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Characterization of low-density polyethylene films incorporated with different concentrations of montmorillonite

Caracterização de filmes de polietileno de baixa densidade incorporados com diferentes concentrações de montmorilonita

Caracterizatión de películas de polietileno de baja densidad incorporadas con diferentes concentraciones de montmorillonita

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Resumo

Este trabalho teve como objetivo avaliar o efeito da incorporação de diferentes concentrações de montmorilonita em filmes de polietileno de baixa densidade. A incorporação do nanocomposto Cloisite® 10A nos filmes foi realizada utilizando-se uma extrusora modular mono rosca. Após a incorporação de seis concentrações de argila montmorilonita organofílica (0,5, 1,0, 1,5, 2,0, 2,5 e 3,0%, p / p) aos filmes, suas propriedades ópticas, mecânicas (resistência à tração, elongação e módulo de Young) e de barreira a gás (CO_2) foram investigadas. O grau de esfoliação foi avaliado através da análise de difração de raios-X. A incorporação de nanopartículas de montmorilonita aumentou a opacidade dos filmes. Quanto às propriedades mecânicas, os filmes incorporados com montmorilonita apresentaram redução nos valores de resistência à tração e elongação, enquanto o módulo de elasticidade não apresentou diferença significativa. Eles também mostraram aumento na permeabilidade a CO_2. A análise de difração de raios X não constatou formação de nanocompósitos. Observou-se, portanto, que os filmes tiveram suas propriedade afetadas com a incorporação da argila montmorilonita.

Palavras-chave: Nanocomposto; Argila; Matriz polimérica.

Abstract

This work aimed to evaluate the effect of incorporating different concentrations of montmorillonite in low-density polyethylene films. The incorporation Cloisite® 10A nanocomposite in the films was carried out using a single screw extruder modular. After the incorporation of six concentrations of organophilic montmorillonite clay (0.5, 1.0, 1.5, 2.0, 2.5 and 3.0%, w/w) to the films, their optical, mechanical (tensile strength, elongation and Young modulus), and gas barrier (CO_2) properties were investigated. The degree of exfoliation was evaluated through the analysis of X-ray diffraction. The incorporation of nanoparticles from montmorillonite increased the opacity of the films. Regarding their mechanical properties, the incorporated films with montmorillonite presented reductions in tensile strength and elongation values, whereas the modulus of elasticity did not show any significant difference. They also showed an increase in the permeability of CO_2. The analysis of X-ray diffraction showed no formation of nanocomposites. It was observed, therefore, that the properties of the films were affected by the incorporation of montmorillonite clay.

Keywords: Nanocomposite; Clay; Polymeric matrix.

Resumen

Este trabajo tuvo como objetivo evaluar el efecto de incorporar diferentes concentraciones de montmorillonita en películas de polietileno de baja densidad. La incorporación del nanocompuesto Cloisite® 10A en las películas se realizó utilizando una extrusora modular mono husillo. Después de la incorporación de seis concentraciones de arcilla de montmorillonita organofílica (0.5, 1.0, 1.5, 2.0, 2.5 y 3.0%, p / p) a las películas, se investigaron sus propiedades ópticas, mecánicas (resistencia a la tracción, el alargamiento y el módulo de Young) y la barrera a los gases (CO2). El grado de exfoliación se evaluó mediante análisis de difracción de rayos X. La incorporación de nanopartículas de montmorillonita aumentó la opacidad de las películas. En cuanto a las propiedades mecánicas, las películas incorporadas con montmorillonita mostraron una reducción en los valores de resistencia a tracción y alargamiento, mientras que el módulo de elasticidad no mostró diferencia significativa. También mostraron un aumento en la permeabilidad al CO2. El análisis de difracción de rayos X no mostró la formación de nanocomposites. Se observó, por tanto, que las propiedades de las películas se vieron afectadas por la incorporación de arcilla montmorillonita.

Palabras clave: Nanocompuesto; Arcilla; Matriz polimérica.

1. Introduction

Since the nineteenth century the increased demand for higher quality and safety of food packaging by consumers has resulted in industry changes in this sector. This has resulted in an increase in the use of polymers due to their advantages when compared to traditional materials, as well as the development of polymers incorporated with nanoparticles (Silvestre et al., 2011).

