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MORINGA OLEIFERA OIL AND POLYMER IN MIXTURES WITH COMMERCIAL POLYMERS – COMPARING STUDY

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ABSTRACT

Composites may be obtained by the mixture of two materials, which one of them is a polymer, with the objective of produced a material with a specific characteristics. Vegetable oils (VO) are mixture with conventional polymer to improve biodegradation capacity without loss mechanical properties. VOs polymers are an alternative to conventional polymers on composites. Previous studies showed that the mixture of Moringa *oleifera* polymer (PMO) with polyethylene (PE) and biodegradable polymer (PB) producing a composite with high biodegrading capacity maintaining mechanical properties. M. *oleifera* oil (MO) was obtained from extraction of seeds in and presents higher concentration of instauration fatty acids as oleic acid (71%) which has high stability and favors the polymerization process. The polymerization assisted by microwaves technology can be used in organic synthesis as a function of the process efficiency and the increase in the selective heating rate. The polymerization of MO assisted by microwaves produced a polymer (PMO) with molecular weight higher than 50,000 g.mol⁻¹. This manuscript shows comparing results for biodegradation behavior and mechanical properties of mixtures by MO or PMO with low density polyethylene and the commercial biopolymer composed by poly (butylene adipate-co-terephthalate) (PBAT) with poly(lactic acid) (PLA). It was verified that MO presented a plasticizer behavior on mixture LDPE/PBAT/PLA (PM) and PMO presented a compatibilizer behavior with PM. It was also verified that mixtures with PMO presented better biodegradation and mechanical properties than those composed by MO.

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INTRODUCTION

Biopolymers can be based in renewable or synthetic resources by fermentative process or chemical reactions (Vieira *et al.*, 2011). The poly (lactic acid) (PLA) is produced by fermentative process and the poly (butylene adipate-co-terephthalate) (PBAT) is produced by polycondensation reaction (Akrami, 2016).

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PLA is having attracted attention due to its stiff, biodegradation and biocompatibility characteristics (Al-Itry *et al.*, 2014). PLA is also one of the most explored bioplastics due to its high modulus and tensile strength that are similar to poly (ethylene terephthalate) (PET), however PLA is brittle which limited its applications. PBAT is a co-polyester that presents similar mechanical behavior to polyethylene besides being biodegradable, hydrophobic and flexible (Al-Itry, 2014; Marinho *et al.*, 2015; Al-Itry *et al.*, 2012). Mixtures formed by biopolymers with conventional polymers are not always miscible and is necessary the use of some compatibilizer as additives, vegetable oils, plasticizers and co-polymers.

Vegetable oils (VOs) are a promising raw material for the production of polymers (Miao *et al.*, 2014). They are base for monomers with similar structure of the petroleum. VOs don't show similar structure of polymers, but they are base for synthesise of polymers as polyurethane, polyester and polyolefins (Miao *et al.*, 2014). VOs can be polymerized by several types of chemical reactions or by thermal process such as microwave irradiation (Aguilera *et al.*, 2016; Sinnwell, 2007). The microwaves irradiation (MW) technology is used in organic synthesis and presents advantages as safety, speed, effectiveness and rate enhancement by selective heating (Da Porto, 2016; Zhang, 2004; Leveneur *et al.*, 2014; Yu, 2007). In most studies, the reactions assisted by MW had reduced the reaction time and increase the rate reaction in compare with conventional heating (Da Porto, 2016). Polymerization under MW is becoming common for synthesis of polyesters, biodegradable polymers and ring open reactions due to the increasing of reactions rates and decreasing reaction times (Gaines, 2015).

