

Development of composites based on a blend of recycled HDPE/post-consumer PET and bentonite clay

Desenvolvimento de compósitos à base de blenda de PEAD reciclado/PET pós-consumo e argila bentonita

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Aline Muniz Lima

Master in Materials Science and Technology

Institution: Universidade do Estado do Rio de Janeiro (UERJ)

Address: Avenida Manuel Caldeiras de Alvarenga, 1203, Campo Grande,

CEP: 23070-200, Rio de Janeiro – RJ

E-mail: aline-muniz15@hotmail.com

Gabriel de Sousa Barros

Materials Engineering Undergraduate

Institution: Universidade do Estado do Rio de Janeiro (UERJ)

Address: Avenida Manuel Caldeiras de Alvarenga, 1203, Campo Grande,

CEP: 23070-200, Rio de Janeiro – RJ

E-mail: gabrielbarros2204@gmail.com

Mônica Cristina Celestino Santos

Bachelor in Technologist of Polymers

Institution: Universidade do Estado do Rio de Janeiro (UERJ)

Address: Avenida Manuel Caldeiras de Alvarenga, 1203, Campo Grande,

CEP: 23070-200, Rio de Janeiro – RJ

E-mail: monicauezo@hotmail.com

Marceli do Nascimento da Conceição

Doctor of Science in Metallurgical and Materials Engineering

Institution: Centro de Tecnologia Mineral (CETEM)

Address: Av. Pedro Calmon, 900, Rio de Janeiro - RJ, CEP: 21941-908

E-mail: mconceicao@cetem.gov.br

Daniele Cruz Bastos

Ph.D. in Metallurgical and Materials Engineering from the Metallurgical and Materials Engineering Program at Coppe by Universidade Federal do Rio de Janeiro (UFRJ)

Institution: Universidade do Estado do Rio de Janeiro (UERJ)

Address: Avenida Manuel Caldeiras de Alvarenga, 1203, Campo Grande,

CEP: 23070-200, Rio de Janeiro – RJ

E-mail: danielebastos@uezo.edu.br

Patricia Soares da Costa Pereira

PhD in Polymer Science and Technology from the Macromolecules Institute
Institution: Universidade do Estado do Rio de Janeiro (UERJ), Universidade Federal do
Rio de Janeiro (UFRJ)

Address: Avenida Manuel Caldeiras de Alvarenga, 1203, Campo Grande,
CEP: 23070-200, Rio de Janeiro – RJ
E-mail: patricia.soares.pereira@uerj.br

Elaine Vidal Dias Gomes Libano

PhD in Polymer Science and Technology from the Macromolecules Institute
Institution: Universidade do Estado do Rio de Janeiro (UERJ), Universidade Federal do
Rio de Janeiro (UFRJ)

Address: Avenida Manuel Caldeiras de Alvarenga, 1203, Campo Grande,
CEP: 23070-200, Rio de Janeiro – RJ
E-mail: elaine.libano@uerj.br

ABSTRACT

The recycling process is the best way to manage waste polymers. From an economic standpoint, more investigation is needed of the whole recycling process to enhance the competitiveness in these systems. A first step is improved interaction and interfacial adhesion of blends. Polyethylene (PE) and polyethylene terephthalate (PET) are incompatible polymers, so their blends in general have poor properties. Compatibilization is thus a necessary step to obtain blends with good mechanical and barrier properties. In this work, blends of recycled (high density polyethylene) (HDPE) and post-consumer PET, with organophilic bentonite clay acting as compatibilization agent at the interface of the two polymers, were obtained. The proportions of HDPE/PET/bentonite (0, 5, 10 and 15% weight percentage of bentonite) were processed by extrusion. The processed materials were characterized regarding density (ASTM D792-13), melt flow index (MFI, ASTM D1238-13), hardness (ASTM D2240–10), infrared absorption spectroscopy (FTIR), X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The results indicated the occurrence of some compatibilization.

Keywords: bentonite, polymer blends, composites, HDPE, PET.