The incorporation of nanocomposites into polymers improves the quality of the packaging relating to its containment, protection, preservation and marketing (Chaudhry et al., 2008; Sanguansri & Augustin, 2006). The development in the application of nanoparticles in polymers aims to reinforce their mechanical, thermal and barrier properties. However, studies reveal that this incorporation usually presents a weak interaction between the interfaces of the component (Luduena et al., 2007). These interactions can be achieved by the organofilization of the compounds in order to ensure better compatibility between the polymeric matrix and clays (Baruel et al., 2018; Paiva et al., 2008).

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Among the nanocomposites, we highlight montmorillonite (MMT), a clay mineral with the formula $M_x(Al_{4-x}Mg_x)Si_8O_{20}(OH)_4$ (Paiva et al., 2008).It exchanges inorganic cation for ammonium ions to improve its compatibility with polymers (Dutta & Singh, 2015; Paul et al., 2003).

The incorporation of polymer nanoclay in food packaging has presented improvements especially in the packaging of processed meat, cheese, confectionery products, and cereals. It can also be used in extrusion processes to form fruit coating, in the packaging of juices, dairy products and in the production of bottles for beer and carbonated beverages. The polymeric matrices used for the incorporation of nanocomposite include polyamides, polyolefins, polystyrene, ethylene vinyl acetate, polyethylene, among others (Barbalho, 2018; Romanzini et al., 2017; Decker et al., 2015; Chaudhry et al., 2008).

Thus, this study aimed to evaluate the mechanical, barrier and optical properties as well as the degree of exfoliation of films incorporated with different concentrations of montmorillonite (0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0% w/w).

2. Methodology

For the development of films incorporated with clay for use in food products, it was used a low-density polyethylene resin (LDPE) polymer matrix, from Braskem, Brazil. The effect of incorporating Cloisite® 10A nanocomposite, provided by Buntech Input Technology, Brazil, on the properties of the films and the degree of exfoliation was evaluated.

Films with a thickness between 95 and 105 micrometers were obtained by the extrusion process using a single screw extruder modular (Model AX 16: 26 Plastics AX, Brazil) with three temperature zones (170, 180 and 190 ºC). Seven treatments were carried out including the control LDPE (no nanocomposite) and six films containing different concentrations of montmorillonite (0.5, 1.0, 1.5, 2.0, 2.5 and 3.0% w/w).

The opacity was analyzed using a colorimeter (Hunterlab ColorQuest) with the Universal Software Version 3.6. Where: Op = (Opn/Opb) x 100%, where: Op = Opacity of the film (%); Opn = opacity of the film overlaid on a black background; Opb = opacity of the film overlaid on a white background.

First, the specimens were cut (5 cm x 2 cm), kept at a temperature of 23 °C ± 2 °C and relative humidity of 50% ± 5% for 48 hours. They were then submitted to traction using a universal mechanical testing machine (INSTRON 3367 Series, Grove City, USA) at a speed

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of 500 mm/min. After the tensile tests, the values of strength (MPa), elongation (%), and modulus of elasticity (MPa) were calculated (ASTM, 2010).

For the measurement of the gas barrier property, the specimens were first to cut into a 7 cm diameter circular shape and kept at a temperature of 23 °C ± 1 °C in a desiccator for 48 hours (ASTM, 2009). The carbon dioxide permeability (CO_2) of the specimens was evaluated using a *permeability testing machine* (Labthink, model PERME Vac 1, China).

