Adding value to pulp industry waste: Bio-based thermosetting epoxy resins using Kraft lignin

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Introduction

- Lignin is one of the tree main structural components of lignocellulosic biomass and is the second most abundant natural phenolic/aromatic polymer.
- Kraft lignin (KL), a low value side product of the pulp and paper industry, is currently highly underutilized despite its highly phenolic and hydroxylated surface.
- Due to its phenolic structure, lignin can be used either as-received or after chemical or mechanical treatment/fractionation, as a curing agent or as a filler/additive in various thermoset polymers like epoxy resins or can be depolymerized to produce phenolic monomers for down-stream polymerization to resins

Aim of the study

The use of various Kraft lignin types (as received or after treatment/fractionation) as curing agents and filler/additives towards the production of epoxy resin composites and the testing of their mechanical properties via tensile strength measurements. The epoxy system used was commercial diglycidyl ether of bisphenol A (DGEBA) epoxy resin and polyetheramine Jeffamine D-230 and D-2000 as curing agents

Experimental

Glycidylized Lignin

- Reaction of KL in methanolic NaOH solution at 70 °C, 20 min
- Addition of epichlorohydrin
- Reaction at 80 °C for 3 h

Ball Milled Lignin

Dry Ball Milling of Kraft Lignin
- 400 rpm

Milling time: 2 and 8 hours

Micro-Lignin

Kraft Lignin dispersed in EtOH (5 wt.% suspension)
- 1 h stirring, T_{room}
- 2 h sonication, T_{room}

Kraft Lignin As Filler/Additive

Lignin + DGEBA

stir. &sonic, 1h at 55 °C

Epoxy-Lignin suspension

→ **Curing Agents Jeffamine D-230 and D-2000**

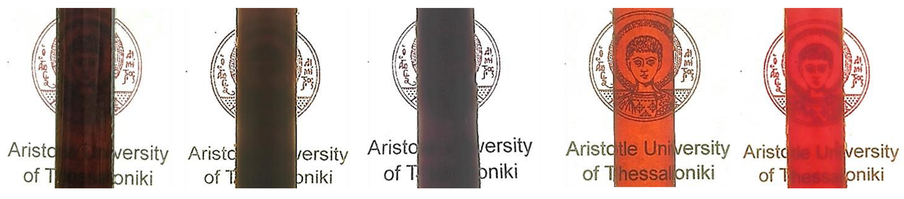
degassing, 10 min at 60 °C

Curing:
1st step: 75 °C for 3 h
2nd step: 125 °C for 3 h

Polymer Composites-Kraft as Filler/Additive

Glassy Composites (Jeffamine D-230)

Rubbery Composites (Jeffamine D-2000)

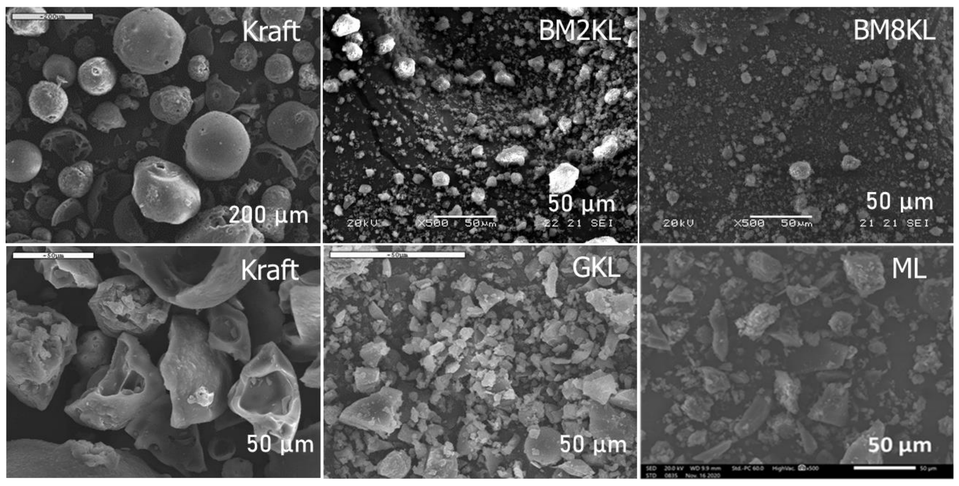


Kraft Lignin (KL) | Ball Milled Lignin (BM2KL) | Ball Milled Lignin (BM8KL) | Glycidylized Lignin (GKL) | Micro-Lignin (ML)

