

Homogeneous Modification of Cellulose in Ionic Liquid with Succinic Anhydride Using *N*-Bromosuccinimide as a Catalyst

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The homogeneous chemical modification of cellulose with succinic anhydride was investigated in a solvent system containing 1-butyl-3-methylimidazolium chloride ionic liquid and dimethylsulfoxide using *N*-bromosuccinimide (NBS) as a catalyst. The results showed that the degree of substitution of the succinylated cellulosic samples, in the range of 0.24–2.31, noticeably increased as compared with the products without any catalysts, indicating that NBS was a novel efficient catalyst for cellulose succinylation in ionic liquids. Fourier transform infrared and solid-state cross-polarization/magic angle spinning ¹³C NMR spectroscopies also provided evidence of succinylation reaction. The results indicated that the reaction of hydroxyl groups at C-6, C-2, and C-3 positions in cellulose occurred. The thermal stability of the succinylated cellulose was found to decrease upon chemical modification.

KEYWORDS: Cellulose; ionic liquid; homogeneous modification; *N*-bromosuccinimide

INTRODUCTION

Cellulose, the major constituent of all plant materials, is a homopolymer composed of D-glucopyranose units linked by β (1→4) glycosidic bonds (1). It is nontoxic, renewable, biodegradable, and modifiable and has great potential for an excellent industrial material (2). In recent years, there has been an increasing trend in society toward more efficient utilization of renewable resources for the production of various products (3), and considerable efforts are now being made in the research and development of cellulose as the basic material for industrial applications. Chemical modification of cellulose could introduce functional groups into the macromolecules to substitute the free hydroxyl groups in the heterogeneous phase or homogeneous phase, which continues to play a dominant role in improving the overall utilization of cellulosic polymers. Homogeneous functionalization has been one focus of cellulose research for a long time because more uniform and stable products could be obtained in the homogeneous phase than the heterogeneous phase (4). However, the intrinsic lack of solubility of native cellulose in water and most organic solvent systems constitutes a major obstacle for cellulose homogeneous modification. The efficient dissolution of cellulose is a long-standing goal in cellulose research and development (5).

Recently, as desirable green solvents and reaction media for a wide range of processes, ionic liquids (ILs) have received

significant attention (6). In 2002, ILs including 1-butyl-3-methylimidazolium chloride ([C₄mim]Cl) were first reported as nonderivatizing solvents for cellulose by Swatloski et al. (7). In 2003, a room-temperature ionic liquid (RTIL), 1-allyl-3-methylimidazolium chloride (AmimCl), was synthesized and proved to have outstanding capability for dissolving cellulose (8). In 2005, another RTIL, 1-(2-hydroxyethyl)-3-methylimidazolium chloride (HemimCl), was reported as the nonderivatizing solvent for cellulose by Luo et al. (9). More recently, it was reported that a series of alkylimidazolium salts containing dimethyl phosphate, methyl methylphosphonate, or methyl phosphonate ([C₂mim][[(MeO)(R)PO₂]] could successfully dissolve cellulose at high concentrations and at room temperature (10). The dissolution of cellulose in ILs has opened a novel platform for the utilization of cellulose materials. Moreover, the dissolution of other biopolymers such as hemicelluloses (11), lignin (12), starch (13), and wood (14, 15) in ILs has also been reported over the last several years, indicating the increasing interests in utilization of ILs in the field of bioresource chemistry around the world. On the other hand, the investigation into the use of ILs as reaction media for cellulose functionalization has also been reported. Cellulose acetylation with liquid acetic anhydride or acetyl chloride has been extensively studied because of easily established homogeneous reaction systems (5, 16, 17). Furthermore, ILs can also be used as reaction media for carbanilation of cellulose with phenyl isocyanate (16, 18), acylation with lauroyl chloride (16), and perpropionylation with propionic anhydride (18). Moreover, similar acylation reactions have been carried out in ILs on wood (19). However, it is much

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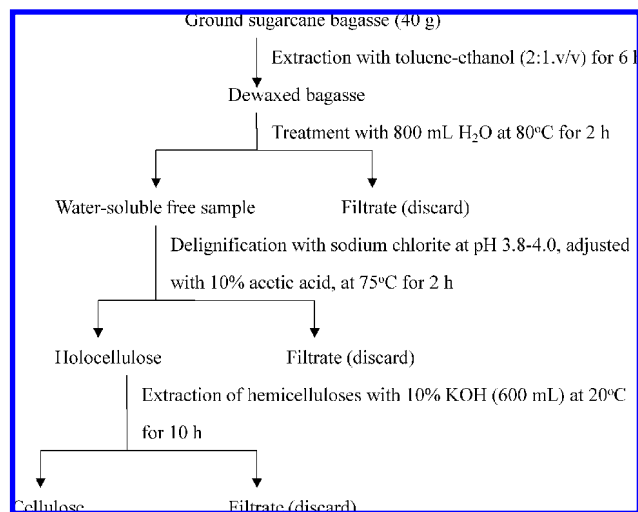


