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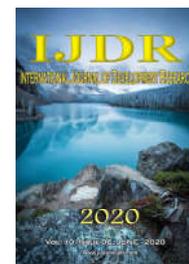
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RESEARCH ARTICLE

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A STUDY OF RECYCLED HIGH-DENSITY POLYETHYLENE WITH MICA ADDITION: INFLUENCE OF MICA PARTICLE SIZE ON WETTING BEHAVIOR, MORPHOLOGICAL, PHYSICAL, AND CHEMICAL PROPERTIES

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ABSTRACT

This work aimed to study the influence of mica (muscovite type) particle size in recycled high-density polyethylene (rHDPE) matrix on wetting behavior and morphological, physical, and chemical properties. The r-HDPE/mica composites were compounded with a fixed ratio of 85/15 (weight percentage), varying the mica particle size (45, 75, 100 and 125 μm). The rHDPE and composites were fed into a twin-screw extruder. The materials were characterized regarding particle size, density, hardness, melt-flow index and morphology, variously using scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), water contact angle measurements and water absorption measurements. A slight increase in density of composites was observed. The efficacy of the mica to increase the resistance was observed by the hardness results. MFI results suggested alignment of the muscovite particles induced by the extrusion process, corroborating the SEM images. X-ray results indicated the mica contributed to increase the crystallinity of the matrix. For composite materials, the contact angle increased around 17% with mica addition, independently of mica particle size. The absorbed water decreased with mica addition in relation to the matrix and decreased more with larger particle sizes.

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INTRODUCTION

Polyolefin-based materials are widely used in several industries, including packaging, foods, automotive etc. Due to its low density, low cost, good thermal and chemical stability, and ability to be recycled, high-density polyethylene is the most used polyolefin in the world. The production of virgin plastic increases the pressure on natural resources and its disposal poses serious environmental problems. An estimated 83,000 million metric tons of virgin plastics have been produced to date, of which only 21% have been reclaimed for recycling or incineration (Curtzwiler et al., 2019; Lapčik et al. 2018). One challenge to the recycling thermoplastics is the loss of mechanical, chemical and thermal properties with each recycling step (Curtzwiler et al., 2019; Lapčik et al. 2018). The potential use of mica in polymeric matrix has also been studied for a long time due to its excellent thermal, electrical,

chemical and fire resistance. In most applications, the percentage of mica in the matrix polymer is less than 50% (Verbeek, 2002; Kuelpmann et al. 2005). Through filling, reinforcing, or blending, it is possible to obtain performance of recycled polyolefin-based material similar to that of the corresponding virgin material (Curtzwiler et al., 2019; Lapčik et al. 2018; Verbeek, 2002; Kuelpmann et al. 2005). Physical and mechanical properties of particle-filled polymer composites depend on size, shape and distribution of particles in the matrix and good adhesion at the interface (Bose and Mahanwar, 2005). In the study of Lapčik et al. (2018), two fillers, mica and wollastonite, increased Young's modulus of elasticity with increasing filler concentration in virgin HDPE composite. In the case of HDPE/mica, the same improvement was also found for the upper yield point with higher filler concentration, indicating greater stiffness. However, for the HDPE/wollastonite composites, the opposite trend was

observed. Kuelpmann *et al.* (2005) prepared composites of virgin HDPE and glass or mica particles with different aspect ratios and surface modifications. The aspect ratio of the mica platelets was evaluated by analysis of SEM micrographs. They observed that surface treatment of the mica particles had practically no influence on the results in the investigated loading range (volume fraction $\leq 7\%$). Li *et al.* (2016) investigated a novel ethylene-vinyl acetate (EVA) composite for cables and insulated wires, prepared by incorporating glass powder, mica powder and organo-modified montmorillonite in the EVA. The results demonstrated that the formed ceramics had excellent mechanical properties and insulativity.

