

Structure and Properties of Novel Regenerated Cellulose Films Prepared from Cornhusk Cellulose in Room Temperature Ionic Liquids

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Received 22 December 2008; accepted 13 August 2009

DOI 10.1002/app.31273

Published online 1 December 2009 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Cornhusk cellulose was regenerated using the ionic liquids viz., 1-allyl-3-methylimidazolium chloride (AmimCl) and 1-ethyl-3-methylimidazolium acetate (EmimAc). The cast cellulose films were characterized by FTIR, WAXD and SEM techniques. Their mechanical properties were also studied. These studies indicated that AmimCl and EmimAc are good solvents for the regeneration of cornhusk cellulose. The regenerated cornhusk cellulose (RCC) was found to be cellulose (II) with dense structure. The films cast from AmimCl exhibited good mechanical properties; the tensile modulus and strength

were as high as 6 GPa and 120 MPa respectively, whereas these values for those films cast using EmimAc were found to be 4.1 GPa and 47 MPa respectively. Further, it was observed that after regeneration, the solvents could be effectively recycled. Thus a novel nonpolluting process of forming RCC films from agricultural waste was developed. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 547–554, 2010

Key words: cornhusk cellulose; regenerated cellulose film; agricultural waste; ionic liquids

INTRODUCTION

Continued massive consumption of fossil hydrocarbon reserves presents serious energy crisis and environmental problems to the world. Petroleum resources are estimated to be exhausted within the next 50–100 years and hence, there is a need for alternative materials for petroleum products.¹ Science and technology tends to move toward renewable raw materials and more environmentally friendly and sustainable resources, and cellulose may be a good candidate for this purpose.²

Cellulose as a linear polysaccharide is the most abundant renewable organic material with a host of current and potential uses. Starting with dissolving pulp as a purified raw material, cellulose is converted industrially into regenerated materials (fibers, films, food casings, membranes, sponges, etc.) and cellulose derivatives (ethers and esters). However, chemical processing of cellulose is extremely

difficult in general because it cannot be molten and is insoluble in common solvents due to its high developed hydrogen bond networks and partially crystalline structure.³ Nowadays, the regenerated cellulose materials including fibers and films, remain predominantly produced by the well-known viscose process having lasted for 100 years. The viscose route, however, is technologically complex and shows significant problems, for example, it brings about considerable pollution to the air, water sources, and land, and it may also pose threat to human health due to the carbon disulphide air pollution. During the last 30 years, the use of *N*-methylmorpholine-*N*-oxide (NMMO) as a new organic solvent for cellulose opened up new perspectives for manufacturing cellulose materials and this process ranks among the environmentally friendly processes.⁴ However, despite the industrialization of NMMO solvent system, it has certain disadvantages associated with its use, such as the requirement of activation before dissolution, instability, the side reaction of the solvent itself without an antioxidant, difficult for recycling and its high cost as well. Therefore, the search for excellent and environmentally friendly solvents for cellulose continues.

A common theme in literature on cellulose dissolution is the need to simplify multistep and time

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Contract grant sponsor: National Basic Research Program of China; contract grant number: 2009CB219901.

consuming processes required to prepare cellulose solutions in nonderivatizing solvents.⁵ Recently, it is reported that room temperature ionic liquids (ILs) can effectively dissolve cellulose without any activation or pretreatment steps.⁶ In contrast to traditional volatile organic compounds, ILs have many attractive properties, such as good chemical and thermal stability, nonflammability and immeasurable low vapor pressure.⁷ ILs, 1-butyl-3-methylimidazolium chloride (BmimCl)⁸ and 1-allyl-3-methylimidazolium chloride (AmimCl)⁹ have been reported to be suitable solvents for cellulose. More recently, the IL, 1-ethyl-3-methylimidazolium acetate (EmimAc)¹⁰ has also been used as a highly efficient nonderivatizing solvent for cellulose. Compared with the alkylimidazolium chloride ILs, the acetate ILs have remarkable low boiling points and lower viscosity.

