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Use of Compression Molding for the Production of Ternary Films Based on Poly (Lactic Acid), Montmorillonite, and Oregano Essential Oil

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Abstract: Poly (Lactic Acid) (PLA) is considered one of the most promising polymers. However, neat PLA films have limitations. An effective strategy to overcome these problems is incorporating clay or essential oils. Cloisite 30B (C30B) is an organoclay widely used to improve the properties of polymers. Notably, the development of films incorporating Oregano Essential Oil (OEO) has attracted significant attention. Compression molding manufactured neat PLA, PLA/C30B, and PLA/C30B/OEO films in this manuscript. The visual evaluation indicated that the films had a good surface finish, and the films thickness varied between 0.15–0.19 mm. The moisture content increased with the incorporation of C30B and OEO. Optical microscopy showed a good distribution of clay particles. The transparency of the films increased with OEO, while with C30B, it presented greater opacity. Incorporating C30B/OEO in the PLA matrix is a promising film proposal that can be directed to the packaging sector. However, other analyses must be done better to understand the films performance for such an application.
Keywords: Compression molding; Films; Oregano essential oil; Organoclay; Poly (Lactic Acid).

1. Introduction

The food industry has focused on developing plastic films for food packaging due to their low cost, low density, and excellent practicality [1–4]. Traditional food packaging has contributed considerably to food distribution systems. However, this model has become increasingly ineffective, requiring the development of innovative packaging [5–7]. Nowadays, scientific research has focused on producing new packaging containing additives with antimicrobial or antioxidant activity [8, 9], and the evidence is that Essential Oils (EOs) are the most used natural additive to increase the shelf life of foods [10–12]. EOs are natural aromatic, antioxidant, and antimicrobial substances extracted from plants through physical processes, and their composition may vary according to their origin [13–15]. EOs of different spices, such as oregano, basil, cloves, rosemary, thyme, garlic, ginger, cinnamon, fennel, and nutmeg, extend the shelf life of packaged foods. According to Ordoudi et al. [16], the quality and certification of authenticity of EOs must be ensured through reliable analysis methods, such as gas chromatography. Among EOs, Oregano Essential Oil (OEO) (*Origanum vulgare*) has efficient antimicrobial activity against various food-borne microbial strains [17–19].

Current scientific research focuses on producing sustainable packaging materials based on polymers from renewable sources, considering consumer health concerns and recurring environmental issues [20, 21]. Typically, biodegradable polymers consist of aliphatic and aromatic polyesters, demonstrating exponential growth in several increasingly sustainable applications [22]. For these reasons, biodegradable packaging materials have been drawing attention as an alternative to films produced from non-renewable resources [23–27]. Poly (Lactic Acid) (PLA), an aliphatic polyester obtained from carbohydrate sources such as corn starch and sugar cane, appears as one of the most promising biodegradable polymers to replace conventional polymers in the production of films [28–30].

Despite many desirable characteristics, PLA performance is unsuitable for packaging due to its relatively low thermal stability, poor gas barrier properties, low ductility, and toughness. These properties, however, can be improved by incorporating layered silicates into the polymer matrix. Montmorillonite (MMT) is one of the most used layered silicates in developing polymer/clay-based materials, as it can improve the mechanical and barrier properties of polymer matrices. MMT has layers of the 2:1 type, with a layer height of almost 1 nm and a distance between layers of less than 1 nm. The 2:1 clay forms when one aluminum octahedral layer occurs between two silica tetrahedral layers [31–35]. The modification of MMT with quaternary ammonium surfactants is called “Cloisite”, with Cloisite 30B (C30B) being widely used in biodegradable polymers [36–38]. Research on the production of ternary systems with a combination of natural additives and clays in biodegradable polymer matrices is still emerging, creating an ideal scenario for research in this area.

The materials used in this manuscript were approved by the “Food and Drug Administration” (FDA), meaning they can be used in the formulation of food packaging without putting human health at risk. According to the “European Food Safety Authority” (EFSA), incorporating up to 12% organophilic montmorillonite clay in the packaging is permitted and not considered a health risk [39]. Therefore, incorporating C30B and OEO in the PLA matrix is an exciting option. Since this is a promising system for application in food packaging, this manuscript aims to develop PLA/C30B films with and without adding OEO. The characteristics of films containing these additives were compared to those of neat PLA films. The films produced were analyzed by visual evaluation, thickness, moisture content, optical microscopy, and visible light barrier (transmittance) were reported.

