Production of colloidal lignin particles: a statistical study

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Motivation and Objectives

The development of colloidal lignin particles (CLPs) is a promising strategy to overcome the challenges of lignin application, such as its poor water solubility [1]. The antisolvent precipitation is a widely used technique to produce CLPs, presenting several advantages, however, studies using this technique present some drawbacks.

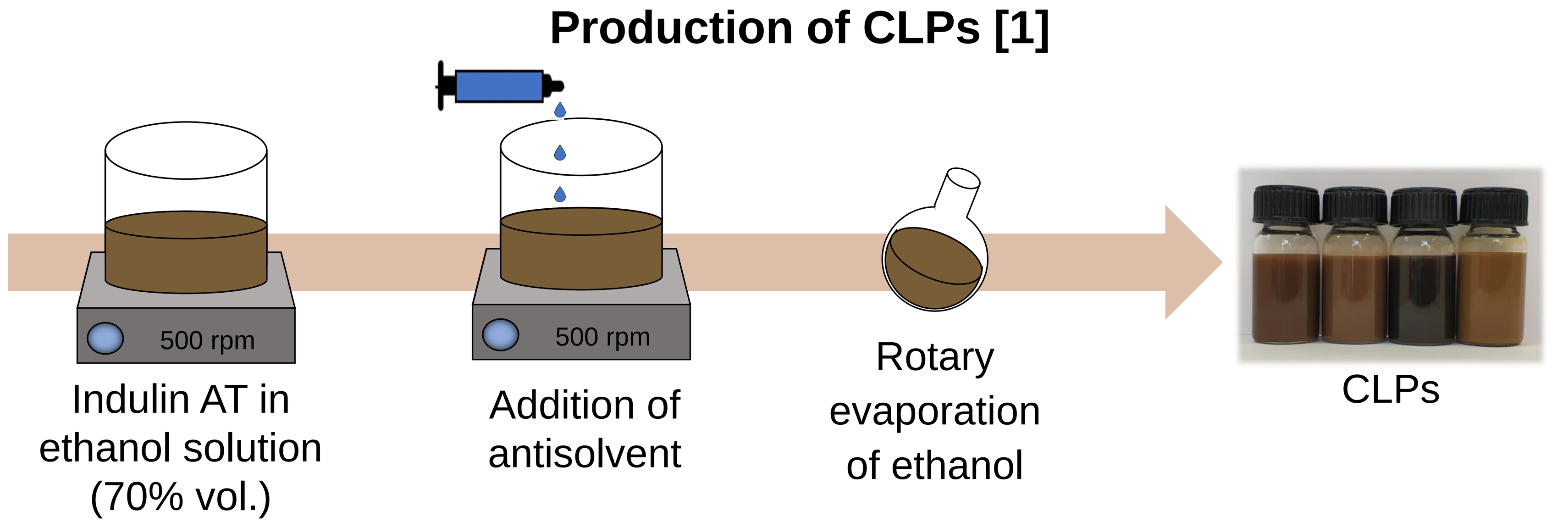
Advantages	Drawbacks
Simple	Low particle concentration
High particle yield	Toxic solvents
Controllable size and morphology	
Low cost	

To produce concentrated and stable CLPs through the antisolvent precipitation technique

Increase knowledge on process variables and their effects on particle characteristics

Methodology

Production of CLPs [1]



Study of process variables effects
Fractional Factorial Design (FFD) 2^{4-1}

Assay	Decodified values: Initial lignin concentration (g/L)	Antisolvent pH	Final ethanol concentration (% volume)	Antisolvent addition rate (mL/min)
1	10	4	15	6
2	50	4	15	14
3	10	8	15	14
4	50	8	15	6
5	10	4	45	14
6	50	4	45	6
7	10	8	45	6
8	50	8	45	14
9	30	6	30	10
10	30	6	30	10
11	30	6	30	10

