUSSION

4.1 Particle analysis of the soapstone waste

Image analysis systems capture a two-dimensional projection of the particle profile and calculate various particle size and shape parameters (Ulusoy and Kursun, 2011). The particle size is determined in terms of circle equivalent (CE) diameter, which is the diameter of a circle with the same area as the projected area of the particle image. In the analysis of the soapstone waste powder, a total of 177862 dispersed particles were measured. Descriptive statistics of the CE diameter can give a better insight to the particle size distribution. Thus, the main statistic descriptors where summed up in Fig. 1, which shows the cumulative number distribution and histogram of the CE diameter. The mean diameter of the particles was 10.62 $\pm$ 8.00 μm. The percentiles D_n indicate that n% (by number) of particles have a size equal to or less than that corresponding value. Even though the dispersed particles were identified in the 0.20 μm - 158.17 μm range, 99% of the particles had diameters less than or equal to 39.11 μm, thus, values above this can be considered as outliers.
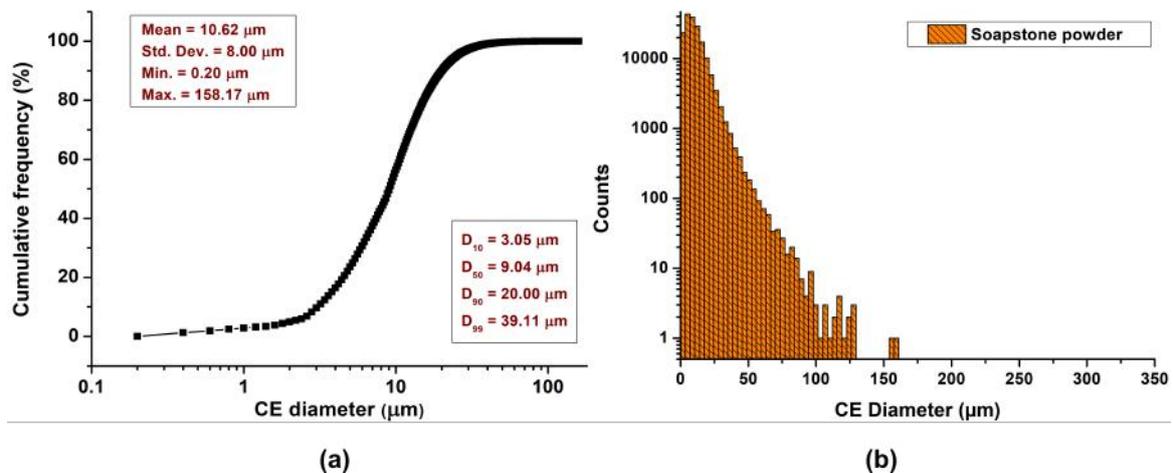

Figure 1. Particle analysis of the soapstone waste powder: (a) number distribution and (b) histogram of particle size.

The shape of the particles can be described by the circularity and feret ratio parameters. The first parameter is more sensible to irregularities in the object's outline, while the latter is very sensible to the object's elongation. The ferret ratio is calculated as width/length ratio and values lie in the range 0 - 1, with 1 being the ratio of a circle. The circularity is calculated as:

$$Circularity = \frac{2 \times \sqrt{\pi \times Area}}{Perimeter} \tag{6}$$

with 1 being the circularity of a perfect circle, while a very narrow object has a circularity close to 0. Figure 2 shows the histogram and descriptive statistics of both shape parameters of the soapstone waste particles. The median circularity of 0.90 indicates regularity in the object's outline, while the values of the feret ratio indicate that the particles are slightly elongated.

