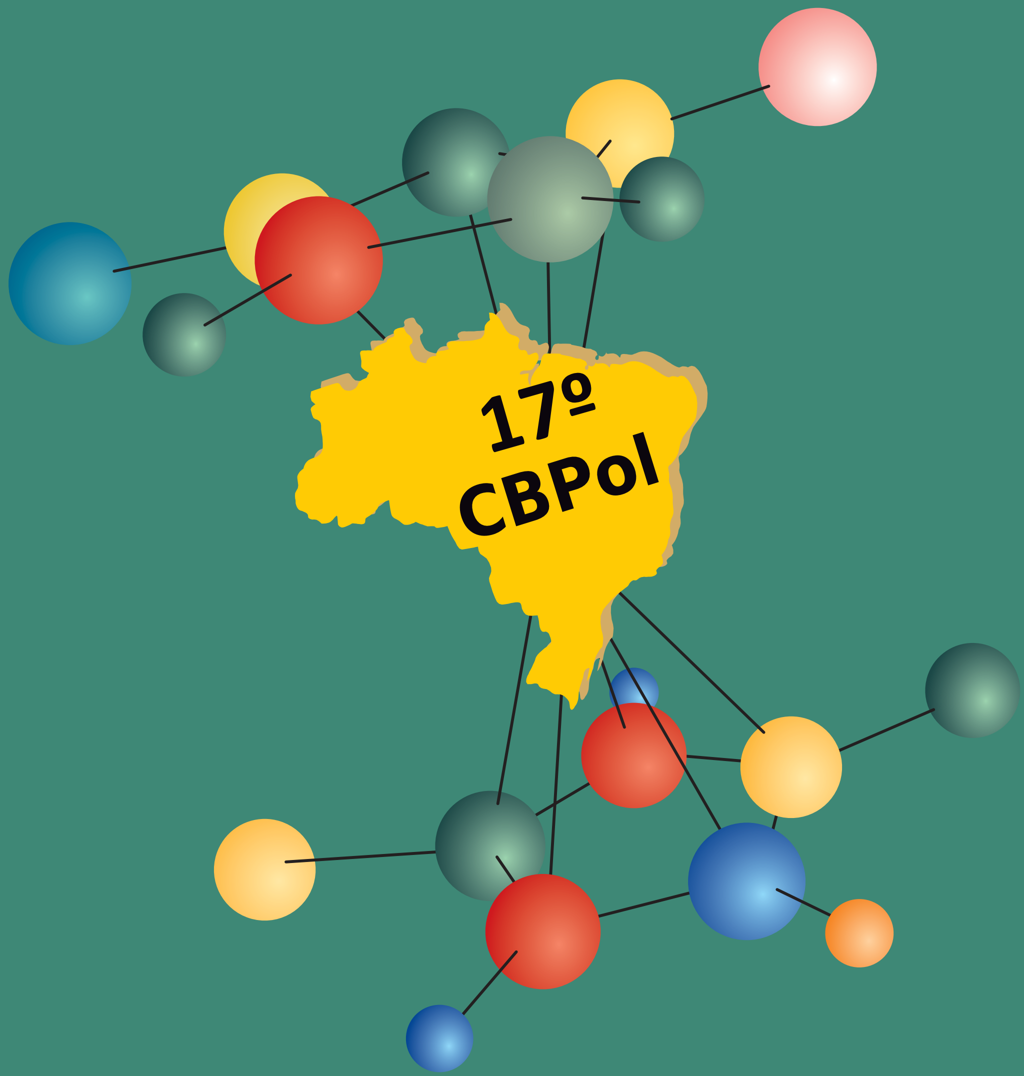

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RICE HUSK FILLED PLA/PBAT COMPOSITES FOR FOOD CONTAINERS: PROCESSABILITY, RHEOLOGICAL AND THERMAL PROPERTIES

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Abstract - This study investigated the potential of using polylactic acid (PLA), polybutylene adipate-co-terephthalate (PBAT), and rice husk (RH) composite as alternative materials for food containers. The composite was prepared using an internal mixer and characterized by torque vs. time mixing curves, melt flow index (MFI), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). The results showed that adding PBAT and rice husks improved the thermal stability of the composite and decreased the melting temperature and crystallinity degree. The MFI decreased with the incorporation of rice husks, indicating good filler dispersion in the matrix. Since it is a byproduct of rice production, rice husk-filled composites can reduce the cost of material production and minimize environmental impact during the packaging life cycle. These findings suggest that the PLA/PBAT/RH composite shows potential as a sustainable alternative to traditional food packaging materials.
Keywords: *PLA, PBAT, rice husk, biodegradable, food packaging.*

