Production of cellulose from sugarcane bagasse for adsorption of copper ions

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Abstract

The presence of metal ions in drinking water as well as high concentrations of heavy metals in wastewater are serious issues that warrant greater scrutiny. As such, utilizing natural materials to adsorb metals from aqueous solutions is an attractive solution as it is inexpensive. Therefore, the present study evaluated the efficacy of utilizing cellulose derived from sugarcane bagasse (SB) as a bio-adsorbent precursor to remove copper ions (Cu$^{2+}$). This study aimed to determine the optimum acid to SB ratio by combining varying acid concentrations at different temperatures during acid hydrolysis for cellulose isolation. A microscope as well as Fourier-transform infrared (FTIR) spectroscopy were used to observe the morphology and structure of these bio-adsorbent precursors, respectively. Atomic absorption spectrometry (AAS) was then used to determine the efficacy of the bio-adsorbent precursors by measuring the Cu$^{2+}$ concentrations, and percentage of Cu$^{2+}$ removed. Microscope image analysis showed that SB had rigid fibre bundles prior to acid hydrolysis. This changed to a smoother surface post-hydrolysis as the binding materials ruptured. FTIR spectroscopy showed an average peak of 3,400 cm$^{-1}$ within the 3,500 to 3,000 cm$^{-1}$ spectral bands with varying peak intensities, which alluded to the presence of different concentrations of O-H bonds depending on the nature of the treatment. More significant peaks between the 1,500 to 1,000 cm$^{-1}$ range were observed in the hydrolysed SB than in the raw SB, indicating the successful removal of hemicellulose and lignin. In the adsorption experiment, SB that had been hydrolysed with 3 M H$_2$SO$_4$ sulphuric acid provided the highest adsorption efficiency (86.6%) after 24 h. Hence, it can be concluded that cellulose derived sugarcane bagasse is a promising ‘green’ adsorbent for the removal of copper ions from an aqueous solution.

Keywords: Adsorbent; Cellulose; Heavy metal; Hydrolysis; Sugarcane bagasse

1. Introduction

An increase in the amount of environmental pollutants is an inevitable by-product of industrial growth in developing countries. As such, development often comes with considerable costs, for instance, a loss of natural resources as well as an inability to preserve the natural state of the environment. Water pollution is a foremost issue of the various types of pollutions caused by urban advancements. Industrial effluents containing common heavy metals such as copper, cadmium, lead, nickel, and chromium are an environmental and public health concern as they are non-biodegradable, toxic, and bioaccumulative in nature [1]. These heavy metals pose health risks at cumulated concentrations such as high blood pressure, fatigue, speech disorders, sleep disorders, behavioural disorders, increased allergic reactions, autoimmune diseases, vascular occlusion, and memory loss; in humans [2]. In Malaysia, the
Department of Environment (DOE) enforces strict regulations for the industrial effluents discharged upstream of raw water intake points. For instance, the allowable amount of copper is only 0.20 mg/L, a considerably low amount. Regardless, it is still essential to develop effective heavy metals removal methods that improve water quality.

Heavy metal removal techniques can be categorized as physical, chemical, or biological. Although each technique possesses advantages and disadvantages depending on the systems used, a combination of these techniques is often employed for better efficiency. The most common methods of heavy metal removal are coagulation, membrane filtration, adsorption, electrochemical treatment, electrodialysis, ion exchange, photocatalysis, biological treatment, and oxidation [3–5]. Of these methods, adsorption is regarded as the most efficient and economical, and it also has a highly flexible design and operation. Furthermore, adsorption also generates its adsorbents which is economically useful [6]. Studies have investigated utilizing adsorption in heavy metal removal without causing further environmental degradation. A popular solution is to re-evaluate and transform solid waste into valuable materials such as bio-adsorbents via various preparation and production techniques.

Biomass solid waste is advantageous as it is naturally occurring and inexpensive [7]. Apart from being an emerging “green technology”, the utilization of biomass as a solution to minimizing pollution and solid waste is also being investigated. As agricultural waste is a potential bio-adsorbent, biomass will never be in short supply in countries with highly active agricultural sectors. Examples of agricultural waste include raw cotton linter [8], corn stover [9], eucalyptus pulp [10], peanut shell [11], rice husk [12], water hyacinths [13], and sugarcane bagasse (SB) [14,15]. These lignocellulosic materials contain varying compositions of cellulose, lignin, and hemicellulose, depending on the source. Of the aforementioned lignocellulosic materials, SB contains the best balance of the three components [16], which makes it a viable bio-adsorbent. Therefore, SB that has been converted into a bio-adsorbent could potentially be applied in wastewater treatment.