M. oleifera oil (MO) is extracted from seeds of *Moringa oleifera* Lam, which is a small tree of northern India (Bhutada *et al.*, 2016). MO presents the oleic acid as fatty acid with higher concentration close to 70%. Oleic acid has high stability due to its low instauration that favors the polymerization process (Fakayode, 2016). VOs can be used as pro degrading additives for commercial polymers but, according to literature, sometimes they had reduced its crystallinity and thermal resistance (Bicalho *et al.*, 2011; Finzi-Quintão *et al.*, 2017). The mixture of VOs with commercial polymers has the objective of producing a material with highest biodegradation capacity (Fang, 2002; Sharmin, 2015). The biodegradation process is the capacity of environment to absorb organic material. It is based in three stages: bio deterioration is the stage where exist the activity of microorganisms; bio fragmentation refers to a cleavage of polymer chain due to action of abiotic or biotic factor, such as metals based additives; and assimilation that also occurs due to these same factors (Emadian, 2017; Tabasi, 2015; Ryan, 2015; Funabashi *et al.*, 2007; Pivsa-Art, 2016). The assimilation stage occurs when small pieces of polymer can be transported into cytoplasm of microorganism to produce energy, biomass and metabolites (Kumar Sen, 2015). The respirometry test can be used to quantify the carbon dioxide during to assimilation process (Montagnolli *et al.*, 2009; Ojeda *et al.*, 2009). *Moringa oleifera* polymer (PMO) presented a compatibilizer behavior when was mixed with low density polyethylene (PE) and PBAT/PLA (PB) to produce a composite with highest biodegradation behavior and mechanical properties adequate to production of plastic bags (Finzi-Quintão *et al.*, 2017). *Moringa oleifera* oil (MO) as other VOs presents a plasticizer behavior in the same mixture and in this manuscript the results of mixtures of PM with PMO and MO are compared. In this study we compared the biodegradation behavior and mechanical properties of films composed by MO and PMO with a blend of LDPE/PBAT/PLA in order to evaluate the effect of the oil and its polymer as additive in ternary blends.

MATERIAL AND METHODS

***Moringa oleifera* derivatives:** MO was extracted from seeds of *Moringa oleifera* during 8 hours in Soxhlet extractor in presence of hexane. PMO was obtained by polymerization MO assisted by microwave irradiation.

The polymerization of MO was carried out in a microwave domestic oven with 0.85 kW of potency. This process was carried out during 16 hours, such as 50 mL of MO was submitted to microwaves during 1 hour, cool down of 24 hours up of to complete 16 hours (Finzi-Quintão *et al.*, 2017).

Samples preparing: LDPE was obtained from transparent food bags and biodegradable polymer (PB) from market bags. Mixtures (10g) were produced by *casting* process mixing moringa compound (MO or PMO) with PM in presence of 100 mL of xylene. The mixture of polymers and solvent was heating up to 110 °C with manual stirring during 1 min every 5 minutes. After 120 minutes, the heating was stopped and the mixture stirred until reduce part of excess solvent. After 36-48 hours, the solvent was completely evaporated and the composite was pressed in a hot press machine at 2.5 ton, during 5 minutes and 150 °C. PMO and MO were mixed with PM in specific weight proportions, mixtures were composed by 15%w (weight) of PMO or MO, 35%w of PE and 50%w of PB (M15-35-50 and P15-35-50). The controller film was composed by 50%w of PE and 50%w of PB.

PEPB was carried out by mixture of 2.5g of PE and 2.5 g of PB, according to procedure described above. The weight proportion of MO and PMO in mixtures with PE and PB was investigated considering the highest presence of moringa compound in relation of PE and PB, when moringa compound rate (MC) increased, PE rate decreased maintaining the PB's concentration. [18]. This manuscript compare mechanical, thermal, morphological and biodegrading properties of better compositions of mixtures of MO and PMO with commercial polymers to verify the interaction of these moringa's compounds with commercial polymers. According previous studies better compositions are 15% of MO or PMO, 35% of PE and 50% of PB and named for moringa oil as M15-35-50 and moringa polymer as P15-35-50.

Characterization Analysis: *Thermogravimetric analysis (TG/DTA)* was carried out in TA Instruments, SDT 2960 Simultaneous DTA-TGA model, 20 °C.min⁻¹, in inert (N₂) atmosphere, interval: 20-700 °C. *Gel permeation chromatography (GPC)* was carried out in a Shimadzu LC-20AD Model, solvent: THF, column: 1 Waters linear e 1 Shimadzu GPC 803, flow: 1.0 mL.min⁻¹, injection: 20 µL, conc.: 0,2 % (p/v). *Fourier Transform Infrared Spectroscopy (FTIR)* was carried out on FT-IR System Spectrum GX/Perkin Elmer, wavenumber range of 4000-400 cm⁻¹, resolution: 32cm⁻¹, scans: 1, using 2 mg samples in 600 mg KBR. *Tensile strength test (TST)* was carried out on EMIC DL-2000, Trd18, speed shift: 500 mm/min, load: 200 kgf. ASTM-D1708-13. *Scanning Electron Microscopy (SEM)* was carried out in a Jeol 1200 with EDS (Energy Dispersive X-Ray Detector) (EDX) and Wide X-ray Fluorescence (WXRf), in a X-Ray Fluorescence spectrophotometer, EDX 720/800HS, HV: 25.0kV, det: SE, SEM MAG: 100x – 4 kx.

