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Dissolution and Delignification of Bamboo Biomass Using Amino Acid-Based Ionic Liquid

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Abstract In the present work, the dissolution of bamboo biomass was tested using a number of ionic liquids synthesized in laboratory. It was observed that one of the synthesized amino acid-based ionic liquids, namely 1-ethyl-3-methylimidazolium glycinate, was capable of dissolving the biomass completely. The dissolved biomass was then regenerated using a reconstitute solvent (acetone/water) and was characterized using Fourier transform infrared spectroscopy, X-ray diffraction, and scanning electron microscopy. The results were compared to preconditioned bamboo biomass. The regenerated biomass was found to have a more homogenous macrostructure, which indicates that the crystalline form and structure of its cellulose has changed from type I to type II during the dissolution and regeneration process.

Keywords Bamboo biomass · Amino acid ionic liquid · Dissolution · Regeneration · Cellulose crystallinity

Introduction

The utilization of lignocellulosic biomass as a feedstock for bioconversion into fermentable sugars, an intermediate which is eventually converted into biofuel, has become increasingly important due its abundance and low price. In addition, lignocellulosic biomass is also the primary source for cellulose, hemicelluloses, and lignin. Cellulose is known to be widely used for fiber and membrane production, paper and paint manufacturing, and polymer processing industries [1, 2]. While hemicellulose and lignin can be depolymerized to produce starting materials for phenolic resin, epoxy, and furan resin production [3].

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The main barrier in utilizing lignocellulosic biomass for biofuel production has been the limitation in its pretreatment process due to the presence of covalent cross-linkages between lignin and carbohydrates in the plant cell wall and the crystallinity of the cellulose [4]. In order to overcome this limitation to enable further processing to be conducted on the lignocellulosic biomass, various methods have been used but each has its own drawbacks. Biological methods require long treatment time [5], while physical methods, such as mechanical milling, require significant energy and capital investment. The effectiveness of the physical methods is also doubtful for complete removal of lignin. On the other hand, chemical methods (acid or base treatment) are costly and not environmentally benign [6]. Physiochemical methods such as steam explosion, even though are considered as very promising methods, require high pressures/temperatures and use of catalysts [7].

Ionic liquids are organic salts with melting point less than 100 °C. Structurally, ionic liquids are made of cation and anion combination, and these affect their physicochemical properties. Given the huge number of possible combinations, their physicochemical properties can be tuned for specific applications, and this has been one of their main advantages. In addition, it has negligible vapor pressures, non-flammable nature, non-explosive, and is thermally stable over a wide range of temperature. It is also easily recyclable and due to the presence of ions, it can also behave as electrolytes [8, 9]. Due to these environmental benign properties, ionic liquids are seen as relatively greener solvent for the processing of lignocellulosic biomass. It has been reported that a number of ionic liquids have the ability to dissolve cellulose and lignocelluloses biomass [10]. Ionic liquids with imidazole type cation and chloride-, phosphate-, and acetate-based anions have been studied in the past [11–15]. Findings have shown that ionic liquids with acetate-based anion (having high hydrogen bond basicity) were found to be more effective for dissolution of biomass compared to the chloride based [11].

However, till now very few ionic liquids have been found to dissolve biomass. Therefore, the present work has been dedicated to discovering some new ionic liquids for dissolution of biomass. In this work, three different types of ionic liquids, namely 1-ethyl-3-methylimidazolium glycinate (EmimGly), 1-ethyl-3-methylimidazolium trifluoroacetate (EmimTFA), and choline propionate (Fig. 1), have been tested for dissolving bamboo biomass. In the study, irrespective to compare with other known efficient ionic liquids, the effects of different cation types, anion side chain, and hydrogen bond basicity of the ionic liquids were investigated during the dissolution process of the biomass. In order to confirm the dissolution process, the pretreated biomass material after regeneration using a reconstitute solvent mixture, i.e., acetone/water, was characterized and for any structural changes, using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) and was compared to preconditioned bamboo biomass.

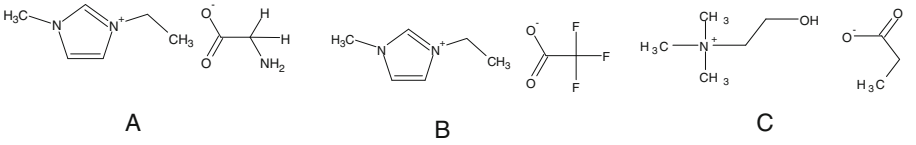

Fig. 1 The molecular structure of **A** 1-ethyl-3-methylimidazolium glycinate, **B** 1-ethyl-3-methylimidazolium trifluoroacetate, and **C** choline propionate

Experimental Procedure

Materials and Preparation

All the starting materials used are of analytical grade. The chemicals were obtained from (1) Merck: 1-ethyl-3-methylimidazolium hydrogen sulfate ($EmimHSO_4$), glycine, ethanol, and propionic acid; (2) Sigma Aldrich: acetonitrile, $Ba(OH)_2$, choline hydroxide (46%), EmimTFA, and indulin AT. For water, the Millipore grade deionized water was used. For the biomass material, the bamboo (*Gigantochloa scortechinii*), a native plant of Malaysia, locally known as buluh Semantan was obtained from a local market and was used. The bamboo was ground into particle of different sizes (125 μm to 1 mm). Before using the bamboo powder in the dissolution process, it was refluxed for 6 h with acetone to remove extractive (soluble nonstructural materials such as nonstructural sugars, nitrogenous material, chlorophyll, and waxes), and then the bamboo powder was dried in an oven at 105 °C for 3 h and the moisture content was determined using HR Halogen Moisture Analyzer (Mettler Toledo).