Introduction

The widespread use of non-biodegradable materials for packaging applications has led to growing concerns about environmental pollution [1]. The accumulation of plastic waste in the natural ecosystem poses a threat to the environment and has implications for human health and well-being. To address these concerns, it is crucial to explore sustainable alternatives and adopt eco-friendly practices that can help reduce the negative impact of packaging waste on our planet [2].

Biodegradable and compostable materials have emerged as potential alternatives to conventional plastics to address these concerns [1,3]. Polylactic acid (PLA) and polybutylene adipate terephthalate (PBAT) are two biodegradable polymers extensively studied for food packaging applications [1]. However, these materials often have limitations in terms of their mechanical and barrier properties, which can affect their suitability for specific food packaging applications. Also, PLA/PBAT blends can be more expensive than traditional petroleum-based plastics, which may limit their widespread adoption in the food packaging industry [1]. Modifying the composition of the polymer blend, such as incorporating different types of polymers or adding natural fillers, can improve the properties and minimize production costs [3].

A rice husk is a natural sheath formed around rice grains during growth [4]. Rice husks have gained attention as a natural filler in polymer composites due to their abundance, low cost, and desirable properties. As a byproduct of rice production, their use in composites provides a sustainable and environmentally friendly solution [4, 5]. Incorporating rice husks in polymer matrices can modify their mechanical properties, final weight, and resistance to weathering and also make the final product more economically competitive [4]. Overall, using rice husks in polymer composites shows great promise in providing sustainable and high-performance materials for various applications.

In recent years, researchers have explored blending natural fillers with biodegradable material to overcome the costs of polymer processing and improve the properties of biodegradable polymers for food packaging applications [1]. In this study, we investigate using rice husk as a natural filler in a PLA/PBAT composite for food containers. We evaluate how thermal and rheological are affected by the composition of the composite material and assess its potential as a sustainable and biodegradable food packaging material. The results of this study are significant for developing environmentally friendly food packaging materials that can tackle the environmental impact of conventional plastic packaging in the food industry.

Experimental

Materials

The materials used in this study were PLA (Ingeo™ Biopolymer 4043D), PBAT (Ecoflex® F Blend C1200), and rice husk (RH) provided by local producers. The PLA/PBAT blends and composites with rice husk were prepared using a mixing chamber (Thermo Scientific HAAKE™ Rheomix OS) at a temperature of 170°C, 60 rpm and a mixing time of 8 minutes. Therefore, the samples were pressed at 170°C under 6 ton to form the sheets used in the compression molding processing. All the specimens were able to be molded in food trays. The formulations studied were named BL1, BL2, CO1, CO2, CO3, and CO4. The compositions of these samples are presented in Table 1.

Table 1 – Composition of PLA/PBAT/rice husks blends.

Sample	PLA (%)	PBAT (%)	RH (%)
BL1	90	10	0
BL2	80	20	0
CO1	80	10	10
CO2	70	10	20
CO3	70	20	10
CO4	60	20	20

Characterization

Thermogravimetric analysis (TGA) was carried out using a TA Instruments TGA Q50 thermogravimetric analyzer. The samples were heated from room temperature to 930°C at a heating rate of 20°C/min under a nitrogen atmosphere with a 10 mL/min flow rate. Each test consumed samples of about 15 mg.

Differential scanning calorimetry (DSC) was conducted using a TA Instruments DSC Q20 calorimeter. The samples were heated from 25°C to 200°C at a heating rate of 10°C/min, then cooled to 25°C at a cooling rate of 10°C/min, and finally reheated to 200°C at a heating rate of 10°C/min under a nitrogen atmosphere with a 50 mL/min flow rate. About 5 mg of the sample was used for each test. The degree of crystallinity (X_c) of PLA was calculated using Eq. 1[6].

$$X_c = \left(\frac{\Delta H_m - \Delta H_{cc}}{\Delta H_{m_{100}}(w_{PLA})}\right) \times 100 \quad (1)$$

Where ΔH_m the melting enthalpy of the tested sample, ΔH_{cc} is the cold-crystallization enthalpy of the tested sample, $\Delta H_{m_{100}}$ is the theoretical melting enthalpy of the 100% crystalline (93 J/g) [6] and w_{PLA} is the weight fraction of the PLA in the sample.