4.2 Microstructural characterization of the composites

Figures 3 and 4 show the 3D rendering of the three composite samples, where is possible to visualize the distribution of reinforcement throughout the samples. Another aspect that was noticeable in the 3D renderings was the void content. Within the resolution of the micro CT used, no voids were identified in the 90/10 and 80/20 samples, however, as can
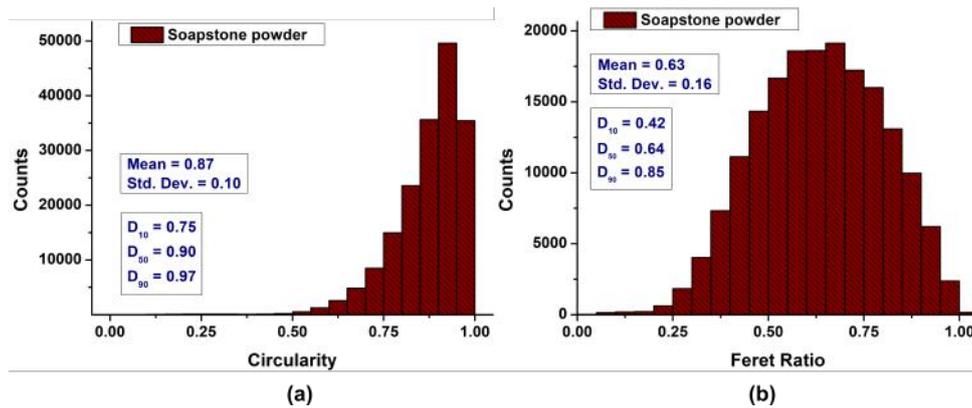
Figure 2. Particle analysis of the soapstone waste powder: histogram of (a) circularity and (b) feret ratio.

be seen in Figure 5, which shows the orthographic projections of the 70/30 sample, voids were formed in the central region of this sample during processing. The formation of voids can be explained by the increase in the polymer's melt viscosity caused by the soapstone particles, which was reported in a previous paper as being higher in the 70/30 ratio of the HDPE/soapstone composites (de Sousa *et al.*, 2021). Higher viscosity hinders the complete filling of the mold cavity and could cause this type of defect.
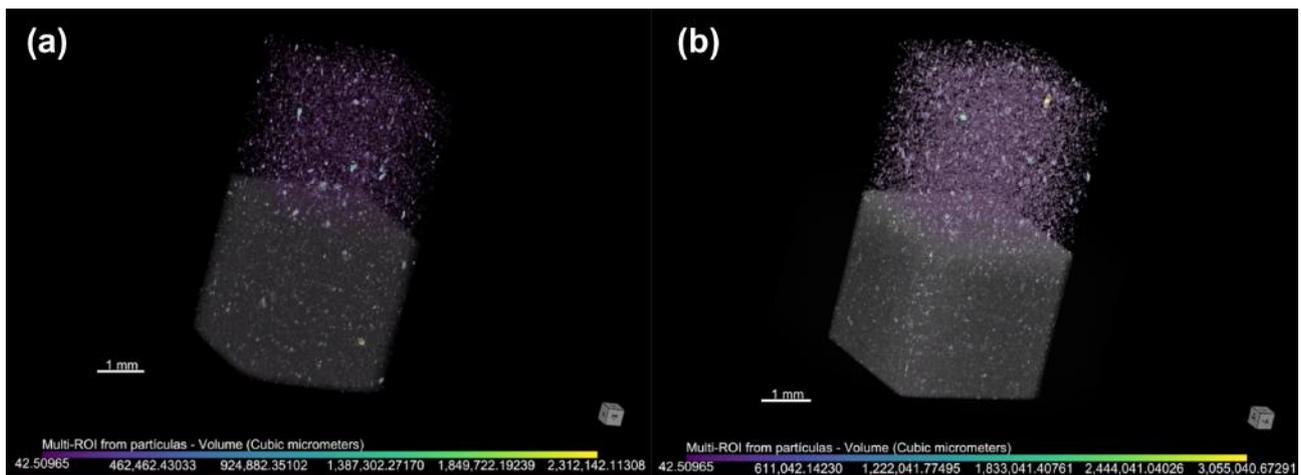

Figure 3. 3D rendering of (a) 90/10 and (b) 80/20 composite samples. Particles are colored according to the volume scale, the polymer is shown only on the bottom half in color gray.

Through image processing techniques it was possible to quantify the particles and voids in the different composite samples. The particle's size was measured in terms of feret diameters. The feret diameter can be defined as the distance between the two parallel planes restricting the object perpendicular to that direction. The minimum and maximum feret diameters are, respectively, the shortest and longest distance between any two points along the object boundary at an arbitrary angle. The mean feret diameter is defined as the mean value of the feret diameters over a sufficient number of orientations. In the Dragonfly software, measurements for the mean feret diameter are taken every 5 degrees. The calculated values for the minimum, maximum and mean feret diameters, as well as the volume of the particles in the different composite samples are listed in Tab. 1.

Table 1. Size measurements of soapstone particles identified in different HDPE composite samples. (Uncertainties represent the standard deviation).