The constituent of interest in the adsorption process is cellulose. With an estimated 700 billion tons produced annually, naturally occurring cellulose is one of the most abundantly available organic materials on the earth [17]. However, cellulose has a rigid structure that requires several steps to isolate it from its source. These isolation techniques include (1) mechanical methods such as steam explosion, mechanical defibrillation, ultrasonication, and high-pressure homogenization; (2) chemical methods such as acid hydrolysis and ionic liquid treatment; and (3) biological methods such as enzyme technology [18–20]. As plant cellulose is particularly tough due to its internal semi-crystalline structure, acid hydrolysis is the most established, efficient, and commonly used isolation technique [21]. Strong acids such as sulphuric acid (H₂SO₄) are commonly used to isolate cellulose at low temperatures.

Synthetic adsorbents are not only expensive but non-biodegradable on occasion. Furthermore, fresh raw materials need to be processed to produce synthetic adsorbents, which is hardly eco-friendly. While some studies have focused on pre-treatments for cellulose production (green options), other studies have evaluated and compared the performance of various cellulose sources as precursor adsorbents. Furthermore, the industry has most likely delayed the adoption of cellulose as an adsorbent for the removal of pollutants from wastewater due to a lack of research on the optimum parameters of cellulose production. Thus, it is essential to obtain the highest amount of cellulose from the SB by evaluating the most suitable process setting for the hydrolysis process. Therefore, this study aims to determine the optimum acid to SB ratio and the best temperature for cellulose production during acid hydrolysis. The ability of the prepared bio-adsorbent to remove copper from an aqueous solution was then investigated.

2. Methodology

2.1. Material

Sugarcane bagasse (SB) was obtained from a local market in Kuching, Sarawak, Malaysia. Sodium chloride (NaClO₂) powder (80%, Macklin), sodium sulphite (Na₂SO₃) powder (97%, Merck), sodium hydroxide (NaOH) pellets (95%, R&M Chemicals) and dimethyl sulphoxide solution (DMSO) (99%, Sigma-Aldrich) were used to bleach as well as remove lignin and hemicellulose from the SB. The sodium chloride, sodium sulphite, and sodium hydroxide were prepared using distilled water to obtain 1 M solutions. Sulphuric acid (98%, R&M Chemicals) was diluted to 1 M, 2 M, and 3 M concentrations for the acid hydrolysis process. For the adsorption experiment, copper solutions were prepared using copper(II) sulphate salt (95%, R&M Chemicals). The reagents used in this study were of analytical grade and were used without further purification. The adsorption of Cu²⁺ was also carried out using charcoal activated carbon (Friendemann Schmidt) for comparison.

2.2. Preparation of sugarcane bagasse

The SB was first dried in an air-drying oven (Memmert, UF750 Plus) at 75°C for 24 h to remove all traces of moisture. It was then ground in an industrial grinder (Retsch, SM100) which was equipped with a 250 µm aperture metal grate. The resulting SB powder was divided into multiple batches of 20 g each then stored for further use.

2.3. Cellulose preparation from SB powder

The systematic procedure outlined by Mandal and Chakrabarty [22] was used to isolate the cellulose from the SB in two batches. Each batch of SB powder was first divided into 10 g batches as bleaching involves a lot of frothing, and too much SB powder would have caused an overflow. The SB powder was bleached at a ratio of 20 g SB to 500 mL NaClO₂ (1 M) at 70°C for 5 h. The colour changed from yellow to off-white during the bleaching process. The mixture also became thick and dense at the end of the process as the lignin was removed. The concoction was then filtered through a cotton filter bag and washed with distilled water until a neutral pH was achieved. 500 mL Na₂SO₃ (1 M) was then added to the resulting residue and boiled at 70°C for 5 h to completely remove lignin and partially remove hemicellulose. The mixture was then filtered, once
again, through a cotton filter bag and washed with distilled water until a neutral pH was achieved. The remaining material was subjected to alkali treatment with 500 mL of NaOH (1 M) for 5 h to remove the remaining hemicellulose. The extracted residue was then filtered through a cotton filter bag and thoroughly washed with distilled water until it achieved a neutral pH. The remaining cellulose materials were left to air-dry overnight before added to 100 mL DMSO in an 80°C water bath for 3 h. The resulting mixture was filtered through a cotton filter bag and washed until a neutral pH was obtained then air-dried.