Contact angle: was carried out by observing of 1 mL of deionized water deposited over the film during 10 min, 20 min and 40 min. According to behavior of drop, film can be classified as hydrophilic or hydrophobic material. *Free biodegradation test:* samples with 0.6 g were buried in containers (1.500 cm³) with compostable soil during 6 months. The container was in ambient temperature and received 100 mL of water every 5 days.

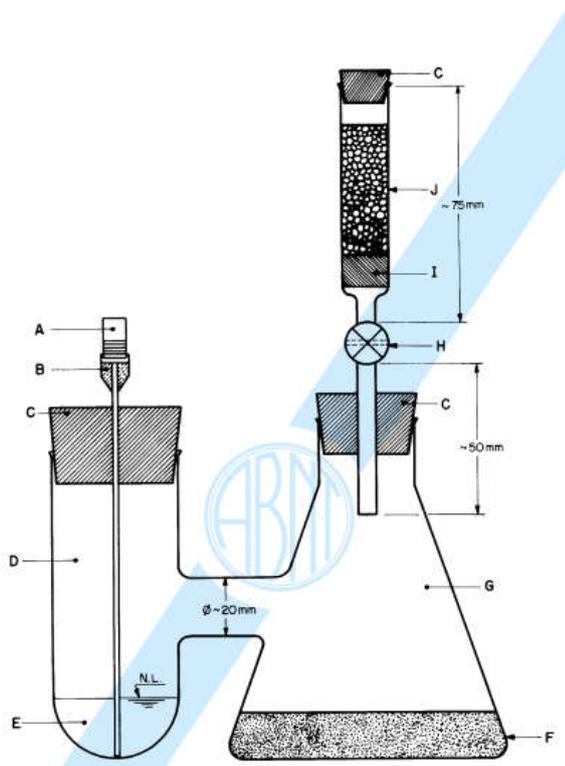


Figure 1- Bartha Respirometer (A- cannula cap; B- cannula (\emptyset ; between 1 mm and 2 mm) with cannon Luer; C_ Rubber stopper; D – Side arm (\emptyset 40 mm ~ H ~ 100 mm); E-KOH solution; F- soil; G- Erlenmeyer flask (250 mL); H – Valve I; I – support (glass or cotton wool); J – ascarita filter (\emptyset 15 mm ~ H ~ 40 mm)) [29]

Table 1. Characteristics numbers of MO and PMO

Value	MO	PMO
Acidity index	0.19	0.53
Iodine index	47.80	24.80
Peroxid index	40.25	91.00

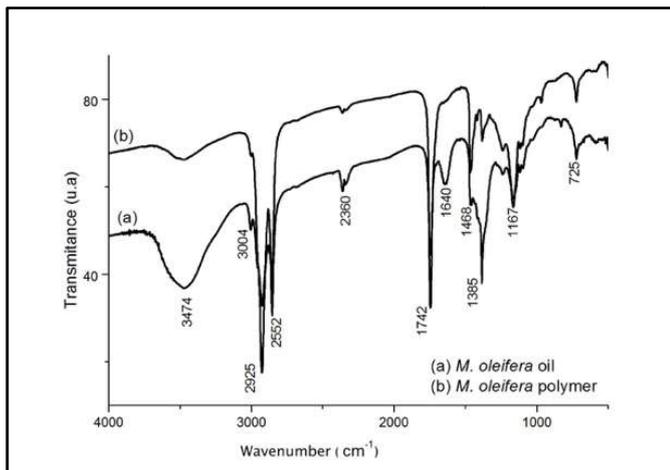


Figure 2 - IV spectra for MO and PMO

The biodegradability capacity of samples was determined by difference of weight before and after incubation. *Bartha Respirometer analysis* was performed according to NBR 14283 – 1999, which specifies the procedures to use the equipment (Figure 1). According to NBR, 0.6 g of samples was buried in 50 g of compostable soil (Figure 1F), under controlled temperature (28°C), free CO₂ during 9 weeks. The biodegradation was verified all day by quantifying CO₂ production with titration of KOH (Figure 1E) converted in