The effects of particle size and particle size distribution on the properties of mica-filled nylon-6 was investigated by Bose and Mahanwar (2005). Composites of nylon-6 with varying concentrations (5 to 40 wt%) of mica were prepared by twin-screw extrusion. The composite had improved mechanical, thermal and dielectric properties with addition of filler. The mica with larger particle caused better improvement than the smaller particles, which might have been due to the continuity of filler particles even at higher loading. The mechanical properties of composite were found to be a function of the particle size, dispersion and particle size distribution. Silva *et al.* (2013) developed a virgin HDPE/CaCO₃ composite by varying the aspect ratio of CaCO₃. Statistical analysis showed that increase in particle size decreased the impact resistance by promoting the formation of agglomerates. On the other hand, the composites with smaller particles had impact resistance properties like those of pure HDPE, in addition to maximum rigidity. The aim of this work was to study the influence of mica (muscovite type) particle size in recycled high-density polyethylene (rHDPE) matrix on the wetting behavior, morphological, physical and chemical properties of extruded composite. The composite materials were processed in a twin-screw extruder. Formulations were compounded at a fixed rHDPE/mica ratio of 85/15 (weight percentage), with varying mica particle size: 45, 75, 100 and 125 μm . The materials were characterized according to particle size, density, hardness, melt-flow index (MFI) and morphology by scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), water contact angle (WCA) measurements and water absorption measurements.

EXPERIMENTAL PROCEDURE

Materials

The mica used in this work was supplied by the Center for Mineral Technology (CETEM) and was collected in the region of Borborema-Seridó (straddling the states of Rio Grande do Norte and Paraíba).

The muscovite was processed using the table gravity concentration method followed by passage through a knife mill (Santos *et al.*, 2011) to obtain particle size below 2 mm without impurities. The plastic lumber waste (recycled high-density polyethylene or r-HDPE) used in this work was provided by Companhia Municipal de Limpeza Urbana (Comlurb, the municipal sanitation company in the city of Rio de Janeiro). The process to prepare the material for use in the composite initially consisted of manual separation to eliminate undesirable coarse residues, followed by magnetic separation to remove metal particles (Martins *et al.*, 2019).

Composite preparation: The mica was milled again to obtain four maximum particle sizes: 45, 75, 100 and 125 μm . Thereafter, the rHDPE was manually cut into smaller sizes. The rHDPE/mica composites (85/15% by weight), with varying mica particle size (μm) were: rHDPE, rHDPE/mica45, rHDPE/mica75, rHDPE/mica100 and rHDPE/mica125. Material with each formulation was fed into a twin-screw extruder (Teck Trill DCT model) equipped with 10 temperature zones, ranging from 170 to 195 °C, from the feed to die (rotating at 90 rpm). Finally a water jet at 25 °C cooled the extruded material, which was immediately pelleted. For characterization, the pellets with each particle size were pressed at 190 °C and 7 ton for 300 seconds and cooled in a cold press for 60 seconds.

Characterization

Particle size measurement: The mica particle size distribution was determined by the laser diffraction technique using a Malvern Mastersizer 2000 analyzer. The measurements were carried out in distilled water. The nominal diameters D (0.1), D (0.5) and D (0.9) were calculated by averaging the mean values of each measurement.

Density, hardness and melt index flow (MFI): The samples (rHDPE, HDPE/Mica45, HDPE/Mica75, HDPE/Mica100 and HDPE/Mica125) were characterized according to density (ASTM D792), hardness (ASTM D2240) and melt-flow index MFI (ASTM D1238).

Scanning electron microscopy (SEM): The SEM analysis was performed using a Hitachi TM3030 Plus microscope to observe specimens coated with silver. Cryogenically fractured transversal sections of the composite samples were assessed, and the images were obtained at 3000 x magnification.

X-ray diffraction (XRD): The XRD analysis was performed with a Rigaku Miniflex X-ray diffractometer using Co-K α radiation (35 kV/40 mA) and 2 θ range from 5° to 80°.

FT-IR analysis: Fourier-transform infrared spectra were acquired using a Nicolet 6700 FTIR spectrometer (Thermo Scientific). The samples (mica, rHDPE and composites) were mounted on an attenuated total reflectance (ATR) accessory equipped with a ZnSe crystal prior to scanning. The spectra were obtained with accumulation of 120 scans and resolution of 4.182 cm^{-1} .