Figure 1. Scheme for the isolation of cellulose from SCB.

more difficult to achieve homogeneous media for cellulose modification with solid derivatizing reagents. Heinze et al. (5) proposed carboxymethylation of cellulose in $[C_4mim]Cl$. However, carboxymethylated cellulose obtained according to this method had a relatively low DS, and an increase of the dosage of carboxymethylating reagent did not increase the DS. The reason for this unusual result is still unclear. Homogeneous tritylation of cellulose was also achieved in $[C_4mim]Cl$ with trityl chloride in the presence of pyridine, resulting in trityl cellulose with a lower DS than acetylation (20). In addition, homogeneous acylation of cellulose was carried out to produce cellulose ester with lower DS in AmimCl with *pyro*-pheophorbide and stearic acid, respectively (20). In our laboratory, homogeneous modification of cellulose with solid succinic anhydride (SA) (21, 22) was also investigated without any catalysts. However, succinylation was more difficult than acetylation, and the obtained cellulose derivatives had relatively low degree of substitution (DS). Recently, it has been demonstrated that *N*-bromosuccinimide (NBS) is a novel, highly effective catalyst for acetylation of alcohols under mild reaction conditions (23). Therefore, we investigated the possibility of developing a new catalyst system for the derivatization of cellulose with SA in the presence of NBS in a solvent system containing ILs. NBS is an inexpensive and commercially available reagent that is traditionally used as an oxidizing agent or brominating agent (23). In present study, succinylation of cellulose was discussed in a BmimCl/dimethyl sulfoxide (DMSO) system using a molar ratio of 4:1 SA:andhydroglucose (AGU) units in cellulose and a weight ratio of NBS/SA between 0 and 20%. The modified cellulose was then characterized by Fourier transform infrared (FT-IR) and solid-state cross-polarization/magic angle spinning (CP/MAS) ^{13}C nuclear magnetic resonance (NMR) spectroscopies as well as thermal analysis.

MATERIALS AND METHODS

Materials. Sugar cane bagasse (SCB) was obtained from a local sugar factory (Guangzhou, China). It was dried in sunlight and then cut into small pieces. The cut SCB was ground and screened to prepare 20–40 mesh size particles (450–900 μm). IL BmimCl was obtained from the Chemer Chemical Co., Ltd. (Hangzhou, China) and used as received. All of other chemicals used were of analytical grade and obtained from Guangzhou Chemical Reagent Factory (China).

Isolation of Cellulose. A scheme for the isolation of cellulose from SCB is shown in Figure 1. The dried SCB powder was first dewaxed with toluene–ethanol (2:1, v/v) in a Soxhlet apparatus for 6 h. The

dewaxed SCB was then soaked in distilled water (800 mL) at 80 °C for 2 h. The insoluble residue was delignified with sodium chlorite at pH 3.8–4.0, adjusted by 10% acetic acid, at 75 °C for 2 h. The residue was collected by filtration, washed with distilled water and ethanol, and then dried in a cabinet oven for 16 h at 50 °C. The holocellulose obtained was extracted with 10% KOH (600 mL) at 20 °C for 10 h. After filtration, the residue was washed thoroughly with distilled water until the filtrate was neutral, then washed with 95% ethanol, and dried in an oven at 50 °C for 16 h.

Homogeneous Modification of Cellulose in BmimCl. The succinylated cellulosic derivatives containing free carboxylic groups were prepared by homogeneous reaction of SCB cellulose with SA using NBS as a catalyst in the solvent system containing IL BmimCl and DMSO. Dried cellulose (0.405 g) was added to BmimCl in a three-necked flask, and the mixture of cellulose/BmimCl was stirred at 80 °C up to 10 h to guarantee the complete dissolution of cellulose. Then, the flask was continuously purged with gaseous N_2 . To reduce viscosity and achieve a suitable mixing, 5 mL of DMSO was added to this mixture at temperatures of 90, 95, 100, 110, and 120 °C, respectively. To the cellulose solution in the BmimCl/DMSO system, SA and NBS, previously dissolved in 5 mL of DMSO, were added at temperatures of 90, 95, 100, 110, and 120 °C with the molar ratio of 4:1 SA/AGU in cellulose and a weight ratio of NBS/SA at 1, 2, 5, 10, and 20%, respectively. The mixture was heated in an oil bath under N_2 atmosphere with continuous magnetic stirring for 30, 45, 60, 75, 90, 120, and 240 min, respectively. After the required time, the resulting mixture was slowly poured into 150 mL of isopropanol with stirring to stop the reaction. The solid was filtered, washed thoroughly with isopropanol to eliminate ILs, unreacted anhydride, NBS, and byproduct, and then dried in a vacuum at 50 °C for 16 h.

