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Optimization of Lignin Separation Process from Black Liquor Using Strong Acids and Supercritical CO₂

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ABSTRACT

Black liquor is the major waste from pulp and paper industry. Generally, it contains the amount of lignin fraction around 10-20% depending on the pulping process. The extracted lignin can be efficiently converted to several value-added products. In this work, lignin was comparatively extracted from black liquor by acidification and suband super-critical CO₂. For the first method, lignin precipitation was achieved by decreasing alkali black liquor pH from 10 to 2 with strong acid (H₂SO₄ and HCl) then filtrate with Glass fiber filter to separated solid and liquid phase. In the second technique, the acidification using CO₂ under sub and supercritical conditions which obtain pH 5 then use membrane to separated liquid and solid phase. The lignin precipitated percentages from each technique were measured and optimized for enhancing the highest yields of precipitated lignin.

Keywords: Lignin; Black liquor; Acidification process; supercritical CO₂; lignin Precipitation

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OPTIMIZATION OF LIGNIN SEPARATION PROCESS FROM BLACK LIQUOR USING STRONG ACIDS AND SUPERCRITICAL CO₂

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Abstract

Black liquor is the major waste from pulp and paper industry. Generally, it contains the amount of lignin fraction around 10-20% depending on the pulping process. The extracted lignin can be efficiently converted to several value-added products. In this work, lignin was comparatively extracted from black liquor by acidification and sub- and super-critical CO₂. For the first method, lignin precipitation was achieved by decreasing alkali black liquor pH from 10 to 2 with strong acid (H₂SO₄ and HCl) then filtrate with Glass fiber filter to separated solid and liquid phase. In the second technique, the acidification using CO₂ under sub and supercritical conditions which obtain pH 5 then use membrane to separated liquid and solid phase. The lignin precipitated percentages from each technique were measured and optimized for enhancing the highest yields of precipitated lignin.

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1. Introduction

Nowadays, the problems about energy and environment are the topics with high global concerns, therefore, renewable biomass energy was increasing interest as sustainable alternative energy. Usually, the biomass is the raw material for pulp and paper industry, which generate the black liquor. Normally, it contains the amount of lignin fraction around 10-20% then, lignin was removed to extract for another products. The structure of lignin is very heterogeneous so, the fractionation process is an important step to obtain specific type of lignin. In the present work, lignin was obtained from black liquor resulting from laboratory. The lignin was comparatively extracted from black liquor by acidification and sub- and super-critical CO₂. Obtaining lignin-rich by both methods were characterized in term of chemical properties.

2. Literature Review

Garcia *et al.* [1] investigated about the acid precipitation of black liquor and selective the precipitation. The achieved precipitation of black liquor is decreasing pH from 12.64 to 0.72 and by adding sulphuric acid (98 %, w/w). The experiment was achieved at pH 0.72 and percentage of lignin 52.36%. Fredrix and Hans [2] studied the filtration properties of lignin suspension which was precipitated at different pH and temperature. The results of the yield increased when the pH decreased. The yield of precipitated lignin decreased when the reaction temperature increased. Nadja carchet *et al.* [3] studied an organosolv lignin, extracted in organic acid media, or called Biolignin. In their work, they have developed the procedure to acetylate lignin using supercritical carbon dioxide as a solvent in term of the supercritical conditions. Kouisni *et al.* [4] investigated separation of lignin from black liquor by oxidizing, acidifying and filtering the acidified black liquor. The

process provides larger lignin particles and higher dry solid content than from corresponding non-oxidized, acidified black liquor.

3. Research Methods

The starting material is black liquor provided from laboratory. The black liquor was separated to liquid and solid fractions. To extract lignin from alkali black liquor, first step is acidification process of alkali black liquor. In this work, strong acids and carbon dioxide was used acidified. First method, the strong acids *i.e.* H_2SO_2 and HCl was used to decrease pH from 10 to 2 at atmospheric pressure for precipitated lignin rich. Second method, using CO_2 under sub- and super-critical conditions are studied in a Parr reactor to decrease pH value of alkali black liquor. After lignin precipitation, the black liquor will be separated into solid and liquid fractions by the filtration process. The black liquor from strong acid precipitation were filtrated with Glass fiber filter $1.2\ \mu\text{m}$ (Whatman) and, black liquor from CO_2 acidification were filtrated with ceramic membrane (10 kDa).

4. Discussion and Conclusion

The effect of acid types (*i.e.* H_2SO_4 and HCl) on the precipitation yield of lignin from black liquor under different reaction times was carried out. The highest percentage of lignin precipitation achieved at the reaction time of 24 hours and using the sulfuric acid are presented in Fig.1. The temperature at $80\ ^\circ\text{C}$ archived the highest percentage yield of lignin were shown in Fig 2. The acidified process that using CO_2 at super-critical condition shown the higher percentage of lignin yield than sub-critical condition.

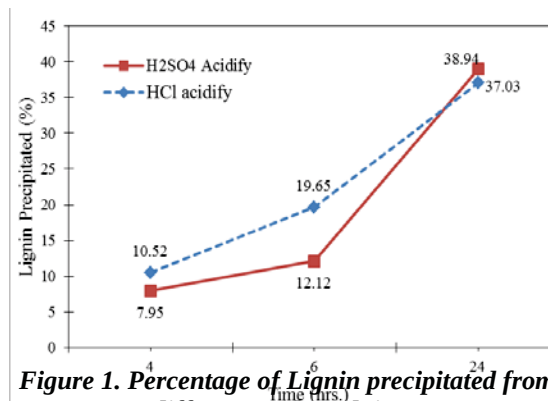


Figure 1. Percentage of Lignin precipitated from different acids and times

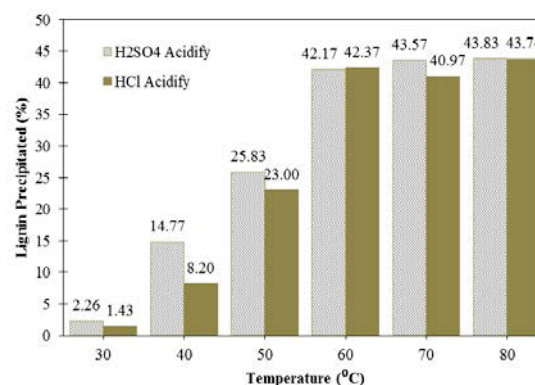


Figure2. Percentage of Lignin precipitated from different acids and temperature.

5. References

- [1] García, A., Toledano, A., Serrano, L., Egüés, I., González, M., Marín, F., & Labidi, J. (2009), Characterization of lignins obtained by selective precipitation. *Separation and Purification Technology*. 68(2): pp. 193-198.
- [2] Fredrik, O., Hans, T. (2007), Filtration properties of lignin precipitated from black liquor. *TAPPI JOURNAL*. 6(7): pp. 3-9.
- [3] Cachet, N., Camy, S., Benjelloun Mlayah, B., Condoret, J. S., & Delmas, M. (2014), Esterification of organosolv lignin under supercritical conditions. *Industrial Crops and Products*. (58): pp. 287-297.
- [4] Kouisni, L., Fang, Y., Paleologou, M., Ahvazi, B., Hawari, J., Zhang, Y., & Wang, X. M. (2011), Kraft lignin recovery and its use in the preparation of lignin-based phenol formaldehyde resins for plywood. *Cellulose Chemistry and Technology*. 45(7): pp. 515.