Sample	Number of particles	Min Feret (μm)	Max Feret (μm)	Mean Feret (μm)	Volume (μm^3)
90/10	540696	13.13 ± 6.28	28.61 ± 17.39	22.78 ± 13.81	4.17E+03 $\pm$ 1.23E+04
80/20	721335	14.70 ± 8.64	31.63 ± 22.26	24.84 ± 17.39	5.75E+03 $\pm$ 1.83E+04
70/30	657450	15.43 ± 10.33	34.14 ± 27.25	26.94 ± 21.40	7.42E+03 $\pm$ 2.62E+04

The method used in this work does not have enough resolution to identify different particles within a cluster, however an approximation can be made by analyzing the size of each item identified as soapstone in the composite. Considering the previous analysis performed on the dispersed soapstone powder, reported in Section 4.1, in which 99% of the soapstone
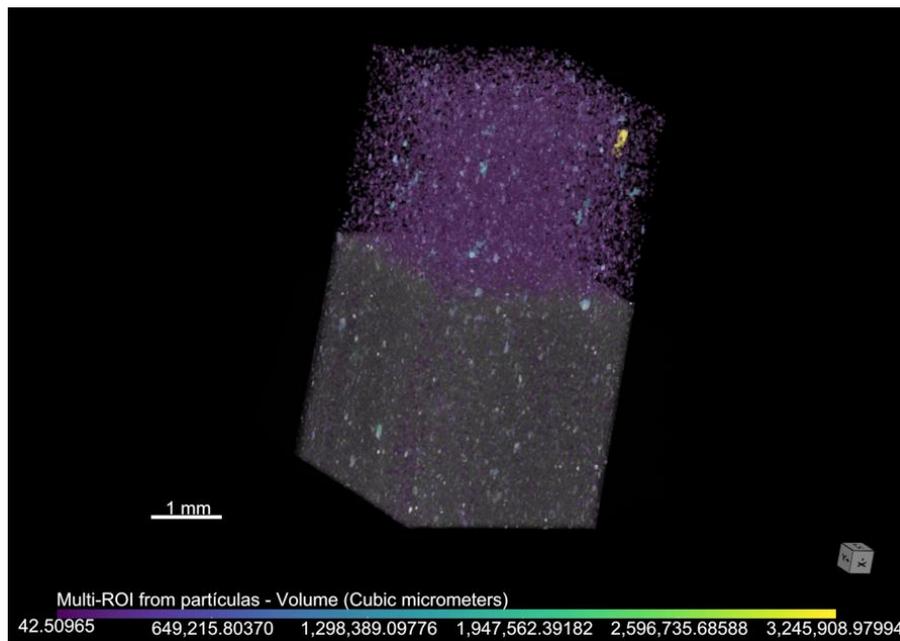
Figure 4. 3D rendering of the 70/30 composite sample. Particles are colored according to the volume scale, the polymer is shown only on the bottom half in color gray.
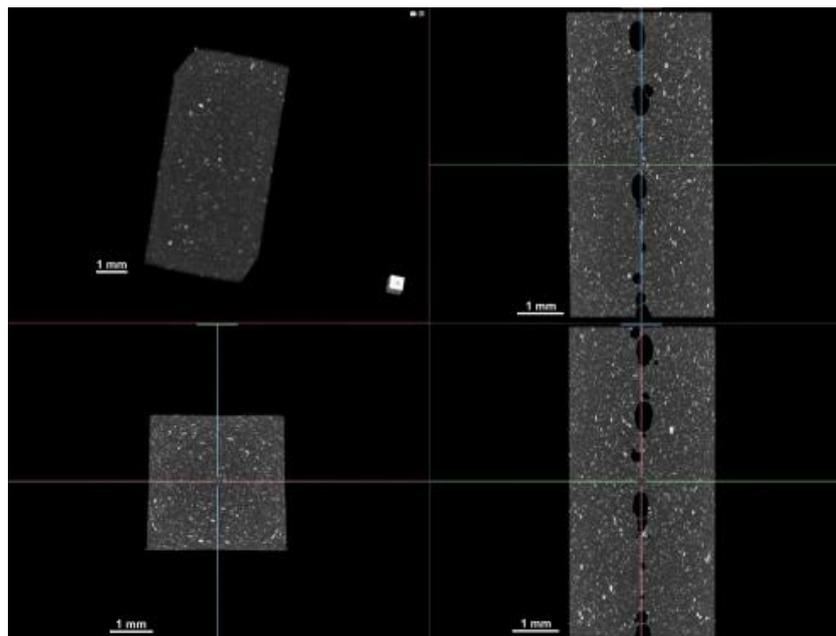