DS Determination. The DS of cellulosic preparations was determined by a direct titration method (24, 25). A known weight of the sample was dissolved in 10 mL of DMSO by stirring at 75 °C for 30 min. After the mixture cooled, 5–6 drops of phenolphthalein indicator was added. This solution was titrated against 0.01 M standard NaOH solution until a permanent pale pink color was seen. The DS was calculated by using the following equation:

$$DS = \frac{162 \times (V_{NaOH} \times C_{NaOH})}{m - 100 \times (V_{NaOH} \times C_{NaOH})}$$

where 162 g/mol is the molar mass of an AGU, 100 g/mol is the net increase in the mass of an AGU for each succinoyl substituted, m is the weight of sample analyzed, V_{COOH} is the volume of standard NaOH solution consumed in the titration, and c_{NaOH} is the molarity of standard NaOH solution.

Spectroscopic and Thermal Characterization. The FT-IR spectra of the cellulose and succinylated cellulosic derivatives were recorded on an FT-IR spectrophotometer (Nicolet 510) from finely ground samples (1%) in KBr pellets in the range 4000–400 cm^{-1} . Thirty-two scans were taken for each sample with a resolution of 2 cm^{-1} in the transmission mode. The solid-state CP/MAS ^{13}C NMR spectra were obtained on a Bruker DRX-400 spectrometer with 5 mm MAS BBO probe employing both CP and MAS, and each experiment was recorded at ambient temperature. The spectrometer operated at 100 MHz. The acquisition time was 0.034 s, the delay time was 2 s, and the proton 90 °C pulse time was 4.85 μs . Each spectrum was obtained with an accumulation of 5000 scans.

Thermal analysis of the cellulose and succinylated cellulosic samples was performed using thermogravimetric analysis (TGA) and differential thermal analysis (DTA) on a simultaneous thermal analyzer (SDT Q600, TA Instrument). The apparatus was continually flushed with nitrogen. The sample weighed between 8 and 12 mg, and the scans were run from room temperature to 550 °C at a rate of 10 °C/min.

RESULTS AND DISCUSSION

Modification and the DS. Esterification of cellulose represents one of the most versatile transformations as it provides access to a variety of biobased materials with valuable properties. Acetylation of cellulose with acetic anhydride or acetyl

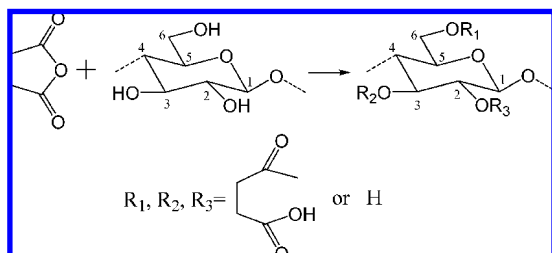


Figure 2. Reaction of SCB cellulose with SA using NBS as a catalyst.

Table 1. DS of Succinylated Cellulose with SA in $[C_4mim]Cl/DMSO$ System Using NBS as a Catalyst

succinylation conditions					succinylated cellulose	
cellulose (%) ^a	molar ratio ^b	NBS/SA (%)	temperature (°C)	reaction time (min)	sample no.	DS
2.0	4:1	0	100	60	1	0.24
2.0	4:1	1	100	60	2	1.27
2.0	4:1	2	100	60	3	1.84
2.0	4:1	5	100	60	4	2.31
2.0	4:1	10	100	60	5	1.55
2.0	4:1	20	100	60	6	1.23
2.0	4:1	2	100	30	7	0.92
2.0	4:1	2	100	45	8	1.31
2.0	4:1	2	100	75	9	1.92
2.0	4:1	2	100	90	10	1.99
2.0	4:1	2	100	120	11	2.03
2.0	4:1	2	100	240	12	1.73
2.0	4:1	2	90	60	13	1.67
2.0	4:1	2	95	60	14	1.74
2.0	4:1	2	110	60	15	1.98
2.0	4:1	2	120	60	16	2.25

^a Concentration of cellulose in ILs during dissolution was 2.0% by weight. ^b Molar ratio of SA to anhydroglucose in cellulose was 4:1.

