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## Material Properties

## PBAT/kraft lignin blend in flexible laminated food packaging: Peeling resistance and thermal degradability

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## ABSTRACT

This work evaluates the peeling resistance of PBAT-kraft lignin (KL) blend films as a laminated food packaging layer. PBAT-KL blend films were obtained by extrusion with 0, 1, 3, 5, and 10 wt% KL. The blend homogeneity and miscibility were investigated by atomic force microscopy (AFM) and dynamic mechanical analysis (DMA), respectively. Contact angle measurements were carried out to quantify changes in surface energy. Multilayer composites were obtained by lamination, in which PBAT-KL blend films were bonded to a polyethylene layer using polyurethane adhesive. Peeling specimens were subjected to aging at 60 °C to evaluate the effects of KL addition on the practical adhesion and PBAT degradation. The DMA results indicated polymer miscibility, and the contact angles increased upon KL incorporation. PBAT-KL could be a synergetic solution for food packaging that can integrate the reuse of industrial waste, mechanical properties, flexibility, peeling resistance, and the well-known biodegradability of PBAT.

## 1. Introduction

Adhesion and composites are closely related since composite phases are linked by adhesive interactions between phases. Composite applications have been increasing in recent years, and adhesion has been receiving special attention [1], particularly in industrial areas such as food packaging, in which composites play an important role. Most materials currently used for food packaging are non-degradable, including polyethylene and poly (ethylene terephthalate), which persist for many years after disposal [2]. It is therefore vitally important to use environmentally and eco-friendly materials instead of traditional plastics for applications in which plastics are used for short periods and then discarded.

Among commercially available biodegradable polymers, poly (butylene adipate-co-terephthalate) (PBAT) is a promising alternative for packaging applications due to its good processability and biocompatibility [3,4]. PBAT is a flexible synthetic aliphatic-aromatic copolyester that features good thermal and mechanical properties, including comparable tensile properties to those of low-density polyethylene (LDPE) [5–7]. However, its relatively high water vapor permeability [8] and high cost restrict its use commercially. To improve the applicability of PBAT, several studies have reported blends with other polymers [9–12], nanofillers [3,13,14], and biomass [5,8] to address the increasing demand for sustainable and renewable resource utilization. Lignin is a

promising and advantageous raw material in this regard.

Lignin is an abundant natural polymer with a cross-linked and complex phenolic structure. As a derivative of native lignin, kraft lignin (KL) is available at a large scale as a by-product of pulping processes such as paper manufacturing by the kraft process. It is normally treated as waste, and its reuse is widely desired for high-value applications. Several studies have reported on the antioxidant [15], antibacterial [16], antimicrobial [17], and barrier [18] properties of lignin, which are important for its potential use in food packaging. It is worth mentioning that industrial lignins including Kraft and Sulfite processes have sulfur residual content and inherent odor, what can be a drawback for active films. However, edible films were prepared from fish skin gelatin and lignin derived from Sulfite process, which proved its antioxidant capacity and low cytotoxicity [19]. The lignin structure contains many functional groups in different portions (phenolic and aliphatic hydroxyls, carbonyls, carboxyls, and methoxyl [20]). These groups enable interaction and compatibility with appropriate polymeric matrices.

The incorporation of KL can help to overcome the disadvantages of PBAT by increasing its vapor permeability resistance. The practical adhesion of PBAT can be improved or at least maintained due to the polarity of lignin. In addition to these benefits, the antioxidant properties lignin mean that KL incorporation can also prevent the degradation of PBAT and thus the degradation of flexible laminated packaging, as long the packaging is not discarded.

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**Table 1**  
Compositions under study.

Code	PBAT (wt%)	KL (wt%)
PBAT_0	100	0
PBAT_1	99	1
PBAT_3	97	3
PBAT_5	95	5
PBAT_10	90	10

The aim of this work is to investigate the behavior of PBAT/KL films as a laminated food packaging layer. As a first step, the miscibility of PBAT and lignin was investigated by atomic force microscopy (AFM) and dynamic mechanical analysis (DMA). The practical adhesion and composite degradability were evaluated by peeling tests before and after aging. This study improves the feasibility of using biodegradable and industrial waste polymers for food packaging applications as a sustainable and environmental alternative.

## 2. Materials and methods

### 2.1. Materials

Technical KL (hardwood – eucalyptus) was supplied by Suzano Papel e Celulose Brasil (Brazil). This lignin was obtained as a byproduct of pulp and paper manufacturing (brown color; pH 3.8; 95% solid content; 2% ash content). Lignin supplier kindly provided the following KL data: mass-average ( $M_w$ ) and number-average ( $M_n$ ) molar mass of 3360 g/mol and 2278 g/mol, respectively; polydispersity of 1.475 ( $M_w/M_n$ ); glass transition temperature ( $T_g$ ) of 106.85 °C; 1.33 mmol/g of aliphatic hydroxyl, total phenolic hydroxyl content of 3.93 mmol/g, which is divided into syringyl (1.81 mmol/g), condensed guaiacyl (0.95 mmol/g), uncondensed guaiacyl (0.99 mmol/g) and *p*-hydroxyphenyl (0.18 mmol/g) units.