RESUMO

O processo de reciclagem é a melhor maneira de gerenciar resíduos poliméricos. Do ponto de vista econômico, é necessária mais investigação de todo o processo de reciclagem para aumentar a competitividade desses sistemas. Um primeiro passo é melhorar a interação e a adesão interfacial das misturas. O polietileno (PE) e o poli(tereftalato de polietileno) (PET) são polímeros incompatíveis, de modo que suas misturas em geral apresentam propriedades ruins. A compatibilização é, portanto, uma etapa necessária para a obtenção de blendas com boas propriedades mecânicas e de barreira. Neste trabalho, foram obtidas blendas de polietileno de alta densidade (PEAD) reciclado e PET pós-consumo, com argila bentonita organofílica atuando como agente de compatibilização na interface dos dois polímeros. As proporções de PEAD/PET/bentonita (0, 5, 10 e 15% em peso de bentonita) foram processadas por extrusão. Os materiais processados foram caracterizados quanto à densidade (ASTM D792-13), índice de fluidez (MFI, ASTM D1238-13), dureza (ASTM D2240–10), espectroscopia de absorção na região do infravermelho (FTIR), difratometria de raios X (XRD) e microscopia eletrônica de varredura (MEV). Os resultados indicaram a ocorrência de alguma compatibilização.

Palavras-chave: bentonita, misturas poliméricas, compósitos, PEAD, PET.

1 INTRODUCTION

The use of polymeric materials is increasing and multiple millions of tons are discarded into the environment. Thus, it is necessary to develop studies to reuse these waste materials, because when they are not sent for recycling, they take decades or even centuries to decompose. The accumulation of these solid wastes in the environment causes degradation and harms the entire terrestrial ecosystem (MONTEIRO *et al.*, 2020; MASSON and LIMA, 2021).

According to Resolution 01/1986 from Brazil's National Council for the Environment (CONAMA), an environmental impact is "any change in the physical, chemical and biological properties of the environment caused by any form of matter or energy resulting from human activities that directly or indirectly affect: I – the health, safety and well-being of the population; II – social and economic activities; III – the biota; IV – the aesthetic and sanitary conditions of the environment; and V – the quality of environmental resources" (CONAMA, 1986).

Solid waste is defined, by Brazilian Law 12,305 of August 2, 2010 as "discarded material, substance, object or good resulting from the activities of humans in society whose final destination occurs, is proposed to occur or is obliged to occur in the solid or semisolid state, as well as gases contained in recipients and liquids whose particularities make their discharge in the public sewer system or water bodies unfeasible, or that require technically or economically unfeasible solutions in light of the best available technology" (BRAZIL, 2010).

Thermoplastic polymers are materials of great relevance to today's society, but their disposal is still a major problem. Polymeric materials such as high-density polyethylene (HDPE) and polyethylene terephthalate (PET) are the classes of thermoplastics consumed the most and constitute a large portion of post-consumer plastic waste, especially from the packaging sector (JASSIM, 2017; TANAKA *et al.*, 2018). Indeed, packaging materials account for the majority of municipal solid waste around the world, making the disposal and destination of these materials a major environmental problem (BNDES, 2012). It is estimated that by 2025, 2.42 billion tons of plastics will be discarded per year (RIGAIL-CEDEÑO *et al.*, 2019).

There are numerous environmental impacts caused by the improper disposal of these materials, so increased recycling is necessary to minimize these impacts (COELHO *et al.*, 2021). An interesting and promising alternative from an environmental point of view, in addition to adding value to waste, is the development of blends, enabling the creation of materials with intermediate properties between the pure constituents (CHEN *et al.*, 2017; SHAHRAJABIAN and SADEGHIAN, 2019). Polymer blends are physical mixtures of two or more polymers without any chemical reaction between them. These materials have a synergistic effect resulting from the mixture, which is revealed by the improvement of properties in relation to those of homopolymers and copolymers (DANTAS, 2011; ORÉFICE *et al.*, 2004).

