# Proceedings of the 2<sup>nd</sup> International Conference on Circular Packaging



Slovenj Gradec and online 9<sup>th</sup> and 10<sup>th</sup> September 2021

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#### FOREWORD BY THE EDITOR

#### Dear Readers,

When we organized the conference for the first time, we were very aware that climate change and linear business models are damaging the environment. Since then, the still-running current pandemic has changed our world even more. Climate change went even worse, and humanity put the quest for circularity/sustainability a little bit aside, but the two crises, the enviromental and the health, must be addressed continuously. It also changed the way we work, communicate, travel and also how are we attending conferences. That is why this conference is organized in hybrid form with online and onsite (this year in Slovenj Gradec) interaction between speakers and audience. The format of the next conference, due to the uncertainty of constant changes, is a question to be answered.

Nevertheless, I am proud that we have grown from the first time and increased the number of submitted articles and authors presented in this Proceedings. The topics of new and bio-based materials, sustainable and circular business models, EOL solutions to sustainable printing and converting operations are the main focal point of many pieces of research. I hope it helps people from the industry make that leap (which is not easy!) from the linear to circular business and material flows.

No man is an island is a quote that is ideal for these challenging times. The idea that human beings do badly when isolated from others and need to be part of a community to thrive is essential for solving all challenges and organizing a conference in these uncertain times. I want to thank all the authors, sponsors, companies, reviewers and the organizing team which worked hard to prepared the conference and the proceedings.

Together we can overcome hardships and make this world more sustainable. So let us close the circle together!

In Ljubljana/Slovenj Gradec, 9<sup>th</sup> of September 2021 PhD Igor Karlovits

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## MODIFIED LIGNIN/PLA COMPOSITES FOR PACKAGING APPLICATION

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**Abstract:** The present work proposes modification reactions such as oxypropylation of lignin hydroxyl groups to boost the hydrophobicity of the lignin polymer. Modified lignin/PLA composite with different weight percentage compositions were produced through a mixing and injection molding process. The produced composites mechanical (tensile, Young's modulus), thermal (Glass transition temperature) and chemical (type of interaction and bonding between the lignin and PLA matrix) properties were examined. The weight percentage of modified lignin in the composites was optimized based on the resulting properties.

Keywords: lignin, polylactic acid, oxypropylation, composites

#### 1 INTRODUCTION

Globally, 42% of total polymer consumption goes for packaging applications. The number of polymers (poly lactic acid, polyethylene, polypropylene, etc) used in food packaging is noticeably high due to convenience over traditional glass and metal packaging. Among them, polylactic acid (PLA) is a biopolymer, widely used in plastic films, bottles and biodegradable medical devices (Huang et al., 2018). However, PLA has some limitations such as slow crystallization, poor thermal stability and high cost which render it unsuitable for food storage applications requiring long shelf life. We suggest that these limitations can be

overcome through incorporation of biopolymers like lignin. Lignin can be obtained as a low-cost byproduct from agricultural and forest biorefineries and contains several different functional groups (hydroxyl, carbonyl, and carboxyl groups). However, in this form lignin has some compatibility issues with PLA due to the polar functional groups present (Laurichesse & Avérous, 2014). Modification of these polar groups within lignin can significantly minimize compatibility issues with the PLA matrix and enhance mechanical and physicochemical properties of PLA without compromising biodegradability.

#### 2 MATERIAL AND METHODS

#### 2. 1 Lignin modification using Oxypropylation

Kraft Lignin (5g) was first placed into a 250mL round bottom flask. Then, 30 g of propylene carbonate and 0.133 g of NaOH were added into the lignin and stirred using magnetic stirrer at 170 °C for 3 hours. After the reaction time, the product was recovered through precipitation using acidified water (pH=2) followed by filtration through cellulose membrane filters.