Recently, the main source for commercial cellulose production are cotton linters and wood pulp (WP). This fact makes the raw materials expensive to obtain. In contrast, the low-cost lignocellulosic biomass has become attractive as a renewable resource because it is available in large quantities and widely cultivated in the world. Among numerous biomass resources, agricultural wastes, woody biomass, and related resources form promising feedstock for this purpose because they are underutilized. The agricultural straw usually contains more than 30% cellulose, and is of limited commercial value at present. Currently China produces around 700 million tons of agricultural straw annually and most of it is simply burnt in the fields, creating a significant local air pollution problem. Therefore, effective utilization of these commonly available by-products as a source for cellulose will offer significant benefits to the economy and the environment. Besides, the cost of harvesting, handling, storing and transporting biomass resources is relatively low in most developing countries than developed countries.¹¹ At present, development of new and effective methods to convert low value agricultural straw into high value products is critical.

During the past several years, many attempts have been made to prepare cellulose materials from the agricultural straw, such as sugarcane bagasse,¹² wheat straw,¹³ rice straw,¹⁴ and rice hulls.¹⁵ Sang et al.¹⁶ prepared the regenerated cellulose fibers from rice straw cellulose with a diameter of 10–25 μm by wet spinning in rice straw cellulose/ NMMO solution. The tensile strength of all the as-spun fibers was between 0.28 and 0.40 GPa and the initial modulus between 11 and 13 GPa. Though tensile strength of the regenerated rice straw fibers is in a similar range as the commercial cellulose fibers, their initial modulus is relatively higher than that of commercially regenerated fibers. However, most previ-

ous studies about cellulose production were based on the traditional chemical methods or cellulose solvents.

Cornhusk is the large agricultural by-product that results from sweet corn processing. In fact, corn is one of the major crops in the world. In 2002, world corn production was about 640 million tons. Associated with corn production, every year about 45 million tons of cornhusk is produced, which consists mainly of 38.2% cellulose, 44.5% hemicellulose, and 6.6% lignin.¹⁷ Hence, it represents an abundant and largely unused cellulose source because of its high growth rate and adaptability to various soil types. Recently, Reddy and Yang¹⁸ developed a fiber extraction method from cornhusk and obtained cellulose fibers with mechanical properties similar to that of the common textile fibers. Their studies indicated that cornhusk might be a good cellulose source for high-value material production.

The main objective of the present study is to establish an environmentally friendly method for the preparation of regenerated cellulose materials from low-value agricultural residues replacing the traditional high-grade dissolving pulp. In this article, the cornhusk was used to prepare cellulose, and the obtained cornhusk cellulose (CC) was dissolved and regenerated in AmimCl and EmimAc solvents, and the films were cast from the resulting solutions. The details of the dissolution process of cellulose in the two ILs, and the formation of the regenerated cellulose materials are presented in this article. To evaluate the suitability of CC for industrial applications, the properties of regenerated cornhusk cellulose (RCC) were compared with regenerated cellulose from high-grade dissolving WP. Moreover, the recovery of ILs after the dissolution and regeneration is also discussed.

EXPERIMENTAL SECTION

Materials and reagents

Cornhusk was obtained from AnHui province, China. It was first dried under the sun, and then cut into small pieces (2–3 cm). The microcrystalline cellulose samples (Vivapur 101, with purity of 99.5%) were purchased from Fluka; Cotton linter was purchased from drugstore in Beijing, China; WP and cuprammonium hydroxide (Cuam) solvent were obtained from Baoding Swan Chemical Fiber Group, China. All the cellulose samples used were dried in vacuum at 100°C for 12 h before using. The ILs AmimCl and EmimAc were synthesized as described in our previous work.⁹ Their chemical structures are shown in Figure 1. All other chemicals used were of analytical grade and obtained from

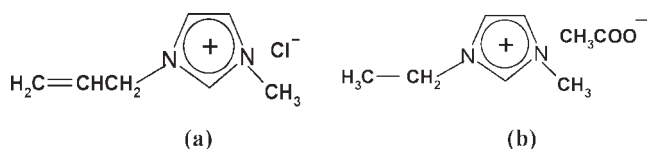


Figure 1 Chemical structure of AmimCl (a) and EmimAc (b).

