Isolation and Characterization of Cellulose Obtained from Bagasse Pith by Oxygen-Containing Agents

Xin Gao, Ke-Li Chen, Heng Zhang, Lin-Cai Peng, and Qi-Xing Liu

The environmentally-friendly procedure of oxygen treatment (O) and peroxide treatment with oxygen and anthraquinone as a catalyst (Po-AQ) was studied for isolation of cellulose from bagasse pith (BP) mainly consisting of parenchyma cells. The optimal conditions were: 20% alkali dosages with 12% BP consistency at 120 °C for 3 h under 0.6 MPa initial oxygen pressure for the O step; then 8% H₂O₂, 20% NaOH, and 0.3% AQ charges with 8% BP consistency at 120 °C for 6 h under the same oxygen pressure for the Po-AQ step. And the optimum process was: BP pretreated by cool-water extraction was firstly O-treated, and after finishing O treatment, intermediate pulp which was manually separated from effluent, was continuously processed by Po-AQ treatment. Based on the conditions above, this process can yield 37.1% of cellulose sample, containing 7.01% hemicelluloses and 0.57% lignin. The representative samples were characterized by gas chromatography (GC), Fourier transform infrared spectrometry (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM).

Keywords: Bagasse pith; Parenchyma cell; Cellulose isolation; Oxygen-containing treatment

Contact information: a: Faculty of Environmental Science and Engineering, Kunming University of Science and Technology, Kunming, 650500, PR China; b: Faculty of Chemical Engineering, Kunming University of Science and Technology, Kunming, 650500, PR China;
* Corresponding author: chenkeli_prof@sina.com

INTRODUCTION

Bagasse pith (BP) is a waste product from the depithing of bagasse. The latter is often utilized for papermaking, and the former is usually used as the complementary boiler fuel in cane mills as well as bagasse pulp mills because of the dominant presence of non-fibrous parenchyma cells in BP. It is a fact that the annual output of about 60 million tons of BP should be considered as an important biomass resource. Based on the similar composition of BP to depithed bagasse (Hamzeh et al. 2013; Huang et al. 2008), its application has the potential to become more valuable than the simple burning. It is noticed that cellulose microfibrils in the parenchyma cell walls are less uniform in diameter and less aligned than those in fibrous cells (Yu et al. 2008). As a result, the cellulose from pith parenchyma cells has a less dense super-molecular structure, leading its ideal properties to further derivative modification. Therefore, this inexpensive source provides an opportunity for producing value-added products, such as cellulose-related derivatives, cellulose composites, and so on. The isolation of pure cellulose in environmentally safe ways only from fibrous materials, such as rice husk (Rosa et al. 2012), barely straw (Sun et al. 2005), and bagasse (Sun et al. 2004) has been addressed in the scientific literature but not yet from bagasse pith.

Efficient processes have been developed to isolate cellulose from biomass, such as the organosolv method (Sun et al. 2004), steam explosion (Chen et al. 2007; Jiang et al. 2008).
2011), chlorine-free pulping and bleaching process (Xu et al. 2006), combined chemical and enzymatic extraction (Reddy and Yang 2006), etc. However, these methods always contain two or multi-step treatments, usually including the chemical pretreatment before cellulose isolation in order to achieve relatively complete removal of lignin and hemicellulose. With the development of cellulose solvents such as N-methylmorpholine N-oxidemonohydrate (Roder and Morgenstern 1999), LiCl/N,N-dimethylacetamide (McCormick et al. 1985), ionic liquids (Song et al. 2010), and sodium hydroxide aqueous solutions with or without urea/thiourea added (Cai and Zhang 2006; Zhang et al. 2002), alternative methods for isolating cellulose from raw materials are based on the dissolution of cellulose. But there are still some limitations for industrial application due to the high cost of chemicals, long period of cellulose dissolution, strictly controlled operation, and effluent-disposal problems (Zhong et al. 2013).