chloride is the most common method for cellulose derivatization. However, modification of cellulose with linear chain acylation reagents such as anhydrides produces an undesired byproduct, namely, the corresponding carboxylic acid. This acid must be removed from the reaction following modification. On the other hand, modification with cyclic anhydrides such SA does not yield a byproduct. Furthermore, the reaction results in a pendant carboxylic moiety attached to the cellulose via a covalent ester bond, providing a site upon which further reactive chemistry is possible. However, our previous studies showed that succinylation of cellulose in ILs such as BmimCl and AmimCl could only result in succinylated cellulose with a lower DS than 0.53 and 0.22, respectively (21, 22), suggesting a decrease of the succinylation efficiency with SA as compared to acetylation. In the present study, NBS was used as a catalyst in the succinylation reaction of SCB cellulose in the $[C_4mim]Cl/DMSO$ system to improve modification efficiency. Usually, SA reacts with cellulose O–H groups to form the monoester as shown in **Figure 2** (26). After cellulose was dissolved in $[C_4mim]Cl$, the viscous solution obtained was diluted by DMSO to achieve a suitable and clear mixing. Solid SA and NBS dissolved in DMSO were added to the solution to achieve homogeneous reaction. The effects of NBS dosage, reaction temperature, and reaction duration on DS of the succinylated cellulose are listed in **Table 1**.

As the data show in **Table 1**, an increase of NBS dosage from 0 (sample 1) to 1 (sample 2), 2 (sample 3), and 5% (sample 4) (based on SA) resulted in noticeable improvement of the DS of succinylated samples from 0.24 to 1.27, 1.84, and 2.31, respectively. Apparently, NBS could significantly improve the efficiency of succinylation of cellulose in the $[C_4mim]Cl/DMSO$ system. However, further improvement of NBS dosage

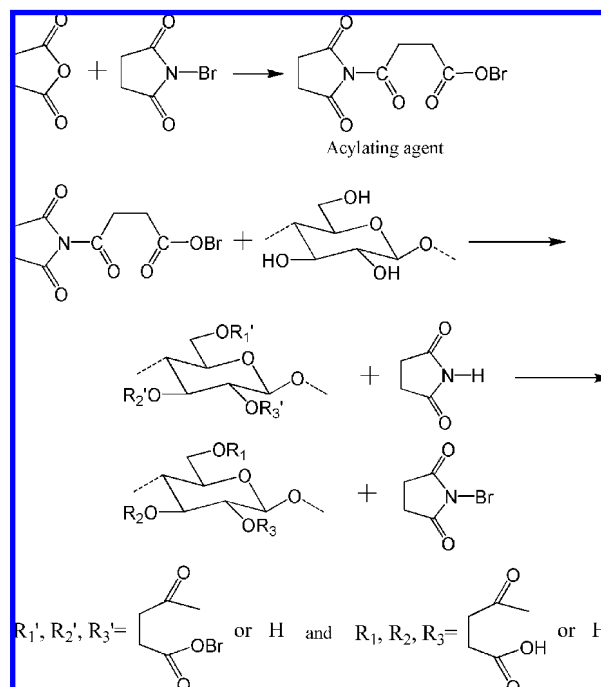


Figure 3. Mechanism of succinylation of cellulose using NBS as a catalyst.

from 5 to 10% (sample 5) and to 20% (sample 6) led to a decrease in the DS of the cellulosic products from 2.31 to 1.55 and 1.23, respectively. The reason of the decrease in the DS could probably be due to the formation of diester by cross-linking of the produced free carboxylic groups in monoesters with other free hydroxyl groups of cellulose. These results suggested that NBS could significantly improve the reaction efficiency of SA and hydroxyl groups in cellulose. However, the succinylated products never reach a DS of near 3.0. The maximum DS observed was only about 76% of the theoretical value. In this respect, it was found that the efficiency of the esterification decreased with the increasing chain length of the anhydrides. Cellulose acetates with a DS in the range from 2.5 to 3.0 are easily accessible within 2 h at 80 °C in a complete homogeneous procedure (5). On the other hand, the esterification of cellulose with the fatty acid chloride lauroyl chloride leads to cellulose laureates with the DS decreasing to 0.34–1.54 under the same conditions in ILs (16). These reports indicated that increasing alkyl chain length of the anhydride resulted in a lower cellulose ester yield, as occurred in our present study on the succinylation of sugar cane cellulose in ILs. The possible mechanism of succinylation and the actual role of NBS are not clear. However, a plausible explanation is that NBS might act as a source for Br^+ , which in turn activates the carbonyl groups of SA to produce the highly reactive acylating agent, as shown in **Figure 3**. This acylating agent reacts with hydroxyl groups of cellulose, which upon elimination of NBS, produce succinylated cellulose (27). However, at this time, the precise role of NBS is not clear, and the actual role of this reagent should be further studied in detail.