Beijing Chemical Reagent Factory (China) and used as received.

Isolation of cellulose from cornhusk

Cellulose was obtained from cornhusks by a chemical extraction.¹⁹ Cornhusks were treated with 5% aq. nitric acid solution and heated to reflux for 3 h with 10% of cornhusks by weight in the acid solution. The treated slurry was washed with water to remove the dissolved substances and the coarse fibers obtained were heated to reflux for 1.5 h using 1.6% aq. sodium hydroxide solution. Sequentially, about 10 mL of 30% H₂O₂ was added to bleach the cellulose at 75–80°C for 0.5 h. After bleaching, the residue was filtered and washed thoroughly with water until the filtrate was neutral. Finally, the resultant white powder was dried in a vacuum oven at 70°C for 24 h, and then kept in a desiccator for analysis.

Nitric acid is a strong oxidizer that it can oxidize lignin in the cornhusk to nitric-lignin, which can dissolve in the post-treated NaOH aqueous solution. Furthermore, hydrogen peroxide has been widely used for many years to bleach pulps with high lignin composition, although it is unstable in alkaline conditions and readily decomposes. This however, generates other active radicals such as hydroxyl radicals (HO[•]) and superoxide anion radicals (O₂^{•-}), which participate in the delignifying and bleaching, and so it has been successfully used to isolate lignin and hemicellulose from agricultural residues.²⁰

Preparation of RCC films and solvent recovery

In a typical procedure for preparation of RCC film, 0.6 g CC sample was dispersed into 14.4 g IL solvent, and the mixture was heated at different temperatures in the range of 80–120°C for different intervals varying between 2 and 12 h under vigorous mechanical stirring. Finally, a clear cellulose solution with about 4 wt % of polymer was obtained. The resulting solution was cast onto a glass plate to give a thickness of 0.5 mm, the air bubbles were removed by keeping the glass plates in vacuum oven, and then immediately coagulated in fivefold amount of

distilled water to obtain a transparent regenerated cellulose gel. The cellulose gel was then washed three times with distilled water until the residual IL was removed. Finally, the cleaned gel was clamped between two glass plates, vacuum dried at 60°C for about 24 h, and the transparent regenerated cellulose film was obtained. The recovery of ILs was accomplished by evaporating water, and their purity was checked by ¹H-NMR spectroscopy.

Characterization

Physicochemical characterization of cornhusk cellulose

The content of α -cellulose in cornhusk cellulose was determined by the method of mercerization,²¹ the CC sample (1 g) was soaked in 17.5% NaOH aqueous solution at 20 \pm 0.1°C for 45 min, the residue was filtered off and washed thoroughly with water until the filtrate was neutral. Finally, the filtrate was dried in a vacuum oven at 70°C for 24 h. This method could remove most hemicellulose and noncellulose carbohydrate, and generated about 94.1 wt % of α -cellulose.

The DP of the CC and RCC was determined by Ubbelodhe viscometer using Cuam as solvent at 25°C. A cellulose sample of 20 mg (exactly weighted) was dissolved in 5 mL Cuam solution mixed with 5 mL distilled water under magnetic stirring for 20–40 min at room temperature. Then the transparent cellulose-cuam aqueous solution was obtained. The viscosity average DP of cellulose was estimated from their relative viscosity [η_r] in Cuam solution using the following equations:²²

$$\eta_r = \frac{t}{t_0} \cdot K$$

$$DP = \frac{2000 \eta_r - 2000}{0.29 C \eta_r + 0.71 C}$$

where:

η_r = relative viscosity.

t = flow time of the solution (s).

t_0 = flow time of the solvent (s).

K = instrument constant $K = 1.0$.

C = concentration of the solution (g/100 mL).

FTIR of the CC and RCC

The CC and RCC samples were ground into powder, and vacuum dried for 24 h. The IR spectra of the samples were recorded using a Fourier transform IR spectrometer (FTIR PE-2000, United States). The test specimens were prepared by the KBr-disk method.

