



Separation of lignosulfonate from spent liquor of neutral sulphite semichemical pulping process via surfactant treatment



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ARTICLE INFO

Article history:

Received 13 January 2015

Received in revised form 2 July 2015

Accepted 8 July 2015

Available online 10 July 2015

Keywords:

Lignosulfonate
Separation
Purification
NSSC process
Biorefinery

ABSTRACT

The spent liquor, SL, of neutral sulphite semi chemical pulping process contains lignosulfonate and hemicelluloses, but it is currently treated in the wastewater treatment of the process. This treatment decomposes the lignosulfonate. However, lignosulfonate can be extracted for a better use, but the extraction process is not an easy task. The main objective of this work was to extract lignosulfonate from SL via treating the SL with surfactant. The results showed that the acidification of SL to pH 1.8 caused 6% lignosulfonate, 53% hemicelluloses, and 7% COD removals from SL. Acidification followed by dodecyltrimethylammonium chloride, DTAC, treatment of SL at the optimal conditions of 22 °C, 1.8 pH and 10 mg/g of DTAC/SL yielded 40% lignosulfonate, 78% hemicelluloses, and 25% COD removals. Model solutions were prepared via dissolving commercial lignosulfonate and xylan in solutions or purifying SL via membrane dialysis in order to investigate the impact of SL components, i.e. hemicelluloses and residual cooking chemicals, on the isolation of lignosulfonate from SL. The model analyses confirmed that the presence of impurities, i.e. residual cooking salts, significantly affected the isolation of lignosulfonate from the SL, but the presence of hemicellulose had a minor effect in extracting lignosulfonate from SL via DTAC treatment. In addition, the elemental analysis confirmed that DTAC and lignosulfonate had a ratio of 0.584 mol/mol in precipitates.

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1. Introduction

Neutral sulphite semi chemical (NSSC) pulping process is widely used for producing corrugating medium paper [1]. The spent liquor (SL) of this process contains lignocelluloses. Currently, the SL of the NSSC process is treated in the wastewater systems or incinerated in a fluidized bed reactors, which can help recover the cooking chemicals [2], but the lignocelluloses in the SL are wasted (i.e. underutilized) in either case.

To help increase the profitability of this pulping process, value-added products can be generated from lignocellulosic materials dissolved in the SL. However, the SL is dilute and its utilization in its present form is uneconomical. It will be necessary to recover the lignocellulosic materials from the SL in an economically viable manner in order to produce value-added chemicals from the lignocellulosic materials of SL. Once isolated, lignosulfonate can be modified to produce adsorbents, foam composites, polymer films, adhesives, and stabilisers [3].

However, there is limited information available in literature on the isolation of lignosulfonate from SLs. In the past, ultrafiltration was proposed as a method to recover lignocellulosic materials from SLs, and the results indicated that a single stage ultrafiltration could remove 80% of the COD, which indirectly shows that lignocelluloses were considerably separated from the SL [4]. However, ultrafiltration is susceptible to fouling issues and thus it may have operational difficulties [4,5]. Pretreatment steps such as centrifugation followed by microfiltration [6] or surface modification (coating) of ultrafilters may help the membrane technology [7,8] and minimize the fouling problem [5]. However, the pretreatment step or surface coating may be expensive.

The flocculation of lignocelluloses with cationic polymers was studied on various SLs in the past [9–11]. In one study, the flocculation of lignocelluloses in the SL of NSSC process using cationic polymers such as polyethyleneimine (PEI) and polydimethyl diallyl ammonium chloride (PDADMAC) was studied in single and dual polymer systems [12]. The study revealed that, under optimum conditions of pH 6, 30 °C, and 15 mg/g PEI/SL, 37% of both lignosulfonate and hemicelluloses were removed from SL. In this case, the dual polymer system led to lignosulfonate and hemicelluloses removals of 47% and 50%, respectively, under the conditions

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of pH 6, 30 °C, and 7.5 mg PEI and 7.5 mg/g PDADMAC/SL weight ratio [12].

Surfactants are amphiphilic materials with a hydrophilic head, which may be anionic, cationic, or non-ionic, and a hydrophobic tail consisting of different hydrocarbon moieties [13]. Recently, a study on the pre-hydrolysis liquor (PHL) of a kraft-based dissolving pulp production process showed that overliming followed by the use of dodecyltrimethylammonium chloride (DTAC) surfactant reduced 43.6% of lignin and 27% of hemicellulose from the PHL [14]. Since the lignocellulosic materials of PHL and SL are different, the results generated via treating PHL with DTAC cannot help predict the impact of SL treatment with surfactants. Once surfactants are added to SL, the cationic or non-ionic head of the surfactants may associate with the sulphate group of lignosulfonate through ionic or ion-dipole interactions, which may increase the overall hydrophobicity and size of lignosulfonate in SLs.

In the past, the complex formation between lignosulfonate and cationic polymers [15,16] and between lignosulfonate and cationic surfactants were studied [17,18], but all on purified lignosulfonate solutions. As the spent liquors of the NSSC pulping process contains hemicelluloses, salts and other impurities, the results of these papers may not help in developing a process for separating lignocelluloses from industrially produced SL of the NSSC process. The main novelty of this work is the investigation on the use of surfactants as a means for isolating lignocelluloses from industrially produced SL. The first objective of this work was to study the performance of various surfactants in isolating lignosulfonate from the SL of an NSSC process. This is the first report on the use of a surfactant in the spent liquor of NSSC process for extracting lignosulfonate. The outcomes of this analysis helped select the most effective surfactant, DTAC, for further study. The second objective of this study was to optimize the treatment conditions so that DTAC could effectively extract lignosulfonate from SL. To investigate the impact of hemicelluloses and salt on lignosulfonate removal, model solutions were also prepared and the impact of DTAC in isolating lignosulfonate from these model solutions were compared with that from industrially produced SL.