2. Material and methods

2.1 Materials

PLA trade name of Ingeo™ Biopolymer 2003D, produced by NatureWorks (Minnetonka, Minnesota, EUA), was used as a matrix. The filler employed was a commercial organoclay provided by Southern Clay Products (Gonzales, Texas, USA), labeled Cloisite 30B. This organoclay has a cation-exchange capacity (CEC) of 90 meq/100 g clay and is organophilized with the surfactant MT2EtOH (methyl, tallow, bis-2-hydroxyethyl, quaternary ammonium chloride). According to the FERQUIMA Technical Report (Vargem Grande Paulista, São Paulo, Brazil), the OEO employed has carvacrol (72%), gamma-terpinene (4.5%), linalool (4%), para-cymene (4%), and thymol (2%) as its main components.

2.2 Preparation of PLA/C30B systems and production of films

Before processing, PLA and C30B were dried in an air convection oven operating at 60 °C for 24 h [40]. A PLA/C30B (85%/15% w/w) masterbatch was prepared in an internal mixer (Rheomix 3000, HAAKE™) with a fill factor of 70% and roller-type rotors at 180 °C, 50 rpm for 7 min. These operational conditions were chosen based on Zembouai et al. [41]. The material obtained was ground in a knife mill from PROJEMAQ Engenharia. The ground material was dried at 60 °C for 24 h in an oven and then diluted in the polymer matrix in quantities necessary to produce systems with 2, 4, and 6% w/w C30B in a bench-top single screw extruder (26 L/D), Model AX-16, AX Plásticos, operating with a temperature profile of 170, 175, and 180 °C and screw speed of 50 rpm. Figure 1 shows the pelleted materials according to the defined C30B contents.

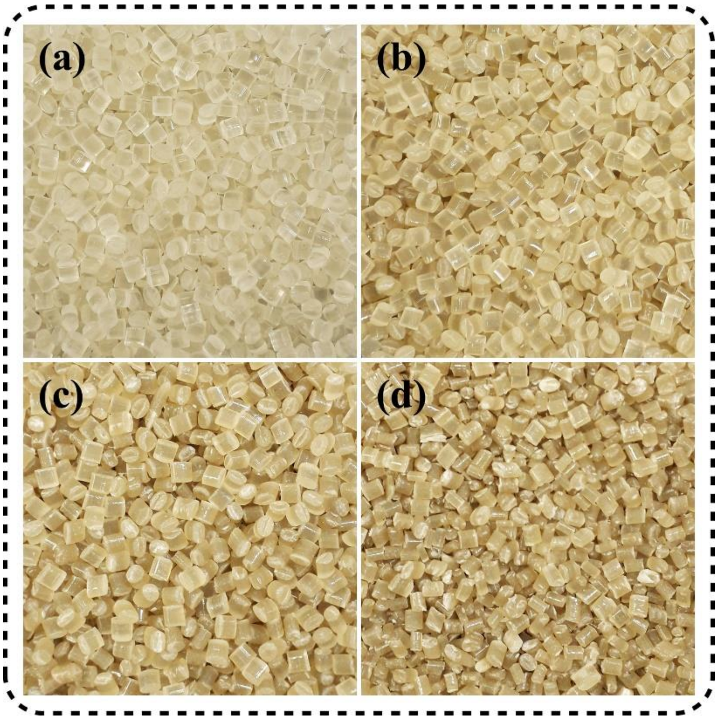

Figure 1. Systems: (a) neat PLA, (b) PLA/2%C30B, (c) PLA/4%C30B, and (d) PLA/6%C30B.

Table 1 shows the materials employed, their contents, and coding. The contents of C30B and OEO used were determined based on Ketkaew et al. [42].

Table 1. Systems produced and respective coding.

Systems	Content			Coding
	PLA (%)	C30B (%)	OEO (%)	
Neat PLA	100	0	0	P
PLA/2%C30B	98	2	0	P/2C
PLA/4%C30B	96	4	0	P/4C
PLA/6%C30B	94	6	0	P/6C
PLA/2%C30B/2%OEO	96	2	2	P/2C/2O
PLA/4%C30B/4%OEO	92	4	4	P/4C/4O
PLA/6%C30B/6%OEO	88	6	6	P/6C/6O

For the production of films using compression molding, the concentrates were dried in an air circulation oven at 60 °C for 24 h. The material needed for the production of each film was deposited between two Teflon sheets positioned over an aluminum plate and then compression molded at 170 °C in a hydraulic press (Model SL-11/15, Solab, Brazil) operating for 2 min at atmospheric pressure and, subsequently, at 4 tons for 2 min before demolding. The film produced was allowed to cool for 1 min at room temperature. The same procedure was used to manufacture the films without and with OEO. For the films incorporated with the OEO, an additional step was carried out: the previous contact of the pellets contained in a container with the OEO.