Polymer blends stand out for the wide range of possible applications, to the extent their physical, chemical and mechanical properties can be altered, depending on the concentration of the constituents, to achieve desirable performance of the final product by offering an attractive balance of these properties. In the context of the present work, HDPE can modify the properties of PET to achieve high impact resistance and good resistance to chemical solvents. On the other hand, PET can improve the mechanical and thermal properties of HDPE. However, the immiscibility between both leads to the formation of blends with low adhesion and high surface tension, generating phase separation and resulting in a material with low mechanical properties. These properties can be improved by adding a compatibilizing agent to act at the interface of the two polymers (UTRACKI, 1989; WANG *et al.*, 2019).

In recent years clays have been used as compatibilizing agents of immiscible polymer blends to improve their properties. Clay is an abundant and inexpensive material. The most used clays are bentonites, especially montmorillonite (MMT), since they have adequate lamination capacity and high aspect ratio. However, it is necessary for the clay to be organically modified by a surfactant in order to promote compatibility with the polymer. Organophilization is one of the main means to promote the dispersion and exfoliation of clay in the polymer matrix (AUBRY, 2019; DE ALMEIDA *et al.*, 2018; LU *et al.*, 2018; MEDERIC, *et al.*, 2018; MÉLO *et al.*, 2014). In the process of obtaining composites, the addition of organophilic bentonite aims to improve the properties of the HDPE/PET blend.

Given this context, we proposed to obtain composites from blends of recycled HDPE and post-consumer PET, with organophilic bentonite clay acting as a compatibilization agent at the interface of the two polymers, thus helping to reduce the

negative environmental impact of these residues. We used different proportions of recycled HDPE, post-consumer PET and bentonite.

2 EXPERIMENTAL PROCEDURES

2.1 MATERIALS

Recycled HDPE was provided by the recycling company Peterlu Indústria e Comércio de Plásticos Ltda., located in Seropédica, Rio de Janeiro State. Green post-consumer PET from soft drink bottles was provided by the company REPET, São Paulo. The organophilic bentonite (Bento) was obtained in the in the laboratories of Rio de Janeiro State University (UERJ). All materials were oven dried at 70 °C before being used.

2.2 COMPOSITES PROCESSING

The HDPE/PET/Bento composites were processed in a co-rotating twin-screw extruder (Teck Tril, model DCT 20-40), with zone temperatures ranging from 190 to 250 °C from the feed to the die outlet and rotation of 30 rpm. The percentage of HDPE/PET used was 50/50 (by mass). The percentages of clay used in the composites were 5, 10 and 15% by mass. After the extrusion of the materials, the extrudate was crushed and conditioned.

2.3 PREPARATION OF SPECIMENS

The specimens for the different tests (physical-mechanical, structural and morphological) were stamped from plates obtained by compression in a bench press (Marconi model MA 098). These plates were obtained using a temperature of 300 °C, pressure of 9 tons for 1 hour and cooling in a cold press for 9 minutes, thus obtaining the films of the materials.

2.4. CHARACTERIZATION

2.4.1. Melt flow index (MFI), density and hardness

The materials were characterized according to melt flow index, MFI (ASTM D1238-13), density (ASTM D792-13) and hardness (ASTMD2240–13).

2.4.2 Fourier-transform infrared spectroscopy (FT-IR)

The HDPE/PET/Bento composites were characterized by Fourier-transform infrared spectroscopy (FTIR), with a Nicolet 6700 FTIR spectrometer (Thermo

Scientific). The samples were mounted on an attenuated total reflectance (ATR) accessory equipped with ZnSe crystal prior to scanning. The spectra were obtained with an accumulation of 120 scans and resolution of 4.182 cm^{-1} .

2.4.3 X-ray diffraction (XRD)

The crystal structure of the HDPE/PET blend and the HDPE/PET/Bento composites was investigated by X-ray diffractometry using a Bruker-AXS D8 Advance Eco diffractometer. The diffractograms were obtained using voltage of 40 kV and current of 25 mA, with a sweep angle between $4^\circ - 70^\circ$.

