



# PROCESSING AND MORPHOLOGY OF POLY(HYDROXYBUTYRATE-CO-VALERATE)/ LIGNOSULFONATE COMPOSITES

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In recent years, increased interest in polymers that are environmentally friendly, such as poly(hydroxybutyrate-co-valerate) (PHBV) has been observed due to the rapid rise in the use of polymers, which has impacted negatively on the environment. However, the high cost of PHBV and its brittleness limit its use to a few exclusive applications. The utilization of lignins in biodegradable materials is receiving increasing attention in order to find alternatives to petrochemicals. Traditionally, lignins have been incorporated into various polymeric materials as low cost filler. In this work, lignosulfonate was used as filler in poly-(hydroxybutyrate-co-valerate) (PHBV)-based composites. The composites containing 90:10, 80:20, 70:30 and 60:40 (w/w) of PHBV/Lignosulfonate were prepared by melting mixing. Torque vs time was measured during the processing of materials. A decrease of torque was observed in function of filler content. The morphology of materials was investigated by Energy Dispersive Spectroscopy (EDS). Through EDS the domains of lignosulfonate in the PHBV matrix were identified and the absence of any physical contact between both components was observed, indicating that the interfacial adhesion between filler and matrix was poor.

## Introduction

The use of synthetic polymers has increased in many different industrial sections, mainly in the package industry, due to the high strength, lightweight, low cost, easy processibility, and good water-barrier properties. However, the large volume of plastic materials produced and their fast discard in landfills have caused many problems in the environment due to their non-biodegradability.

Poly(hydroxybutyrate-co-valerate) (PHBV) has been recognized as a potential environment-friendly substitute for traditional plastics<sup>1</sup>. It is produced via bacterial fermentation and can be easily degraded to water and carbon dioxide under different environmental conditions. However, high cost, slow crystallization rate, relative difficulty in processing and high degree of crystallinity are drawbacks for wide PHBV use<sup>1,2,3</sup>.

The addition of filler frequently contributes to obtain a more competitive price and a general improvement in the load-bearing capability<sup>2</sup>. In this way, lignins have been incorporated into various polymeric materials as low cost filler. Enormous amounts of industrial lignin and derivatives are produced as byproducts of papermaking<sup>4</sup>.

In this work, composites based on PHBV and lignosulfonate were prepared by melt mixing. The processing and morphology of composites were examined.

## Experimental

### Materials

PHBV containing HV units of 15 % and a density of 1.263 g/cm<sup>3</sup> at room temperature was supplied by Biocycle. The lignosulfonate Vixilex-SD was supplied by Melbar Produtos de Lignina Ltda., as a brown powder and density of 1.085 g/cm<sup>3</sup>. Both materials were used as received.

### Preparation of PHBV/Lignosulfonate Composites

The composites based on PHBV and lignosulfonate were prepared by mechanical mixing in Haake Rheocord 90 internal mixer equipped with counter rotating rotors at 175°C for 5 min and 50 rpm. The lignosulfonate was added 2 min after the PHBV addition to the mixing chamber. The final compositions of the PHBV/Lignosulfonate composites were 90:10, 80:20, 70:30 and 60:40 (w/w). The materials were ground and flat plaques of 2 mm thick and 5 x 2 cm dimension were prepared by compression molding at 160 °C and 235 MPa.

### Characterization

Torque vs time was measured during the processing of materials.

The morphology of the composite 90:10 (PHBV/Lignosulfonate) was studied by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) in a Jeol JSM-T3000 microscope operating at 20 kV. The sample for morphology study was prepared by cryogenic fracture in liquid nitrogen. The fracture surface

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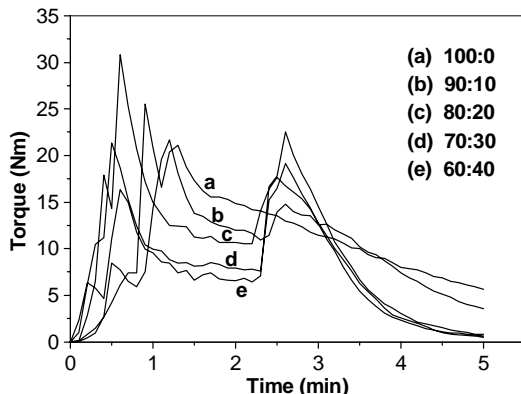
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of the sample was sputtered with Pd/Au alloy in a Balzer MED 020 machine.

