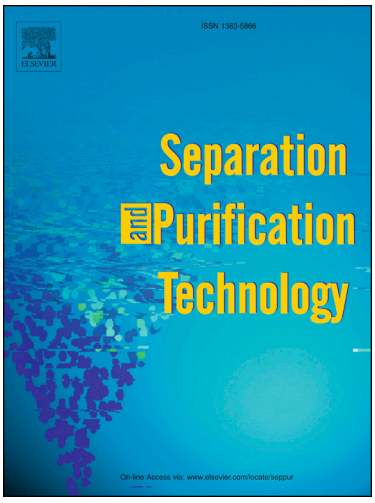

Solubility and fractionation of Indulin AT kraft lignin in ethanol-water media

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Keywords: lignin, fractionation, solubility, molar mass, phenolic, hydroxyl

Highlights

- **Identified a solubilization maximum at 60 wt% ethanol**
- **The molar mass distribution of the fractions can be adjusted by the solvent's ethanol concentration**
- **The phenolic hydroxyls in lignin can be concentrated by removing the water-soluble fraction**
- **The phenolic hydroxyls in lignin can be concentrated by removing the ethanol-insoluble fraction**

Abstract

Lignin solubility is a varying property, as depending on the type of lignin and its origin, its solubility in different solvents will differ. This is due to the highly heterogeneous nature of lignin. Solubilizing lignin could improve its potential valorization by making it more conducive to chemical reactions. Solvent fractionation could be utilized as the first step in lignin refining to adjust some of its properties before further processing. It is known that kraft lignin is completely soluble in alkaline solvents such as aqueous NaOH. The purpose of this study was to assess the solubility of Indulin AT lignin in aqueous ethanol solvents and to determine the useful properties of the resulting fractions: namely, molar mass distribution and phenolic hydroxyl groups. The highest concentration of lignin was achieved in 60 wt% ethanol solution with 235.89 g/L at a solid-to-liquid ratio of 300 $g_{Lignin}/L_{Solvent}$. The original lignin had a mass average molar mass of 4.7 kDa. When only water was utilized, the average molar mass of the dissolved fraction was between 1-2 kDa and the molecular size distribution was mostly between 0.1-1 kDa. When using pure ethanol, the molecular size distribution ranged from 0.1 kDa to 10 kDa, with a mass average molar mass between 1-1.3 kDa. With an ethanol content in the solvent higher than 90 wt%, the bigger molar mass molecules (>10kDa) could be separated as the insoluble fraction. Additionally, the polydispersity of both fractions decreased with an ethanol concentration above 80 wt%. The hydroxyl group content of the insoluble fraction was higher when fractionating with less than 20 wt% ethanol, whereas with 20 wt% ethanol and higher, the soluble fraction retained most of the phenolic hydroxyls. Fractionating lignin prior to use or further processing may be potentially beneficial if the lower molar mass molecules can be used as they are, leaving only the higher molar mass molecules to be further processed or burned for energy.

1. Introduction

The solubility of lignin is a varying property. Due to the highly heterogeneous nature of lignin, its solubility in different solvents will differ depending on the lignin type and origin. In simple compounds, solubility can be as straightforward as identifying the polarity or lack thereof of the compounds to assess their solubility (e.g., NaCl in water). In polymers, however, several factors come into play: solubility may be affected by polarity, degree of polymerization, and cross-linking, as well as the pH of the solvent [1]. It is especially challenging to determine solubility in lignin, which is a highly heterogeneous material [2]. It is well known that kraft lignin such as Indulin AT is completely soluble in alkaline solvents, e.g., aqueous NaOH. Indulin AT kraft lignin is also