2.3 Film characterization

Tactile and visual analysis of the macroscopic films were performed to select those with good surface finish, without impurities, holes, scratches, and bubbles around the clay particles.

Sample thickness was determined according to the methodology adapted from the manuscript by Souza et al. [43]. A thickness gauge (Model 130.125, DIGIMESS, São Paulo, Brazil) with a 0.01 mm graduation and an accuracy of ±0.02 mm was used to determine film thickness. The analysis was performed on five samples (3.0 x 3.0 cm) for each system, and the thickness was measured at ten different locations of each sample: two in the central part and eight along its perimeter. Average results are reported.

The moisture content (MC) of the films was determined by sample mass loss after drying in an oven as described (adapted) in Medina-Jaramillo et al. [44] and Song et al. [45]. Film samples (1.7 x 1.7 cm) were weighed before and after drying at 105 °C for 24 h, and their MC was determined using Equation 1. Reported results are an average of five measurements per system.

$$\text{Moisture content (\%)} = \frac{(w_i - w_f)}{w_i} * 100 \qquad (1)$$

where: w_i and w_f are, respectively, the sample mass (g) before and after drying.

Optical microscopy (OM) was performed in a binocular optical microscope (Model ICC50 E, Leica Microsystems) operating in the transmission mode, 100x magnification, and 200 μm scale. The analyzed samples were taken from the region between the films perimeter and center.

Light transmission measurements (T%) were performed on a Cary 60 UV-Vis Spectrophotometer (Agilent Technologies) operating at 400 and 700 nm wavelengths. The methodology employed was adapted from the manuscript of Jahed et al. [46]. Film samples (4.0 x 1.0 cm) were cut out from a film sheet, and their thickness was measured at three different locations (at the ends and in the center) in each sample. The average results of ten measurements are reported here.

3. Results and discussion

3.1 Visual evaluation

The visual evaluation of the produced films aimed to verify possible surface imperfections, and the compression molding processing parameters were adequate, allowing the production of films with reduced surface defects. However, residues (impurities) and imperfections (scratches and small holes) were observed in some films. The residues supposedly come from the mixing step between the polymer and the clay through an internal mixer or may be associated with the intrinsic processing conditions in the compression molding step. As seen in Figure 2, the films showed flexibility and did not show brittle behavior when handled, indicating that they have mechanical strength for a possible application in packaging. According to Dong et al. [47], film flexibility is an essential feature in polymer films for food packaging.

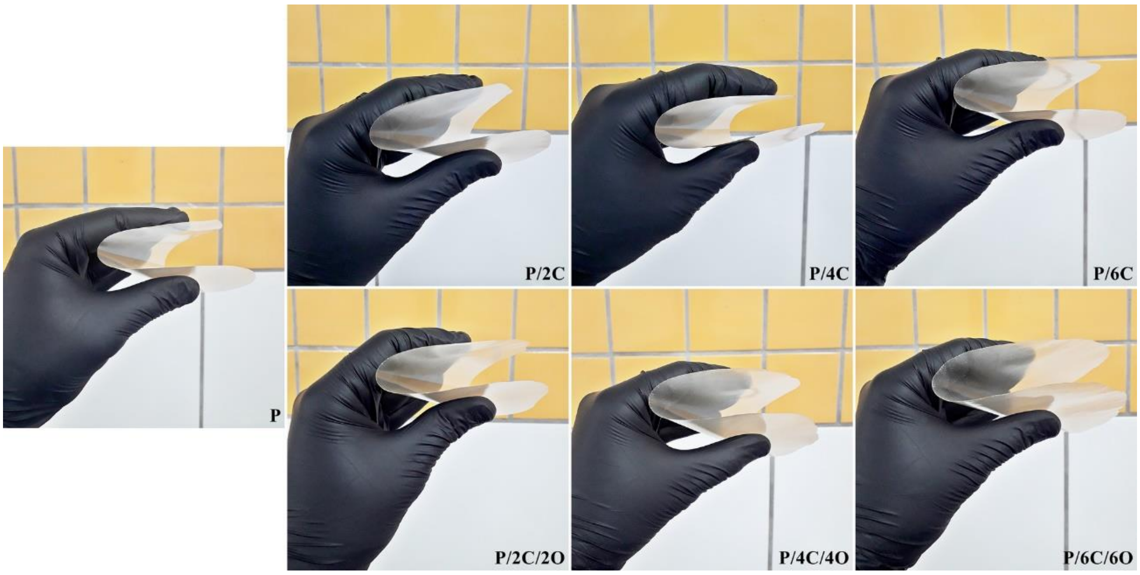

Figure 2. Flexibility of the compression molded films.