PBAT was supplied by BASF Brazil under the trade name Ecoflex® F Blend C1200 (mass density of 1.26 g cm<sup>-3</sup> at 23 °C, melt flow rate (MFR) of 3.3 g·10 min<sup>-1</sup> at 190 °C, 2.16 kg, melting point 115 °C). Corona-treated LDPE films were supplied by Nold Polytech (Brazil). Bicomponent polyurethane (PU) adhesive (diisocyanate Loctite Liofol LA 9526 and biopolyol LA 6145, supplied by Henkel, Brazil) was used for bonding the polymers films.

### 2.2. PBAT and PBAT-KL film preparation

KL and PBAT were dried at 80 °C and 60 °C, respectively, for 24 h before use. KL (0%, 1%, 3%, 5% and 10% by mass) was incorporated into PBAT prior by pre-mixing the compounds in a kinetic type K-mixer (model MH-100, MH Equipment Ltd). The neat PBAT and pre-mixtures

were extruded in a twin-screw extruder (THERMO HAAKE Minilab Rheomex, model CTW5, L/D = 24). The extrusion conditions used included a temperature profile of 132, 135, 135, 138, 138 and 140 °C from the feeder to the die and 60 rpm. After extrusion, the pellets were dried at 80 °C for 8 h and thermo-compressed under a heating press (140 °C, 50 N, 90 s) sandwiched between two Teflon sheets and a steel mold of 0.3 mm. Table 1 summarizes the compositions.

### 2.3. PBAT and PBAT-KL film characterization

#### 2.3.1. Dynamic mechanical analysis (DMA)

The elastic storage and loss moduli of the biopolymers were obtained by DMA using a DMA8000 thermal analyzer (Perkin Elmer). Measurements were carried out in flexural mode in the temperature range of –60 to 110 °C with a frequency of 1.0 Hz and heating rate of 3.0 °C·min<sup>-1</sup>. The dimensions of the specimens were 30 × 12 × 0.3 mm. The measurements were repeated three times for each condition.

#### 2.3.2. Atomic force microscopy (AFM)

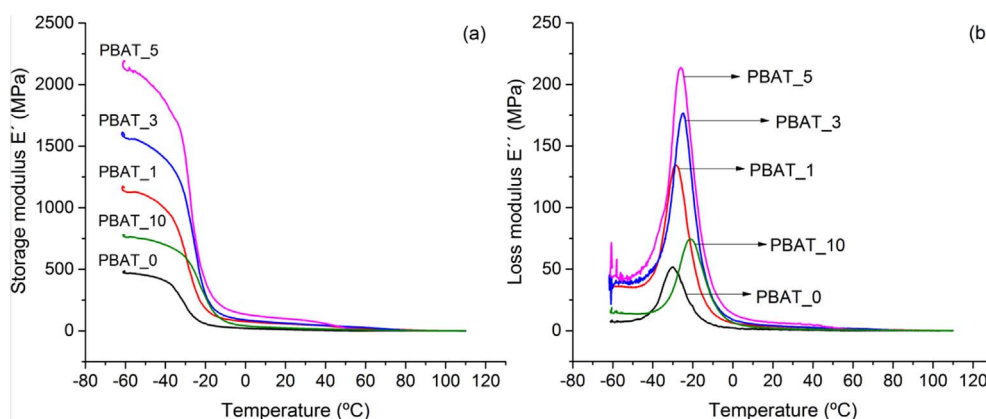
The biopolymers were characterized in air by AFM using a NanoScope IIIa controller (Bruker) in force modulation mode. The controller was operated in tapping with scanning areas of 2.5 × 2.5 μm, a scan rate of 1.0 Hz and images with 256 lines were obtained, resulting into a point to point distance of approximately 9.7 nm. PBAT and PBAT-KL films were individually positioned between stainless steel plates and cut using a high precision diamond knife. The cut transversal surface was used to obtain AFM micrographs.

#### 2.3.3. Contact angle

The surface energies of the biopolymers were determined from contact angle measurements using distilled water and diiodomethane solvents with a test drop volume of 10 μL. The contact angles were measured by the sessile drop method with a Drop Shade Analyzer DSA 100 (Krüss). The measurements were repeated 10 times for each condition taken at a different position on the films. The free surface energy was calculated according to the literature using two liquids with different polarity: water ( $\gamma_s = 72.80$  mN/m,  $\gamma_s^d = 21.80$  mN/m,  $\gamma_s^p = 51.00$  mN/m) and diiodomethane ( $\gamma_s = 50.80$  mN/m,  $\gamma_s^d = 50.80$  mN/m,  $\gamma_s^p = 0.00$  mN/m) [21–23].

### 2.4. Specimen lamination

The lamination process was carried out manually according to a previous study [24]. PBAT and PBAT-KL films (thickness of 0.3 mm) were cut into 14 × 28 cm segments using a template. PU adhesive, mixture of diisocyanate and biopolyol (stoichiometric condition), was dissolved in ethyl acetate at a mass proportion of 50%. Biopolymers



**Fig. 1.** (a) Storage modulus  $E'$  and (b) Loss modulus  $E''$  of neat PBAT and PBAT-KL blends.

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