The melt flow index (MFI) was measured according to ASTM D1238-13 standard on a Ceast Modular Melt Flow Tester with a 2.16 kg load and at temperatures of 170, 190, and 210°C. The melt flow index was calculated as the mass of the polymer flowing through a capillary of a specified length and diameter over a specified time.

Results and Discussion

The torque vs. mixing time curves obtained during the preparation of the PLA/PBAT/RH composites are shown in Fig.1. The total torque corresponds to the area under the curve. The mixing torque suffered no alteration as PBAT content in the blends increased, indicating an equal viscosity of the melt for the blends. Overall, the composites filled with rice husk presented higher stabilized torque – measured around 5 min of mixing - compared to the blends with equal PBAT content, which indicates that RH can increase the viscosity of the melt. Thus, the final torque observed in composite CO4 may be due to the higher contents of PBAT (20%) and rice husk (20%).

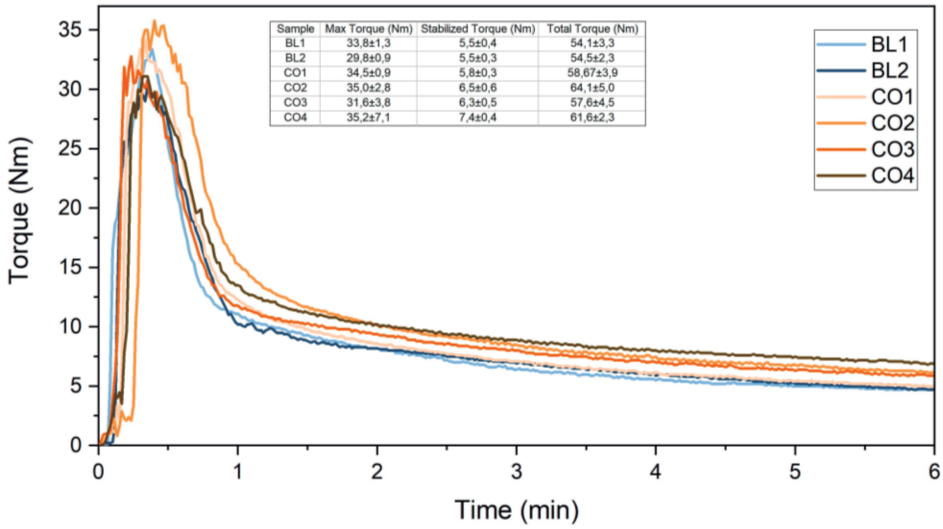


Figure 1 – Torque vs. mixing time of PLA/PBAT blends and RH filled composites.

The MFI of the pure materials, PLA/PBAT blends, and RH filled composites is shown in Fig. 2. The MFI is an indirect indication of the viscosity of the melt; the higher the MFI, the lower the viscosity. All the samples reported lower viscosity in the higher testing temperature (210°C) due to increased molecule kinetic energy. Compered to neat blends, the addition of rice husks led to a decrease in the MFI, indicating an increase in the viscosity of the melt, as already observed during the mixing of samples. This decrease in the MFI may be attributed to the high aspect ratio and low surface energy of the rice husks, which hinder the movement of the polymer chains and reduce the flowability of the melt.