Holding the molar ratio of SA/AGU at 4:1, the ratio of NBS/SA at 2%, and the reaction temperature at 100 °C, the DS of the cellulosic derivatives reached 0.92 within only 30 min (sample 7), 1.31 within 45 min (sample 8), 1.84 within 60 min (sample 3), 1.92 within 75 min (sample 9), 1.99 in 90 min (sample 10), and 2.03 in 120 min (sample 11). This increment in the DS of the succinylated cellulose with reaction time could be due to the increase rate and time of collision of SA with

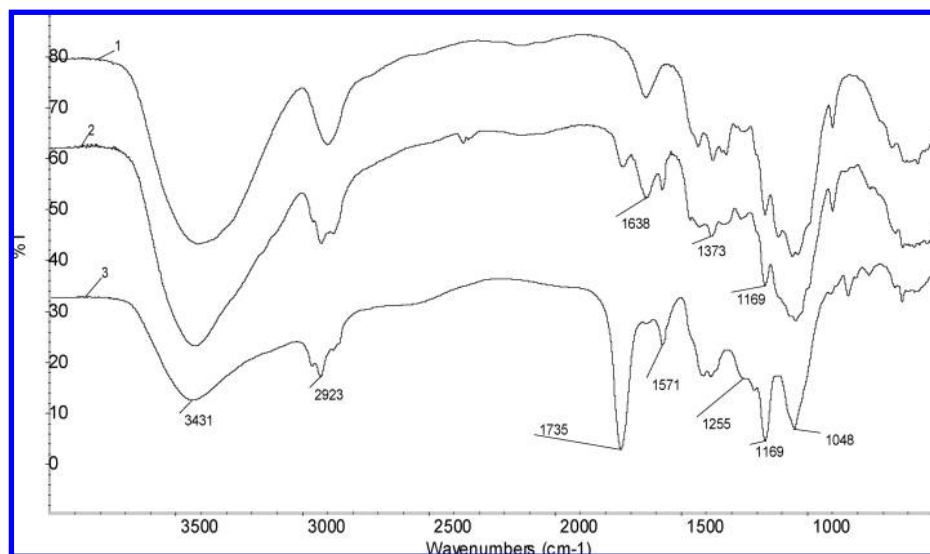


Figure 4. FT-IR spectra of unmodified cellulose (spectrum 1) and succinylated cellulose without any catalyst (sample 1, spectrum 2) and with NBS catalyst (sample 5, spectrum 3).

cellulosic molecules. The longer the reaction time is, the higher the collision rate and time of reactants are, and the more completely the reaction proceeds. However, prolonging the reaction time from 120 to 240 min (sample 12) led to a decrease in the DS of cellulose products from 2.03 to 1.73, probably due to the further reaction of the produced free carboxylic groups with other free hydroxyl groups in cellulose. Furthermore, raising the reaction temperature from 90 (sample 13) to 95 (sample 14), 100 (sample 3), 110 (sample 15), and 120 °C (sample 16) resulted in an increase in the DS of the products from 1.67 to 1.74, 1.84, 1.98, and 2.25, respectively, suggesting the favorable effect of temperature on succinylation reaction.

FT-IR Spectra. The most convenient method for the elucidation of structural features of cellulosic products is FT-IR spectroscopy. **Figure 4** illustrates the FT-IR spectra of unmodified cellulose (spectrum 1), succinylated cellulose without any catalyst (spectrum 2, sample 1), and succinylated cellulose with NBS catalyst (spectrum 3, sample 5). The absorbances at 3431, 2923, 1638, 1373, 1169, and 1048 cm^{-1} seen in spectrum 1 are associated with native cellulose. A strong band at 3431 cm^{-1} corresponds to the O–H stretching, and that at 2923 cm^{-1} originates from the C–H stretching. The band at 1638 cm^{-1} is attributed to the bending mode of the absorbed water. The peak at 1373 cm^{-1} corresponds to the O–H bending. The absorption band at 1169 cm^{-1} originates from the C–O antisymmetric bridge stretching (28). A strong peak at 1048 cm^{-1} arises from the C–O–C pyranose ring skeletal vibration (29). In comparison, spectra 2 and 3 for succinylated cellulose provide evidence of succinylation by showing the significant changes of three bands at 1735, 1571, and 1169 cm^{-1} . The former band is indicative of absorption by carbonyl group in carboxyl and esters. In general, the absorption by carbonyl bonds in esters gives peaks at 1750 cm^{-1} , and one in carboxylic acid exhibits a band at 1712 cm^{-1} (30). The two bands are strongly overlapped and therefore result in a peak centered at 1735 cm^{-1} . The absorption band at 1571 cm^{-1} originates from antisymmetric stretching of carboxylic anions (26), indicating the formation of monoester. The intensity of the absorption band at 1169 cm^{-1} for C–O antisymmetric stretching increased after succinylation, suggesting that the esterification reaction does occur. These results indicated that the monoester of succinylated cellulose was formed in the BmimCl/DMSO system under the conditions

given. Clearly, as compared with the spectrum 2 for succinylated cellulose without any catalyst, the intensity of the bands at 1735, 1571, and 1169 cm^{-1} is significantly improved in spectrum 3 for cellulose modified with NBS catalyst, indicating enhanced succinylation efficiency of cellulose catalyzed with NBS. As expected, the absence of peaks at 1850 and 1780 cm^{-1} in spectra 2 and 3 for succinylated cellulose confirmed that the products are free of the unreacted SA (31). In addition, the intensity of the broad peak at 3431 cm^{-1} for the O–H stretching decreased, which was probably due to the formation of diester by further cross-linking of produced free carboxylic groups with other free hydroxyl groups of cellulose, resulting in the decreased content of hydroxyl groups. In comparison with spectra 1 and 2, a sharper peak at 1048 cm^{-1} in spectrum 3 was observed after succinylation using NBS as a catalyst. This change was probably due to partial hydrolysis and degradation of cellulose molecules during succinylation in the presence of water produced in the cross-linking of free carboxylic groups with the surrounding free hydroxyl groups of cellulose.