K₂CO₃. After 19 weeks, the weight and morphology of samples was analyzed. Each respirometer must be prepared with 50 ± 1g of soil (humidity controlled between 50% to 70%) (Figure 1-F) disposal on Erlenmeyer flask (Figure 1-G). On side arm (Figure 1D) was deposited 10 mL of KOH (0.2 N) solution using the Cannula with cannon Luer (Figure 1 – B and Figure 1-E) closed with the cannula cap (Figure 1-A). KOH solution reacted with CO₂ produced by residue's biodegradation forming K₂CO₃ used in titration of CO₂. Before close the system was passed pressure are through ascarite filter (Figure 1-J) during 5 minutes, maintaining the cannula cap open. The ascarite filter prevents passage of CO₂ and cleans the system of CO₂. To close system NBR indicated the sequence: first - close the cannula cap, second - close valve 1 (Figure 1-H) and in the third step rubber stopper (Figure 1-H). Bartha respirometer must in a temperature-controlled greenhouse (28 °± 2°C) until completely CO₂ production stooped. Systems with residues/soil were provided in triplicate and also the controller (soil) systems.

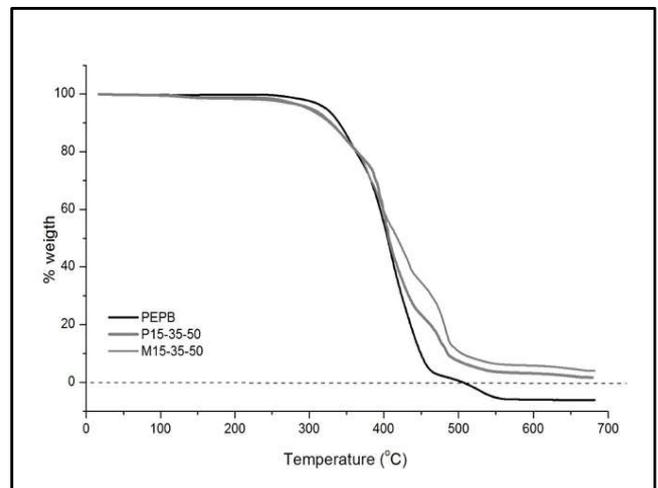


Figure 3 - TGA curves for films composed by PEPB, P15-35-50 and M15-35-50

Table1 - TGA results for PMO, PE, PB, M15-35-50 and P15-35-50

	T _i (°C)	T _d (°C)	T _{max} (°C)
PMO	352.1	402.5 / 411.4	411.4
PE*	429.0	479.1	479.1
issPB*	342.0	360.6 / 388.3	360.6
PB (plastic bags)	347.9	353.9/ 412.2	412.2
PEPB	254.2	405.2 / 431.7	405.2
M15-35-50	307.0	124/254.4/377.8/ 390.2/432.5/483.8	483.8
P15-35-50	333.8	124/399.8/ 494	399.8

* BASF

The titration of K₂CO₃ started after 15 days of respirometers incubation in temperature-controlled greenhouse. The solution presented in Erlenmeyer flask was discarded and substituted by 10 mL of KOH (0,2 N). Day to day, the solution of K₂CO₃ was titration to determinate BI for polymeric residue. According to NBR, CO₂ and biodegraded carbon are calculated as equations bellow. CO₂ production was calculated for soil/residue respirometer (Eq. 1) for controller system (eq. 2) (A: measure of HCL 0,1 N used for white's titration (mL); B: measure of HCL 0,1 N used for respirometer solution's titration (mL); 50: conversion factor for CO₂; f_{HCL}: conversion factor for HCL 0,1 N).

$$\text{mol } CO_{2(\text{soil/sample})} = (A - B) \times 50 \times f_{HCL} \quad (1)$$

$$\text{mol } CO_{2(\text{soil control})} = (A - B) \times 50 \times f_{HCL} \quad (2)$$

CO_{2b} production due to biodegradation was calculated by difference between systems (eq.3)

$$CO_{2b} = \text{mol } CO_{2(\text{soil/sample})} - \text{mol } CO_{2(\text{soil control})} \quad (3)$$

According to NBR, 50% of biodegraded carbon was converted to CO_2 and 50% was incorporated on soil as biomass and due to this, the effective biodegraded carbon is calculating by Equation 4.

$$C_b(\text{mol } C) = 2 \times CO_{2b}(\text{mol } CO_2) \quad (4)$$

Technical information for PLA/PBAT indicate its completely degradation at 180 days. According to NBR 14283-1999, residues which reduced higher than 30 % off its mass under controlled respirometry producing cumulatively CO_2 can be considered degradable and mineralized when C_b constant. Considering technical information for PLA/PBAT that indicate its completely degradation at 180 days, this manuscript present degradation results after 14 weeks of test in Bartha Respirometer.