2.4. Cellulose isolation using acid hydrolysis

The resulting mixture was then divided into 1 g samples and diluted with varying H₂SO₄ concentrations of 1 M, 2 M, and 3 M at a fixed temperature of 80°C and an agitation rate of 300 rpm. This was performed to ascertain the optimum acid concentration required to yield the highest amount of cellulose. The second batch of cellulose was isolated at a fixed agitation rate of 300 rpm and at varying temperatures of 65°C, 75°C, and 80°C. The final product was filtered from the suspension using Whatman® filter papers.

2.5. Characterization

2.5.1. Microscope image analysis

Image analysis by microscope (Olympus, Model: BX41-PDP72) was used to capture images of the cellulose surface at 20X and 40X magnification powers.

2.5.2. Fourier-transform infrared spectroscopy

Fourier-transform infrared (FTIR) spectroscopy was used to determine the functional groups present in the dried SB-derived cellulose. The spectra were scanned from 500 to 4,000 cm⁻¹.

2.6. Adsorption experiment

Copper (II) sulphate salt was dissolved in distilled water to obtain a standard copper solution. A batch experiment approach was employed in which 0.1 g of dried cellulose was placed in a conical flask containing 100 mL of the copper solution. The flask was then placed in a thermostatic shaker at 30°C at 150 rpm for 24 h to obtain equilibrium adsorption. Samples were collected at 30-min intervals during the first 2 h. An atomic absorption spectrometer (AAS) (Hitachi, Z-2000) was used to measure the concentration. The percentage of Cu²⁺ removed was calculated using Eq. (1) where \( C_i \) is the initial concentration of the Cu²⁺ and \( C_{eq} \) is the residual concentration of Cu²⁺ left in the sample after 24 h.

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\text{Percentage removal of Cu}^2+ = \frac{C_i - C_{eq}}{C_i} \times 100 \tag{1}
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3. Results and discussion

3.1. Cellulose extraction using acid hydrolysis

Fig. 1 depicts how cellulose was extracted from its raw state via the acid hydrolysis process. Fig. 1a shows the SB that was purchased from a local market. Fig. 1b shows the dried SB that was ground in an industrial grinder to obtain an SB powder with a uniform size of approximately 250 µm. The SB powder was subjected to a series of pre-treatments prior to acid hydrolysis in order to bleach and remove lignin and hemicellulose. Fig. 1c depicts the bleaching process as the first pre-treatment step. After 5 h, the colour of the SB changed from yellow to off-white. DMSO is then treated to swell the biomass matrix and ensure better acid diffusion for cellulose disintegration [23]. As seen in Fig. 1d, the lignin and hemicellulose removal process decreased the mass of the SB powder from 10 g to approximately 6 g. Acid hydrolysis was then performed for 10 h under five varying conditions; three varied H₂SO₄ concentrations of 1 M, 2 M, and 3 M as well as two different temperatures of 65°C and 75°C; until the suspension seen in Fig. 1e was obtained. As depicted in Fig. 1f, the suspensions were then filtered and dried to produce the substance shown in Fig. 1g. The mass of cellulose obtained by the 1 M H₂SO₄ sample was 0.73 g. This was heavier than the mass of cellulose obtained using the 2 M H₂SO₄ and 3 M H₂SO₄ samples which were 0.435 and 0.264 g, respectively. Hydrolysing 1 M H₂SO₄ at varying temperatures of 65°C and 75°C resulted in a significant reduction in cellulose mass compared to cellulose obtained by hydrolysing 1 M H₂SO₄ at 80°C. Although a significant amount of solid treated cellulose was found after 10 h, it was not completely broken down into a cellulose suspension. Other studies have found that performing acid hydrolysis for 1 h at 70°C resulted in partial hydrolysis [24]. Therefore, higher temperatures are required for lower acid concentrations.