Figure 5. 3D rendering with orthogonal views of the 70/30 composite sample. Voids identified in the central region of the sample.

particles had a diameter equal to or smaller than 39.11 μm, it is reasonable to use this value as an upper limit for the mean feret diameter of individual particles. By analyzing Fig. 6, that shows the histogram and descriptive statistics of the mean feret of the soapstone particles in each composite sample, it is possible to conclude that a considerable number of particles had larger diameters than that limiting value, which indicates that the great majority of these items are, in fact, agglomerated particles. Using this approximation, it was possible to classify particles into two categories: individual particles and clusters, as shown in Tab. 2. The percentage of particles in clusters increased with filler content, from 10% by number in the 90/10 up to 17% in the 70/30.

The variability of the amount of reinforcement is better characterized by the the volume fraction and its spatial distribution. Therefore, the volume fraction of particles and voids, as well as the parameters defined in Section 2.1, were used to quantify the amount of clustering in each composite sample. The resulting values are listed in Tab. 3. These results reveal that both the volume fraction of particles in clusters (ζ) and the volume fraction of clusters in the composites (γ)
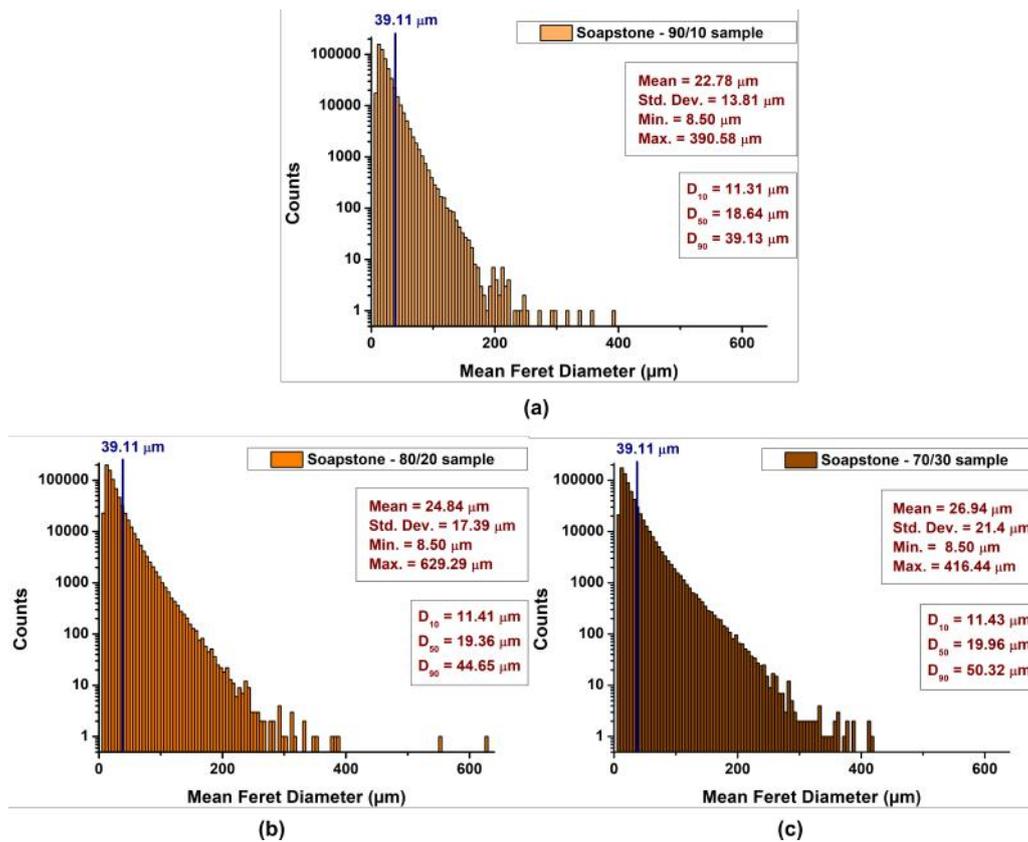
Figure 6. Mean feret diameter histograms of the soapstone filler identified in (a) 90/10, (b) 80/20 and (c) 70/30 samples, respectively.

Table 2. Classification of the soapstone particles in different HDPE composite samples.