Water contact angle (WCA) measurements: The wettability of the materials' surface (rHDPE and composites) was examined through water contact angle measurements using a Ramé-Hart NRL A-100-00 goniometer.

The evolution of the droplet shape was recorded with a CCD camera every 15 s for a period of 225 s for each sample, at room temperature.

Water absorption: The water absorption test of rHDPE and composites was carried out according to ASTM D-570. Three conditioned specimens (20 mm x 20 mm) were placed in a container of distilled water maintained at temperature of 23 \pm 1°C, resting on edge and entirely immersed. At the end of 24 h, the specimens were removed from the water one at a time, all surface water was removed with a dry cloth, and they were immediately weighed with an analytical balance (Marte, UX4200H model) with precision of 0.001 g.

RESULTS AND DISCUSSION

The results of particle size distribution of the four mica samples studied here are shown in Table 1. These samples were initially ground and classified by dry sieving to maximum particle sizes of 45, 75, 106 and 125 μm . The results in Table 1 reveal a particle size distribution that is desirable for the production of composites, since fine particles embedded in a polymer matrix act as structural filler (Lapčík *et al.* 2018).

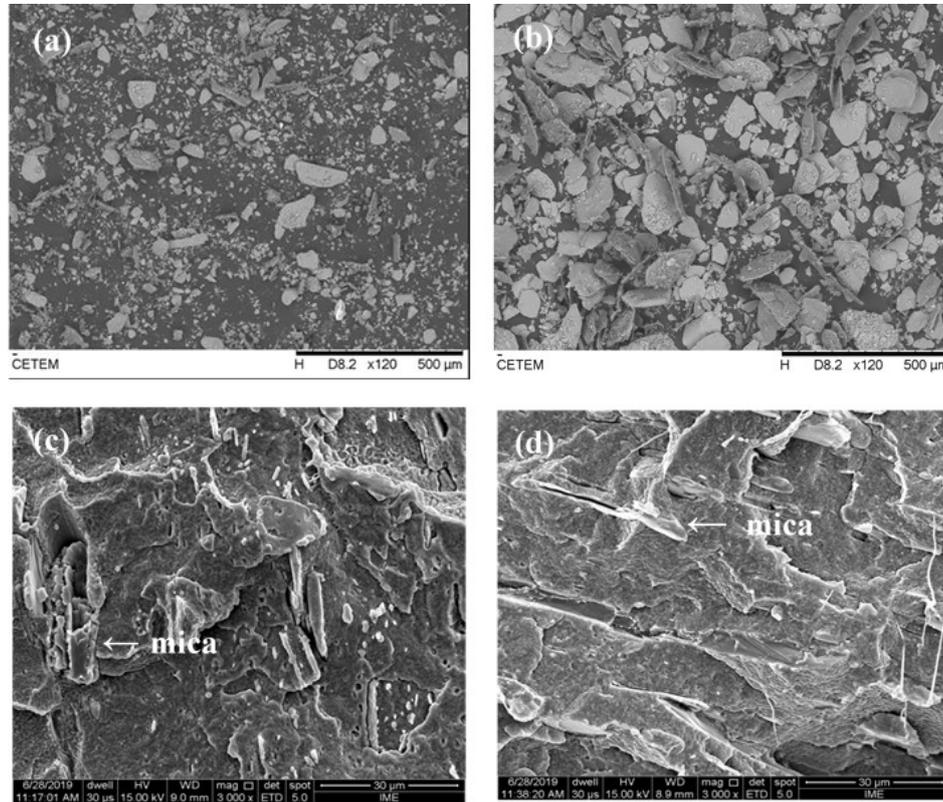


Figure 1: SEM micrographs of mica 45 μm (a), mica 100 μm (b) and fractured surfaces of rHDPE/mica75 (c) and rHDPE/mica100 (d).