TABLE I
Solubility and DP of Cellulose Sample in AmimCl and EmimAc (dissolving Temperature 80°C)

Cellulose		Solubility in			
		AmimCl		EmimAc	
Type	DP	^a Max. solubility (wt %)	^b Dissolution time (min)	Max. solubility (wt %)	Dissolution time (min)
Avicel	200	25	15	25	15
Wood pulp	516	15	30	16	25
Cotton linters	726	6	55	9	45
Cornhusk cellulose	509	14	35	15	30

^a The maximum solubility for cellulose at 80°C.

^b Time for dissolving 4 wt % cellulose in IL at 80°C.

WAXD of the CC and RCC

The natural CC was ground as powder, and the RCC films were cut into strips of 10 mm long and 15 mm wide, and vacuum dried for 24 h before analysis. The X-ray diffraction patterns with CuK α radiation ($\lambda = 1.5406$) at 40 kV and 200 mA were recorded in the range of $2\theta = 5^\circ\text{--}40^\circ$ with an X-ray diffractometer (D/MAX-2500, Rigaku Denki, Japan).

Morphology of the RCC

Scanning electron micrographs were recorded with a JSM-6700F scanning electron microscope of JEOL. RCC film was frozen in liquid nitrogen, immediately snapped, and then vacuum dried. The free surface (side in direct contact with the coagulant) and the fracture surface of the film were sputtered with gold before observation.

NMR characterization of the ionic liquids

The structure of fresh AmimCl, EmimAc and recycled ones were determined by ¹H-NMR spectroscopy (Bruker AV-400 at room temperature) in DMSO-*d*₆.

Mechanical testing

The average Young's modulus (*E*), tensile strength (σ_f) and elongation at break (ε_f) of the RCC films were measured on a universal testing machine (Instron 3365, UK) at a crosshead speed of 5 mm min⁻¹. The specimens with length 70 mm and width 3–5 mm were used in this test. A grip distance of 40 mm was maintained. For each category, at least five samples were tested and the average values were reported. Similar conditions were maintained for all the samples in the test.

RESULTS AND DISCUSSION

Dissolution of CC in ILs

In our previous studies, we reported that the commercially available cellulose samples, such as microcrystalline cellulose, WP, and cotton linters, with the DP in the range from 200 to 1000 could be readily dissolved in AmimCl.⁹ The recently developed EmimAc was also found to be an appropriate solvent for cellulose, that could dissolve cellulose in high concentration.¹⁰ In this article, the dissolution of CC, as well as WP and cotton linters, in AmimCl and EmimAc was examined.

Table I presents the solubility of different types of cellulose in AmimCl and EmimAc at the temperature of 80°C. From this table, it is obvious that the amount of dissolved cellulose in IL decreased with increasing DP of the cellulose sample. For example, the maximum solubility of microcrystalline cellulose in AmimCl was as high as 25%, while with increasing the DP of cellulose samples to 516 (WP) and 726 (cotton linter), the amount of dissolved polymer in ILs decreased to 15% and 6% respectively. Further, the dissolution rate of cellulose samples in either AmimCl or EmimAc was found to be quite rapid. For example, the dissolution of WP with a DP of 516 could be achieved in AmimCl within 30 min at 80°C without any pretreatment, and even for cotton linters with DP of 726, the dissolution time was just less than 1 h. In the case of CC, also a similar trend was observed. Moreover, it was also observed that the solubility of cellulose and dissolution rate in EmimAc was slightly higher than in AmimCl, which may be due to its relatively low viscosity that facilitated good dispersion of cellulose.