2.4.4 Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) analysis was performed using a Hitachi model TM3030Plus microscope to observe specimens coated with gold. Cryogenically fractured cross-sections of the samples were assessed, and the images were obtained at 2000 x magnification, at 15 kV.

3 RESULTS AND DISCUSSION

Figure 1 shows the average MFI values of the HDPE/PET blend and the blends generated by the incorporation of clay (bentonite). A decrease in MFI was observed for all clay mixtures, with the lowest value being 19.4 g/10 min in the HDPE/PET/Bento5% mixture. Machado *et al.*, (2021) observed similar MFI reduction results for post-consumer polymer compounds and clay. These results were probably associated with the restriction of the movement of the chains, and consequently of the flow caused by the presence of the clay (Libano *et al.*, 2018).

Figure 1. MFI of HDPE/PET blend and composites HDPE/PET/Bento.

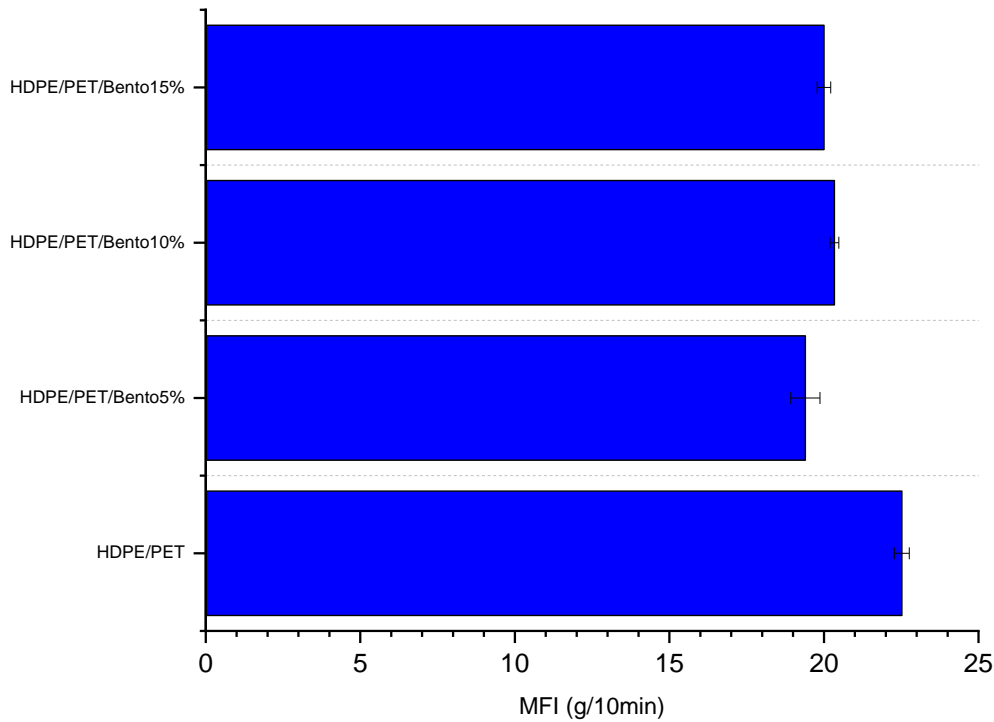


Figure 2 shows the average density values of the HDPE/PET blend and the blends generated by the incorporation of clay (bentonite). The density increased with bentonite addition, which can be attributed to good matrix/filler adhesion. The good interaction is probably related to the additives present in the recycled HDPE, which act as plasticizers (Fernandes *et al.*, 2020). The highest density value was found in the HDPE/PET/Bento10% blend.

Figure 2. Density of HDPE/PET blend and composites HDPE/PET/Bento.

