

Lignins-first flexible biorefinery: design of an integrated valorization process

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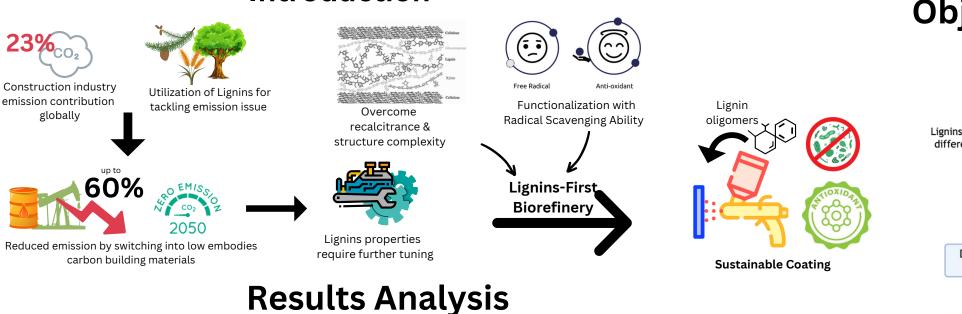
Lignins-First Flexible Biorefinery: Valorization Process

Design for Sustainable Construction Material

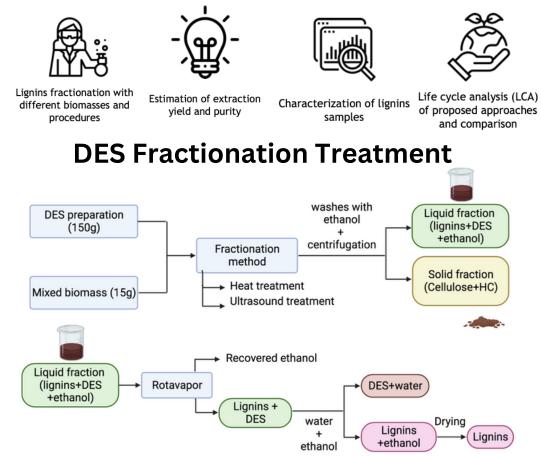
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Introduction



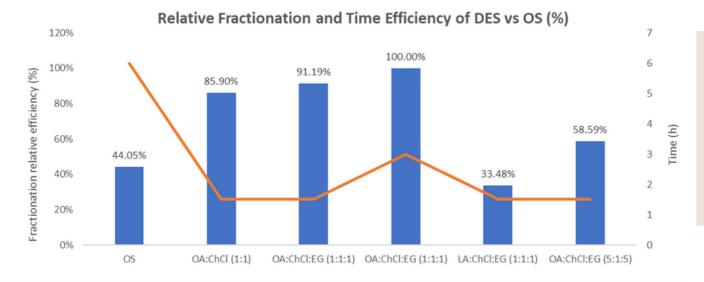
Objectives and Experimental Plan



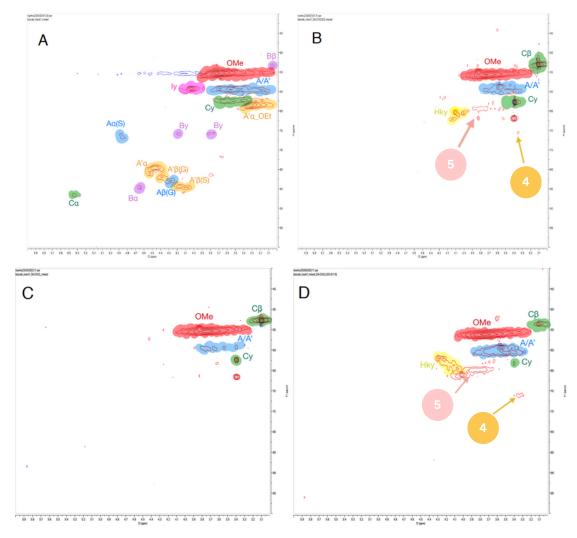
Flexibility through the integrated approach

- Fractionation similar rates for mixed biomass and individual biomasses (95.44%)
- Lower Mw obtained through an integrated approach
- Wider range of available products with mixed biomass fractionation

Relative performance of DES fractionation of the biomass



DES effect on Isolated Lignins



• The considerable drop in duration period and increase in the relative fractionation efficiency set DES as the preferred treatment for lignins isolation.

• While DES lignins present "thinner" grains" and darker colors, OS lignins were found much"rougher" and lighter colors. This can be doomed in the former case's acidity conferred by the employed

solvent.

80423-mixed-os-ATR

 Different fractionation treatments presented distinct textures and colors when comparing OS and DES-isolated lignins.