The effect of temperature and time taken for the dissolution on the DP value of RCC was also investigated and the results are presented in Table II. From this Table, it is evident that the DP of the RCC films regenerated from AmimCl decreased with increasing the dissolution time. For example, as the dissolving

TABLE II
Regeneration Conditions and Mechanical Properties of RCC Film

Code	Sample	ILs	Temperature (°C)	Time (h)	DP	Tensile strength Σ (MPa)	Young's modulus E (GPa)	Elongation at break ϵ (%)
A1	CC	AmimCl	80	4	410	112 \pm 13	6.01	3.8
A2	CC	AmimCl	80	8	394	119 \pm 13	6.55	4.1
A3	CC	AmimCl	80	12	386	108 \pm 15	7.10	2.4
A4	CC	AmimCl	100	4	362	85 \pm 14	4.92	5.6
A5	CC	AmimCl	120	4	300	78 \pm 12	5.05	2.1
A6	WP	AmimCl	80	4	433	100 \pm 11	5.30	3.0
R1	CC	AmimCl ^a	80	4	400	109 \pm 10	5.45	5.0
E1	CC	EmimAc	80	2	370	47 \pm 4	4.11	1.4
E2	CC	EmimAc	80	4	350	41 \pm 6	4.55	0.6
E3	CC	EmimAc	80	8	270	40 \pm 9	3.49	2.6
E4	CC	EmimAc	80	12	193	40 \pm 8	3.33	2.5
E5	CC	EmimAc	100	4	240	45 \pm 11	4.42	1.3
E6	CC	EmimAc	120	4	200	39 \pm 10	3.13	1.6
E7	WP	EmimAc	80	4	353	35 \pm 4	4.57	0.6
R2	CC	EmimAc ^a	80	4	370	48 \pm 8	5.52	0.5

^a The recycled IL.

time increased from 4 to 8 h, the DP of RCC decreased slightly from 410(A1) to 394(A2); and a further increase of the time to 12 h did not cause any appreciable decrease of DP (A3). However, with the increase of the temperature, the DP value significantly decreased. For example, an increase in temperature from 80 to 100°C led to a decrease of DP of RCC from 410 (A1) to 362 (A4) and the loss in DP became more pronounced as the temperature was increased to 120°C (A5). It is implied that the effect of temperature on the degradation of cellulose was greater than that of the dissolving time. The same tendency is observed for the dissolution of CC in EmimAc. It is also worth noting that the DP of regenerated cellulose from WP in AmimCl and EmimAc was 433 (A6) and 353 (E7) respectively, which lied in the same range as RCC regenerated under the similar conditions.

FTIR analysis

FTIR spectroscopy was used to obtain direct information on chemical changes of cellulose during dissolution and regeneration. Figure 2 illustrates the FTIR spectra of the original CC (spectrum a), the RCC films prepared from EmimAc (spectrum b) and AmimCl (spectrum c). Obviously seen from spectrum a, the CC extraction procedures removed most of the lignin as evidenced by the disappearance of the lignin-associated absorbance at 1600 and 1510 cm^{-1} .²³ Further, it can be observed that the three spectra were quite similar and no new peaks appeared in the regenerated samples, indicating that there was not any chemical reaction between the ILs and the CC occurred during the dissolution and

coagulation processes. In other words, both AmimCl and EmimAc were nonderivatizing solvents for cellulose.

Generally, in the spectra of cellulose and regenerated cellulose, the bands at 3665–3000 cm^{-1} (–OH stretching), 2903 cm^{-1} (–CH stretching) are sensitive to the crystalline state of the samples. From Figure 2, it can be observed that the maximum absorption of strong hydrogen-bonded –OH stretching for CC was located at 3401 cm^{-1} , while that for RCC shifted to higher wavenumber (3437 cm^{-1} and 3448 cm^{-1}). Accordingly, the –CH stretching mode at 2903 cm^{-1} also shifts to lower wavenumber at 2892 cm^{-1} and 2896 cm^{-1} . These results indicated that a crystal

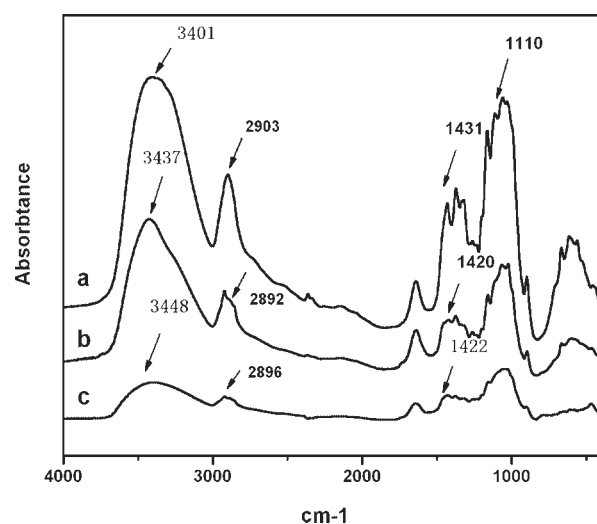


Figure 2 The FTIR spectra of original CC and RCC. (a) original CC, (b) RCC from EmimAc/CC solution(E1), (c) RCC from AmimCl/CC solution (A1).