RESULTS AND DISCUSSION

Moringa oleifera polymerization: According to GPC analysis, MO presents molecular weight close to 1217 g.mol^{-1} and PMO is composed by a mixture of components with high molecular weight up to $50,000 \text{ g.mol}^{-1}$. The increased of molecular weight after microwaves irradiation indicated that polymerization of oil occurred which was also confirmed with iodine number, peroxide number and acidity number (Table 1). The increased of acidity value indicated highest presence of free H^+ due to broken of double bond which was confirmed on expressive reduction of iodine value. The polymerization of MO was verified using FTIR (Figure 2) spectra due to the reduction of intensity transmittance band at 3474 cm^{-1} which refers to OH of triglycerides hydrolysis that composed fatty acids [18]. The transmittance band at 1640 cm^{-1} which refers to double bond ($C=C$) is not present in PMO confirming the polymerization process. Thermal analysis indicated that the presence of MO or PMO enhancement the thermal resistance of mixture of PE with PB. Thermal analyses (Figure 3) showed enhancement of thermal stability in temperatures higher than $400 \text{ }^\circ\text{C}$.

PEPB, M15-35-50 and P15-35-50 presented same melting point of polyethylene at $124 \text{ }^\circ\text{C}$ as seen in the thermal differential analyses. The presence of PLA was observed at $254.4 \text{ }^\circ\text{C}$ of M15-35-50 and PEPB curves; however in this last one there is a principal stage of oxidation at $390.2 \text{ }^\circ\text{C}$ with a large base that indicates an interaction of PBAT with PE (Table 2). The M15-35-50 DTA curve (**Error! Reference source not found.**) indicated the presence of fatty acids (oleic acid, linoleic acid and linolenic acid) in smaller peaks near to $400 \text{ }^\circ\text{C}$ ($390.2 \text{ }^\circ\text{C}$ and $432.5 \text{ }^\circ\text{C}$), which pointed out the interaction of PLA/PABT with MO and with PE, indicated by temperatures near to $483.8 \text{ }^\circ\text{C}$. P15-35-50 presented thermal behavior similar to PEPB and better definition for peaks of temperature. The temperature of principal peak of PEPB was

reduced from $412.2 \text{ }^\circ\text{C}$ to $399.8 \text{ }^\circ\text{C}$ what indicates the interaction of PB with PMO. Tensile Stretch Test (TST) for samples PEPB and M15-35-50 presented similar results of maximum stress and rupture strain (). MO had improved the ductility with similar tensile stress of PEPB, meanwhile PMO improved both tensile stress value and ductility. P15-35-50 presented better results for maximum stress comparing to M15-35-50. The presence of MO and PMO had reduced the stiff of PEPB. PEPB presented higher stiff than mixtures with moringa compounds. MO had improved the ductility with similar tensile stress of PEPB; meanwhile PMO improved both tensile stress value and ductility.