Synthesis of Ionic Liquids

1-Ethyl-3-Methylimidazolium Aminoethanic Acid Salt (EmimGly)

The ionic liquid EmimGly was synthesized by the following procedures. In the first step, EmimOH aqueous solution was synthesized from 1-ethyl-3-methylimidazolium hydrogen sulfate by adding $EmimHSO_4$ (0.05 mol) into equimolar aqueous solution of $Ba(OH)_2$ (dissolved in boiling water) and stirred for 12 h. In the second step, the solution was filtered and EmimOH containing filtrate was neutralized with equimolar aqueous solution of glycine by stirring at room temperature for 12 h. After neutralization, water was evaporated off under soft vacuum at 50 °C. The excess amount of glycine was precipitated by adding ethanol solvent. After filtration, the ionic liquid was first dried using vacuum rotary for 6 h and was further dried in a vacuum oven.

Choline Propionate

Choline propionate was synthesized using a neutralization method. Propionic acid (0.11 mol) was added slowly dropwise into an aqueous solution of choline hydroxide (46%, 0.1 mol) under cooling condition. The solution was stirred continuously for 12 h at room temperature. The ionic liquid obtained was dried using rotary vacuum evaporator followed by further drying in a vacuum oven.

Study on the Effect of Temperature on Water Content of Ionic Liquid over Time

In a typical example, EmimGly ionic liquid was poured into a reagent bottle and was exposed to the room humidity level overnight to obtain maximum saturation point. It was then placed in an oil bath and heated on a hot plate with magnetic stirrer at 300 rpm, while still exposed to room humidity. The selected temperature of 120 °C for the process was monitored by inserting a thermometer inside the oil bath. The water content of the ionic liquid was measured by coulometric Karl Fischer titrator, DL 39 (Mettler Toledo) at every 30 min interval for 6 h (360 min).

Dissolution and Regeneration of Biomass Using Ionic Liquids

A 5% (0.25 g, *w*/*w*) of preconditioned bamboo powder (125 μm particle size with moisture content of 3%) solution was prepared in each of the ionic liquids (5 g), prepared earlier (EmimGly, choline propionate), and EmimTFA. Initially half (2.5%) of the bamboo sample and ionic liquid (5 g) were charged in reagent bottle, after a while the remaining sample (2.5%) of biomass was added. The solution was heated at 120 °C for a known time (8 h for EmimGly and 24 h both for choline propionate and EmimTFA, respectively) with stirring speed of 400 rpm for the purpose of dissolving the biomass. After the biomass has dissolved, the resultant bamboo/IL slurry was poured into a conical flask containing a mixture of acetone/water (7:3 *v*/*v*) to regenerate the dissolved material. The acetone in the mixture was used to solubilize lignin and also to enhance the precipitation of cellulose-rich material along with water. The conical flask was sealed with parafilm and the mixture was stirred at room temperature for 3 h. The formed precipitate of cellulosic-rich material was separated using vacuum filtration. The dissolved lignin in the filtrate was subsequently precipitated by evaporating off the acetone at ambient conditions and also adjusting the pH of the solution to approximately 2 to 3 by adding 0.5 M HCl. After precipitation, the lignin was also separated using vacuum filtration. To recover the ionic liquid, the solution was neutralized with 0.5 M NaOH solution, and the ionic liquid was recycled by filtration of NaCl and subsequently evaporation of water. The same dissolution and regeneration procedures were used for above-mentioned ionic liquids.

Lignin Determination

The sample approximately 0.1 g was treated with 2 ml of 72% (*v*/*v*) sulfuric acid at room temperature for 2 h, followed by dilute acid (4%) at 121 °C for 1 h (in autoclave: pressure 2 bar). The solution was filtered and the precipitate was used for determination of acid-insoluble lignin by gravimetric method, after correction of ash content at 575 °C for 3 h. The acid-soluble lignin was calculated from UV absorbance measured by UV–Vis spectrophotometer (SHIMADZU) at 205 nm with an extinction coefficient value of 110 L/g cm [16].

Characterization of Materials

FTIR spectra for all the solid samples were taken using SHIMADZU 8400S at wavenumber setting ranging from 400 to 4,000 cm^{-1}. The crystalline structure of the bamboo powder and the regenerated cellulose-rich materials were analyzed using powder XRD, model Bruker D8 Advance horizontal X-ray diffractometer equipped with Cu anode, at room temperature. The samples were scanned within 10.00–35.00° 2θ in step mode with a step of 0.01° and a rate of 1° min^{-1}.