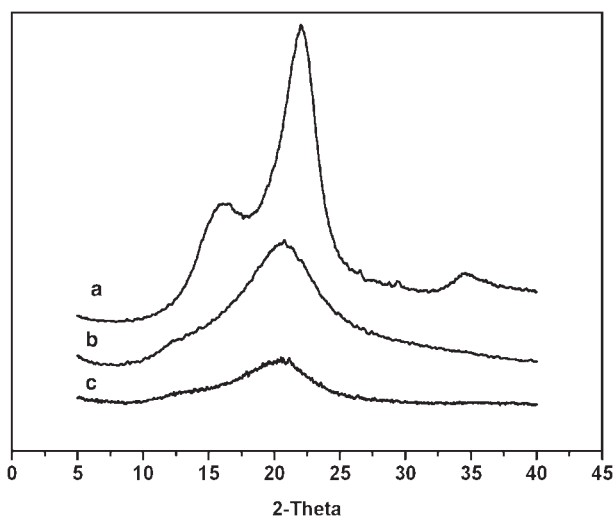


Figure 3 The WAXD patterns of original CC and RCC. (a) original CC, (b) RCC from AmimCl/CC solution (A1), (c) RCC from EmimAc/CC solution (E1).

transformation of cellulose I to cellulose II had occurred in the present case. Furthermore, the diminishing of the absorption band at 1110 cm^{-1} in the RCC, which was strong in the spectrum of CC, also confirmed this crystal transformation. In addition, the absorption band at 1431 cm^{-1} for the original CC, which was assigned to the $-\text{CH}_2$ scissoring motion, was weakened and shifted to lower wave number at 1420 cm^{-1} and 1422 cm^{-1} for the RCC, indicating the destruction of an intramolecular hydrogen bond involving O at C_6 .²⁴

WAXD analysis

The X-ray diffraction patterns of the original CC and RCC films regenerated from AmimCl and EmimAc separately are shown in Figure 3. It can be observed that the X-ray diffraction patterns of original CC (spectrum a) exhibited the crystalline lattice of cellulose I, with typical diffraction peaks at $2\theta = 16.0^\circ$ and 22.6° .²⁵ The diffraction patterns of the RCC films (spectra b and c) from the two ILs showed a broad peak at around $2\theta = 20.0^\circ$ and a small broad peak at $2\theta = 11.8^\circ$ corresponding to the crystalline form of cellulose II. These results indicate that the crystal transformation of cellulose I to cellulose II occurred during the dissolution and regeneration process. The same phenomenon also occurred for cellulose dissolution and regeneration in most other known solvent systems for cellulose.^{16,26} Compared to the original cellulose, the intensity of diffraction peaks of these regenerated cellulose films reduced significantly. Crystallinity was calculated by the ratio of the integral intensity in the profiles of the crystalline peaks to the total scattering intensity, and the results showed that the crystallinity of these RCC samples

was lower than that of the native CC. For example, the crystallinity of native CC was 59.6%, while for RCC samples A1 and E1, their crystallinity was 49.5% and 47.9%, respectively. This result indicates that, during the dissolution process, the original crystalline form and most of the inter- and intramolecular hydrogen bonds of cellulose were destroyed, and the rapid coagulation and regeneration of cellulose from the IL solutions might lead to the difficulty in recrystallization of cellulose chains.

Morphology of RCC films

As AmimCl and EmimAc were completely miscible with water in any ratio, the regenerated cellulose materials were easily obtained by coagulation in water. The IL/cellulose solution was cast onto a glass plate and then coagulated in water. After removing ILs and drying completely, a transparent cellulose film was obtained.