Responses
- Particle size (Laser Diffraction Analysis, D50 number-based distribution)
- Zeta potential (Nano-ZS Zetasizer)
- Color (Colorimeter CR-400 Konica Minolta, L*a*b* CIELAB space)
- Emulsifying potential (Mixture of CLPs and Miglyol 812, vortex for 1 minute). Emulsified Layer (EL %) calculated as Equation 1:

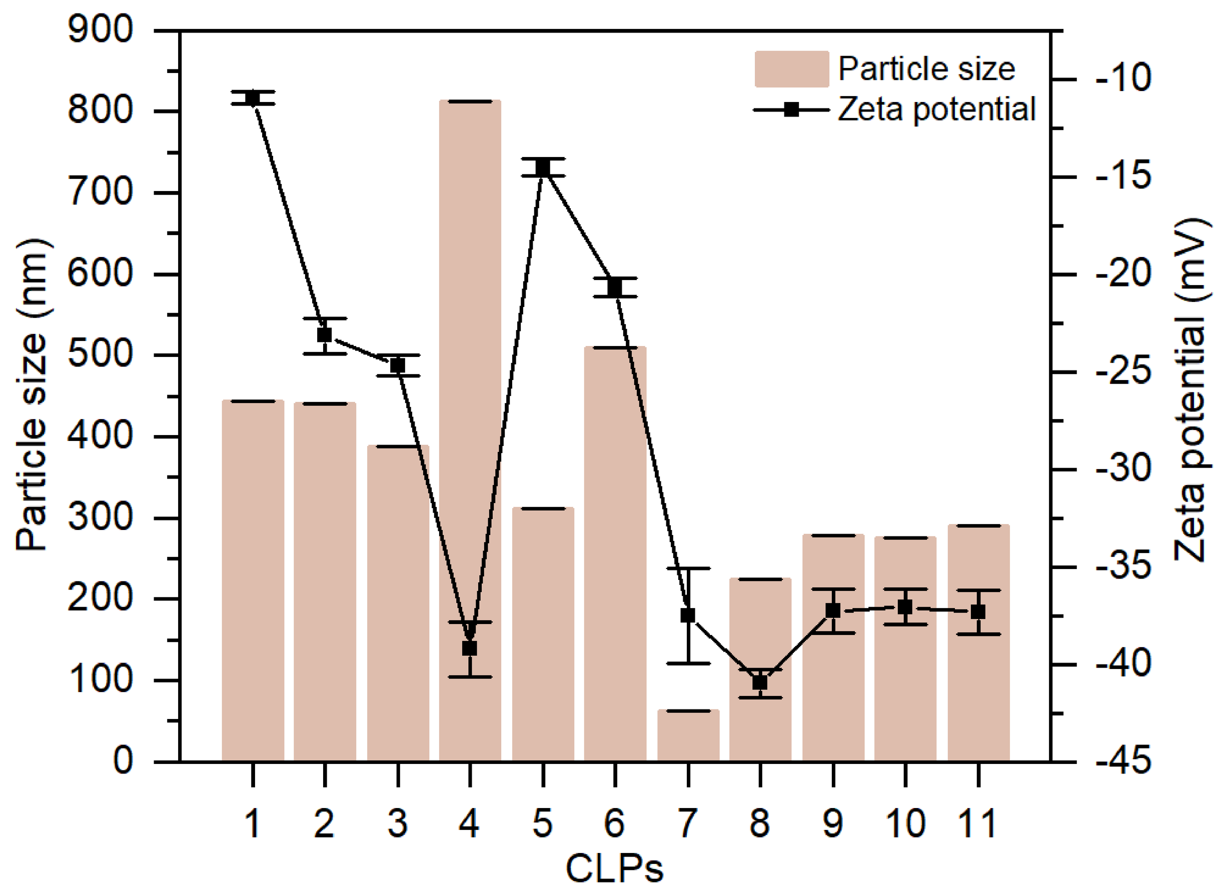
$$EL\% = \frac{Emulsion_{height}}{Total_{height}} \times 100 \quad (1)$$

Conclusions and Future Work

- The **antisolvent pH** and **initial lignin concentration** were **significant variables** affecting the stability, size, and color of CLPs.
- It was possible to produce **stable CLPs** at a concentration up to **50 g/L**
- **CLPs** presented **high potential** as **Pickering emulsions stabilizers**

Ongoing/Future work: production of Pickering emulsions using CLPs as stabilizers for bio-based applications.