Depending on polymer identity, molar mass, degree of crystallinity, and processing conditions, both flexible and rigid polymer films can be manufactured. Flexible films are usually desired in food packaging as the use of rigid, brittle films for these applications is limited [48]. The most common technique used for film production on a laboratory scale is by casting, and some films containing EOs obtained by solution casting are reportedly brittle [49]. In the manuscript reported here, flexible polymer composite films containing OEO were suitably obtained by compression molding. Souza et al. [50] developed cassava starch/cinnamon essential oil films and found that the films became increasingly fragile with higher oil contents. The predominance of a brittle behavior of the film in the presence of higher essential oil contents may be due to a discontinuous structure that produces; as a result, the appearance of voids on the film surface.

One of the main disadvantages of PLA films is their relatively high modulus and low elongation at break [51]. These characteristics can be modified with proper processing conditions and additive incorporation. Byun et al. [52] developed extruded PLA films and obtained a rigid and brittle material. Rhim et al. [53] produced PLA films via compression molding and casting. They concluded that the differences in their mechanical properties were mainly due to the processing technique adopted for their production. The authors also reported that the film flexibility depends on the polymer structure, processing, physical-chemical conditions (temperature, pressure, solvent composition, and concentration), plasticizers, and other additives. The PLA films flexibility in this manuscript is adequate for food packaging applications.

Our data shows that PLA film opacity increased with C30B incorporation and content. Results indicate that, macroscopically, C30B organoclay particles were very well dispersed and well distributed throughout the film, which was attributed to the processing conditions and equipment (internal mixer) employed to produce the films. Salahudeen et al. [54] stated that internal mixers are essential in polymer/clay mixing.

Incorporating OEO into the PLA/C30B films produced fewer uniform films with a larger diameter, supposedly due to the nature of the OEO that acts as a plasticizer [55]. The plasticizing effect of EOs has yet to be thoroughly investigated [56]. However, it is reasonable to assume that the increase in the diameter of the films with OEO is due to its plasticizing effect, as its low molecular weight allows it to occupy the intermolecular spaces between the polymer chains, changing the matrix three-dimensional organization, reducing the secondary forces between them and, consequently, increasing their free volume [57, 58]. After processing, the PLA/C30B films containing OEO (P/2C/2O, P/4C/4O, and P/6C/6O) had a characteristic odor, which indicated that the essential oil was retained in the films. The volatile nature of EOs is one of its main disadvantages, as the oil can exude and affect the flavor of the packaged food due to the migration of its volatile compounds. Although exuding odor, the films obtained in this manuscript did not ooze OEO as the film was not wet or moist to the touch.

3.2 Thickness measurements

The thickness of the films used in packaging plays an essential role in food protection, being a relevant variable in analyzing numerous properties [59]. Figure 3 shows the average thickness value of each film produced.

