

## Design of experiments for bio-based composites with lignosulfonates matrix and corn cob fibers

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### ARTICLE INFO

#### Keywords:

Lignin  
Lignosulfonates  
Corn cob  
Composite  
Mechanical properties

### ABSTRACT

Bio-based composites with lignosulfonates as a matrix and corn cob particles as reinforcement were developed. Their mechanical strength in compression mode was evaluated with determination of Young modulus (E) and ultimate compressive strength ( $\sigma_{\max}$ ). The influence of fiber content, fibers particle size, and compaction pressure applied on the composite during processing was investigated with a design of experiments approach. The model enabled to calculate an optimum for  $\sigma_{\max}$  equal to 18 MPa for a corresponding Young modulus of 0.27 GPa. Particle size was found to be the most influent parameter on mechanical properties responses. In order to understand this dependency, an automated optical particle analysis was performed to measure size and shape of corn cob particles. Distribution leading to the highest  $\sigma_{\max}$  was the one with the less smooth surface of particles (low convexity shape parameter), providing a better mechanical adhesion between fiber and matrix. Increasing compaction pressure and fiber content was in favor of compressive strength. Interface between lignosulfonates and corn cob inside the composites was observed with X-ray microtomography technique. It was noticed that interlocking occurred between the two phases.

### 1. Introduction

Nowadays, men are facing depletion of fossil resources such as petroleum and natural gas. The urgent need for sustainability implies other alternatives to produce energy, materials and fine chemicals. Bearing this in mind, scientists have placed their focus on lignocellulosic biomass as a potential source of raw material (Raschka et al., 2014). The amount of plant biomass on Earth is about  $10^{13}$  tons and 3 % of it is renewed every year as reported by Belgacem and Gandini (2008). Contributions of scientists and engineers will permit probably the emergence during the 21st century of biorefineries that use lignocellulosic biomass entries at industrial scale (Octave and Thomas, 2009; Paleologou et al., 2011; Maity, 2015). Ideally, the whole plant would be used, raw material, by-products and energy flows being optimized. For instance, every by-product would serve as raw material to give value-added products. Octave and Thomas (2009) speak of “industrial metabolism” concept to describe this scheme of process. On a dry-weight basis, woody plants cells are mainly composed of polysaccharides (cellulose, hemicelluloses), and lignin (20–40 % w/w). Lignin is a complex polymer of phenylpropane units. It provides

hydrophobization and strength to plant cell walls. It also protects carbohydrates against biological attack thanks to its anti-oxidative and antimicrobial properties. Lignin and hemicelluloses can be linked together with covalent bonds. This interconnected network of biopolymers is destroyed during pulp treatments (both papermaking and bioethanol production processes). Depending on the delignification process, different technical lignin derivatives are obtained as by-products. Interestingly, technical lignins can be used together with natural fibers to produce bio-based composites that substitute to synthetic ones. Nägele et al. (2002, 2014, 2016) developed plastic-like materials constituted of lignin matrix (30–60 % w/w), fiber reinforcement like hemp, flax, or wood (10–60 % w/w) and additives (0–20 % w/w) by injection-molding. Mean tension at break was 18 MPa and mean modulus of elasticity in tension was 6 GPa. Privas and Navard (2013) studied composites made of 30 % lignosulfonates powder and 70 % natural fibers such as flax, Miscanthus, hemp, straw, and jute by a compression process. Elastic modulus in bending was 7.7 GPa and flexural strength was 24.1 MPa for composite with untreated flax fibers. In this study, lignosulfonates and corn cob natural fibers were chosen as matrix and reinforcement respectively with the objective to build a bio-based