## Results and Discussion

The Haake torque-time curves provide information on the effectiveness of the mixing or compounding, rheological behavior, thermal and shear stability, and the effect of different additives on processability behavior.

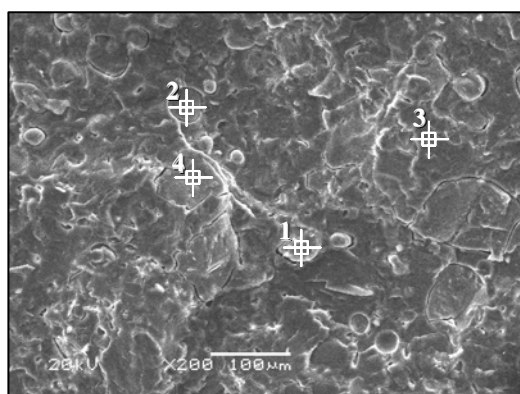
The torque curves of pure PHBV and the composites were showed in Figure 1.



**Figure 1** – Effect of lignosulfonate on the torque obtained during the processing of composites

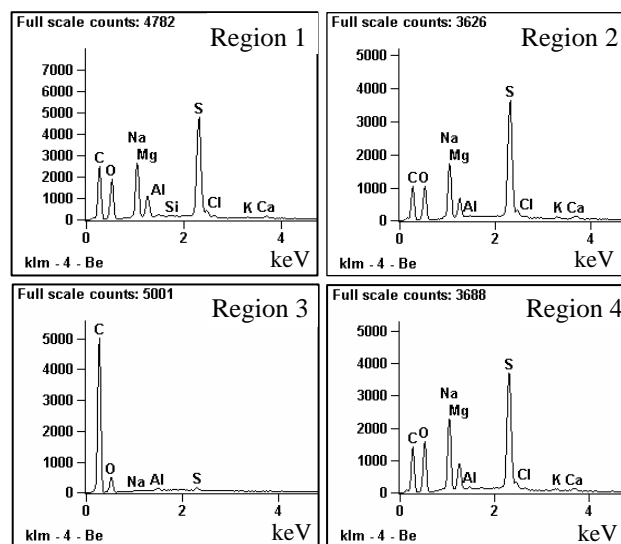
The curve of pure PHBV showed a decrease of torque due its low thermal stability. In the composites decrease of torque values is higher as lignosulfonate amount in composites increase. This behavior was similar as a plastificant or lubricant effect.

The micrograph of fractured surface of 90:10 (PHBV/Lignosulfonate) composite (Figure 2) showed the absence of physical contact between lignosulfonate and PHBV, indicating that the interfacial adhesion between filler and matrix was poor. This was observed in all prepared composites (micrographs not showed).



**Figure 2** – Electron micrograph of fracture surface of 90:10 PHBV/Lignosulfonate composite.

EDS analysis was used to identify lignosulfonate domains disperses in the PHBV matrix. The spectra obtained in EDS analysis (Figure 3) showed chemical elements presents on select regions in the fracture surface (Figure 2).



**Figure 3** – EDS spectra of regions 1, 2, 3 and 4 of 90:10 (PHBV/Lignosulfonate) composite.

The sulphur (S) present only in the lignosulfonate structure was used to identify the regions relative to filler in the PHBV matrix. Thus, regions 1, 2 e 4 (Figure 2) which have a large content of sulphur correspond to domains of lignosulfonate, while the region 3, which did not show S content corresponds to PHBV matrix.

## Conclusion

Results show that is possible to obtain composites based on Vixilex-SD lignosulfonate and PHBV by melting mixing. The addition of lignosulfonate result in a different rheological behavior. The decrease in the composite melt viscosity indicates that composites can be processed in lower temperatures, which could avoid the PHBV degradation during the melt mixing.

Morphological study shows a poor adhesion between lignosulfonate and PHBV. The non-interaction between PHBV and lignosulfonate was anticipated due the hydrophilic characteristics of lignosulfonate filler and hydrophobic characteristics of PHBV matrix.

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