Sample	Individual particles		Clusters		Total particles
	Count	(%)	Count	(%)	Count
90/10	4.87E+05	90	5.42E+04	10	5.41E+05
80/20	6.23E+05	86	9.85E+04	14	7.21E+05
70/30	5.47E+05	83	1.10E+05	17	6.57E+05

increased as the particle loading increased, which indicates that the soapstone particles have a strong tendency of forming agglomerates in the polyethylene matrix, specially at higher filler loadings. This conclusion is in consonance with findings reported by Leong *et al.* (2004), regarding commercial talc particles in a polypropylene matrix.

Table 3. Particle clustering parameters, void and filler content of the composite samples.

Sample	M_p (%)	Φ_p (%)	Φ_v (%)	ζ (%)	γ (%)
90/10	10	3.29	0.00	55.54	1.82
80/20	20	6.19	0.00	67.75	4.19
70/30	30	7.83	0.64	77.06	6.03

M_p, Φ_p: mass and volume fraction of particles in the composite, respectively; Φ_v: volume fraction of voids.

The values listed in Tab. 3 reveal that void content also increased with particle loading. As mentioned before this phenomenon is related with increased polymer viscosity during processing, however, the volume fraction of voids was less than 1% for all samples, which is reasonable for most applications (Saenz-Castillo *et al.*, 2019).

The uniformity of filler distribution was analyzed by dividing the composite samples into three subsets along the length, namely, top, middle and bottom, as shown in Fig. 7. The volume of the particles in each subset was measured and the local parameters described in Section 2.1 calculated. The results are listed in Tab. 4, where the local volume fractions were compared with the total volume fraction of particles (Φ_p) in each sample.

The chi-square test of homogeneity was used to determine whether the volume distribution of particles is the same
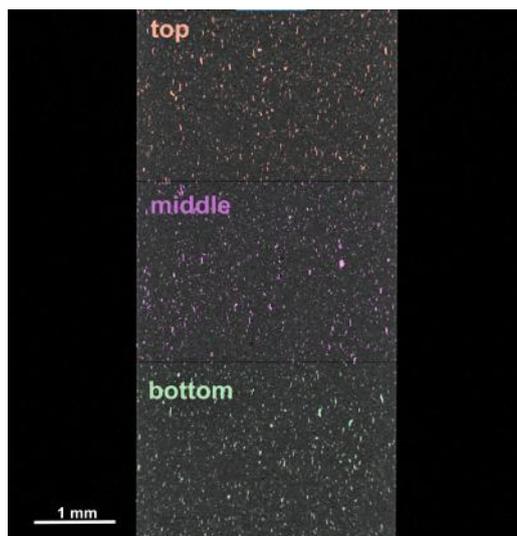
Figure 7. 2D slice of the 90/10 composite showing the division of the sample into three equal-sized subsets: top, middle and bottom.

Table 4. Comparisons between local fractions and the total volume fraction of particles of the composite samples.

Subset	90/10		80/20		70/30	
	ξ_p^{sub}	Φ_p^{sub}	ξ_p^{sub}	Φ_p^{sub}	ξ_p^{sub}	Φ_p^{sub}
Top (%)	33.61	3.31	33.67	6.25	33.63	7.90
Middle (%)	35.03	3.45	34.90	6.48	35.45	8.32
Bottom (%)	31.54	3.11	31.54	5.86	31.04	7.29
Sample (%)	100	3.29	100	6.19	100	7.83

for the subsets, i.e., if the filler distribution in the composite samples can be considered statistically homogeneous. This test measures the amount that the observed counts in the samples deviate from the expected counts. For the fraction of particles in subset (ξ_p^{sub}) the expected values used were 1/n (in this case, 33.33%), and the p-values obtained were less than 0.05 for all the samples. Thus, considering a 95% confidence level, the hypothesis of homogeneity can be accepted. The same analysis was done for the the local volume fraction of particles (Φ_p^{sub}), using the total volume fraction of particles (Φ_p) in each sample as the expected values. The p-values obtained were also less than 0.05 for all samples. Therefore, the volume distribution of particles in all three composite samples can be considered statistically homogeneous in the direction studied.