3.1.1. Microscope image analysis

Microscope images of the various stages of cellulose preparation are shown in Fig. 2. Fig. 2a shows the surface of the alkali-treated SB (ALK-SB) sample where rod-like structures are seen in bundles. These structures still had a well-defined shape, indicating that the lignocellulosic materials were intact. Fibres that were still regularly arranged and bound closely together in bundles could be attributed to the presence of binding materials, such as hemicellulose, lignin, oil, and wax [19]. The surface of the ALK-SB sample was also found to be homogenous in comparison to the surfaces seen in Fig. 2b–f.

The cellulose obtained by hydrolysing 1 M H₂SO₄ (1 M SB) is shown in Fig. 2b. A significant difference was noted between the surface images of the 1MSB and ALK-SB samples. The well-defined bundle structures were completely absent and, instead, replaced by a gel-like rough surface. This could be because further treatment of the cellulose destroyed cell tissues resulting in a rougher surface, where the hemicellulose coated the cellulose fibrils, and lignin filled the vacant areas [25]. Additionally, alkali-treated was claimed as an effective pre-treatment for delignification [26]. Fig. 2c and d show the surface images of cellulose produced using 2 M H₂SO₄ (2MSB) and 3 M H₂SO₄ (3MSB), respectively. No significant differences were observed in comparison to the surface of the ALK-SB sample (Fig. 2a). However, the surfaces of the 2MSB and 3MSB samples were slightly rougher than the 1MSB sample. The cellulose was found to be clumped up together for 2MSB and 3MSB samples. Nano
Fig. 1. Cellulose extraction process, (a) raw sugarcane bagasse (SB), (b) dried and ground SB, (c) alkali pre-treatment, (d) dried cellulose, (e) cellulose in emulsion after acid hydrolysis, (f) filtration of cellulose, and (g) dried cellulose.
fibres are known to coagulate at acid concentrations above 0.2 wt.% [17] which explained the presence of more clumps on the surfaces of the 2MSB and 3MSB samples. Fig. 2e and f show the cellulose obtained by hydrolysis at 75°C and 65°C, respectively. The surfaces of both the SB75 and SB65 samples were rough with a slight mixture of clumped cellulose and structured bundles. This lack of smooth surfaces could be attributed to partial hydrolysis at lower temperatures.

3.1.2. Fourier-transform infrared spectroscopy

Functional groups; such as alkanes, aromatics, esters, alcohols, and ketones; are naturally present in the cellulose, hemicellulose, and lignin of fibrous plants [19]. Moreover, various functional groups present in biomass such as amido groups, carbonyl, phenolic, polysaccharides, amino, carboxyl group alcohols and esters can promote metal chelation and promising for metal removal from wastewater [27]. Although common peak areas were observed in all the samples, the band strengths varied as the SB underwent more treatment. Fig. 3a and b show the spectrum peaks of raw sugarcane bagasse (RSB) and SB post-alkali treatment (ALK-SB) with NaOH, respectively. A small peak was observed around the 3,350 cm⁻¹ spectral bands, with a more significant band in the ALK-SB sample than the RSB sample. Peaks within the 3,175 to 3,490 cm⁻¹ spectral bands for original cellulose alluded to O–H stretching of intramolecular hydrogen bonds [21]. The ALK-SB sample had a broader band in the 3,400 cm⁻¹ spectral bands. The intensity of this band could be attributed to the presence of moisture [28], while a broad band within the 3,500 to 3,200 cm⁻¹ spectral regions indicates the free O–H stretching common in cellulose molecules [22]. None significant peaks were observed within the 3,000 to 2,000 cm⁻¹ range for both the RSB and ALK-SB samples. Additionally, C–H stretching is known to peak around 2,850 to 2,970 cm⁻¹ [21], indicating the absence of the C–H bond. A significant peak was seen at approximately 1,750 cm⁻¹ in the RSB sample. This can be attributed to the stretching of C=O bonds predominantly found in hemicelluloses [15]. This did not occur in the ALK-SB sample as hemicellulose had been removed. The peaks at approximately 1,100 and 1,000 cm⁻¹ in both the RSB and ALK-SB samples were the vibrations of the C–O–C bonds [21].