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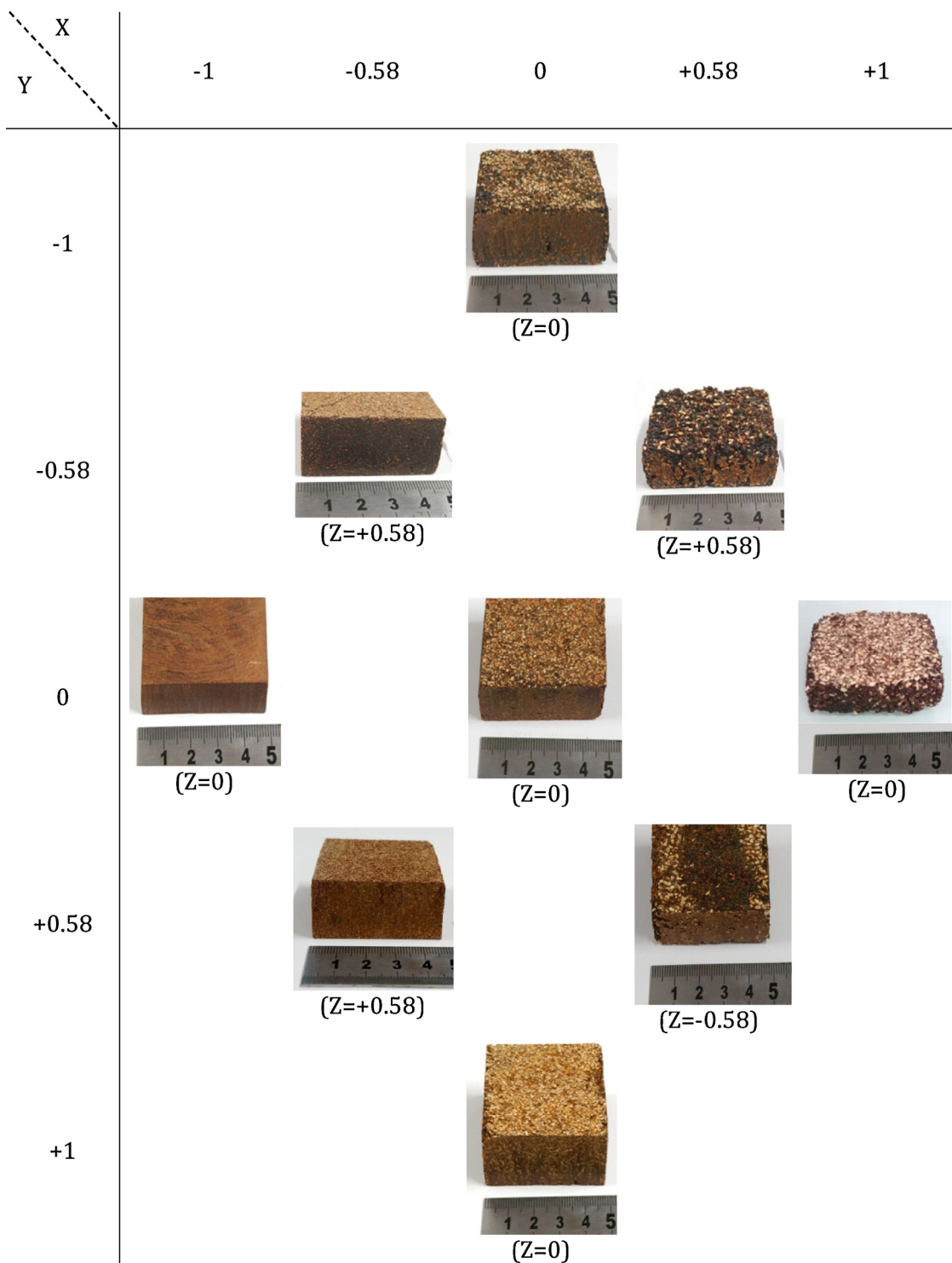


Fig. 1. Samples of bio-based composites from lignosulfonates matrix and corn cob fibers obtained after molding, compaction, and machining. Variables X, Y, and Z are referring to Table 1.

composite. The feasibility of using corn cob fibers as reinforcement in bio-based materials has been previously demonstrated by some authors who tested the mechanical properties in flexion and tension (Luo et al., 2017; Panthapulakkal and Sain, 2007; Luo et al., 2014; Garadimani et al., 2015).

This work aims to establish a proof-of-concept by developing bio-based composites composed of lignosulfonates matrix and corn cob particles that could substitute to current plastics for some applications. Mechanical strength in compression was measured. Several parameters

may impact mechanical properties. The effects of fiber content, fibers particle size, and compaction pressure applied on the composite during processing were investigated. Textural constitution of the composite was evaluated by particle shape analysis and X-ray microtomography observations for a better understanding of the effects of the compression process (Badel et al., 2003, 2008).

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