4.3 Mechanical behavior

The mechanical behavior of the composites was determined by tensile testing. Eight samples of each composition were tested and the results were reported in previous works (de Sousa *et al.*, 2019, 2021). The experimental data from the tensile tests was fitted by the macro-mechanical model described in Section 2.2, using the filler volume fraction (Φ_p) determined in Section 4.2. The value of the shape parameter (A) was set as 2.5, which is the upper limit considering close packing of spherical particles (Turcsányi *et al.*, 1988). The other parameters were adjusted to the curve. Calculations were performed using the method of least squares and the results are shown in Fig. 8.

As can be seen in Fig. 8, the yield strength increased with particle loading and the data was well fitted by the Turcsányi model (r = 0.977). The adjusted value of 6.82 obtained for the *B* parameter is a quantitative indicator that there is good interfacial adhesion between the soapstone particles and the HDPE matrix, which resulted in the improvement in yield strength observed.

5. CONCLUSIONS

A thorough microstructural characterization of soapstone-waste-filled HDPE composites was performed. Automated image analysis of the waste powder attained the particle size and shape distributions, which revealed that most particles had a slightly elongated, circular shape and that 99% of them had diameters ≤ 39.11 μm. X-ray microtomography allowed both qualitative and quantitative analysis of particle distribution, void content and agglomeration in the different composite samples (with 10 wt%, 20 wt% and 30wt% loadings). Micro voids were identified in the 70/30 sample, with 0.64 vol%
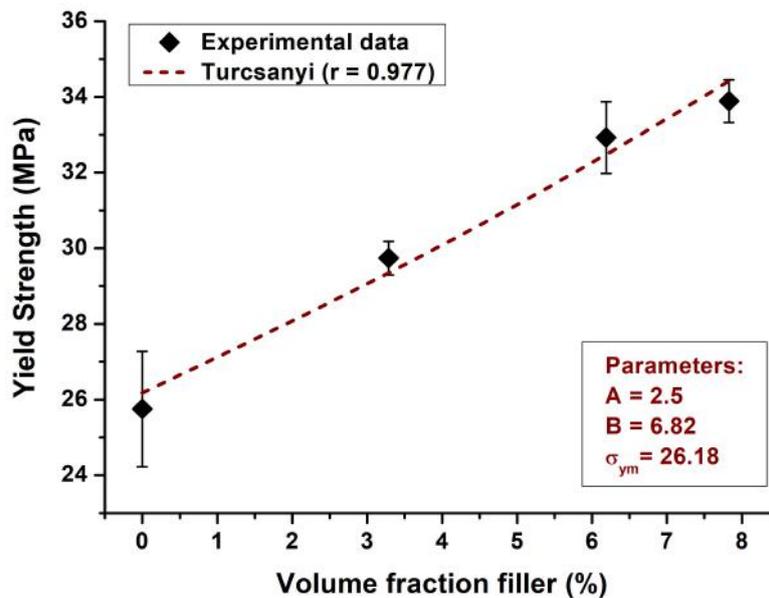
Figure 8. Yield strength as function of filler content of the composites. Data fitted by Eq. (5). (Error bars represent the 95% confidence interval).

void fraction, which was due to increase in viscosity during processing. The value 39.11 μm was used to classify the particles identified in the samples as individual particles or clusters. The analysis revealed that the soapstone particles have a tendency of forming agglomerates in the HDPE matrix, and this tendency increases at higher filler loadings. As mentioned before, the unequal distribution of filler can have relevant effects in this composite's practical behavior. These results suggest that perhaps a few modifications on the processing conditions can improve the distribution of the reinforcement throughout the composites. For instance, an increase in pressure during the injection molding process, could compensate the increased viscosity and improve flow into the mold cavity. Other possible solution is the addition of a coupling agent, aiming to improve the compatibility between phases and promote better dispersion.

The variability of reinforcement in the composite samples was analyzed by dividing the samples into three subsets throughout the length and measuring the volume of the particles in each subset. A statistic test of homogeneity revealed that, despite particle clustering, the distribution of particles can be considered homogeneous for all the composite samples in the direction studied, which is the direction of the applied load in tensile tests.

The relationship between the microstructure and the macroscopic mechanical behavior was done by fitting tensile test data with a macro-mechanical model from the literature. The tensile yield strength of HDPE increased with particle loading, which can be linked with the homogeneous filler distribution observed in the composites microstructure. The analysis allowed a quantification of filler-matrix adhesion and, as a value of 6.82 was obtained for the *B* parameter, it is possible to infer that there is good interfacial adhesion between the soapstone particles and the HDPE matrix.

6. ACKNOWLEDGEMENTS

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