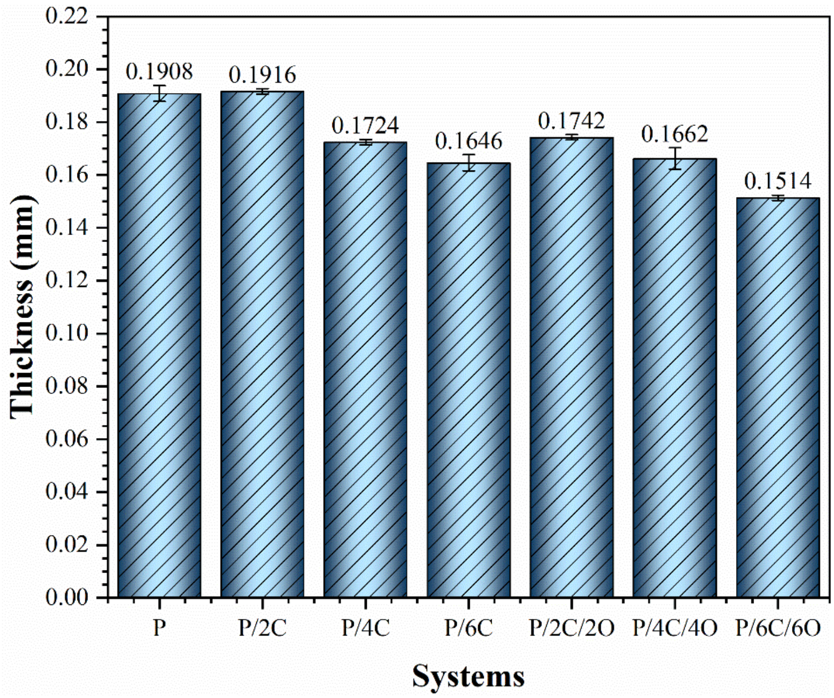

Figure 3. Average thickness of the films produced.

In general, the thickness of the films varied between 0.15–0.19 mm, within the thickness range mentioned by Barlow and Morgan [60]. According to the authors, the thickness of films applied to food packaging varies between 10–250 µm (0.01–0.25 mm) depending on the strength, durability, and barrier function determined by the application. The film thickness must be defined according to its final use, as the food to be packaged is considered.

The average thickness value for the neat PLA films was 0.19 mm. With the incorporation of 2% C30B in the polymer matrix, practically no variation of this value was observed, contrary to the results recorded for the compositions with 4 and 6% C30B that showed reductions of 9.64 and 13.73%, respectively. Incorporating 4 and 6% of C30B in the matrix resulted in larger films than neat PLA and P/2C films. Consequently, the thickness was reduced, suggesting that the C30B particles influenced the flow of polymer chains. According to Wang et al. [61], the alkylammonium chains used in the organophilization of montmorillonite can act as a plasticizer, which may be responsible for the mobility of the polymer chains. However, the authors also reported that this mobility can be influenced by the scission of macromolecular chains that usually occur during intercalation in the molten state. In the molten state, the molecules easily slide past each other, giving the polymer matrix a very high flux under shear [62]. The thickness of the film depends on the composition and parameters used in the processing. In principle, clay particles would be expected to impede the flow of the matrix [63]. Di Maio et al. [64] also observed thickness reduction with increasing clay content.

The incorporation of OEO in the PLA/C30B films resulted in a greater reduction in thickness compared to the neat PLA and clay films, evidencing the influence of OEO on the fluidity of the molten material during processing. A similar result was obtained by Llana-Ruiz-Cabello et al. [65], who developed PLA films incorporated with OEO (2, 5, and 10%) and observed that with increasing OEO content, the thickness was reduced. Rojas-Graü et al. [66] produced apple puree edible films with 0.05, 0.075, and 0.1% OEO. They observed a reduction of about 11.76% in film thickness for the system with 0.1% OEO. Although the OEO reduced the thickness of the films, as previously reported, its use in different matrices also caused an increase in the thickness [67–69].

3.3 Investigation of Moisture Content (MC)

For a material to be used as food packaging, it is essential to evaluate its water retention capacity because when this is high, it can be a relevant factor for the degradation of the packaging that is in contact with food with a high MC [70]. According to Xu et al. [71], this property indicates the empty volume occupied by water molecules in the film structure. For packaging applications, analysis of the films performance in contact with water or water vapor is essential, as the high-water content of the packaging can accelerate the oxidation of the packaged food [72]. The MC of the films was quantified, and the result is shown in Figure 4.

