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Fractionation of corncob biomass towards sustainable valorization in biorefinery processes

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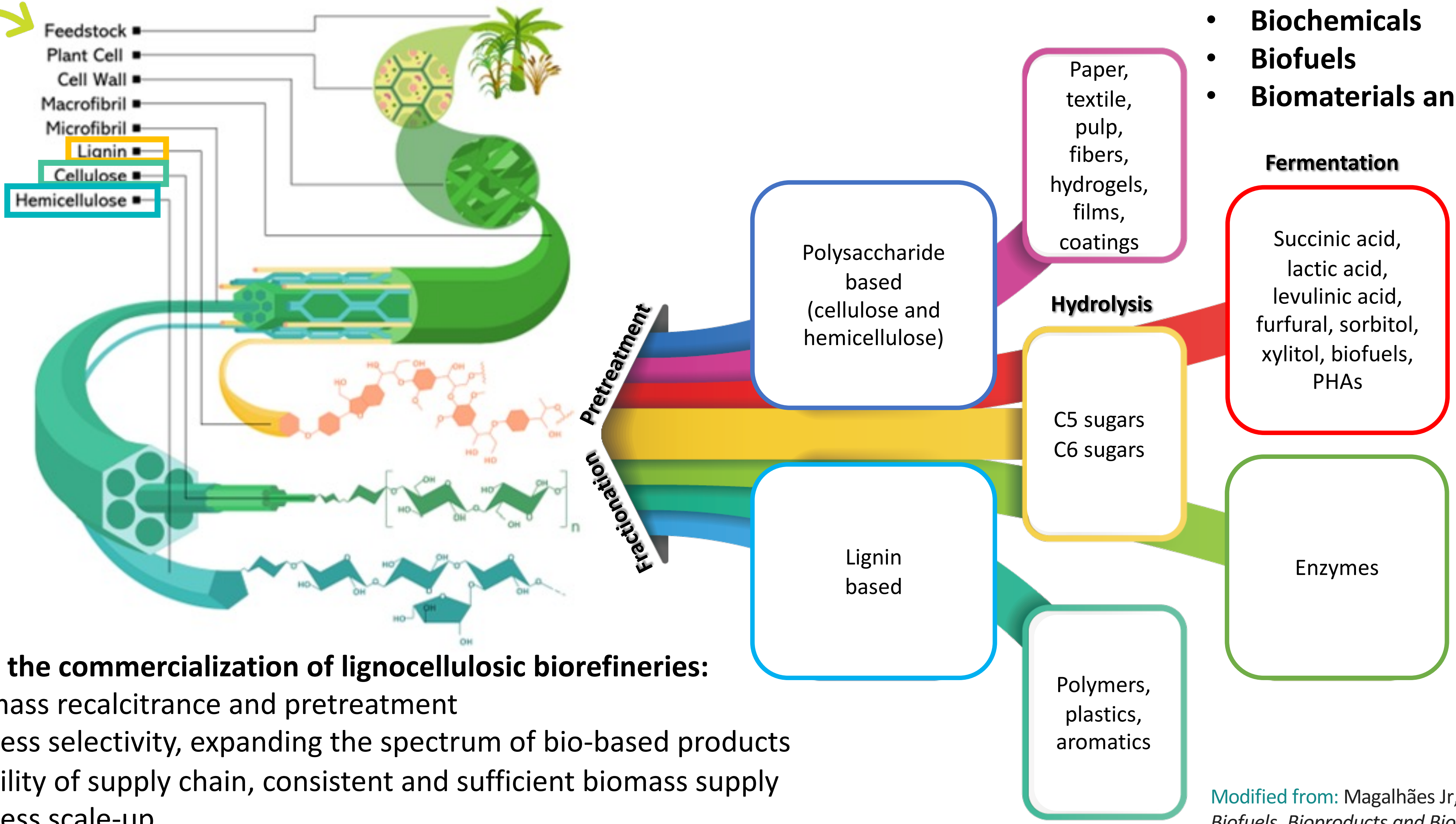
Biochemical valorization of lignocellulosic biomass to value-added products

Primary or secondary residues from agroforestry or from non-food/food crops

- **Biochemicals**
- **Biofuels**
- **Biomaterials and others**



Challenges in the commercialization of lignocellulosic biorefineries:

- Biomass recalcitrance and pretreatment
- Process selectivity, expanding the spectrum of bio-based products
- Viability of supply chain, consistent and sufficient biomass supply
- Process scale-up

Modified from: Magalhães Jr, A.I., et al., 2019. *Biofuels, Bioproducts and Biorefining*, *13*(6), 1505-1519.