The oxygen-containing agents such as oxygen and hydrogen peroxide have been re-explored and utilized in biomass-refined fields. Peroxide and oxygen can form hydroxyl radicals and superoxide anion radicals in alkaline conditions (Shi et al. 2012), both of which can break the linkages between carbohydrates and lignin, leading to the dissolution of lignin and hemicelluloses. Anthraquinone (AQ) has already been a well-known catalyst for delignification, as well as being a polysaccharides protector in alkaline pulping (Hedjazi et al. 2009). Besides, sodium hydrogen (NaOH) plays an indispensable role not only in providing alkaline conditions for delignification but also in the effective hydrolysis of hemicelluloses. These chemicals have been widely used for pulping and/or bleaching of lignocellulosic materials, while utilization of these clean agents for cellulose isolation has not been reported yet.

In the present work we propose the use of BP as a new source for obtaining parenchyma cell cellulose by a clean and convenient process involving O₂, H₂O₂, and alkali as well as anthraquinone (AQ) as a catalyst. To meet the requirement of producing biopolymers (e.g. oxidized cellulose or cellulose acetate) related with BP cellulose in future work, we investigated the influence of primary parameters not only on the yield and on the content of cellulose and lignin, but also on the intrinsic viscosity of the preparations. The structural features and changes of the samples were characterized and compared via Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The aim was to gain comparatively pure cellulose and insight into the structure of parenchyma cell cellulose.

**EXPERIMENTAL**

**Materials**

Bagasse pith was obtained from Yunnan Xinping Nan’en Sugar and Paper Co., Ltd (Xinping, Yunnan Province, China). It was air dried and then sieved with 40 and 60-mesh stainless steel screens. The chemical composition (%, w/w) of the pith is cellulose 41.82%, holocellulose 75.88%, Klason lignin 21.47%, ash 0.59%, and wax 2.06% on a dry weight basis. Part of the pith were extracted with cool water at the conditions of 23 ± 2 °C for 48 h where 0.7 g/L of the mass was dispersed in the cool water. The extracted pith was washed with distilled water to remove dissolved substances and then air-dried. Untreated bagasse pith (UBP) and extracted bagasse pith (EBP) were prepared for the experiments in this study.
Methods

Isolation of cellulose

All experiments were performed in a 500-mesh nylon bag loading to a 0.8-L stainless steel reactor, which was set and rotated in a silicon oil thermo-electrical autoclave. At the O stage, the UBP (20 g) was well mixed with certain alkali and 0.5% MgSO$_4$ and was delignified at different temperatures and times. After this reaction, the reactor was taken out and rapidly cooled down below the ambient temperature in an ice-water bath. At the Po-AQ stage, the cooled intermediates were treated with the 0.5% EDTA at room temperature for 30 min in the reactor. The chelated pulp was continuously reacted after mixing AQ and peroxide with oxygen and additional alkali. After the process, the insoluble residue was collected by filtration, thoroughly washed with distilled water until the pH of the filtrate was neutral, then disintegrated and air-dried.

In order to further increase cellulose purity, the process was improved by such ways as using EBP as starting material and manually discharging effluent from intermediates after the O stage. To quantify errors and confirm the results, each experiment was repeated in duplicate under the same conditions.

Characterization of extractives and water liquor

The yield of cellulose extractives was calculated according to,

Extractive yield (%) = \( \frac{m_S}{m_{BP}} \times 100\% \) \hspace{1cm} (1)

where \( m_s \) and \( m_{BP} \) are the weights of oven-dried sample and BP raw material in g, respectively.

Holocellulose was measured following the procedure originally pioneered by Wise (Abe et al. 2007). Cellulose of the processing samples was prepared by the extraction of holocellulose with 10% KOH for 10 h at 20 °C (Sun et al. 2004). The residual lignin content in the cellulose sample was analyzed by reaction with sulfuric acid, using a standard method as recommended by TAPPI T222 om-88. Both cellulose and Klason lignin contents of the extractives were determined according to,

Cellulose or Klason lignin yield (%) = \( \frac{m_{C/KL}}{m_S} \times 100\% \) \hspace{1cm} (2)

where \( m_{C/KL} \) and \( m_S \) are the weights of oven-dried cellulose (or Klason lignin) and sample in g, respectively.