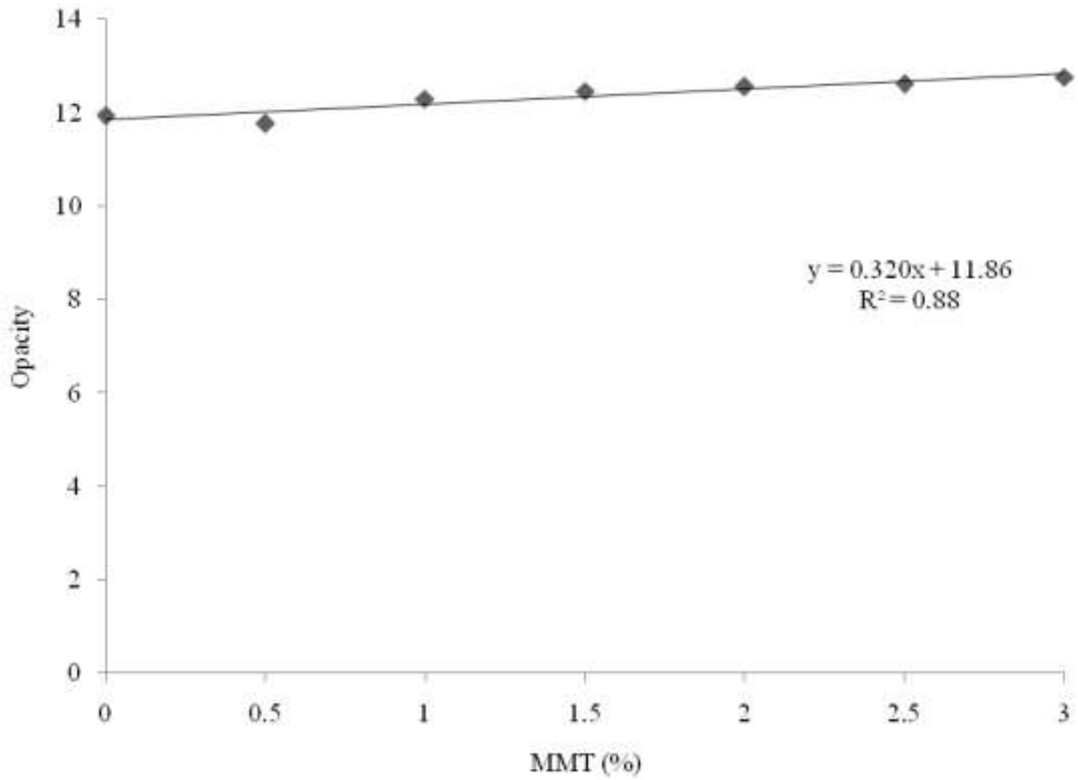
A diffractometer (Lab X XRD 6000, Shimadzu brand) was used to perform X-ray diffraction tests on the films. The films were exposed to a Cu Kα radiation with angular intervals of $2\Theta = 2^o$ and $2\Theta = 10^o$, at a speed of 1°/min, 40 kV of voltage, with an intensity of 20 mA. The basal spacing between the MMT layers was derived from the peak position (d_{001}) observed in the diffractograms, according to the Bragg equation: $2d\ sen\Theta = n\lambda$; where: n = diffraction order; λ = wavelength of the incident radiation; d = distance between atomic planes; Θ = diffraction angle.

The experiment was carried out in a completely randomized design with ten replications performed for each of the mechanical, optical and permeability properties tested. Data obtained from the treatments performed were submitted to regression analysis using the Statistica 7.0 program (Statsoft, 2004). The data from the mechanical tests were submitted to an analysis of variance (ANOVA) and the differences between the treatments were evaluated by the Tukey test, at 5% of probability.

3. Results and Discussion

The opacity of the films ranged from 11.86 to 12.82, with the minimum value recorded in the control and the maximum opacity in the 3% MMT films. The latter presented a significant increase of 8.09% in opacity, showing that an increase in the concentration of nanocomposite results in less transparent films (Figure 1). Earlier studies corroborate with these results, as they observed that the addition of clay caused an increase in the opacity of the films (Ghasemi et al., 2012; Morales et al., 2010).

Figure 1. Opacity of low-density polyethylene films incorporated with 0, 0.5; 1.0; 1.5; 2.0; 2.5 and 3.0 % (w/w) of the Cloisite® 10A nanocomposite.