Oxidation processes in the treatment of the lignocellulosic biomass-the role of alkaline hydrogen peroxide

- Hydrogen peroxide decomposition at pH close to 11.5 and the production of highly reactive radical species, selectively promoting lignin oxidation and depolymerization by cleaving and fragmenting the macrostructure of lignin into fragments with lower molecular weight.

$$H_2O_2 + H_2O \longleftrightarrow \underbrace{HOO^-}_{\text{Hydroperoxyl anion}} + H_3O^+$$

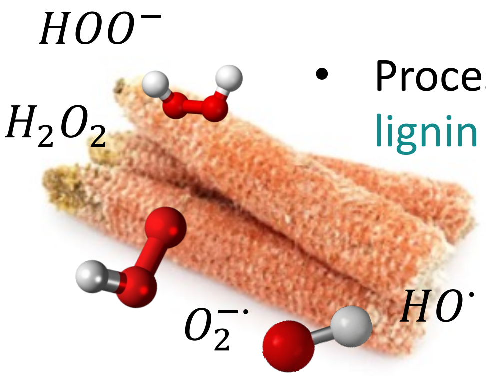
$$H_2O_2 + HOO^- \longleftrightarrow \underbrace{HO^{\cdot}}_{\text{Hydroxyl radical}} + \overbrace{O_2^{-\cdot}}^{\text{Superoxide anion radical}} + H_2O$$

$$HO^{\cdot} + O_2^{-\cdot} + H^+ \longleftrightarrow O_2 + H_2O$$

- For achieving higher process efficiency, longer reaction time and/or higher peroxide concentration are usually required.

- Side effects - depolymerization of hemicellulose and cellulose.

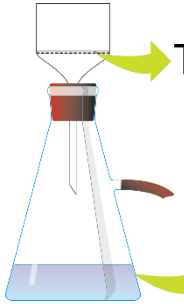
- Process optimization for simultaneous recovery of polysaccharide-rich residue and high-quality lignin fraction - peroxide concentration, reaction time, and temperature.



Methodology

Corncob biomass
H_2O_2 solution pH 11.5

Vacuum filtration
Rinsing with H_2O



Treated solid

Lignin recovering

Precipitation with acid

Taguchi Orthogonal Array L9 was used for pretreatment optimization

Exp. no.	Factor			
	Power (W)	Time (s)	H_2O_2 conc. (%)	Liquid-to-solid ratio
1	100	20	0.5	10
2	100	40	1.5	15
3	100	60	2.5	20
4	200	20	1.5	20
5	200	40	2.5	10
6	200	60	0.5	15
7	300	20	2.5	15
8	300	40	0.5	20
9	300	60	1.5	10

Lignin content

Delignification efficiency

$$\text{Delignification (\%)}=\frac{\text{Lignin}_{\text{untreated}}-\text{Lignin}_{\text{pretreated}}}{\text{Lignin}_{\text{untreated}}}\times 100$$