Viscosity of the preparations was measured by “British Standard Methods for determination of limiting viscosity number of cellulose in dilute solutions, Part 1. Cupriethylenediamine (CED) method (BS 6306: Part 1: 1982)”. The residual alkali in effluent was measured following the procedures given by Shi and He (2003).

The sugar composition of the samples was determined by gas chromatography (GC) analysis of the corresponding alditol acetates according to TAPPI T 249 cm-85.

Fourier transform infrared (FT-IR) spectra of the extractive factions were recorded on a Nicolet 560 spectrophotometer in the range 4000 to 400 cm$^{-1}$ using a KBr disc containing 1% finely ground sample.

The XRD patterns were obtained in an X-ray diffractometer (D/max-2200, Japan), using CuK$\alpha$ radiation at 40 kV and 20 mA. Scattered radiation was detected in the range
of $2\theta = 5$ to $40^\circ$, at a scan rate of $4^\circ$/min. The extent of crystallinity ($C_{IC}$) was calculated by the MDI JADE 5.0 software.

Surface morphology of the UBP and the well-dispersed samples were characterized with a scanning electron microscopy (SEM, XL30 ESEM-TMP, EDAX, Ltd.) on aluminum stubs and coated with a thin layer of palladium-gold alloy.

RESULTS AND DISCUSSION

Effect of Sequential O/Po-AQ Process of UBP

Based on the aim of extensive delignification, the charge of peroxide at the Po-AQ stage was varied in the range from 1% to 15% (weight percentage, oven-dry UBP basis). The effect of the peroxide charge on extractive properties is shown in Fig. 1. The percentage of Klason lignin was decreased significantly by increase of H$_2$O$_2$ charge to 8%. Thereafter, the content of lignin and the yield of cellulose remained constant but with a decrease of extractive yields, which showed that P charges higher than 8% gave no additional benefit. Hence, the comparatively suitable P charge for Po-AQ stage was judged to be 8%, taking the delignification degree and cellulose protection as well as peroxide cost into consideration.

![Fig. 1](image)

Fig. 1. The Klason lignin content and the yields of extractive and cellulose isolated from UBP with 1-15% H$_2$O$_2$ charges under the other conditions of 20% alkali dosage, 12% consistency and 0.5% MgSO$_4$ for 2 h at O stage, and 25% alkali dosage, 0.3% AQ charges, 0.5% EDTA and 8% consistency for 3 h at Po-AQ stage, and initial O$_2$ pressure and temperature of both stages 0.6 MPa, 110 °C

The total NaOH dosage of O and Po-AQ stages was set in the range of 20 to 60% (to raw materials) under the given conditions indicated in Fig. 2a and b. When the NaOH charge of O-stage was 20% (Fig. 2a), increasing the alkali dosage of Po-AQ stage from 0% to 20% was beneficial to enhance the delignification and the contents of cellulose in samples. Further increasing the alkali dosage from 20% to 40% based on pith weight, the percentage of lignin decreased slightly accompanied with extractive-yield loss due to carbohydrate breakdown, especially at the 40% NaOH dosage. Similar effects can be seen at 25% (Fig. 2b) alkali dosage of the O stage.
It is worthwhile to notice the fact that the extractive yields in Fig. 2a were readily lower than those in Fig. 2b under the conditions of the same total alkali charges. A reasonable explanation might be that the stronger delignification at 25% NaOH dosage in the O stage leads to exposing more cellulose to chemicals in the next stage, resulting in an increased accessibility and degradability of cellulose, compared with 20% alkali dosage at the O stage. Therefore, the nearly optimal distribution of NaOH dosages was judged to be 20% in the O stage and 30% in Po-AQ stage.

As stated above, the total alkali dosage of 50% led to high residual alkali (13.86 g/L) in the effluent, which could be utilized for continuously delignifying and hemicellulosic hydrolysis. In view of this, the processing by further extending reactive time was also studied.