2. Materials and methods

2.1. Materials

Industrially produced SL was obtained from an NSSC pulping process that is located in Eastern Canada. The SL was then centrifuged at 3000 rpm for 15 min using a Thermo Scientific Sorvall ST 16 centrifuge prior to further use in order to remove large undissolved particles. Sodium lignosulfonate powder was obtained from Sigma Aldrich and a 5 wt.% model lignosulfonate solution (MLS) was prepared with sodium lignosulfonate using distilled water. Xylan from beechwood was obtained from Sigma Aldrich and a 1.5 wt.% model xylan solution (MXS) was prepared from it using distilled water. Polyethylene glycol sorbitan monolaurate based surfactants: Tween® 20 and Tween® 80, polyethylene glycol hexadecyl ether based surfactants: Brij® 58 and Brij® C10, quaternary ammonium based surfactants: tetrabutylammonium iodide (TBAI) and dodecyltrimethylammonium chloride (DTAC), sodium hydroxide (98 wt.%), sulphuric acid (98 wt.%), 3,5-dinitrosalicylic acid, sodium potassium tartrate (Rochelle salt), phenol, and sodium meta bisulphate were all purchased from Sigma Aldrich. All surfactants were diluted to a 1.0 wt.% solution prior to use. The chemical oxygen demand (COD) kit (K-7365) was obtained from CHEMetrics Inc., USA. Dialysis membranes with a molecular weight cut off of 1000 g/mol were obtained from Spectrum Labs.

2.2. Treatment of SL with surfactants

In one set of experiments, 0.15 g (oven dried) of the different surfactants were added to 20 g SL at its original pH (5.6), and then the solutions were shaken at 100 rpm and 30 °C for 10 min in a Boekel shaking water bath. Previous studies confirmed that flocculation/coagulation of lignocelluloses occurred rapidly and that a period of 10 min would be sufficient for forming lignin/surfactant complexes [10,14]. The samples were centrifuged at 2500 rpm for 10 min in a Thermo Scientific Sorvall ST 16 centrifuge. The lignosulfonate content and the turbidity of the SL were measured before and after the surfactant treatment. DTAC was the only surfactant to exhibit an effective lignosulfonate removal, and thus was selected for further study.

In another set of experiments, the pH of SL was adjusted with sulphuric acid or sodium hydroxide to 1.8, 4, 7, 9, and 11. Then, the pH adjusted SLs were centrifuged at 2000 rpm for 10 min prior to the DTAC treatment. This pH adjustment and centrifugation helped remove some of the lignosulfonate from SL. Subsequently, 0.15 g (oven dried) of DTAC was added to 20 g of the pH adjusted SLs, and then the samples were shaken at 100 rpm in a water bath shaker for 10 min at different temperatures of 22 °C, 50 °C, or 70 °C. The samples were subsequently centrifuged at 2500 rpm for 10 min. The lignosulfonate content and the turbidity of these samples were measured after acidification and DTAC treatment in order to determine the optimum pH and temperature for treating SL with DTAC for lignosulfonate removal. This set of experiments confirmed the optimal conditions of 22 °C and pH of 1.8 for DTAC treatment.

In another set of experiments, various dosages (up to 0.35 g, oven dried) of DTAC was added to 20 g of SLs at pH 1.8 and mixed at 22 °C in the water bath shaker at 100 rpm for 10 min, and then centrifuged at 2500 rpm for 10 min. The lignosulfonate content and the turbidity of the treated samples were measured in order to determine the effect of DTAC dosage on lignosulfonate removal. This analysis confirmed that the optimal DTAC/SL ratio was 10 mg/g.

To determine the impact of DTAC on properties of SL, 0.20 g of DTAC was added to 20 g of SL at pHs 1.8 and 5.6 (original pH) and then the mixture was subjected to shaking in the water bath shaker at 22 °C and 100 rpm for 10 min. Subsequently, the samples were centrifuged at 2500 rpm for 10 min. The lignosulfonate, COD, and hemicelluloses contents as well as the turbidity of the treated SL were measured. Furthermore, the COD of the sample, which was produced via adding 0.2 g DTAC to 1 g of distilled water (control sample), was determined.

2.3. Treatment of model lignosulfonate and xylan solutions with DTAC

In one set of experiments, the pH of model lignosulfonate solution (MLS) and model xylan solution (MXS) was adjusted with sulphuric acid or sodium hydroxide to 1.8, 4, 7, 9, and 11. Then, the pH adjusted MLS and MXS were centrifuged at 2000 rpm for 10 min prior to the DTAC treatment. Subsequently, 20 g of these samples was treated with 0.15 g of DTAC and shaken at 22 °C, 50 °C, or 70 °C in a Boekel water bath shaker at 100 rpm for 10 min. Then, the samples were centrifuged at 2500 rpm for 10 min. The lignosulfonate content and the turbidity of the DTAC-treated MLS were determined afterwards.

In another set of experiments, various dosages (up to 0.35 g) of DTAC was added to 20 g of MLS, mixed at pH 1.8 and 22 °C in the water bath shaker at 100 rpm for 10 min, and then centrifuged at 2500 rpm for 10 min. The lignosulfonate content and turbidity of the DTAC-treated MLS were measured in order to determine the effect of DTAC dosage on lignosulfonate removal from MLS.

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