The effects of NBS dosage, reaction time, and reaction temperature on the DS of cellulosic preparations were also investigated by the peak intensity of succinylated cellulose samples. In **Figure 5**, the intensity of peaks at 1732 cm^{-1} for C=O stretching, 1572 cm^{-1} for antisymmetric stretching of carboxylic anions, and 1165 cm^{-1} for C–O antisymmetric stretching obviously increased with an increase of NBS dosage from 2 (spectrum 1, sample 3) to 5 (spectrum 2, sample 4) and 10% (spectrum 3, sample 5). This trend was different from the DS in **Table 1**, which was presumed to be due to the formation of diester in the BmimCl/DMSO system. On the other hand, the intensity of the bands at 3429 cm^{-1} for O–H stretching decreased with the improvement of NBS dosage, indicating declined content of hydroxyl groups in succinylated cellulose. This decrease of hydroxyl groups is paralleled to the trend of the DS in **Table 1**, suggesting that the cross-linking of produced free carboxyl groups in monoesters with surrounding free hydroxyl groups of cellulose does occur.

CP/MAS ^{13}C NMR Spectra. CP/MAS ^{13}C NMR spectroscopy has been extensively applied to the structural studies of natural materials (32). In present study, the succinylation reaction of cellulose was also studied by solid-state CP/MAS ^{13}C NMR spectroscopy, and the spectra of unmodified cellulose (spectrum a), succinylated cellulose sample 1

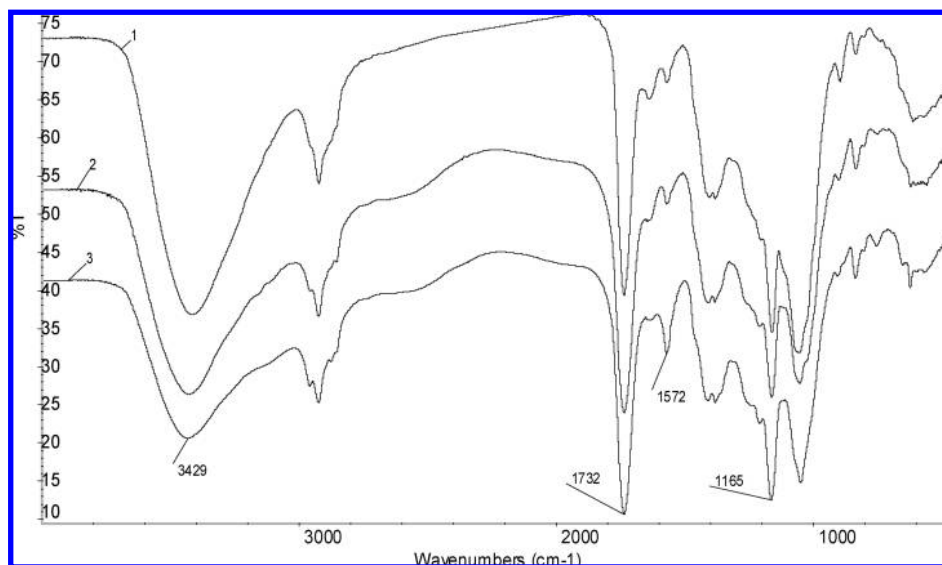


Figure 5. FT-IR spectra of succinylated cellulose prepared with the ratio of NBS/SA at 2 (spectrum 1, sample 3), 5 (spectrum 2, sample 4), and 10% (spectrum 3, sample 5).