With the time of the O stage being 2 h, the effect of Po-AQ time being extended from 2 to 8 h on extractive properties is illustrated in Fig. 3a. It was found that more than 18% hemicellulose was contained in the preparations after 4 to 5 h of processing. Prolonging the time from 6 to 8 h at the second stage seemed to reduce the hemicellulose contents. This indicates that extending time with sufficient active alkali in the Po-AQ stage was beneficial to hemicellulose dissolution. Meanwhile, the hemicellulose degradation seemed to promote the removal of the residual lignin to some extent, implying that the residual lignin might be attached to saccharides by the chemical bonds, namely lignin-carbohydrate compounds (LCC) (Chen et al. 2011; Li et al. 2009).

Additionally, the time division of the O/Po-AQ stages was also studied by varying the O-processing time from 1 to 4 h with the total time being set at 8 h. Under the given conditions as shown in Fig. 3b, reacting for more than 2 h at the O stage increased the lignin contents. This is probably due to the fact that extending time at the first stage implies shortening reaction period at the second one, resulting in insufficient reaction for delignification at the Po-AQ process. This result showed that cellulose purity depends correlatively on the total processing time and the time distribution between the O and Po-AQ stages. The most suitable allocation of the processing time at O/Po-AQ stages was...
thus determined to be 2/6 h, based on the criteria of comparatively lower lignin content and higher cellulose purity according to the Fig. 3b.

![Figure 3](image1.png)

**Fig. 3.** The Klason lignin content and the yields of extractive and cellulose isolated from UBP with time variation under the other conditions of 20% alkali dosage, 12% consistency and 0.5% MgSO4 at O stage, and 8% H2O2, 30% alkali dosage, 0.3% AQ charges, 0.5% EDTA and 8% consistency at Po-AQ stage, and initial oxygen pressure and temperature of both stages 0.6 MPa, 110 °C. (‘x / y’ means the reactive time of ‘x’ h and ‘y’ h at O stage and Po-AQ stage, respectively.)

The effect of the reaction temperature on preparations is shown in Fig. 4. The contents of Klason lignin in extractives decreased with increasing temperature. The reaction at higher temperature would increase the consumption of alkali not only for delignification but also for carbohydrates degradation mainly by “peeling reaction” and “alkaline hydrolysis”. Consequently, increasing the temperature to 130 °C resulted in decreasing the extractive and cellulosic yields to 32.6% and 84.29%, respectively. As a result, processing at 120 °C showed the comparatively low lignin number (0.78%) and high cellulose content (86.02%) of preparation.

![Figure 4](image2.png)

**Fig. 4.** The Klason lignin content and the yields of extractive and cellulose isolated from UBP with 70-130 °C reactive temperature under the other conditions of 20% alkali dosage, 12% consistency and 0.5% MgSO4 for 2 h at O stage, and 8% H2O2, 30% alkali dosage, 0.3% AQ charges, 0.5% EDTA and 8% consistency for 6 h at Po-AQ stage, and 0.6 MPa initial oxygen pressure of both stages.

In order to obtain acceptable cellulose, the effect of AQ dosage on the process was investigated. As shown Fig. 5, the addition of AQ from 0.0 to 0.2% increased cellulose yields and decreased the lignin content from 1.12% to 0.65%. Further increasing the AQ dosage from 0.2% to 0.5% gave rise to the unfavorable phenomena of increasing lignin content accompanied by raising the hemicellulose yield from 13.13% to 17.20%. This is due to the stabilization of the reducing end-groups of the carbohydrates through oxidation to aldonic acids, which are stable to end-wise degradation (Khristova et al. 2006). However, based on the largely accepted concept of the existence of LCC in lignocellulosic materials (Hamzeh et al. 2013), it has been inferred that the enhancement of Klason lignin content is probably attributable to preserving more hemicellulose from alkaline degradation by continuously increasing the AQ dosage.