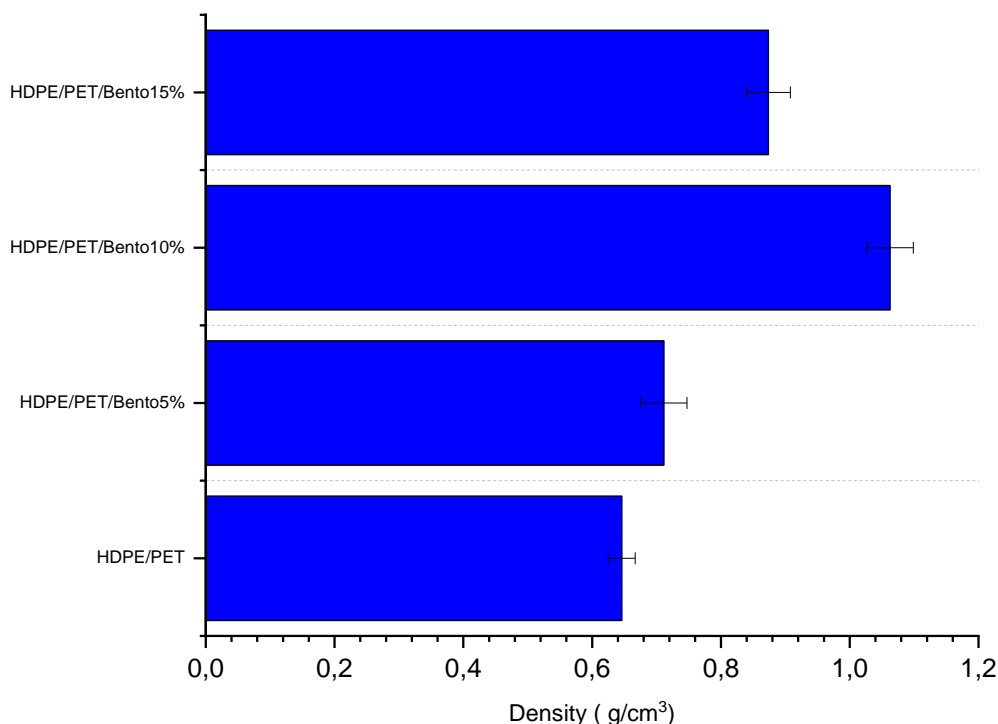
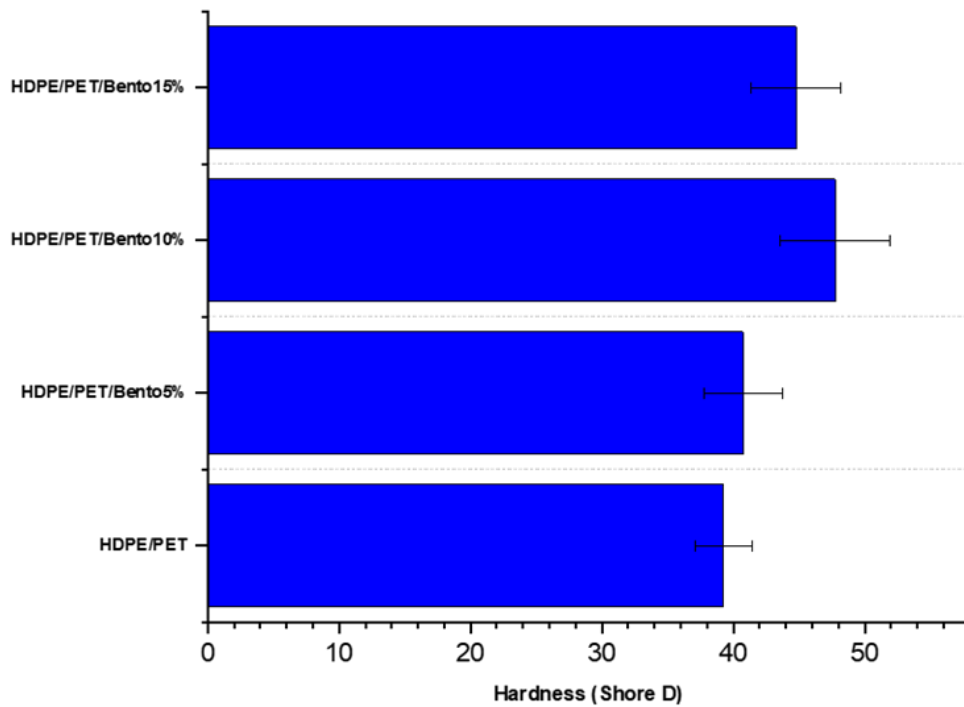