As expected, the average diameters, $D(0,5)$, increased as the particle size increased. However, for the 106 μm and 125 μm samples, the size distribution was quite similar. This phenomenon can be related to the interferences that irregularly shaped particles, such as mica, can cause in measurements by the laser diffraction technique, as reported in the literature (Guzzo *et al.*, 2019). Figure 1 (a-b) shows the SEM images of mica samples with particle sizes of 45 and 125 μm , respectively, indicating the random planar shape of this mineral (Santos *et al.*, 2011). It is also possible to observe the presence of smaller particles in both samples, as described regarding particle size distribution analysis. This is desirable for powder filler because these fine particles are better able to fill the voids in the matrix (Lapčík *et al.* 2018). The rHDPE/mica75 and rHDPE/mica100 composite samples can be seen in Figure 1 (c-d). There was relatively good adhesion between the filler and matrix, with few occurrences of small cavities around individual particles. This good adhesion was also observed in a similar system (polypropylene/mica) studied by Monsorens *et al.* (2017). Besides this, the good adhesion of the rHDPE/mica composites studied in this work can be attributed to the presence of additives in the recycled matrix (Martins *et al.*, 2019). Table 2 summarizes the results obtained for density, hardness and MFI of rHDPE and composites. The density value of rHDPE sample is in accordance with literature data [10].

The composites' density increased slightly with mica addition, which can be attributed to good matrix/filler adhesion, as seen in the SEM micrographs. This good interaction is probably related to the additives present in the recycled material, which act as plasticizers. However, we observed no significant changes in composite samples with mica having different particle size. The small increase in hardness result for the composites in relation to the rHDPE suggests the efficacy of the mica to increase the resistance of the polymeric matrix

against deformation (Lapčík *et al.* 2018; Li *et al.*, 2016). Here we also did not observe significant difference in composite samples with varying mica particle size. Again, this is in line with literature data indicating that the hardness of composites is determined mainly by the load domain (Talgatti *et al.*, 2017). The MFI results showed that the viscosity of the composites decreased with mica addition. This result suggests that alignment of the muscovite particles occurred, possibly induced by the extrusion process, which facilitates the fluidity of the polymer chains (Lapčík *et al.* 2018). The SEM micrographs confirmed this theory, since they revealed the mica particles were horizontally oriented in the direction of the flow induced by the extruder. Furthermore, the literature indicates that muscovite may have a lubricating effect when heated, and our result suggests this effect is more pronounced for larger particles size, as observed for the composites with mica having 100 and 125 μm size (Du *et al.*, 2018). Figure 2 (a-d) presents XRD patterns of pure rHDPE (a), mica (b) and those of the rHDPE/mica (c-d) studied. Diffraction peaks of pure rHDPE, Fig. 2b, are clearly present between the 2θ diffraction angles of 20° and 30° , corresponding to the orthorhombic phase (Peltoa *et al.*, 2017). The typical XRD pattern for muscovite mica (Santos *et al.* 2011) can be seen in Figure 2a. For composite samples, shown in Fig. 2c and 2b, both rHDPE and muscovite peaks can be seen, but the rHDPE peaks are sharper compared to the pure rHDPE patterns,

indicating that the mica contributed to increase the crystallinity of the matrix. This result is supported by the hardness observation, since an increase in crystallinity usually promotes an increase in the rigidity of the material (Lapčák *et al.* 2018). Figure 3 shows the FT-IR results of the samples. The rHDPE peaks in the 2950 and 2850 cm^{-1} region correspond to C-H stretching vibrations; peaks in the 1450-1350 cm^{-1} region correspond to CH_2 bending vibrations; and peaks in the region of 730 to 717 cm^{-1} refer to CH_2 rocking vibrations (Torres *et al.*, 2010; Lazrak *et al.*, 2019).

Table 1. Particle size diameters of mica samples

Mica size fraction (μm)	Particle size (μm)		
	D (0.1)	D (0.5)	D (0.9)
45	3.829	17.189	55.819
75	11.985	52.561	106.247
100	17.153	68.263	131.674
125	13.980	66.328	136.437