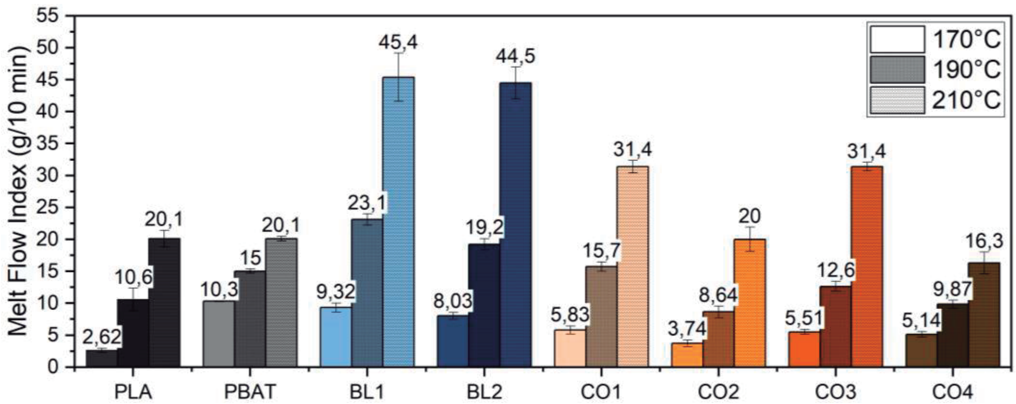


Figure 2 – MFI in three temperatures of pure polymers, PLA/PBAT blends and RH filled composites.

The TGA thermograms of the samples in the study are shown in Fig. 3. It was noticed that PBAT has a higher onset temperature than PLA; thus, the blends and composites with 20%PBAT presented higher thermal stability. PBAT also confers higher residues at 600°C (3.59 wt%), indicating that carbonization occurs more severely for PBAT than for PLA [7]. Albeit CO2 and CO4 showed higher residues, 9.41 wt% and 7.83 wt%, respectively, possibly due to char formation during RH degradation process. The decomposition processes of all the blends and composites could not be distinguished obviously from the TG curves. Two processes of thermal decomposition signaling to PLA and PBAT phases can be observed via DTG curves, where the intensity of degradation each event is related to the weight fraction of the component [7].

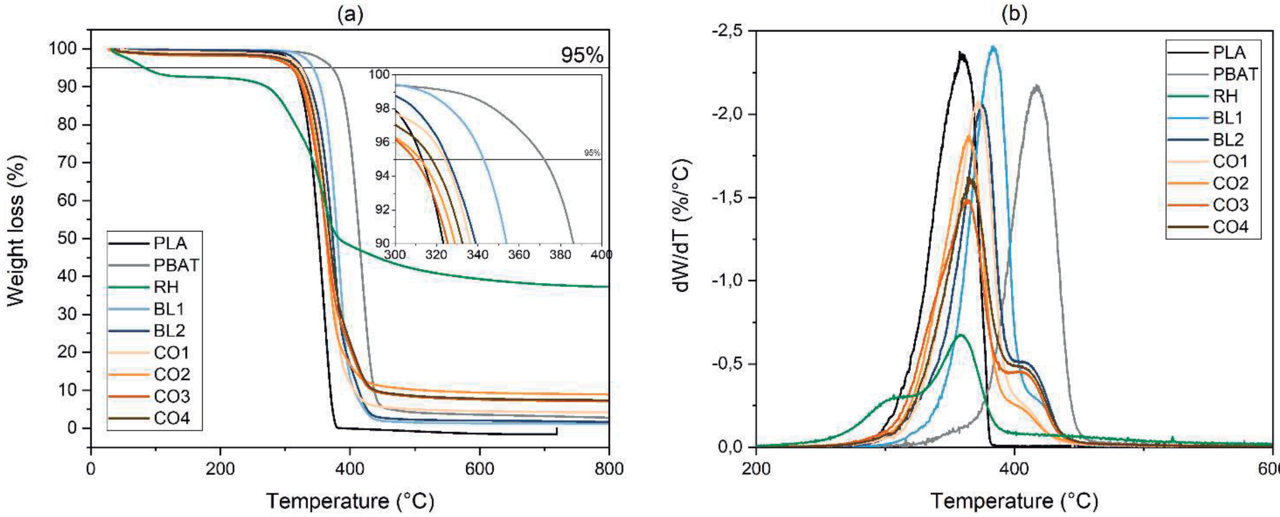
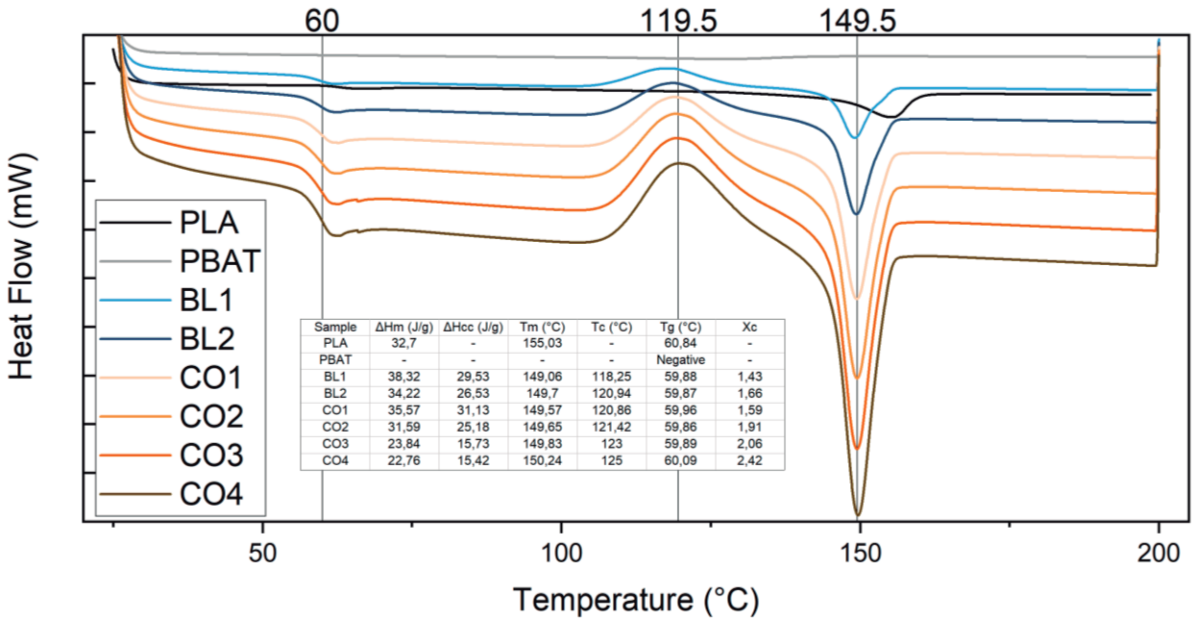