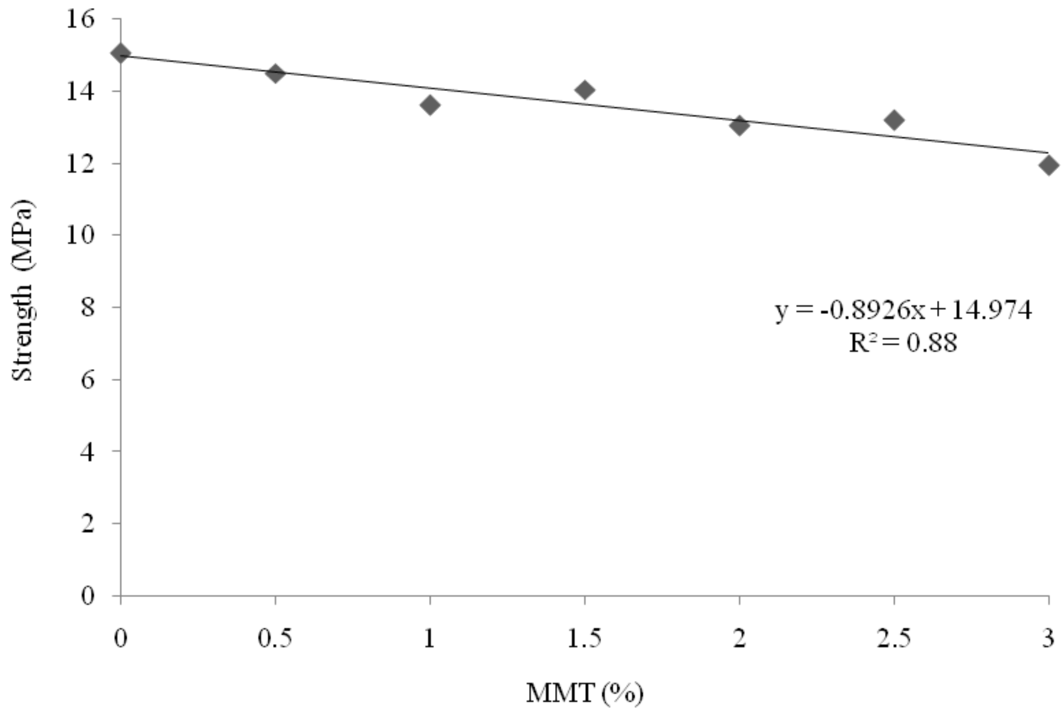


Source: Authors.

The use of more transparent packaging enables a clearer view of the exposed product, so the contents are clearly visible for the consumer. However, some products need protection from the light, which can cause oxidation in some types of food leading to nutrient losses, discoloration, and the development of an odor (Jorge, 2013). These light-sensitive products require the use of opaque packaging, thus enabling the application of montmorillonite clay in the packaging.

The tensile strength ranged from 14.97 to 12.29 MPa from films with 0 to3% of MMT respectively, showing a decrease of approximately 18% (Figure 2). As shown in previous studies, the variation in tensile strength values may be attributed to the breakage of particles or even to an aggregation of the clay layers (Paiva et al., 2006). The decrease in tensile strength was probably a result of the high content of MMT during the preparation of nanocomposites, leading to an aggregation of MMTs in the formed chain (Liu & Tu, 2011). The improvement in the mechanical properties is caused by the polymer-clay interaction and often results in a much greater effect on the modulus of elasticity when compared to the material strength (Zhong et al., 2007). In some cases, variations in tensile strength can be observed (Zehetmeyer et al., 2012).

Figure 2. Strength tensile values (MPa) of low-density polyethylene films incorporated with 0, 0.5; 1.0; 1.5; 2.0; 2.5 and 3.0 % (w/w) of the Cloisite® 10A nanocomposite.



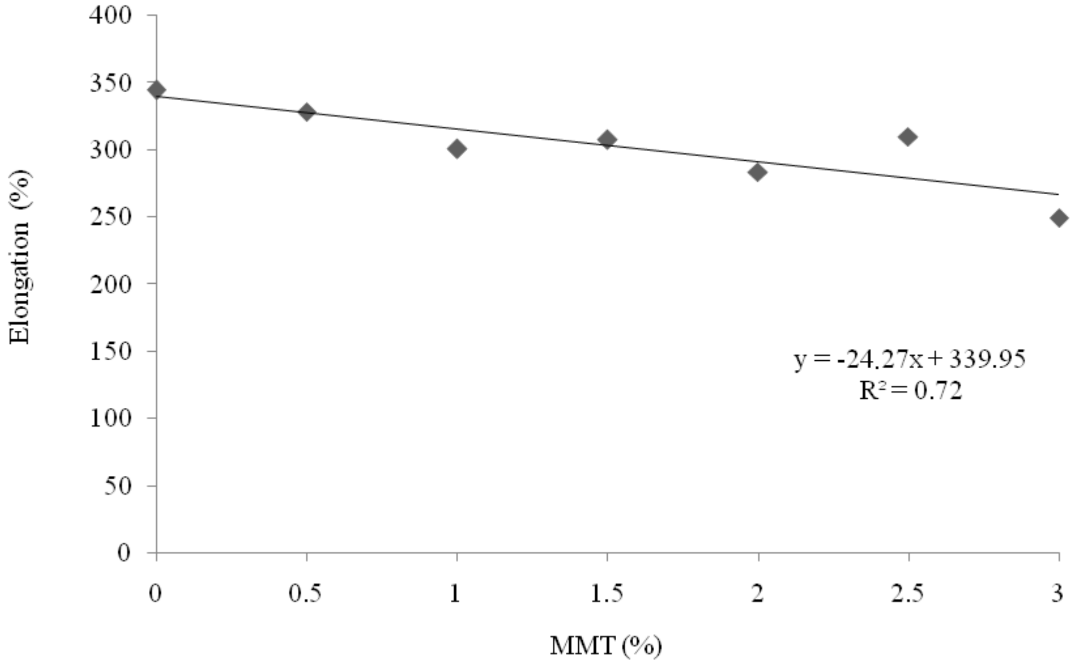
Source: Authors.