Biodegradation

Biodegradation tests aim to verify if the presence of MO and PMO can improve the biodegradation behavior of M15-35-50 and P15-35-50 (Figure 4). M15-35-50 and P15-35-50 presented rough appearance after both biodegradation tests. Both blends were not fragmented even losing mass after biodegradation tests which indicates that fragmentation stage did not occurs in the studied period. Table 3 data present that M15-35-50 and P15-35-50 have similar loss of weight after Bartha respirometer, however, P15-35-50 produced 31% more CO_2 than M15-35-50. On free test P15-35-50 lost 20% more weight than M15-35-50. Previous studies showed that PEPB lost less than 7% of mass during biodegradation tests (Finzi-Quintão, 2016). The SEM micrography of PEPB (Figure 5) showed cracks which confirming stiff behavior indicated by tensile stress test. The morphology for M15-35-50 (Figure 6) showed structures like fibers which are due to interaction of PEPB with the moringa oil. Figure 6 showed a homogeneous morphology for P15-35-50, without fibers or structures what indicated the interaction of PEPB with PMO. The micrography of M15-35-50 after the biodegradation with Bartha Respirometry (Figure 7) showed a spongy aspect with the disappearing of fiber structures showed in Figure 6. The micrography of P15-35-50 after biodegradation showed the presence of structures similar to fibers distributed for all film. The micrography of M15-35-50 would indicated better assimilation process than P15-35-50, but its production of dioxide of carbon is higher than M15-35-50 (Table 4). The presence of fibers after P15-35-50 biodegradation indicated that PMO presented a compatibilizer behavior for PEPB, which improved thermal and mechanical properties. Micrographies of free biodegradation test showed similar results, indicating PMO as a better biodegradation additive. Contact angle test for PEPB and M15-35-50 samples at initial time when angles are close to $90^\circ \pm 3^\circ$ indicating film's hydrophobicity. At initial time P15-35-50 and M15-35-50 presented similar for contact angle. After 20 minutes, the slide of drops for PEPB and M15-35-50 samples were close to $115^\circ \pm 3^\circ$ and for P15-35-50 was close to $126^\circ \pm 3^\circ$. After 32 minutes, all drops stopped moving and at 40 minutes was register $123^\circ \pm 3^\circ$ for PEPB and M15-35-50 samples and $138^\circ \pm 3^\circ$ for P15-35-50 sample. According to contact angle test, all films can be considering hydrophobic and the alteration of drop occurred due to infiltration of the liquid into the pores of the film. Material hysteresis is the ability to slide the drop out of the material and can be obtained by contact angle test. The movement of the drop allows classifying the surface as hydrophobic or not. The surface of the material must be homogeneous, otherwise the movement of the drop can occur due to presence of pores, contaminant or chemical non-homogeneity [Luz, 2008].

Table 2 - Mechanical properties of produced films

	Maximum stress(MPa)	Rupture Strain(%)	Elastic modulus(MPa)
PEPB	8.7 ± 1.2	23 ± 3	103,1 ± 1.3
M15-35-50	8.2 ± 0.7	22 ± 4	33,1 ± 12.3
P15-35-50	9.7 ± 0.6	17 ± 2	51,3 ± 15.7



Figure 4 . Biodegradation Results

Table 3. Biodegradation results

	Free test	Bartha Respirometer	
Sample	Loss weight	Loss weight	CO ₂ (mg)
M15-35-50	33 %	24 %	86,76
P15-35-50	51 %	27 %	114,03