Results

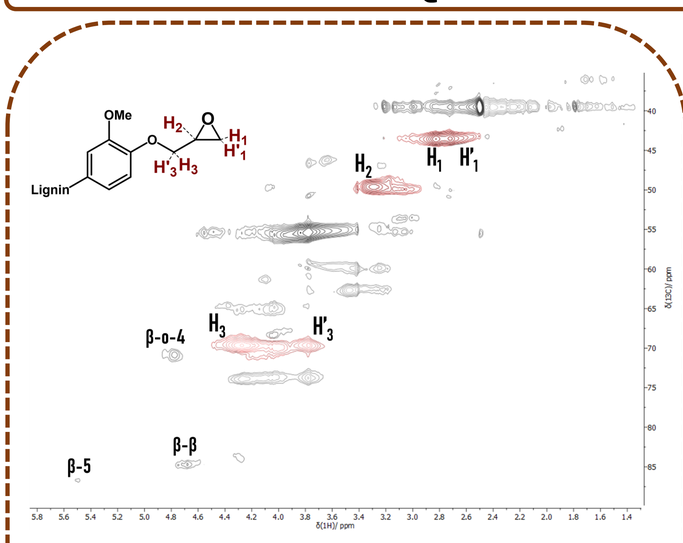
Lignins Characterization

SEM

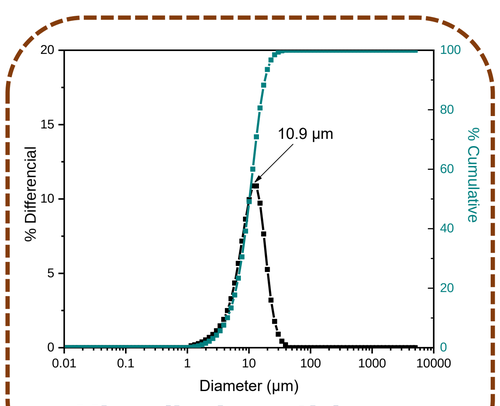


Particle size reduction after chemical and mechanical treatment.

2D HSQC NMR and PSD analysis



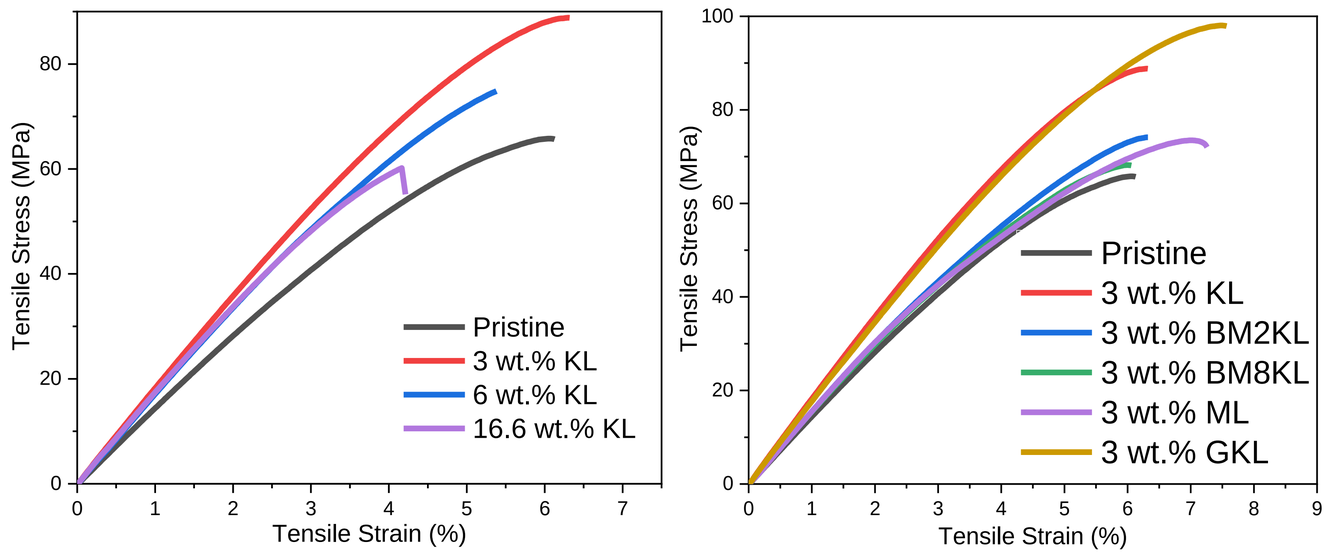
Glycidylation of Kraft lignin is evident from the 2D-HSQC NMR analysis