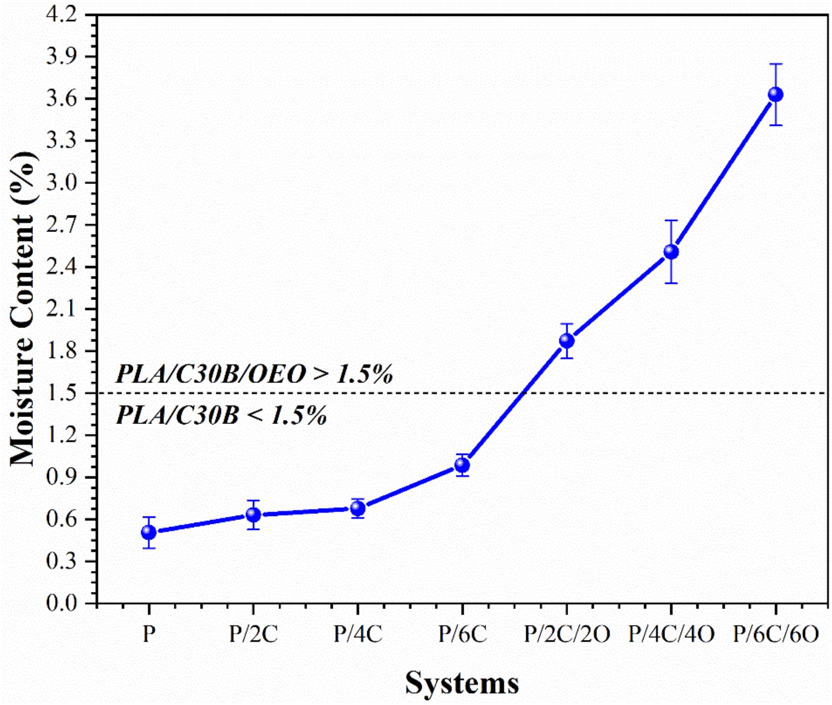

Figure 4. MC of PLA films containing C30B and OEO.

In materials used as food packaging, the water molecules retained in the film structure can influence its physical properties, as they can affect the molecular interactions of the matrix [73]. According to the results obtained, the neat PLA films presented a value of 0.51%, attributed to the hygroscopic nature of the PLA resin [74]. Hygroscopic materials can absorb moisture through hydrogen bonds between water molecules and oxygen groups in the polymer chain. The values corresponding to the MC for PLA resins are around 0.5% [75]. The quantification of MC for films produced from PLA is essential since the main form of degradation of PLA occurs by hydrolysis [76–78]. Despite the relevance of moisture penetration in evaluating the performance, degradation, and life cycle of polymer products, the available literature on moisture transport in PLA to packaging is still quite limited [79].

MC increased by incorporating 2, 4, and 6% of C30B in the matrix, resulting in values of 0.63, 0.68, and 0.99%, respectively. In the same way that PLA has a hydroxyl radical in its chemical structure, C30B also has this radical in the structure of its organic modifier, so with its incorporation, there is a greater availability of hydroxyl to interact through hydrogen bonds with water molecules. After the organophilization treatment, the montmorillonite clay has lower polarity than the natural clay (untreated). However, there are still polar groups on its surface even after the treatment, allowing the interaction through hydrogen bonds of the inorganic clay layers with the water molecules. Therefore, this understanding can also justify the result obtained. The ideal moisture content applied to the package will depend on the type of food being packaged, as each food interacts in a specific way with the package. The high-moisture film is more effective for packing watery food, and the low-moisture film is more effective for packing greasy food [80].

The result obtained for the PLA/C30B films indicated that the formation of strong chemical bonds between the clay and the matrix did not occur. This understanding can be reinforced by the results of Achachlouei and Zahedi [81] and Salarbashi et al. [82], who concluded that the MC decreased with clay incorporation due to strong hydrogen bonds between clay particles and polymer matrix molecules. Moisture influences food spoilage processes, such as lipid oxidation, microbial growth, non-enzymatic browning reactions, and pigment destruction [83].

The incorporation of OEO to the PLA/C30B films resulted in a more expressive increase in the MC of the films, mainly for higher OEO contents, resulting in values of 1.87, 2.51 and 3.63% for 2, 4 and 6% OEO, respectively. Reports on the literature on this subject need to be more conclusive. Although an analogous behavior was observed in the manuscript by Ortiz et al. [84], Lyn and Hanani [85] reported that essential oil incorporation caused a reduction in the films MC. It is believed that different interactions occur between the film matrix and the phenolic compounds present in the OEO. The extent of these interactions depends on the surface characteristics of the matrix employed and leads to an increase, decrease, or no effect on MC. The increase in humidity can cause undesirable effects on the products, namely, cookies lose their crunch, powdered foods agglomerate, and minimally processed vegetables alter their structure due to mass loss [86]. The observed increase in film MC with OEO incorporation was surprising as EOs are considered hydrophobic. Possible explanations for the observed results are: 1) despite being hydrophobic. These oils have some hydroxyl groups in their structure, which could interact with water molecules, and 2) the observed irregularities in film surfaces with OEO addition could assist in trapping water molecules.

3.4 Morphological evaluation by Optical Microscopy (OM)

Figure 5 shows the OM of the surface of the films, which allowed the visualization of the distribution of C30B particles in the PLA matrix of the PLA/C30B films.