Interleaved structures are more easily found in n*anocomposite systems* formed by *melt*, as a result of the low polarity of the polyethylene and the difficulty of inserting their long chains into the clay galleries. Therefore, improvements in tensile properties are minimal (Nikkahah et al., 2009).

The results obtained in this study were different from some reports that showed improvements in the mechanical properties of films containing low clay content, below 3% (w/w) (Pegoretti et al., 2007; Araújo et al., 2006).

The elongation decreased with the increase in the incorporation of clay, showing that the addition of 3% MMT resulted in a decrease of approximately 21% in the elongation of the films when compared to the control film (Figure 3). Studies reported that the addition of clay contributes to the formation of immobilized or partially immobilized polymeric phases, thus interfering with their movement (Paiva et al., 2006). These results were similar to studies that reported reductions in elongation with the addition of clay (Arndt et al., 2017; Hong & Rhim, 2012; Liu & Tu, 2011).

Figure 3. Elongation values (%) of low-density polyethylene films incorporated with 0, 0.5; 1.0; 1.5; 2.0; 2.5 and 3.0 % (w/w) of the Cloisite® 10A nanocomposite.



Source: Authors.

There were no *statistically significant differences between the values of* Young's modulus between the treatments ($p>0.05$). Therefore, there was no change in the stiffness of the films after the addition of clay. These results are related to the weak bonds between the molecules in this region, where elongation and maximum force reactions are reversible (Melito & Daubert, 2011). After the addition of clay, it was expected that the modulus of elasticity values would increase (Ghasemi et al., 2012; Deshmanea et al., 2007; Zhong et al., 2007). However, the non-interference in the stiffness of the films through the addition of nanocomposite was reported by some studies in which Young's modulus remained unchanged (Zehetmeyer et al., 2012; Morales et al., 2010).

In the analysis of barrier properties using CO_2, the permeability rate increased after an increase in the nanocomposite concentration, which shows that the nanoparticle did not act as

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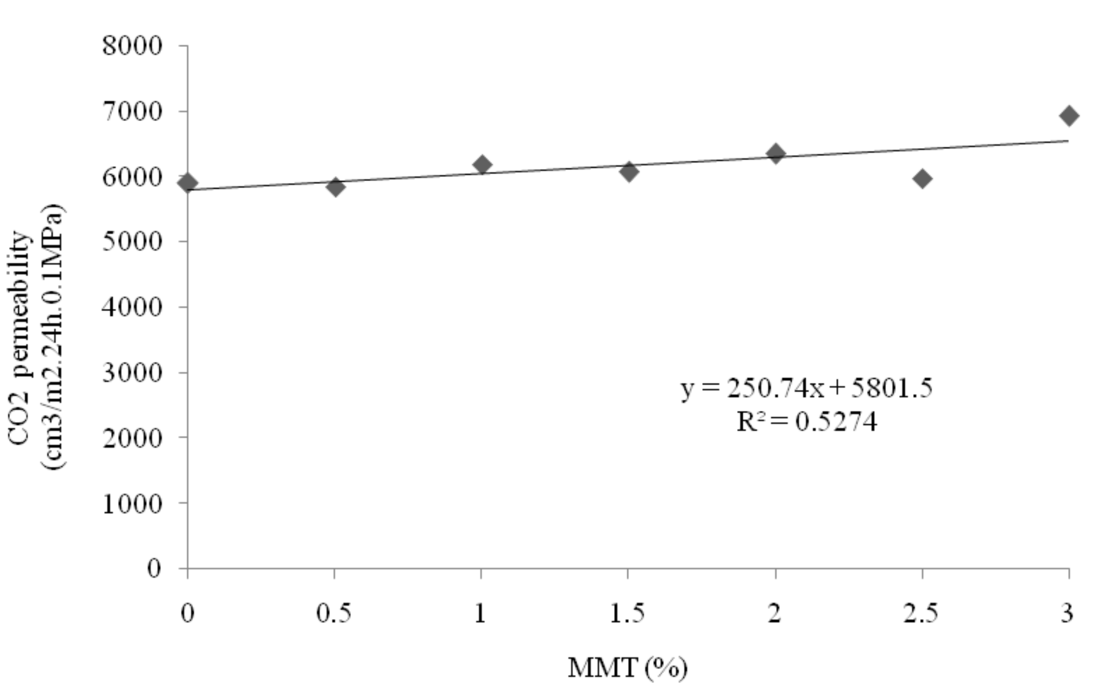
a barrier to the gas passage. In the film containing 3% clay, a 15% increase in carbon dioxide permeability was observed when compared to the control LDPE, indicating that the clay nanoparticles enhanced the gas permeation through the polymer matrix (Figure 4).