![Graph](image)

**Fig. 5.** The Klason lignin content and the yields of extractive and cellulose isolated from UBP with 0.0-0.5% AQ charges under the other conditions of 20% alkali dosage, 12% consistency and 0.5% MgSO₄ for 2 h at O stage, and 8% H₂O₂, 30% alkali dosage, 0.5% EDTA, and 8% consistency for 6 h at Po-AQ stage, and initial oxygen pressure and temperature of both stages 0.6 MPa, 120 °C.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Parameters of improved O/Po-AQ treatments and conventional processes</th>
<th>Extractive properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Raw material</td>
<td>NaOH (Po-AQ, %)</td>
</tr>
<tr>
<td>F0</td>
<td>UBP</td>
<td>0</td>
</tr>
<tr>
<td>F1</td>
<td>UBP</td>
<td>30</td>
</tr>
<tr>
<td>F2</td>
<td>UBP</td>
<td>25</td>
</tr>
<tr>
<td>F3</td>
<td>UBP</td>
<td>20</td>
</tr>
<tr>
<td>F4</td>
<td>UBP</td>
<td>25</td>
</tr>
<tr>
<td>F5</td>
<td>UBP</td>
<td>20</td>
</tr>
<tr>
<td>F6</td>
<td>EBP</td>
<td>25</td>
</tr>
<tr>
<td>F7</td>
<td>EBP</td>
<td>20</td>
</tr>
</tbody>
</table>

Other conditions: 20% alkali dosage, 12% consistency and 0.5% MgSO₄ for 2 h of O stage; 8% H₂O₂, 0.2% AQ charges, 0.5% EDTA, and 8% consistencies for 6 h of Po-AQ stage; Initial O₂ pressure of both stages was 0.6 MPa.
Processing Improvement

On the basis of the O/Po-AQ process discussed above, the improvement of manually discharging O-stage effluent was carried out in order to promote the purity of cellulose isolated from UBP or EBP. The results are shown in Table 1. It was expected that the removal of O effluent could impact the extractive properties to a significant degree. Even the properties of F3 under the milder conditions of 20% NaOH dosage at Po-AQ stage were very close to those under the optimum conditions of 30% alkali charge discussed above. Elimination of the degraded lignin in O effluent resulted in increasing the Po-AQ reactivity for continuously removing the residual lignin and hemicellulose components. Compared with increasing temperature by 5 °C (F4 and F5), the using EBP as starting material (F6 and F7) was more beneficial in that the delignification degree and yields of extractive and cellulose were evidently improved. Lei et al. (2010) suggested that some of the hemicelluloses are originally chemically bound to lignin by a glycosidic linkage. EBP had a more open structure due to removal of some hemicelluloses and migration of lignin to the pith particle surface after extraction facilitated the agents to reach lignin. Therefore, the reaction activity of lignin was thus improved. Therefore the processing of EBP yielded much lower lignin content even though lower alkali charge and temperature were employed. Based on the literature dealing with cellulose modifications (Fan et al. 2013; Hirota et al. 2009), low Klason lignin contents and intrinsic viscosities of samples, especially F6 and F7, are considered to be favorable characteristics for making such derivatives as cellulose acetate or oxidized cellulose.

Additionally, the extractive, which was obtained only from the O process using EBP as raw material, was evaluated. The properties of 74.63% cellulose and 6.25% lignin contents were much less favorable than those from the O/Po-AQ sequential treatment. The O treatment could remove more than 2/3 of total lignin but cause only less than a half of the hemicellulose hydrolysis. Therefore, it is necessary to add the Po-AQ process after O treatment for further removal of the residual hemicellulose and lignin.

GC and Spectroscopic Characterization

*GC analysis of sugars*

The five samples were treated to covert polysaccharides to alditol acetate forms so as to measure neutral sugar composition. It is well known that carbohydrates including cellulose and hemicelluloses are the main components of the secondary layers of the cell wall in lignocellulosic materials (Timell 1967). It was revealed that glucose was a predominant monosaccharide, comprising 85.82 to 93.53% of the total neutral sugars in Table 2, followed by xylose (6.04 to 13.40%), arabinose (0.21 to 0.58%), and mannose (0.10 to 0.20%) with no galactose in the three samples of F1, F3, and F7.