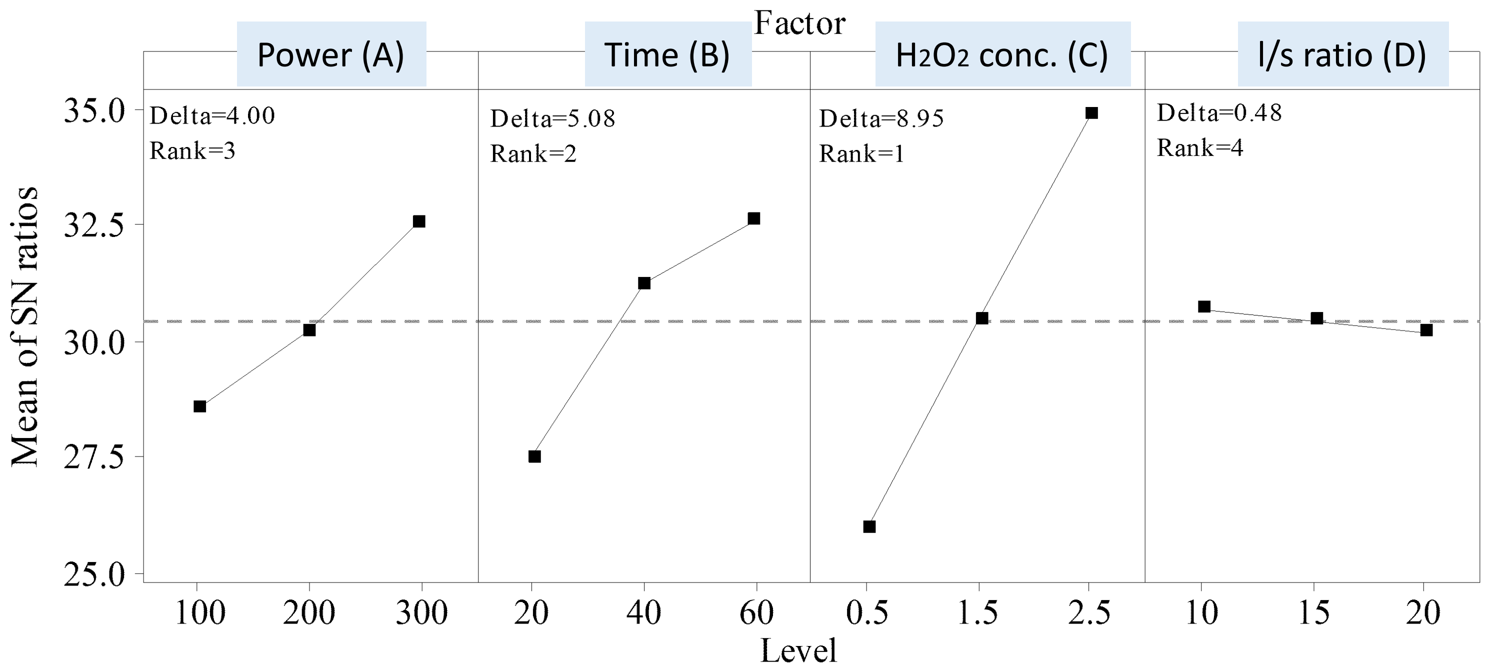
The yield of isolated lignin

Optimal conditions

- Compositional analysis
- XRD
- FTIR

Results

The optimum combination of factors leading to the highest delignification: A3-B3-C3-D1



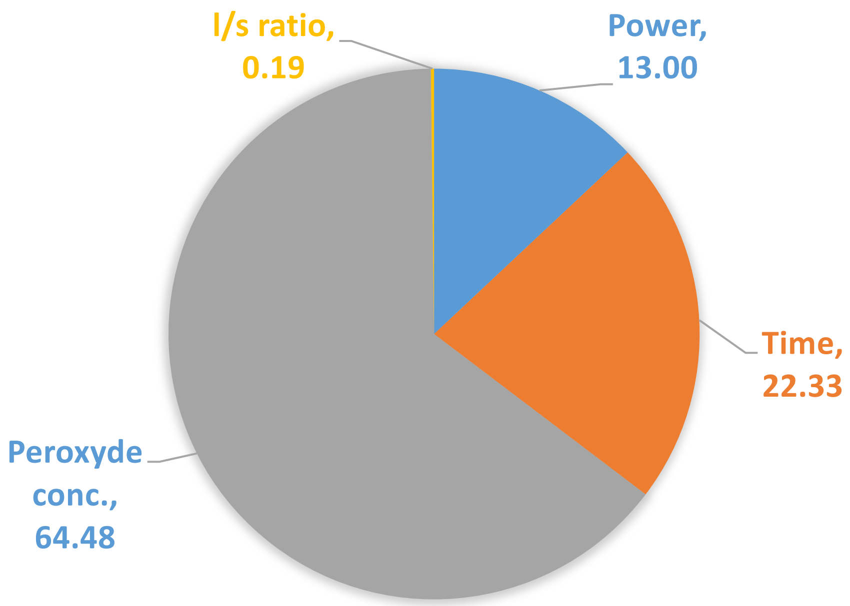
Main effects plot for S/N ratios for delignification efficiency