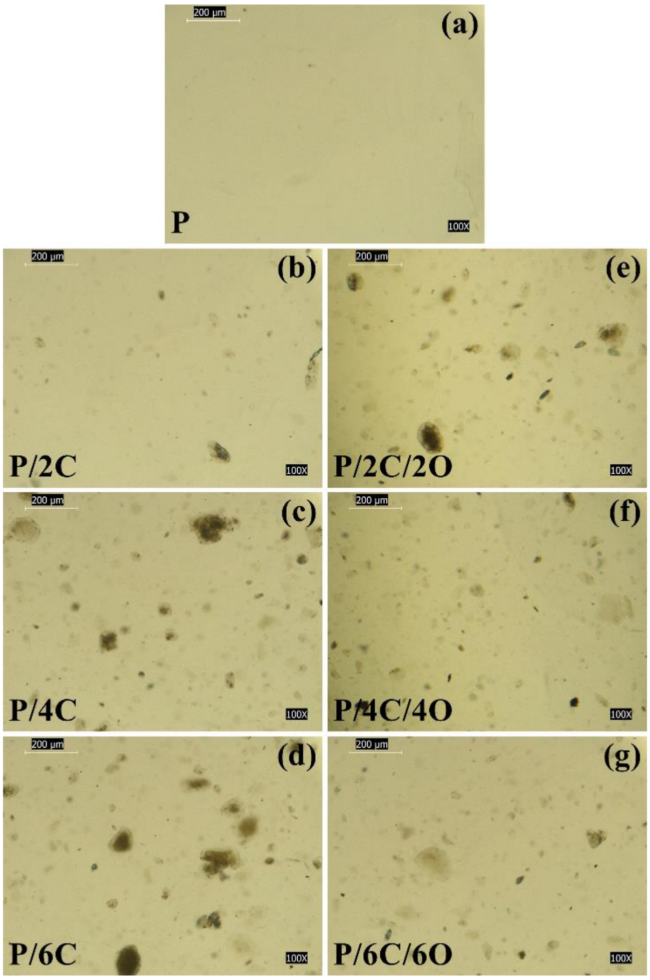

Figure 5. OM images of the film surfaces.

Obtaining a homogeneous dispersion of the material used as reinforcement is one of the most important requirements to improve the properties of polymer/clay materials [87]. The neat PLA sample (Figure 5.a) presented a smooth surface without voids or bubbles; however, some impurities were visualized. Regarding the PLA/C30B samples, there was a similar morphology regarding the absence of bubbles but with agglomerates of clay particles. The increase in clay particle content in the PLA matrix influenced the size and quantity of clay agglomerates. However, all systems showed good distribution conditions along the film area. In the manuscript by Arjmandi et al. [88], montmorillonite did not disperse uniformly in the PLA matrix with a content of 7 phr (parts per hundred resin), which generated agglomerates. Typically, clay agglomerates reduce mechanical properties as they act as stress concentrators. The agglomerates in the matrix reduce the surface interactions between the clay particles and the polymer, resulting in lower tensile strength [89, 90].

Although the results obtained by OM did not allow for a detailed analysis of the effects of OEO incorporation on the morphology of PLA/C30B films, they did indicate that rougher and irregular surfaces were obtained. Scanning Electron Microscope (SEM) studies reported in the literature indicate that essential oil incorporation into polymer matrices led to surface morphological changes on films. Cao and Song [91] found that OEO increased film surface roughness, and Liu et al. [17] concluded that in 3, 5, and 7% of OEO, the films showed grooves and pores on the surface. Therefore, even though the surface analysis of the films surface by OM could not provide indepth conclusions, their contribution was relevant to this manuscript, as they allowed us to observe that C30B particle distribution in the PLA polymer matrix was good.

3.5 Influence of C30B and OEO on visible light transmittance (T%)

For applications in food packaging, optical properties are important factors, as it is desirable to visualize the packaged product, in addition to directly influencing consumers acceptance of the product [92, 93]. Film transparency can be quantitatively determined in light transmittance (T%), a parameter related to the films behavior in transmitting or scattering light [94]. Figure 6 shows the results of the spectrophotometric scan of the films produced through T% measurements.