**Table 2.** Contents of Neutral Sugars (Relative % Different Sample, w/w) in Three Cellulose Preparations, Holocellulose, and BP

<table>
<thead>
<tr>
<th>Sugars (%)</th>
<th>F1</th>
<th>F3</th>
<th>F7</th>
<th>Holocellulose</th>
<th>BP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arabinose</td>
<td>0.21</td>
<td>0.58</td>
<td>0.31</td>
<td>2.13</td>
<td>2.79</td>
</tr>
<tr>
<td>Xylose</td>
<td>7.93</td>
<td>13.40</td>
<td>6.04</td>
<td>21.27</td>
<td>23.29</td>
</tr>
<tr>
<td>Mannose</td>
<td>ND</td>
<td>0.20</td>
<td>0.10</td>
<td>1.29</td>
<td>1.96</td>
</tr>
<tr>
<td>Galactose</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>0.79</td>
<td>1.05</td>
</tr>
<tr>
<td>Glucose</td>
<td>91.84</td>
<td>85.82</td>
<td>93.53</td>
<td>74.52</td>
<td>70.89</td>
</tr>
</tbody>
</table>

ND, not detected.
Based on the results in Table 2, compared with the neutral sugar contents of BP and its holocellulose, the pith parenchyma processed in the O/Po-AQ procedure can provide cellulose with high purity accompanied by hexosan/pentaose-hemicellulosic degradation to the some extent. And the sample F7 isolated from EBP under the milder condition of alkali dosage was a favorable result because it achieved the highest glucose content of 93.53%.

**FT-IR measurements**

FT-IR spectroscopy is an appropriate technique that has been extensively used for obtaining direct information about chemical structures. Figure 6 compares the FT-IR spectra of UBP, pith holocellulose, cellulose, and three preparations (F1, F3, and F7). The dominant peaks of OH-stretching and CH-stretching at approximately 3400 cm\(^{-1}\) and 2900 cm\(^{-1}\) were observed in all spectra. After the O/Po-AQ processing, the peaks of the UBP and holocellulose at about 1730 cm\(^{-1}\), representing either the acetyl and uronic ester groups or the ester linkage of the carboxyl groups of the ferulic and p-coumeric acids of lignin and/or hemicellulose (Chen et al. 2011; Sain and Panthapulakkal 2006), disappeared completely in the spectra of F1, F3, and F7 as well as pith cellulose. This indicates that most of the hemicellulose and lignin were removed from the raw material during the two-step procedure. The intensity of the 1514 and 1448 cm\(^{-1}\) peaks in BP, which are attributed to aromatic ring vibration and C-H deformation vibration of lignin, respectively (Chen et al. 2011; Ciolacu et al. 2012), also disappeared in the three extractives, providing evidence that all the isolating procedures removed most of the lignin. The band at 1633 cm\(^{-1}\) observed in all of the spectra corresponds to the H-O-H stretching vibration of the absorbed water in carbohydrates (Ciolacu et al. 2012). Each spectrum gives a peak at 1423 cm\(^{-1}\) which is due to the CH\(_2\) bending and a band at 1373 cm\(^{-1}\) to the O-H bending. The absorption of 1316 cm\(^{-1}\) arises from the C-C and C-O skeletal vibrations (Pastarova et al. 1994). The absorbance at 1244 cm\(^{-1}\) is indicative of the OH in-plane bending cellulose.

![FT-IR spectra](image)

**Fig. 6.** FT-IR spectra of the preparations of F1, F3, and F7, and cellulose, holocellulose, and raw material of bagasse pith
The 1160 cm\(^{-1}\) band is associated with C-O antisymmetric bridge stretching. The region of 1080 to 1035 cm\(^{-1}\) and the 896 cm\(^{-1}\) relate to C-O-C pyranose ring skeletal vibration and β-glycosidic linkages between glucose units in cellulose, respectively (Alemdar and Sain 2008; Pappas et al. 2002). Therefore, it was implicated that larger amounts of lignin were removed and only a small quantity of alkali insoluble hemicellulose remained after the two-stage isolation.