Particle Size and Zeta Potential



Effect Analysis (FFD 2^{4-1})	Particle size: Effect (nm)	p (<0.10)	Zeta potential: Effect (mV)	p (<0.10)
Mean	398.99	**0.0006**	-26.46	**0.0000**
Curvature	-234.64	0.2952	-21.54	**0.0057**
Initial lignin concentration (10 to 50 g/L)	↑ 196.03	**0.1205***	↓ -9.10	**0.0135**
Antisolvent pH (4 to 8)	-54.53	0.6252	↓ -18.27	**0.0007**
Final ethanol concentration (15 to 45%)	↓ -244.03	**0.0674**	-3.91	0.1689
Antisolvent addition rate (6 to 14 mL/min)	-115.48	0.3209	1.26	0.6259

***Considering the proximity of 0.1205 with the p-value significance level of 0.10 (10%), the initial lignin concentration was considered a significant factor for the particle size response.

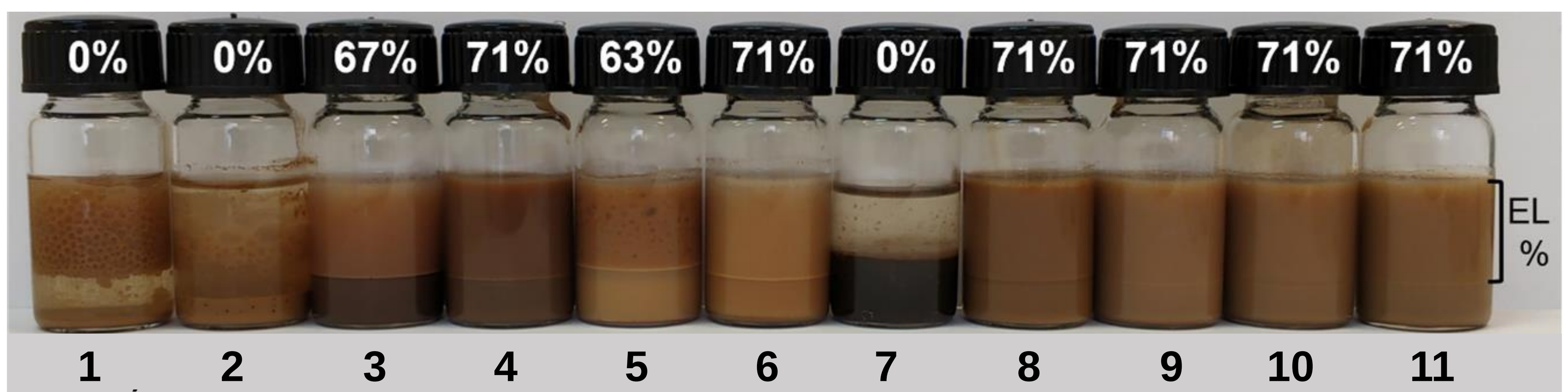
Color of CLPs

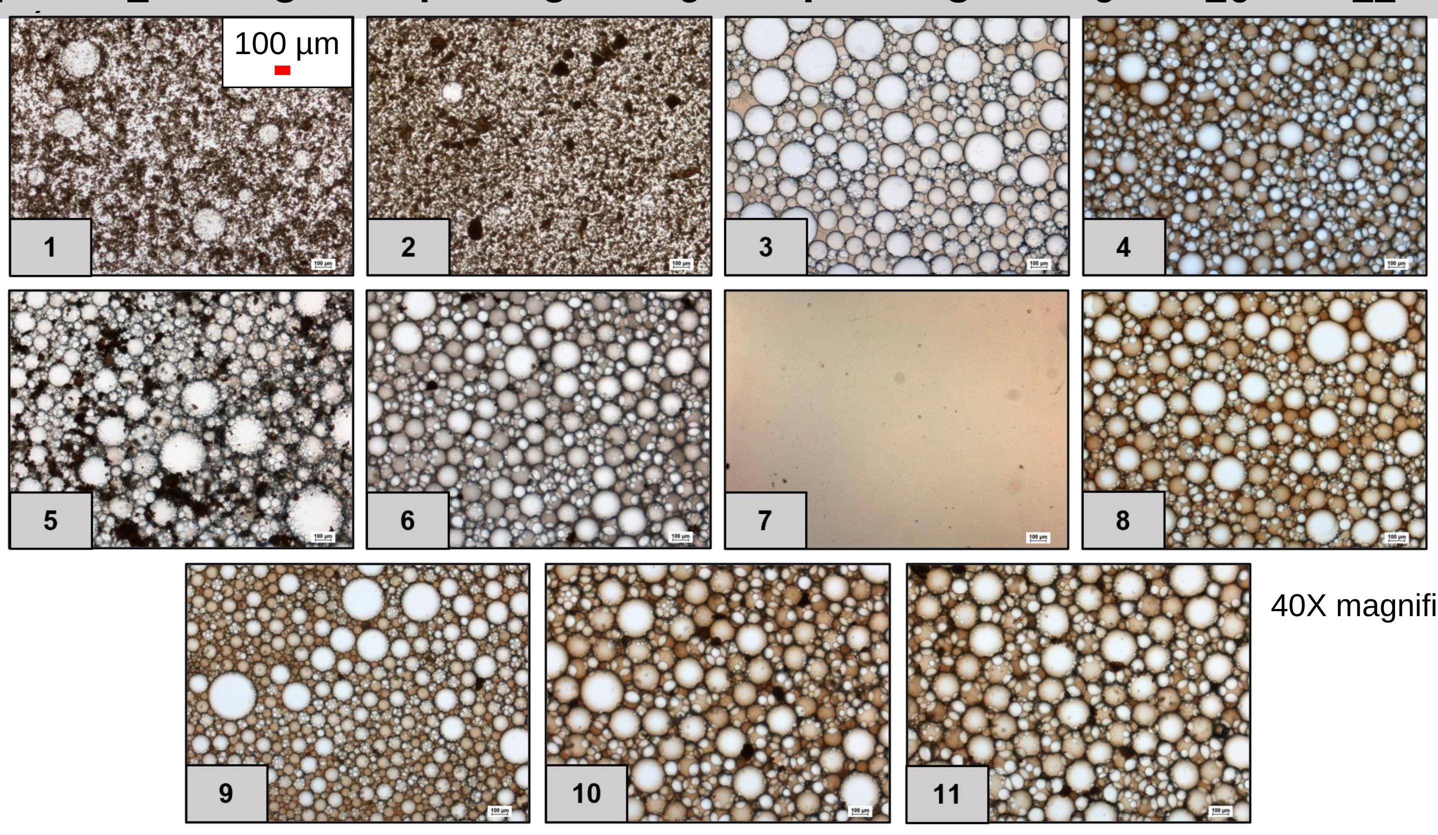
↑ **Initial lignin concentration (10 to 50 g/L)** = ↑ **Luminosity and yellowish tendence**

↑ **Antisolvent pH (4 to 8)** = ↓ **Luminosity** ↑ **brownish tendence**

Emulsifying Potential

Mixtures of CLPs and Miglyol 812 (50/50 vol.)
EL% indicates the percentage of emulsified layer formed for each sample





40X magnification

- Only CLPs 1, 2 and 7 were not able to form an emulsified layer, due to particle instability (CLPs 1 and 2) or low particle size and concentration (CLPs 7)

References and Acknowledgments

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References. [1] Colucci, G., Santamaria-Echart, A., Silva, S. C., Teixeira, L. G., Ribeiro, A., Rodrigues, A. E., Barreiro, M. F. Colloids Surf. A: Physicochem. Eng. Asp., **666** (2023) 131287.