Figure 4. CO_2 permeability values of low-density polyethylene films incorporated with 0, 0.5; 1.0; 1.5; 2.0; 2.5 and 3.0 % (w/w) of the Cloisite® 10A nanocomposite.



Source: Authors.

The barrier property is directly related to the degree of clay exfoliation in the polymeric matrix. The greater extent of organophilic clay intercalation, the lower permeability of the CO_2 (Frounchi et al., 2006). When exfoliation levels are not satisfactory, agglomerated structures are formed which allow the easier permeation of gaseous molecules and vapors.

Studies showed that molecular diffusion is one of the main factors related to the flow of vapor and gases through the polymeric membrane with no cracks. In this case, the oscillations between the polymer segments occur through an empty space, allowing the permeation of the molecules (Mali et al., 2010).

Therefore, the obtained results differ from studies in which the addition of clay acts as a physical obstacle in slowing down or decreasing the gas diffusion through the formed film (Zehetmeyer et al., 2012; Morales et al., 2010; Golebiewski et al., 2008; Zhong et al., 2007).

The peaks obtained from the films and nanocomposite are concentrated in the 4th to 5thregions. The values of 2Θ and basal spacing are also summarized in Table 1.

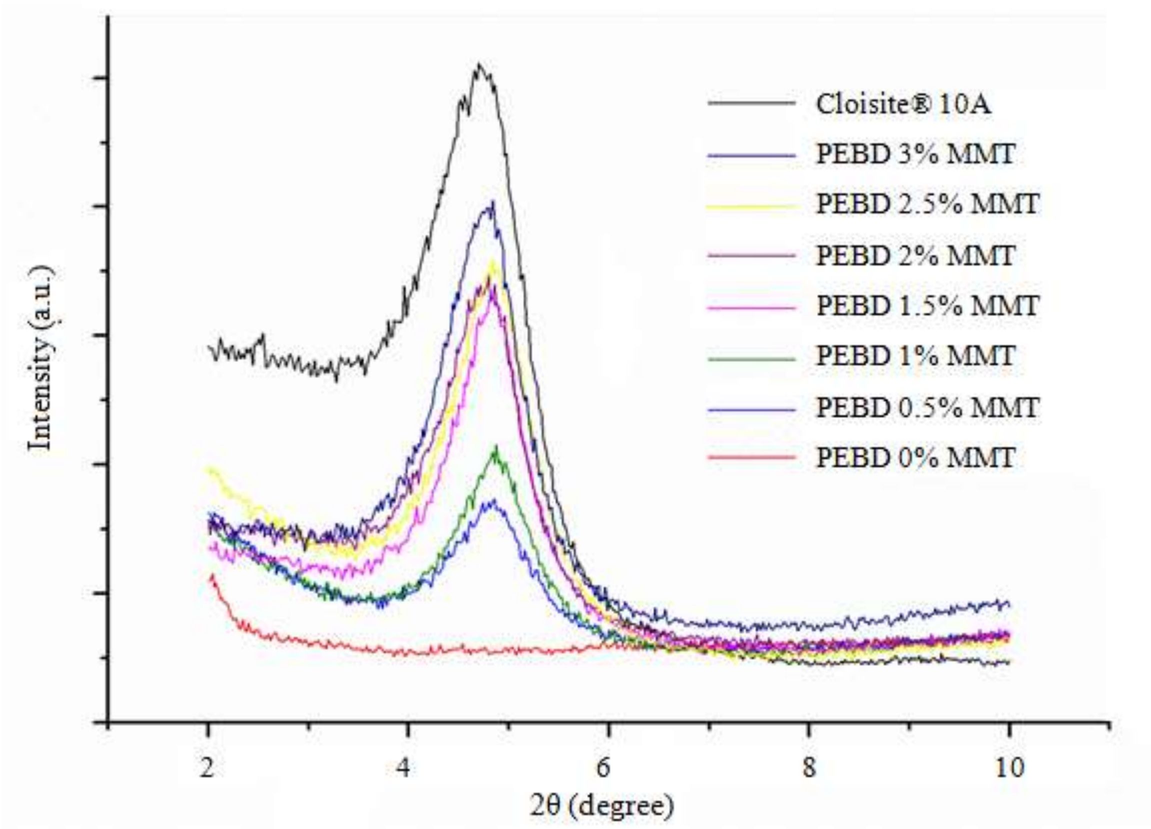
Table 1. Values of 2Θ and basal spacing for the Cloisite® 10A sample and low-density polyethylene films incorporated with 0, 0.5; 1.0; 1.5; 2.0; 2.5 and 3.0 % (w/w) of Cloisite® 10A nanocomposite.