Figure 3 – (a) TG and (b) DTG curves of pure polymers, PLA/PBAT blends and RH filled composites.

Fig. 4 presents the second heating DSC thermograms for the blends and composites. The glass transition, cold crystallization, and melting are distinctly observed in the curves. It is noted that PLA glass transition temperature (Tg) remains constant through the compositions (~ 60 °C), indicating that the other components do not affect the amorphous phase. PBAT has a negative Tg (-30 °C) [6]; thus, the analyses do not observe the event. The addition of rice husk affects the crystallinity phase, where the samples with a higher amount of rice husk (20%) modify the reorganization phenomena of the crystals during the heating run due to the nucleation effect promoted by the presence of the particles. The relative amplitude of cold crystallization and melting peaks determined from DSC measurement showed that these blends were mostly amorphous, with the lower and higher crystallinity degree reported for blend BL1 (1.43%) and composite CO4 (2.42%), respectively.



Sample	ΔHm (J/g)	ΔHcc (J/g)	Tm (°C)	Tc (°C)	Tg (°C)	Xc
PLA	32,7	-	155,03	-	60,84	-
PBAT	-	-	-	-	Negative	-
BL1	38,32	29,53	149,06	118,25	59,88	1,43
BL2	34,22	26,53	149,7	120,94	59,87	1,66
CO1	35,57	31,13	149,57	120,86	59,96	1,59
CO2	31,59	25,18	149,65	121,42	59,86	1,91
CO3	23,84	15,73	149,83	123	59,89	2,06
CO4	22,76	15,42	150,24	125	60,09	2,42

Figure 4 – DSC thermograms of pure polymers, PLA/PBAT blends and RH filled composites obtained from second heating scan.

Conclusions

In conclusion, this study investigated the potential of using PLA/PBAT/RH composites as an alternative material for food containers. The results indicate that adding rice husk to the composites modifies their rheological properties, increasing viscosity and reducing flowability. The composites' thermal properties were also improved, with higher thermal stability related to the amount of PBAT phase. Therefore, the PLA/PBAT/RH composites could be a promising solution for developing sustainable food packaging materials, as they can withstand high temperatures. Further studies are necessary to investigate the mechanical and barrier properties of the composites, as well as their composition for specific applications.

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