Table 2. Density, hardness and MFI of formulations

Samples	Density (g/cm^3)	Hardness (Shore D)	MFI ($\text{g}/10\text{min}$)
rHDPE	$0.815 \pm 0,087$	$73.1 \pm 4,2$	$0.1487 \pm 0,0122$
rHDPE/Mica45	$1.154 \pm 0,087$	$78.8 \pm 2,3$	$0.1547 \pm 0,0165$
rHDPE/Mica75	$1.224 \pm 0,055$	$80.0 \pm 2,5$	$0.2561 \pm 0,0135$
rHDPE/Mica100	$1.106 \pm 0,035$	$80.0 \pm 1,7$	$0.3015 \pm 0,0256$
rHDPE/Mica125	$1.167 \pm 0,033$	$79.8 \pm 2,8$	$0.2833 \pm 0,0106$

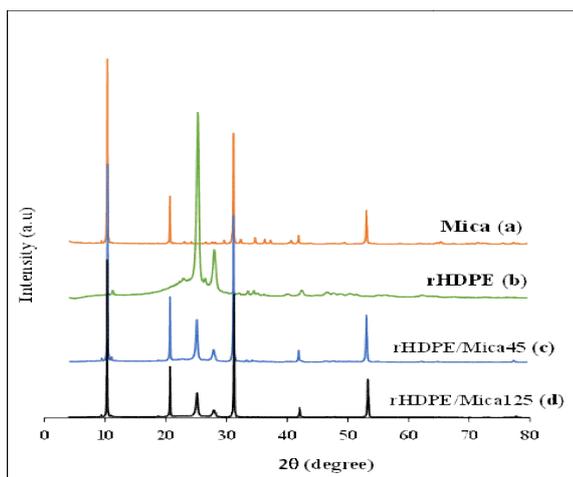


Figure 2. X-ray diffraction patterns of mica (a), rHDPE (b), and rHDPE/Mica45 (c), rHDPE/Mica125 (d) composites

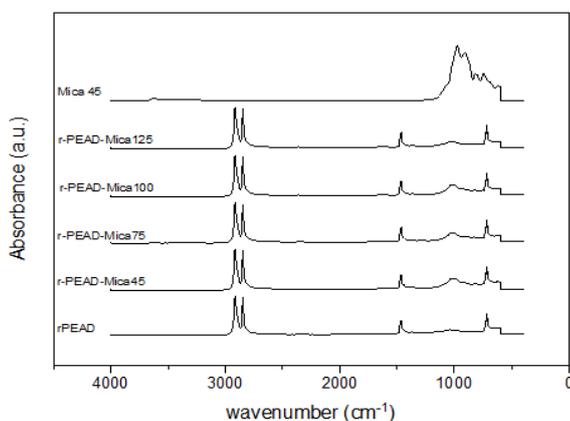


Figure 3. FT-IR spectra of mica, rHDPE and rHDPE/mica formulations

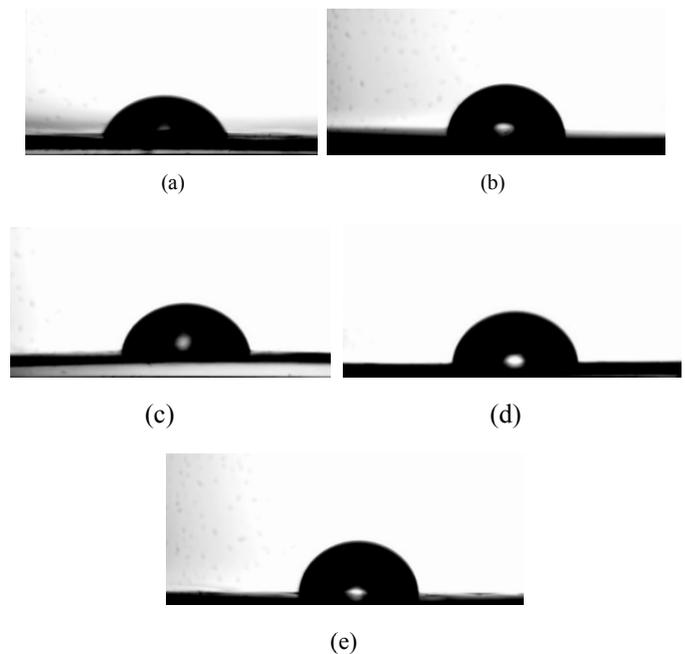


Figure 4. Images of ontact angle with water for: (a) rHDPE, (b) rHDPE/Mica 45, (c) rHDPE/Mica 75, (d) rHDPE/Mica 100 and (e) rHDPE /Mica 125