Sample	**2Θ (degrees)**	**Basal Spacing (Å)**
Cloisite® 10A clay	4.6599	17.811
PE 0	0	0
PE 0.5	4.8129	17.245
PE 1	4.8501	17.113
PE 1.5	4.7980	17.299
PE 2	4.7181	17.592
PE 2.5	4.8166	17.232
PE 3	4.7224	17.560

Source: Authors.

The montmorillonite Cloisite® 10A presented a peak at 4.6599^{o}, which corresponds to the 17.811Å (001) basal plane spacing diffraction (Figure 5). The extrusion of the mixture of clay and polyethylene did not cause a significant change in the peak, remaining close to the clay. The basal spacing also remained close to 17Å. The intensities of the peak increased with increased concentrations of clay. No peaks and curves were observed in the control film, thus highlighting its absence of nanocomposites.

Figure 5. X-ray diffraction of Cloisite® 10A sample and low-density polyethylene films incorporated with 0, 0.5; 1.0; 1.5; 2.0; 2.5 and 3.0 % (w/w) of the Cloisite® 10A nanocomposite.



Source: Authors.

Reductions in peak intensity and the angle implies better nanoparticle dispersion in the matrix. The wider the peak opening, the greater the degree of exfoliation (Khalili et al., 2013).

The results showed that a satisfactory interleaving process between LDPE and Cloisite® 10A clay was not achieved. It can also be said that with the increase in clay, the intercalation process remained practically constant during all concentrations developed, thus forming similar microcomposites. Therefore, during the extrusion process, clumps of clay were formed, which contributed to minimizing the mechanical properties of the films, as also observed by the tensile test. X-ray spectra do not always change considerably when the clay content increased by up to 10% in weight. A small effect on the intercalation and independence of the basal spacing of clay was observed when using this range of nanocomposite incorporation (Zhong et al., 2007; Fornes et al., 2001). Increasing the basal spacing (d_{001}) of clay added films and decreasing the peak values d_{001} indicate penetration of

PE chains between clay layers, suggesting the formation of nanocomposites (Zhou et al., 2012; Morelli & Filho, 2010; Zhong et al., 2007). In general, the disappearance of diffraction peaks indicates possible exfoliation of the clay platelets, with a peak widening considered as the result of partial exfoliation (Zhong et al., 2007).

Some studies showed no nanocomposite formation, whereas the d_{001} plane of *organophilic* clays remained practically unchanged, when compared to the peaks obtained from clays inserted in polymeric matrices (Silva et al., 2011). In order to obtain a nanometric dispersion of silicates in polymeric matrices, strong interactions between the clay and the polymeric chains are required (Sánchez-Solís et al., 2004). The high hydrophobicity of the polyethylene reduces the compatibility of the clay with the polymer chains, requiring the use of a hydrophobic clay to increase their compatibility (Hong & Rhim, 2012).

4. Final Considerations

The incorporation of clay into low-density polyethylene films increased the opacity and reduced the tensile and elongation resistance. The stiffness of the film was not changed with the addition of MMT in the range of concentrations evaluated. The permeability to CO_2 increased and the intercalation between LDPE and Cloisite® 10A clay was not satisfactory. New studies will help to comprehend if the changes caused by the MMT on the properties of the polymer affect the preservation of foods packaged by it.

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