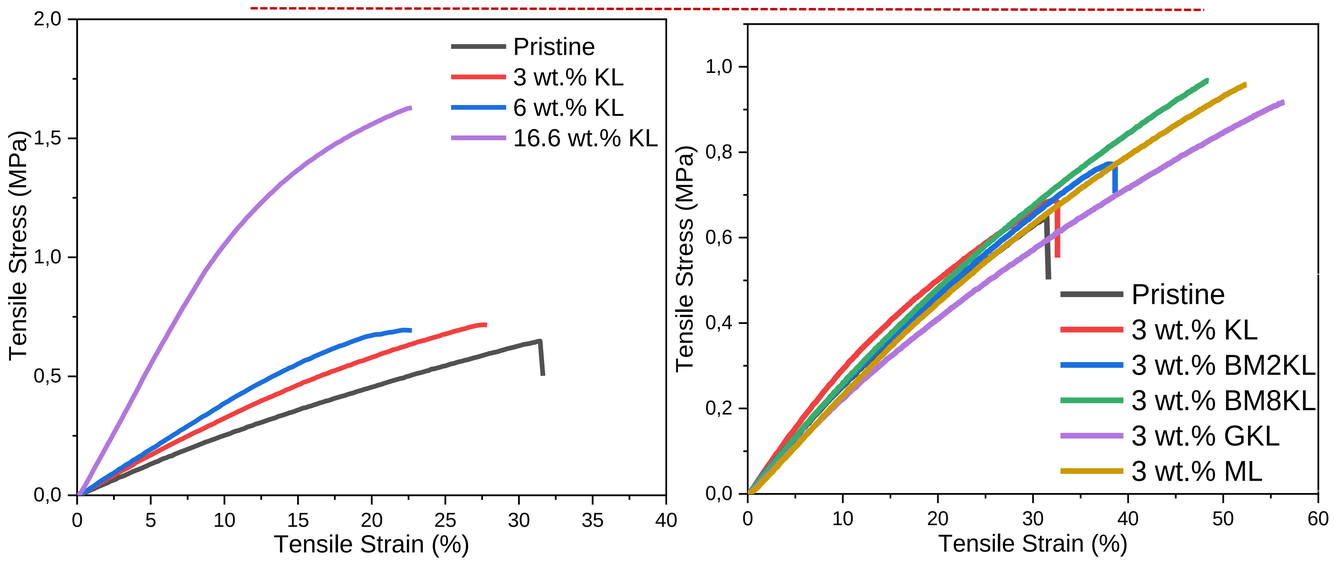
Micro-lignin particles are verified by PSD analysis

Epoxy Polymer Composites Characterization

Glassy Composites



Rubbery Composites



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Conclusions

- **Better dispersion of lignin in both glassy and rubbery epoxy /lignin composites can be achieved when lignin is either chemically or mechanically treated, i.e. glycidylized or micro – lignin.**
- **Mechanical properties of both glassy and rubbery epoxy/lignin composites utilizing initial or treated Kraft lignin as a filler/additive are enhanced.**

References

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