**XRD studies**

XRD measurements for BP as well as extractives F1, F3, and F7 showed similar diffraction patterns with the peaks at 2\(\theta\)=16° and 22.6° in Fig. 7, characteristic of the typical cellulose I type (Lu and Hsieh 2012), which indicates that the crystal structure of samples was not changed during the treatment. However, the crystallinity indexes of the preparations were apparently different. The crystallinity for UBP was 56.41%, whereas those for the samples were significantly higher at 77.35%, 71.79%, and 83.49% for F1, F3, and F7, respectively. The increasing crystallinity of three preparations was probably due to the removal of hemicellulose and lignin existing in amorphous regions, which leads to the realignment of cellulose molecules. The F7 has apparently higher crystallinity than the other extractives of F1 and F3, which is attributed to the highest purity of cellulose following removal of the most hemicellulose contents. These results were also supported by the GC analyses above.

![X-ray diffraction patterns of the three preparations (F1, F3, and F7), and raw material of bagasse pith](image)

**SEM analysis**

The oxygen-based treatments used for cellulose isolation from BP are also expected to induce morphological changes. The surface structures of the three samples (F1, F3, and F7) were investigated by SEM (Fig. 8). At a low (200×) magnification (Fig. 8 a, c, and e), all extractives mainly consisted of two different tissues: translucently lamellar parenchyma cells and shortly fibrous epidermal fibers. However, the observation at higher magnifications demonstrated a structure difference among them. In the F1 prepared with the highest alkali charge of 30% at the Po-AQ stage, Fig. 8b shows that the majority of parenchyma cells were curled and fragmented, probably due to the comparatively strong NaOH effect on BP during the reactions. On the other hand, the
parenchyma surface of F7 (Fig. 8f) became much rougher than that of F1 and F3 (Fig. 8d). This could indicate that the padding and filling components such as hemicelluloses, lignin, and other impurities contained in BP were almost removed, thus exposing more cellulose and showing rugged appearance. This is further evidence that the cool-water exaction prior to the O/Po-AQ sequential treatment was convenient to separate hemicelluloses and lignin from BP.

Fig. 8. Scanning electron micrographs of (a and b) F1, (c and d) F3, and (e and f) F7

CONCLUSIONS

1. A new process of cellulose isolation from BP using an integrated O/Po-AQ sequence has been successfully developed. The cellulose with minor hemicellulose could be
extracted effectively in the improved process, and the optimal conditions were 20% alkali dosages and 12% consistency at 120 °C for 3 h with 0.6 MPa initial oxygen pressure for the O stage; 8% H$_2$O$_2$, 20% NaOH, and 0.2% AQ charges at 120 °C for 6 h with 8% consistency under the same oxygen pressure for the Po-AQ stage.

2. The results of GC and FT-IR indicated that lignin was extensively removed and only a small amount of alkali insoluble hemicellulose remained in the extractives after the two-step isolation. Images of XRD and SEM showed that the crystallinity of the samples and surface roughness of preparations were both enhanced with the increasing cellulose content in the extractives.

3. On the basis of the optimal conditions, the extractive properties with 91.52% cellulose content and 337 mL/g intrinsic viscosity are adequate for application of BP cellulose as a raw material for esterification or oxidation modification. According to SEM images, the morphological structure of parenchyma cellulose is greatly different from epidermal fibers, implying larger specific surface areas and chemical reactivity. In this way the use of bagasse pith as a novel material source makes it possible to obtain parenchyma cellulose with translucently lamellar shape, widening the supply of biomaterials usable for cellulose-based polymers.

ACKNOWLEDGMENTS

This study was financially supported by National Natural Science Foundations of China (21276119, 51363013) and Scientific Research Foundation Project of Yunnan Municipal Education Commission (2012Y537).

REFERENCES CITED


Article submitted: December 10, 2013; Peer review completed: April 13, 2014; Revised version received and accepted: May 12, 2014; Published: May 19, 2014.