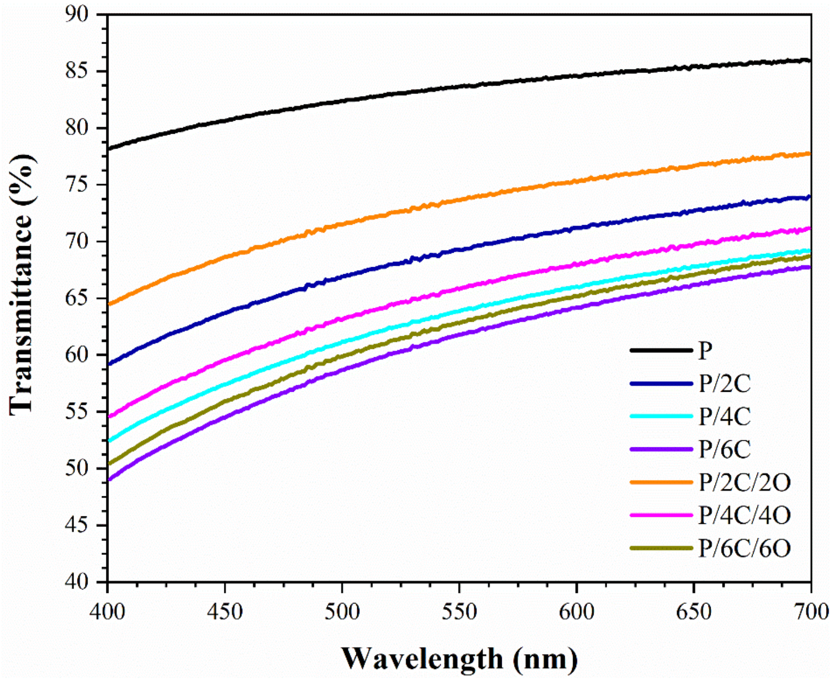

Figure 6. Transmittance of the films in the visible light spectrum (400–700 nm).

It was possible to observe through Figure 6 that the neat PLA films presented the highest T% with an average value of 83.16%, which corroborates the research by Othman et al. [95], who observed higher T% for the film produced from neat PLA compared to films incorporated with montmorillonite and halloysite. High transparency allows consumers to see the packaged food. The results showed that the T% of the films incorporating C30B and OEO in the PLA matrix was reduced, indicating a more opaque profile. The film that presents high opacity may not be the most suitable to be marketed as packaging material [96]. However, the high T% can cause accelerated decomposition of photosensitive foods [97]. The transparent materials applied to the packaging allow light to be transmitted more quickly, causing the loss of nutrients from food, especially milk, fruit juices, and oils [98]. It is important to emphasize that applying transparent or opaque films will depend on the type of food that will be packaged.

The T% of neat PLA films decreased linearly with increasing C30B content in the matrix, corresponding to mean values of 68.46, 62.93, and 60.72% for P/2C, P/4C, and P/6C films, respectively. This reduction in T% can be explained as a result of the clay layers stacking, making it difficult for light to pass through them. The OM (Figure 5) showed that our films had some C30B clusters, which also contributed to the reduction of T%. The films incorporated with 6% of C30B showed the highest opacity. However, this characteristic does not eliminate its potential for application in food packaging, as this opacity can be desired for food protection from light.

It is a fact that using EOs can induce changes in the films appearance and transparency/opacity. For the evaluated systems, it was observed that the average T% value increased to 72.88 (P/2C/2O), 64.94 (P/4C/4O), and 61.84% (P/6C/6O) in relation to the films incorporated only with clay, indicating that the oil improved the transparency of the films. The films incorporated with 2% of OEO showed higher T% than those with 4 and 6%; however, the low C30B content in the matrix also contributed to this result. Differences in the thickness value may have caused differences in the T% of the films with the OEO. The thickness can play an essential role in determining the T% values. However, this result cannot be attributed only to the value of the thickness of the films. It is also essential to consider the C30B content in the matrix.

4. Conclusions

Our data shows that, in general, the films obtained exhibited good surface finish and good flexibility. Films with thicknesses ranging from 0.15–0.19 mm were obtained. OEO films were thinner than neat PLA or PLA/C30B films. MC increased with C30B addition and content, which was more significant in the presence of OEO. OM indicated a good distribution of clay particles in the matrix with few aggregates. However, the effect of OEO addition to the PLA/C30B films was not so evident. Film transparency decreased with organoclay addition and slightly increased with OEO incorporation. Combining C30B and OEO in the PLA matrix for application in food packaging is promising. Nevertheless, other analyses must be conducted to better understand film performance and applicability.

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