(A) Power: **300W**
(B) Treatment time: **60s**
(C) H_2O_2 concentration: **2.5%**
(D) Liquid-to-solid ratio: **10**

- Theoretically predicted optimum response: **75.81%**
- Experimentally obtained delignification: **76.46±2.48%**

Results

The effect of individual factors on delignification efficiency



Factor contribution (%)

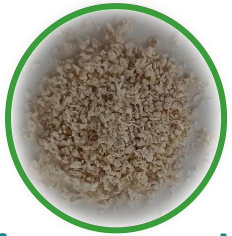
ANOVA for S/N ratio for corncob delignification

Factor	DF	SS	MS	*F*	*P*
Power	2	24.239	12.1196	69.04	0.014^{s}
Time	2	41.625	20.8125	118.55	0.008^{s}
H_2O_2 conc.	2	120.185	60.0926	342.31	0.003^{s}
Residual error	2	0.351	0.1756		
Total	8	186.400			

DF-degree of freedom, SS-sum of squares, MS-mean squares, *F*-Fisher ratio, *P*-probability, C-percentage of contribution.
^{s}significant (P<0.05)

Results

Characterization of the polysaccharide fraction



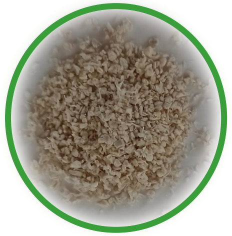
The compositional analysis of pretreated and untreated corncob

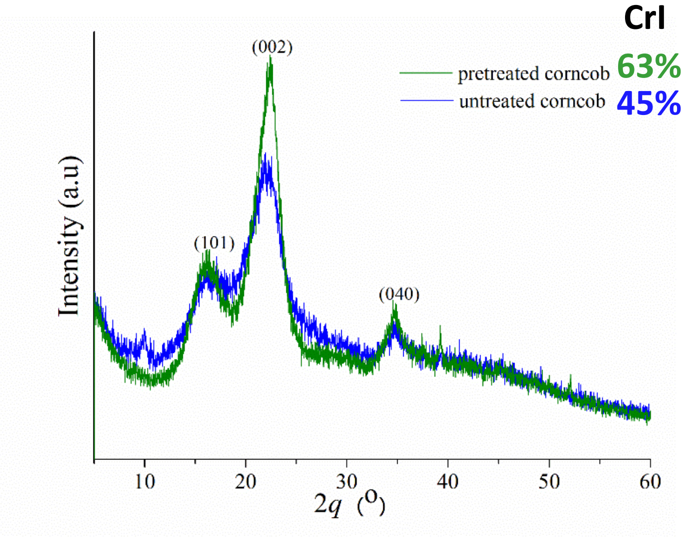
Parameter (% dm)	Pretreated biomass	Untreated biomass
Cellulose	58.60±3.86	37.29±1.47
Hemicellulose	35.28±3.05	40.86±3.39
The total lignin	4.95±0.08	19.76±0.46
Acid-insoluble lignin	4.01±0.09	17.40±0.44
Acid-soluble lignin	0.94±0.01	2.27±0.95
Acetyl-bromide lignin	4.57±0.35	19.42±1.01