To study the morphology of RCC films, SEM micrographs were recorded at $5000\times$ magnification, and their images are shown in Figure 4. It can be observed from micrographs that, for the films regenerated from both AmimCl and EmimAc, the morphology of the dry material fused into a relatively homogeneous macrostructure from the interior to the surface, displaying a rough, but dense texture.

Mechanical properties of RCC films

To examine the suitability of cornhusk cellulose for industrial applications, the mechanical properties of RCC films were determined. The typical stress-strain curves of RCC films prepared in AmimCl (A1) and EmimAc (E1) are shown in Figure 5, and the average Young's modulus E (GPa), tensile strength σ_f (MPa), and failure strain ε_f (%) of the RCC films with different DP are shown in Table II.

From Figure 5, it can be seen that no yield point existed in the tensile process for RCC films prepared in both of the ILs, indicating the brittle feature of RCC films.

Further, it can be noted from Table II that relatively high film strength was attained for the films prepared from AmimCl, even when the DP was as low as 300, suggesting strong intermolecular interactions in the film. Moreover, the mechanical properties of the RCC films were found to be slightly higher than that of the films from WP. For example, under similar conditions, the maximum tensile strength of regenerated cellulose films prepared from WP and CC in AmimCl was 100 (A6) and 112 MPa (A1) respectively. Similarly, for RC films regenerated in EmimAc, the tensile strength was 35 (E7) and 47 MPa (E1), respectively. It should also be noted that both Young's modulus and tensile