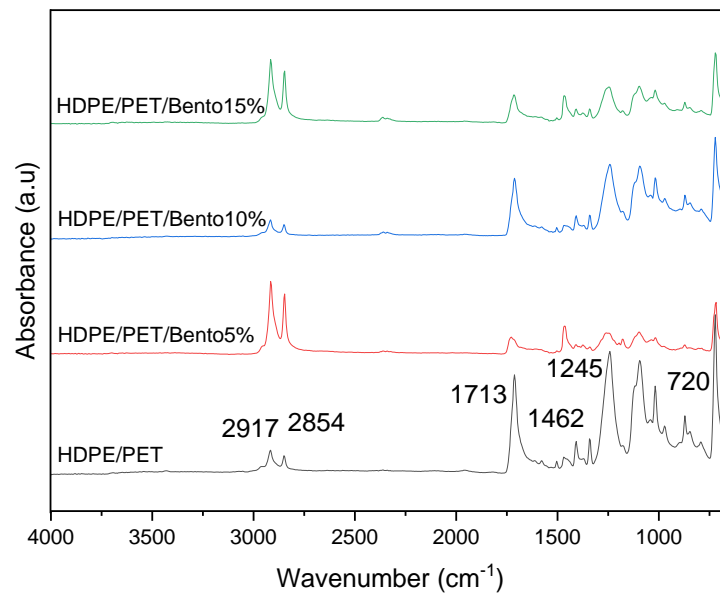
Figure 3 shows the hardness results of the HDPE/PET blend and HDPE/PET composites with different clay contents. There was an increase in hardness of all compatibilized samples in relation to HDPE/PET without compatibilizer, which can be explained by the presence of the compatibilizing agent, favoring the interaction between the chains, making them more difficult to break by the durometer indenter. The same effect occurred in the density analysis of the sample with the highest content of compatibilizer (15%), where there was a decrease in hardness in comparison with the 10% bentonite content, a result that was surprising, since we expected the addition of larger amounts of compatibilizer to produce higher hardness values. However, this fact may have been caused by the poor homogenization of the composites prepared in the extruder due to the low shear promoted by the device. Figure 3 shows the FT-IR spectra of the HDPE/PET blend and HDPE/PET/Bento composites.

Figure 3. Hardness of HDPE/PET blend and composites HDPE/PET/Bento.



The FTIR spectra of HDPE/PET blend and composites HDPE/PET/Bento are presented in Figure 4. The peaks at 2917 and 2854 cm^{-1} denote the $-\text{CH}_2-$ stretching of PE. The peaks at 1462 and 720 cm^{-1} are attributed to the in-plane bending vibration and rocking vibration of $-\text{CH}_2-$ in PE, respectively (Monteiro *et al.*, 2020; Rodrigues *et al.*, 2020). The $-\text{C}=\text{O}$ stretching (at 1725 cm^{-1}) of PET could be observed in the spectra. The $-\text{C}-\text{O}-\text{C}-$ asymmetric stretching appeared at 1251 cm^{-1} and the peak at 720 cm^{-1} denoted the out-of-plane bending vibration peak of $-\text{CH}-$ in the benzene ring (Ravichandran *et al.*, 2016; Li *et al.*, 2018). The addition of the inorganic filler in the polymeric mixture did not promote the appearance of new absorption bands, suggesting the existence of simple physical mixing without chemical reactions involved (Cazan *et al.*, 2018).