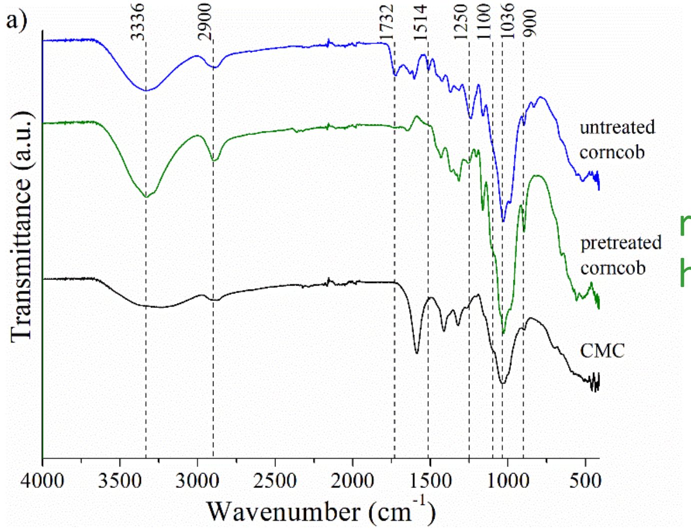
- Lignin 75% ↓
- Hemicellulose 14% ↓
- Cellulose 57% ↑

Results

Characterization of the polysaccharide fraction





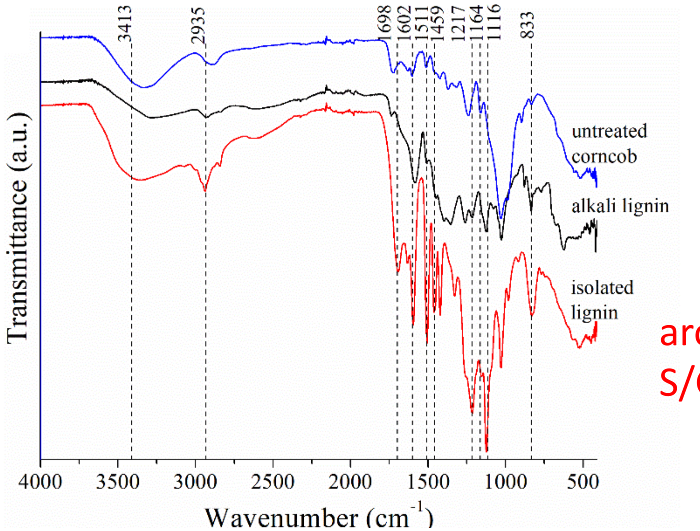


reduced lignin and hemicellulose content

XRD and FTIR spectra of pretreated and untreated corncob

Results

Characterization of the lignin fraction



aromatic structure
S/G type

FTIR spectra of the isolated lignin

- Purity 98.13±0.54%
- Yield 17.85±0.96%

Conclusions

- Microwave-assisted peroxide treatment was successfully optimized by the Taguchi method achieving high selectivity in lignin removal with minimal polysaccharide degradation.

- The peroxide concentration was the most influential factor promoting delignification, followed by treatment time, microwave power and liquid-to-solid ratio. A very short 1-min treatment removed 75% of lignin, resulting in 57% higher cellulose content.

- The purity and yield of isolated lignin suggest the potential integration of lignin extraction into corncob biorefinery which could improve the overall efficiency and economics of corncob processing.

Thank you for your attention!!!

Acknowledgements
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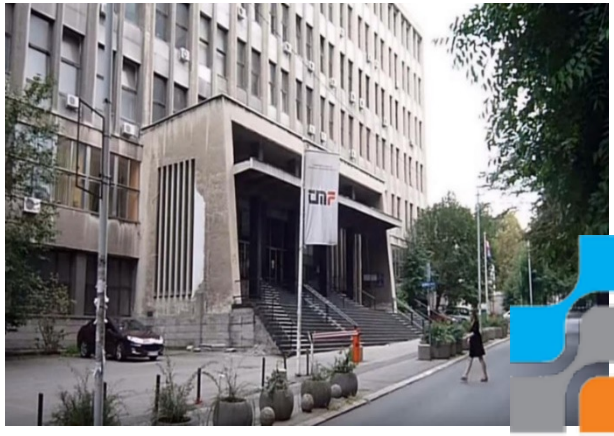

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