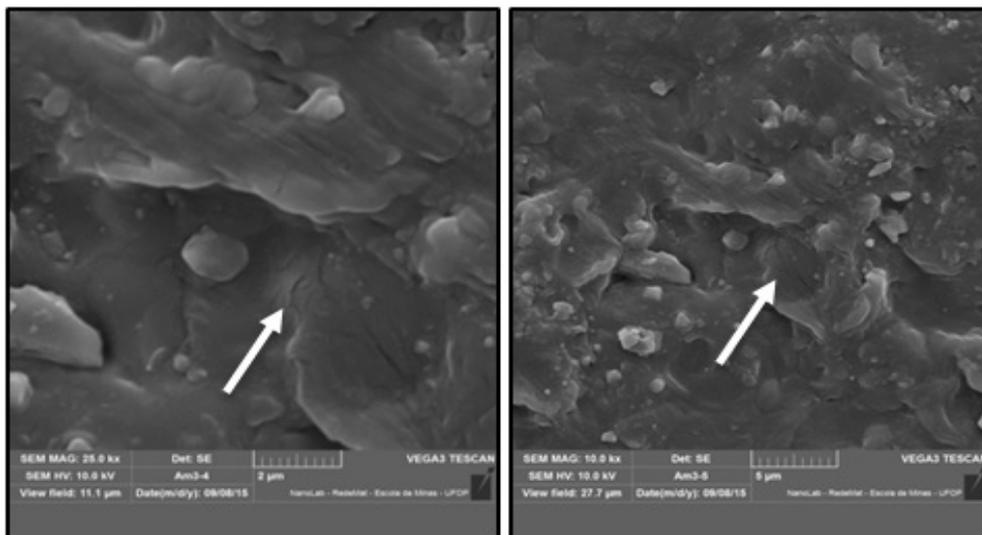


Figure 5. Micrography of PEPB before mixture with moringa compounds

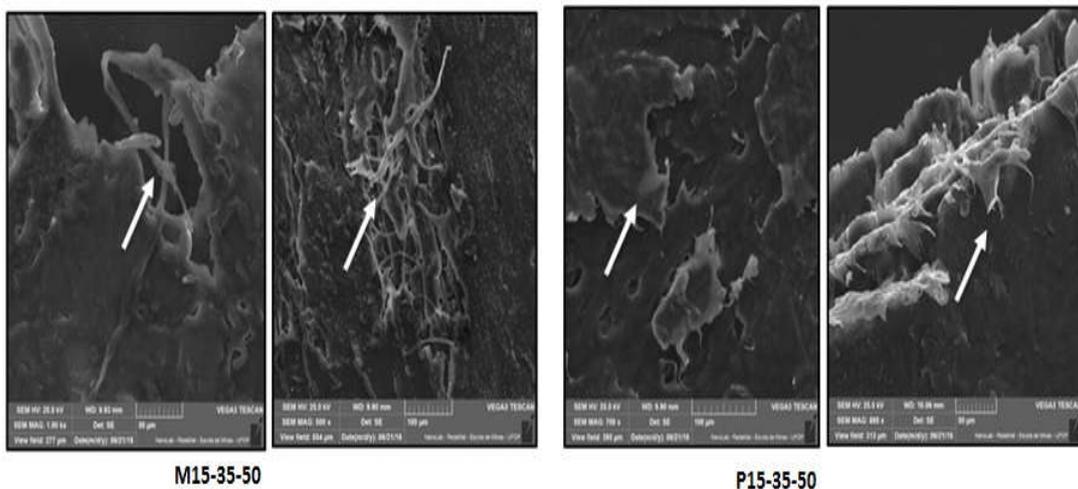


Figure 6. Micrography before Bartha respirometry

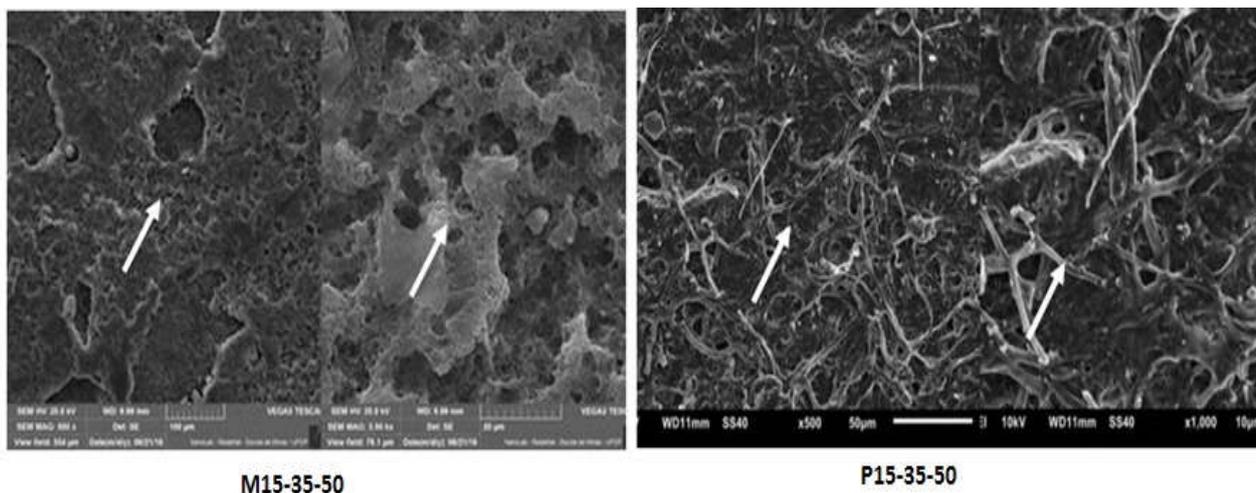


Figure 7. Micrography after Bartha Respirometry

Hysteresis phenomenon for the samples allowed inferring that the material's biodegradation capacity occurred due to action of microorganisms in the porosity. The highest angle of P15-35-50 sample indicated better biodegradation than PEPB and M15-35-50 samples which is confirming by respirometry tests and SEM micrographies.

Conclusion

Respirometry tests showed significant improving of biodegradation behavior using MO and PMO in compare with PEPB. Hysteresis phenomenon allowed inferring that polymer moringa increased the biodegradation capacity due to better porosity for action of microorganisms. Moringa polymer samples presented better results for biodegradation process and mechanical resistance than films with moringa oil.

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