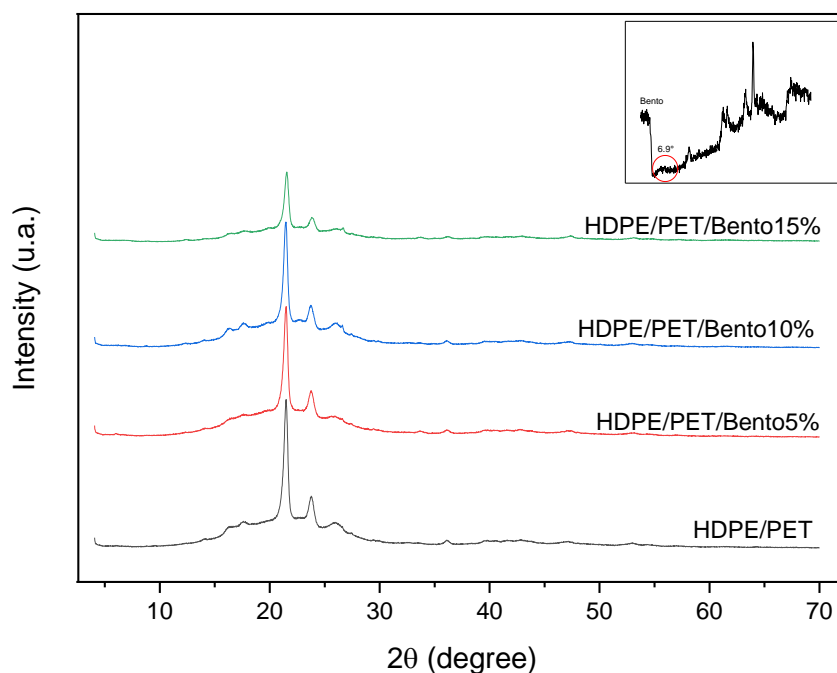
Figure 4. FT-IR spectra for HDPE/PET blend and composites HDPE/PET/Bento.



X-ray diffractometry (XRD) analyses are shown in Figure 5. All the diffractograms show the presence of crystalline peaks corresponding to the HDPE diffraction planes at $2\Theta = 21.4^\circ$ (110) and 23.7° (200). The 2Θ diffraction peak at 25.74° (100) can be attributed to the triclinic PET system (Torres-Huerta *et al.*, 2014; Cazan *et al.*, 2018; Wang *et al.*, 2019). Note that the intense peaks are well defined, which clearly shows the existence of crystallinity in all systems studied.

Bentonite presents a characteristic peak at $2\Theta = 6.9^\circ$, but this peak was not observed in HDPE/PET/Bento composites, suggesting possible intercalation/exfoliation of clay layers in the HDPE/PET matrix (Chen *et al.*, 2017).