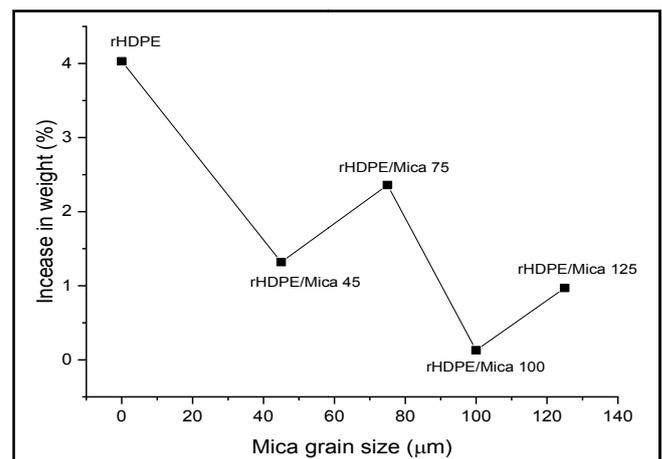


Figure 5. Effect of particle size on the absorbed water of rHDPE/mica composites

The vibrations of the mica group minerals are reported in (Beran, 2002) as: OH stretching vibrations (3750-3550 cm^{-1} region); Si-O stretching vibrations (1200-700 cm^{-1} region); Si-O bending vibrations (600-300 cm^{-1} region). These usually show coupling with stretching and bending vibrations of the cation-oxygen octahedra lying in a similar spectral region. The peaks of the whole composites were unchanged, so it can be inferred there were no chemical interactions between rHDPE and mica. The interphase rHDPE-mica was of physical nature. Table 3 and Figure 4 show the water contact angle measurements and images of the droplets on the materials' surface, respectively. The surface of recycled HDPE indicated hydrophobic behavior, with an angle of 68.6° . A similar result was found by Lazrak *et al.* (2018) for recycled HDPE (67.6°). For composite materials, the contact angle increased around 17% with mica addition, independently of mica particle size. The higher contact angle values for composites in comparison of pure rHDPE showed that muscovite provides a high level of hydrophobicity to rHDPE, near that of virgin HDPE (around 90°) (Badgayan *et al.*, 2019; Wu *et al.*, 2003).

Table 3. Water contact angle measurements of rHDPE and composites

Samples	% by weight	Contact angle (°)
rHDPE	100	68.600 ± 1.064
rHDPE/Mica 45	85/15	81.833 ± 1.119
rHDPE/Mica 75	85/15	83.560 ± 0.515
rHDPE/Mica 100	85/15	82.987 ± 0.569
rHDPE/Mica 125	85/15	83.740 ± 1.518

The effects of mica particle size on absorbed water of rHDPE composites are shown in Figure 5. The absorbed water decreased with mica addition in relation to the matrix (rHDPE). The absorbed water decreased more with larger particle sizes (100 and 125 mm). Smaller particles (45 and 75 mm) have higher surface area to volume ratio than that of larger particles (100 and 125 mm), so they have greater ability to absorb water (Awad et al., 2019).

Conclusion

In this study, the influence of mica (muscovite type) particle size on wetting behavior, morphological, physical, and chemical properties of recycled HDPE were analyzed. The slight increase in composite density revealed by the SEM images suggested good adhesion between mica and rHDPE. The hardness, MFI and XRD results showed that mica addition slightly increased the rHDPE resistance. The water contact angle and water absorption analyses indicated an increase in hydrophobicity of the composite, which is particularly interesting for use in outdoor settings. The FT-IR analysis showed that the mica-rHDPE surface had physical interaction. In general, the results showed that properties of recycled HDPE improved with mica addition. The variation of mica particle size studied did not significantly affect the properties of rHDPE. Since the mica particle size did not significantly affect the properties of rHDPE, we suggest the use of larger particle sizes, which can reduce the final cost of the product. The results obtained corroborate those of several studies related to the improvement of post-consumer polymers, aiming at sustainability.

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