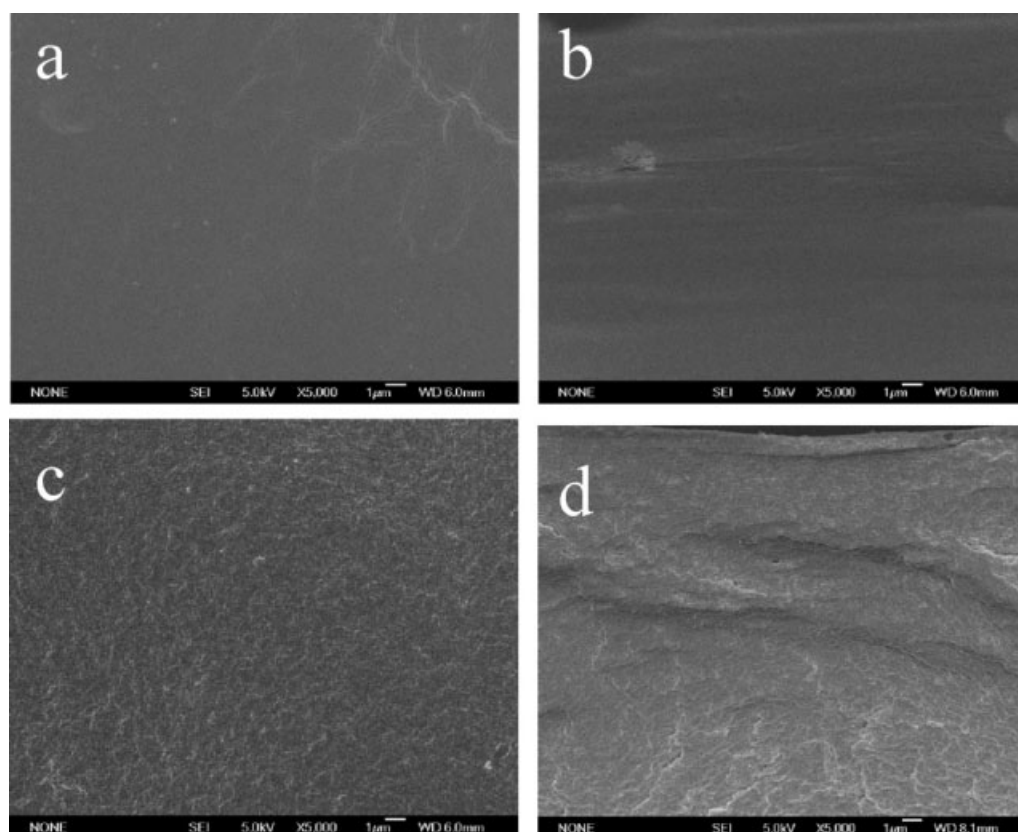


Figure 4 SEM photographs of the free surface and fracture surface of the RCC films. (a) free surface of RCC from AmimCl/CC solution (A1), (b) fracture of RCC from AmimCl/CC solution (A1), (c) free surface of RCC from EmimAc/CC solution (E1), (d) fracture of RCC from EmimAc/CC solution (E1).

strength of the RCC films prepared from AmimCl were obviously higher than those of regenerated cellulose films from EmimAc, suggesting that AmimCl is more suitable for preparing regenerated cellulose films with relatively high mechanical properties. However, even the lower tensile strength value for RCC films obtained in EmimAc was slightly higher than those largely used commercial polyolefin films,

such as polyethylene (PE) and polypropylene (PP), with tensile strength in the range of 20–40 MPa.²⁷ Therefore, these studies indicated that the RCC films can replace both PP and PE for packaging and agricultural purpose. Considering the nonbiodegradable nature of PP and PE, the regenerated cellulose materials are environmentally friendly.

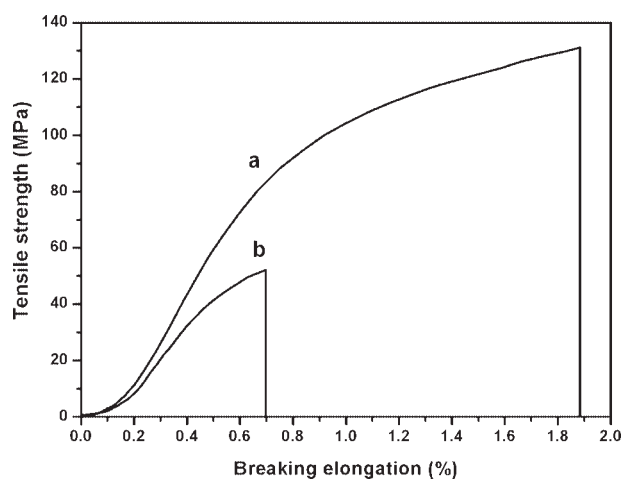


Figure 5 The stress–strain curve of the RCC films for A1 (spectrum a) and E1 (spectrum b).

Recycling of ILs

From the viewpoint of environmental conservation and economics of the process, after the regeneration of cellulose, effective recovery of the solvent ILs is absolutely required. In our study, after the regeneration of CC, the residual ILs in the coagulation bath were distilled under reduced pressure to remove water and give a 99% pure IL as confirmed by ¹H-NMR spectroscopy. The high purity of recovered solvent indicated the feasibility of solvent recycling without further purification. The recovered IL exhibited the similar dissolving capability for cellulose compared with fresh ILs. And RCC with similar performance was obtained under comparable dissolving and regenerating conditions. It is apparent that this advantage can promote the industrial application of ILs.

CONCLUSIONS

Regenerated cellulose films were successfully prepared from cornhusks in two ionic liquids AmimCl and EmimAc. FTIR analysis showed that AmimCl and EmimAc were direct solvents for cellulose. The X-ray diffraction patterns indicated that RCC film has a typical cellulose II crystalline form, and the degree of crystallinity was obviously lower than that of the original cellulose I. The SEM images of the RCC films displayed a homogeneous structure from the interior to the surface, indicating a dense architecture. The RCC films prepared from cornhusks displayed good mechanical properties: the tensile strength of RCC films regenerated from AmimCl with a DP of 410 could reach as high as 112 MPa, a value, which is even higher than that of the regenerated cellulose films obtained from dissolving WP under similar conditions, thus confirming the suitability of cornhusks for high quality use. Based on the fact that the ILs could be effectively recycled after regeneration, this study provides a technically feasible and environmentally acceptable method to prepare regenerated cellulose films using relatively cheap cornhusk as cellulose resources.

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