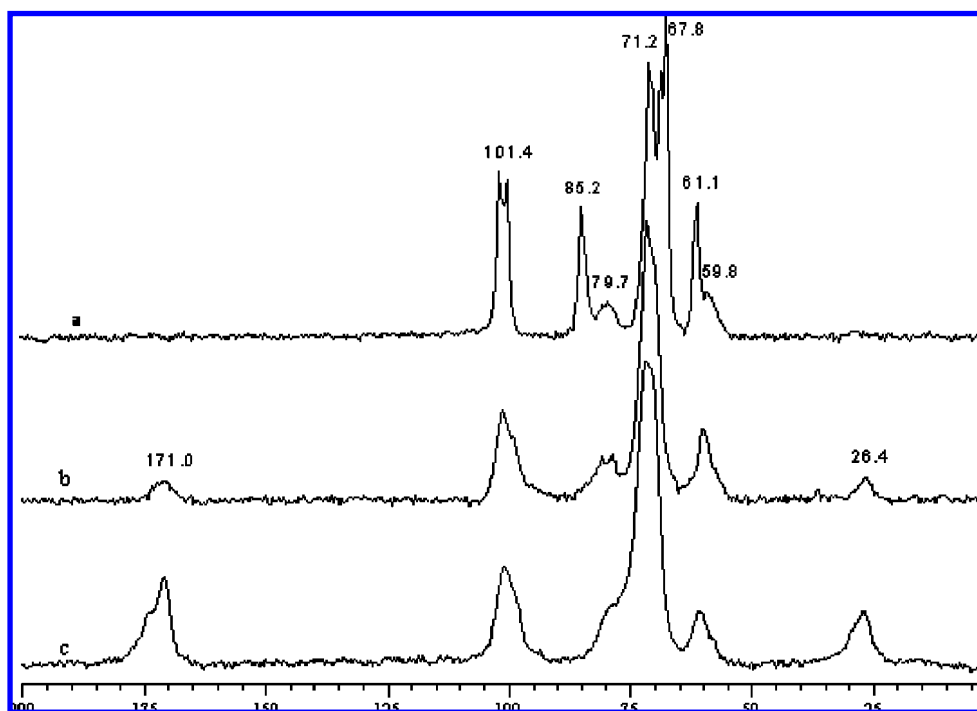


Figure 6. Solid-state CP/MAS ^{13}C NMR spectra of unmodified cellulose (spectrum a) and succinylated cellulose without any catalysts (spectrum b, sample 1) and with NBS as a catalyst (spectrum c, sample 2).

(spectrum b, without any catalysts), and sample 2 (spectrum c, with NBS catalyst) are shown in **Figure 6**. In spectrum a, all noticeable signals are distributed in the region between 50 and 110 ppm for the carbons of carbohydrate. The signals at 101.4 (C-1), 85.2 (C-4 of crystalline cellulose), 79.7 (C-4 of amorphous cellulose), 71.2 (C-5), 67.8 (C-2 and C-3), 61.1 (C-6 of crystalline cellulose), and 59.8 ppm (C-6 of amorphous cellulose) are all observed (33). After succinylation in the $[\text{C}_4\text{mim}]\text{Cl}/\text{DMSO}$ system, the signals at 85.2 ppm for crystalline C-4 and 61.1 ppm for crystalline C-6 disappeared in spectra b and c, which indicated that the crystalline structure of the cellulose was completely disrupted during the dissolution and functionalization, suggesting that the succinylation reaction occurs in the homogeneous phase. Evidently, the presence of the signals at 171.0 ppm for the

carboxylic group and 26.4 ppm for the methylene group also provided evidence of succinylation, which indicated that the reaction shown in **Figure 2** does occur. The three free hydroxyl groups at the C-6, C-2, and C-3 positions are the main reactive sites in cellulose. As shown in **Figure 6**, the intensity of the signal for C-6 decreased after succinylation, and that at 67.8 ppm for C-2 and C-3 also decreased, which indicated that the succinylation reaction at the C-6, C-2, and C-3 positions does occur. Clearly, as compared with spectrum b for cellulose sample 1 succinylated without any catalysts, the intensity of the signals at 171 ppm for carboxylic group and 26.4 ppm for methylene group significantly increased and that at 59.8 ppm for C-6 noticeably decreased in spectrum c for succinylated cellulose with NBS