As seen in Fig. 3a–c, the peak differences between the RSB and ALK-SB samples were compared against peaks in the SB samples after acid hydrolysis using 1MSB, 2MSB, and 3MSB. Significant peaks were observed at approximately 3,300 cm⁻¹ in the 1MSB, 2MSB, and 3MSB samples. This indicates strong vibrations of O–H hydroxyl groups [19]. The O–H bonds stretching appeared to intensify in samples produced with higher acid concentrations. Similarly, the lack of significant peaks between the 3,000 to 1,800 cm⁻¹ spectral bands indicated the absence of or lack of C–H alkyl and alkene groups in all the samples. Absorbance peaks were found within the 1,649 to 1,641 cm⁻¹ region of the cellulose fibres. This was attributed to the O–H bending of adsorbed water [22]. The predominant peaks noted between the 1,636 to 1,641 cm⁻¹ spectral bands of the 1MSB, 2MSB and 3MSB samples indicated the presence of O–H groups from adsorbed water. This peak was less dominant in the SB sample before acid hydrolysis. Another set of dominant peaks was observed at approximately 1,100 cm⁻¹ in all the acid-hydrolysed SB samples which indicated C–O stretching. Spectral peaks between the 1,080 and 900 cm⁻¹ spectral bands could be attributed to interactions between glucose which indicate the removal of hemicellulose and lignin species [29]. A common peak at 1,030 cm⁻¹ was observed in all the samples. This appeared to intensify post-acid hydrolysis, indicating the removal of hemicellulose and lignin.

Fig. 4 illustrates the FTIR peaks of the RSB and ALK-SB with SB acid hydrolysed at 1 M H₂SO₄ at 80°C (SB80), 75°C (SB75), and 65°C (SB65). Within the 3,500 and 3,000 cm⁻¹ range, the presence of O–H stretching is known to peak around 2,850 to 2,970 cm⁻¹ [21], indicating the absence of the C–H bond. A significant peak was seen at approximately 1,750 cm⁻¹ in the RSB sample. This can be attributed to the stretching of C=O bonds predominantly found in hemicelluloses [15]. This did not occur in the ALK-SB sample as hemicellulose had been removed. The peaks at approximately 1,100 and 1,000 cm⁻¹ in both the RSB and ALK-SB samples were the vibrations of the C–O–C bonds [21].
spectra, similar dominant peaks were noted around 3,400 cm⁻¹ in all the hydrolysed samples. This suggested the presence of similar concentrations of O–H functional groups. Similarly, no significant peaks were noted in all the samples between 2,900 and 1,750 cm⁻¹, which alluded to the absence of C–H functional groups. The SB80, SB75, and SB65 samples had highly significant peaks at 1,700 cm⁻¹. This could be attributed to a strong stretching of the C–O functional group connected to acetyl and ester linkages in cellulose [21]. All these three hydrolysed samples exhibited similar significant peaks within 1,500 and 1,000 cm⁻¹ and they have almost similar intensities. Similar to Fig. 3, a distinct peak at 1,030 cm⁻¹ indicates the removal of lignin and hemicellulose, and this was not observed in the RSB sample.

Cellulose from sugarcane bagasse was derived via acid hydrolysis of 1 M, 2 M, and 3 M H₂SO₄ concentrations at
80°C. Although hydrolysis at 75°C and 65°C successfully removed hemicellulose and lignin, the samples had undergone only partial hydrolysis as they did not completely turn into suspensions. This was evidenced by the presence of bundled structures that were observed during the analysis of their surface images. The FTIR spectroscopy results showed a general peak within the 3,500 to 3,000 cm⁻¹ spectral band and varying peak intensities that indicated the presence of different O-H bond concentrations, depending on the type of treatment. As peaks between 1,500 to 1,000 cm⁻¹ indicate the successful removal of hemicellulose and lignin, these were more significant in hydrolysed SB than raw SB. The batch adsorption experiment found that cellulose produced using a 3M concentration of sulphuric acid (H₂SO₄) had the highest Cu²⁺ removal percentage (86.6%) after 24 h. Therefore, it can be concluded that acid concentration had a more profound effect on the performance of the isolated cellulose than temperatures during hydrolysis. Lower temperatures of 75°C and 65°C were unable to hydrolyse the SB in comparison to 80°C completely. As such, a combination of higher acid concentration and higher temperature may expedite the hydrolysis process and produce cellulose that is better suited for Cu²⁺ adsorption.