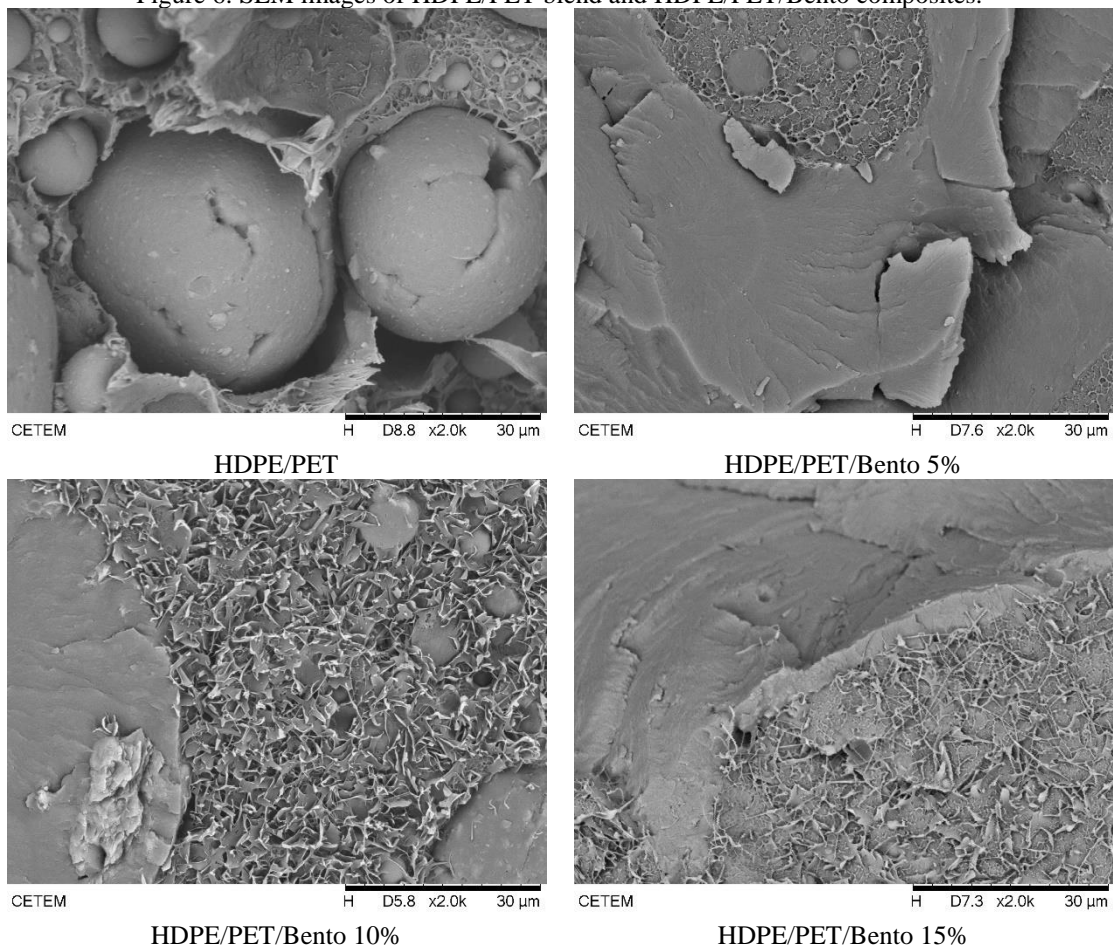
Figure 5. X-ray diffractograms of HDPE/PET blend and HDPE/PET/Bento composites.



The SEM micrograph of the HDPE/PET blend (Fig. 6) confirmed the strong incompatibility evidenced by the X-ray diffraction. The dispersed particles are large, have very different dimensions, and do not show good interfacial adhesion between phases. Some HDPE particles are slightly elongated because of the extrusion process.

The SEM micrographs of the HDPE/PET/Bento composites confirm the hypothesis of the occurrence of some compatibilization. In relation to the HDPE/PET, the micrographs of the HDPE/PET/Bento composites show better adhesion. All these features clearly indicate the occurrence of significant compatibilization between the two phases induced by the presence of bentonite. Similar results have been reported by other authors (Dimitrova *et al.*, 2000; Rosnan *et al.*, 2013, Yousfi *et al.*, 2013).

Figure 6. SEM images of HDPE/PET blend and HDPE/PET/Bento composites.



4 CONCLUSION

Blends of recycled HDPE and post-consumer PET, with organophilic bentonite clay acting as compatibilization agent, were obtained by extrusion. The results are summarized as:

- The MFI decreased for all clay mixtures, associated with the restriction of the movement of the chains, and consequently of the flow, imposed by the presence of the clay.
- The density increased with bentonite addition, which can be attributed to good matrix/filler adhesion.
- There was an increase in hardness of all compatibilized samples in relation to HDPE/PET without compatibilizer, indicating better interaction between the chains in the HDPE/PET/Bento composites.

- The FTIR results indicate that the addition of the inorganic filler to the polymeric mixture did not promote the appearance of new absorption bands, suggesting only physical interaction occurred.
- The XRD results suggest possible intercalation/exfoliation of clay layers in the HDPE/PET matrix.
- The SEM micrographs of the HDPE/PET/Bento composites confirmed the hypothesis of the occurrence of some compatibilization.

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