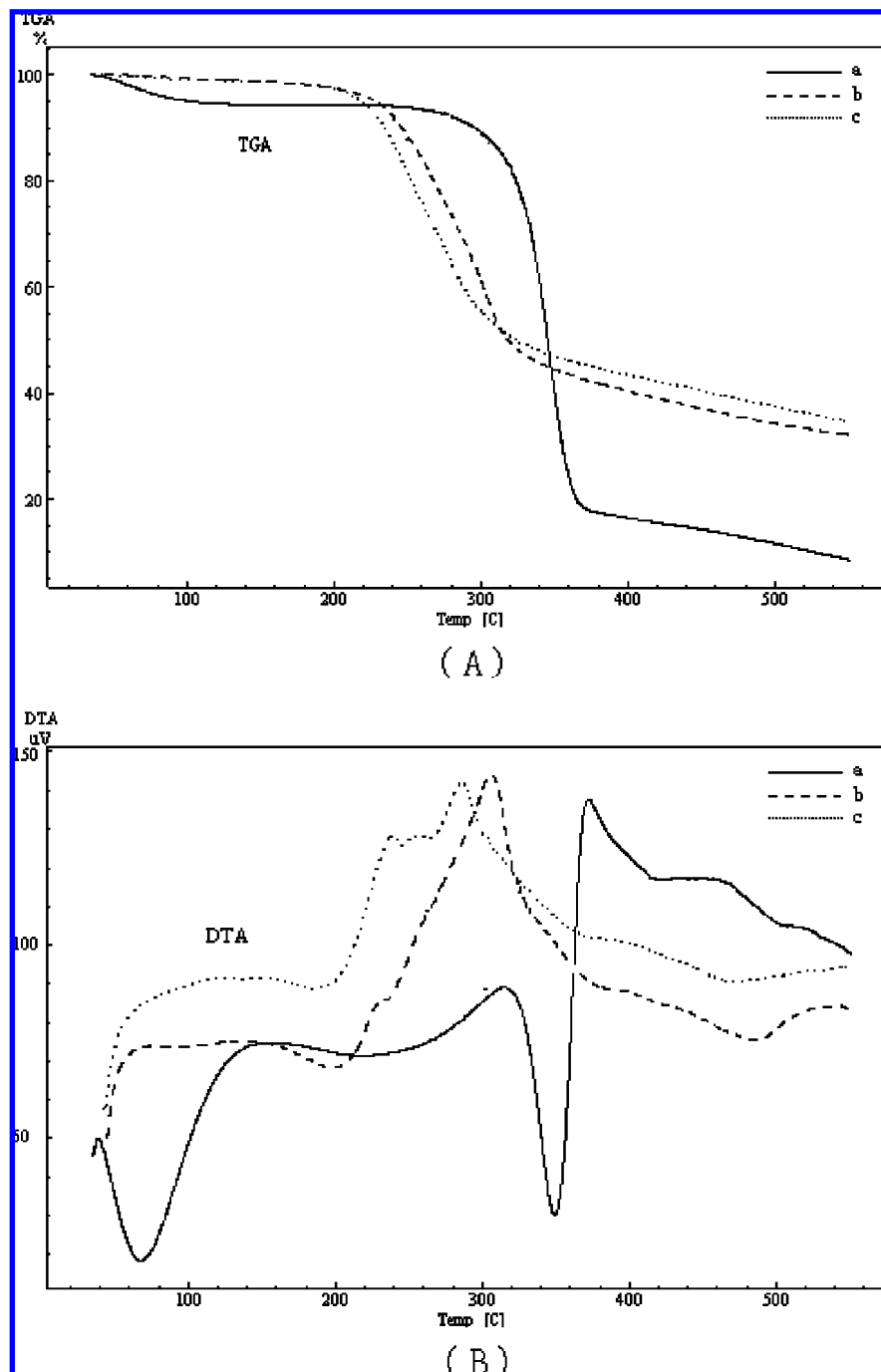


Figure 7. Thermograms of unmodified cellulose (a) and succinylated cellulose prepared with the weight ratio of NBS/SA 5 (b, sample 4) and 20% (c, sample 6).

catalyst, which indicated that NBS could significantly improve the functionalization efficiency of cellulose with SA in the IL BmimCl/DMSO system.

Thermal Analysis. Thermogravimetry is one of the most widely used techniques to monitor the polymers and structural dependence on the thermal degradation of cellulose. This is because the different polymers and supermolecular structures of cellulose behave differently when undergoing thermal degradation (34). **Figure 7** illustrates TGA and DTA curves of cellulose (curve a) and succinylated cellulose samples 4 (curve b) and 6 (curve c). The native cellulose starts to decompose at 240 °C, whereas the succinylated cellulose samples 4 and 6 begin to decompose at 203 and 198 °C, respectively. At 50% weight loss, the decomposition temperature occurs at 341 °C for native bagasse, 310 °C for sample 4, and 315 °C for sample

6. These decreasing trends of decomposition temperature imply that the thermal stability of succinylated cellulose is lower than that of native cellulose. It should be noted that the modified cellulose becomes less stable than the unmodified cellulose, although cross-linking occurs during succinylation. The main reason for the decrease of thermstability of succinoylated cellulose is partial hydrolysis and degradation of macromolecular cellulose, and this degradation has been reported in our previous studies (21, 22).

In conclusion, it is clear from these studies that NBS is a novel efficient catalyst for cellulose succinylation in the ILs BmimCl/DMSO system, and the succinylation efficiency of cellulose is significantly improved with NBS catalyst. The DS of succinylated cellulose samples ranged from 0.24 to 2.31 under the conditions given. FT-IR and solid-state CP/MAS ^{13}C NMR

spectroscopies provided evidence of homogeneous succinylation catalyzed with NBS and confirmed that the succinylation reaction at C-6, C-2, and C-3 positions all occurred. The thermal stability of succinylated cellulose was found to be lower than that of the native cellulose.

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