3.1.3. Adsorption of Cu²⁺ by cellulose adsorbents

The adsorption of copper ions (Cu²⁺) experiments were performed to determine the adsorption capacity of the prepared cellulose samples. Activated carbon was used to compare the Cu²⁺ adsorption capacity of the 1MSB, 2MSB, and 3MSB samples. As the samples that had been prepared at various temperatures (SB80, SB75, and SB65) only underwent partial hydrolysis and possessed structures that were similar to the 1MSB sample, they were excluded from the adsorption experiment. A copper solution with an initial concentration of 255 mg/L was first prepared. The batch adsorption was conducted for 24 h, with samples collected every 30 min during the first 2 h and at the end of the 24 h. Eq. (1) was used to calculate the percentage of Cu²⁺ that was removed. It was observed that the percentage of Cu²⁺ removal significantly increased after 30 min of adsorption. As seen in Fig. 5, the 1MSB, 2MSB, and 3MSB samples removed 45.5%, 48.7%, and 83.3% of Cu²⁺, respectively. Therefore, higher acid concentrations for the acid hydrolysis of SB resulted in higher adsorption of Cu²⁺. Compared to the lower molarity of acid (1 M and 2 M), 3M sulphuric acid (3MSB) provides the best concentration to extract the cellulose and successfully remove the lignin and hemicellulose from SB. Higher molarity of acid hydrolysis (for instance, 72%, which is equivalent to 13.4 M) can completely dissolve the crystalline cellulose resulting in the homogeneous hydrolysis of cellulose, for which the cellulose is converted into several oligosaccharides containing glucose units, mainly cellobiose and glucose [30]. However, having oligosaccharides as the final product is not the aim of this work.

After 30 min of adsorption time, 3MSB samples removed 83.3% of Cu²⁺. The percentage of Cu²⁺ removed by the 3MSB sample only increased to 86.6% after 24 h of adsorption time. Therefore, as the adsorption time extended from 30 min to 24 h, the 3MSB sample only removed less than 5% of Cu²⁺. Another study that used chemically modified SB to remove Cu²⁺ found that the adsorption percentage increased between 5 to 40 min but plateaued between 20 to 240 min [31]. A similar study that used the same amount of SB bio-adsorbent (0.1 g) reported that Cu²⁺ adsorption reached equilibrium at 40 min [32]. Therefore, despite the increased contact time, the active sites of the adsorbents have all interacted with the metal ions within the first few minutes and can no longer be occupied. Comparatively, activated carbon removed only 70.1% of Cu²⁺ within the first 30 min and only 81.8% after 24 h. Therefore, the 3MSB removed more Cu²⁺ than activated carbon. As such, it is safe to conclude that cellulose produced using higher acid concentrations serve as a better adsorbent.

4. Conclusions

Cellulose from sugarcane bagasse was derived via acid hydrolysis of 1 M, 2 M, and 3 M H₂SO₄ concentrations at 80°C. Although hydrolysis at 75°C and 65°C successfully removed hemicellulose and lignin, the samples had undergone only partial hydrolysis as they did not completely turn into suspensions. This was evidenced by the presence of bundled structures that were observed during the analysis of their surface images. The FTIR spectroscopy results showed a general peak within the 3,500 to 3,000 cm⁻¹ spectral band and varying peak intensities that indicated the presence of different O-H bond concentrations, depending on the type of treatment. As peaks between 1,500 to 1,000 cm⁻¹ indicate the successful removal of hemicellulose and lignin, these were more significant in hydrolysed SB than raw SB. The batch adsorption experiment found that cellulose produced using a 3M concentration of sulphuric acid (H₂SO₄) had the highest Cu²⁺ removal percentage (86.6%) after 24 h. Therefore, it can be concluded that acid concentration had a more profound effect on the performance of the isolated cellulose than temperatures during hydrolysis. Lower temperatures of 75°C and 65°C were unable to hydrolyse the SB in comparison to 80°C completely. As such, a combination of higher acid concentration and higher temperature may expedite the hydrolysis process and produce cellulose that is better suited for Cu²⁺ adsorption.

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removed hemicellulose and lignin, the samples had undergone only partial hydrolysis as they did not completely turn into suspensions. This was evidenced by the presence of bundled structures that were observed during the analysis of their surface images. Additionally, lower temperatures (75°C and 65°C) were unable to completely hydrolyse the SB and aspenwood: a statistical approach, BioResources, 7 (2012) 301–316.


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