> C-O-C aliphatic



Isolated lignins by DES treatment in the left (OA:ChCl and OA:ChCl:EG correspondingly) and OS treatment in the right

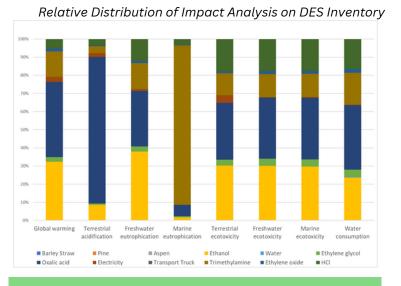
ß-5 conserved

Little reduction in C-H bond availability in DES-treated lignins. It shows the **B-5 bond is less likely to be cleaved** in DES treatment

ß-O-4 Selective Cleavage

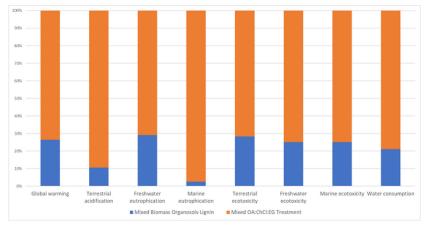
2D HSQC-NMR spectra of aliphatic side chain region of A. OS B. OA:ChCl:EG (1:1:1) C. OA:ChCl (1:1) D. OA:ChCl:EG (5:1:5)

Life Cycle Assessment



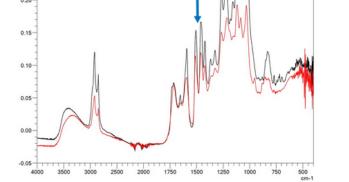
- The DES treatment yields a noticeable LCIA criteria compared to OS. This result can be traced from the inventory analysis, where oxalic acid emits significant carbon emissions from the oxidation of sucrose using vanadium pentoxide.
- In addition, choline chloride production involves the dehydration of Trimethylamine and Ethylene Oxide with HCl, which contributes to major water discharge into the environment.

Comparison on LCIA Relative Distribution



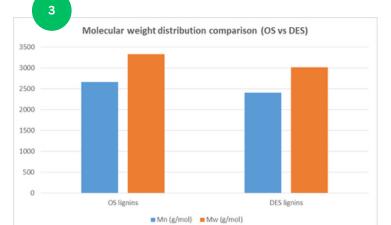
Main Footprints from Respective Treatments





C-H

ATR FTIR spectrums of isolated lignins from OS treatment (black) and OA:ChCl:EG (1:1:1) (red)



A significant drop is observed n C-O-C aliphatic region. It emphasized that the **B-O-4 bond is selectively cleaved** to prevent C-C bond cleavages (B-5, B-B, or 5-5") leading to less condensation.

Molecular Uniformity & Weight Distribution

The Mn of lignins from DES treatment is comparatively lower than OS due to smaller oligomers possibly found with a lower polydispersity index. It indicates that **DES oligomers or monomers are more consistent** in terms of molecular size and weight. Additionally, **lower Mw are identified for the DES** isolation of lignins.

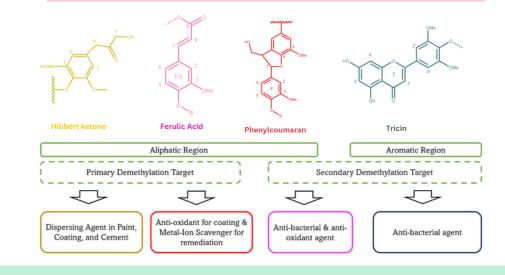
Stabilizer Signal

3

Comparing to current research's result, there is small signals on 70.20/3.32 (A" – EG/A" –GLY) in OA:ChCl:EG 1:1:1 and 70.64/3.34 for OA:ChCl:EG 5:1:5 2D NMR results which are believed as EG traces.

Novel Chemical Bonds Formed by EG Stabilization

These keeper signals were attributed to polyol-incorporated β -O-4 structures. During the fractionation, the acidic DES forms carbocation intermediates via dehydration at the Ca position of lignin. Then, the carbocation intermediate was trapped by the EG to generate polyol-alkoxylated lignins.



Future Perspectives

- Depolymerization of isolated lignins through microbial approaches.
- Estimation of the further effect of functionalization on abundancy of hydroxyl group on lignins product

Conclusions

- **Flexibility** approach through implementation of the **integrated approach**; not only access to wider amount of interest molecules, but also fractionation of lower MW lignins
- **Lignins-first** through **DES fractionation** approach: performance with higher fractionation and time efficiency rates when compared to OS results
- Simultaneous fractionation and functionalization through ternary DES with EG

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