#### 2. 2 Analytical conditions

Fourier transform infra-red (FTIR) spectra of unmodified kraft lignin (KL) and oxypropylated kraft lignin (OKL) were collected on a Bruker spectrometer in an absorbance mode using ATR method. Spectra were recorded in the range between 400 to 4000cm<sup>-1</sup> at a resolution of 4cm<sup>-1</sup> with the accumulation of 32 scans.

Thermal properties of KL and OKL samples were analyzed using Waters TA instrument<sup>™</sup> TGA 5500. The samples (5-10 mg) were tested in the temperature range from 40 °C to 800 °C with a heating rate of 10 °Cmin<sup>-1</sup> under nitrogen atmosphere with flow rate of 25mLmin<sup>-1</sup>.

#### 2. 3 PLA/Lignin composite preparation

PLA/lignin composites were produced through melt blending followed by injection molding. PLA/lignin and PLA/oxypropylated lignin were blended using Thermo scientific<sup>™</sup> HAAKE <sup>™</sup> PolyLab OS Torque Rheometer with the operating condition of temperature at 170 °C, rotation speed of 100 rpm and 8 min of mixing time. PLA with unmodified and oxypropylated lignin blends were produced at different weight percentages (1%, 5% and 10% of lignin). PLA polymer was used as a matrix and different weight percentage of lignin was added as a filler. The produced blends were pelletized using PULVERISETTE

25/19 cutting mill grinder with the pellets size of 4mm thickness followed by injection molded using Thermo scientific minijet pro with the condition of holding temperature at 180 °C and molding temperature at 80 °C (Figure 1).



Figure 1: PLA/Oxypropylated lignin specimen at different weight percentages (1%, 5% and 10%)

#### **3 RESULT AND DISCUSSION**

The chemical and thermal degradation properties of KL and OKL were analyzed using FTIR and TGA analysis. Figure 2 shows the comparison spectra of unmodified lignin (Orange) with oxypropylated lignin (blue). In Figure 2, the region between 3600- 3200 cm<sup>-1</sup> corresponds to the -OH stretching frequency. The addition of ether groups after the oxypropylation increased the band intensity at the region of 2980, 2850 and 1467 cm<sup>-1</sup> which belong to the aliphatic -CH<sub>2</sub> groups. The increase in intensities at 1210, 1080-1000 cm<sup>-1</sup> attributes to newly formed ether bonds.



Figure 2: FTIR spectra of unmodified kraft lignin (KL) and oxypropylated kraft lignin (OKL)

Figure 3 illustrates the thermogravimetric analysis (TGA - plotted between temperature and % weight loss) and derivative thermogravimetric curve (DTG - plotted between temperature and derivative weight %). From the TGA graph in Figure 3a, the degradation of KL appeared to take place in the temperature range of 350 °C and the initial weight loss under 100 °C belonged to the moisture loss. On the other hand, OKL encountered the first degradation around 140 °C. From the DTG result (Figure 3b), the decomposition temperature of KL is found in the range of 370 °C whereas in the case of OKL, the decomposition temperature peaks appeared at 140 °C and 316 °C. The first decomposition temperature could be due to the weak bond cleavages (-C-O-C, -CH<sub>2</sub> and -CH<sub>3</sub>) which were formed after the oxypropylation and the peak at 316 °C corresponds to the breaking of aromatic structure of lignin macromolecule. The obtained results were consistent with the reported values in the literature (Lee et al., 2017).



Figure 3: (a) TGA and (b) DTG curves of KL and OKL samples

#### **4 CONCLUSION**

FTIR, TGA and DTG analysis have confirmed that oxypropylation of the kraft lignin was successful. TGA and DTG results revealed that the decomposition temperature after modification were decreased due to the cleavage of added ether group at the aliphatic and aromatic -OH groups after oxypropylation. The unmodified and oxypropylated lignin were mixed with PLA using different weight percentages (1%, 5% and 10%) and the prepared composites mechanical and barrier properties are under investigation. The obtained results will be discussed during the presentation.

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