SEM images of the solid samples were also taken at ×3,000 magnification using a LEO VP1430 SEM instrument, operated at 15 kV accelerating voltage. Prior to imaging, the samples were sputter-coated with gold to make the fibers conductive, thus avoiding degradation and build up of charge on the specimens.

Results and Discussion

The synthesis of EmimGly ionic liquid using the above-mentioned method is relatively easy compared to the method reported by Fukumoto et al. [17]. In the method reported by

Fukumoto et al., the EmimOH was prepared from EmimBr through anion exchange resin column followed by neutralization with glycine. That method is time-consuming due to slow exchange of Br with OH of the resin, and moreover, large batch of ionic liquids could not be prepared by this method, while the method presented here, which uses $Ba(OH)_2$, requires lesser time than ion exchange resin method for preparation of EmimOH. In addition to shorter synthesis time, this method also allows large batch of EmimGly ionic liquids be prepared by neutralizing the EmimOH with glycine.

The results acquired by NMR and elemental analysis for the above synthesized ionic liquids confirmed their structures:

> EmimGly ^{1}H-NMR (DMSO-d_6): 1.42 (t, 3H), 2.76 (s, 2H), 3.83 (s, 3H), 4.25 (q, 2H), 7.73 (s, 1H), 7.82 (s, 1H), and 9.60 (s, 1H); elemental analysis (%) calculated C 51.87, H 6.8, N 22.68, and O 17.27 and found C 51.43, H 8.49, N 22.55, and O 17.35 (thermal decomposition by TGA is around 225 °C).
> Choline propionate ^{1}H-NMR (DMSO-d_6) 1.133 (t, 3H), 2.198 (q, 2 H), 3.232 (s, 9H), 3.508 (t, 2H), and 4.017 (s, 2H); elemental analysis (%) calculated C 50.88, H 11.59, N 8.47, and O 29.04 and found C 50.62, H 11.76, N 8.40, and O 29.14 (thermal decomposition by TGA is around 210 °C).

It has been reported that dissolution of biomass in ionic liquids depends on various factors such as particle size, density of biomass, ionic liquids-to-biomass ratio, water content in biomass, cooking time and temperature, biomass type, and several others [11, 18]. In consideration of these factors, the following experiments were conducted to study their effect in a more specific and quantitative manner.

The bamboo powder of small particle size (125 μm) was used, as it has been reported that increased dissolution of smaller wood particles is likely due to increased surface area, and more mechanical grinding (breaking down the internal structure) was required to obtain smaller particles [11]. As suggested by Swatloski et al. [10], the ionic liquids with low water content (as shown in Table 1) were used, as it has been reported that excess in water content above 1 wt.% (10,000 ppm) would impair the dissolution capacity of ionic liquid, as water would compete with anionic part of the ionic liquid to form hydrogen bonding with cellulose [10]. It is reported earlier that temperature above 100 °C would not only enhanced the dissolution process by providing higher energy to the dissolution process but it would also reduce the effect of moisture content on the dissolution process [11]. However, the detail study of moisture removal profile at higher temperature is not discussed. In this work, temperature of 120 °C was selected for the dissolution process, and the effect of heating temperature on moisture reduction was observed while heating the EmimGly ionic liquid containing 4.56 wt.% of water at 120 °C for 360 min as shown in Fig. 2. It can be observed that the water content decreases more rapidly at the beginning (up to about 120 min) and slows down after a while before finally stabilizing after approximately 350 min. It was observed that the water content dropped from 4.56 wt.% to about 0.75 wt.% after 350 min of heating. This result confirmed

Table 1 Water content (parts per million) of ionic liquids measured by coulometric Karl Fischer titrator, DL 39 (Mettler Toledo)

EmimGly	EmimTFA	Choline propionate
587 ppm	452 ppm	508 ppm

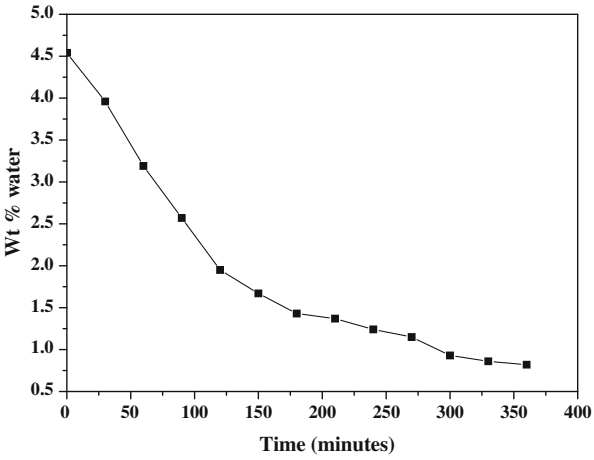

Fig. 2 Effect of heating time (120 °C) on water content in EmimGly ionic liquid

that under such relatively high temperature, it will facilitate not only the dissolution process of the bamboo biomass but also removes the water from the mixture thus reducing its moisture content.

Generally, the biomass components, i.e., cellulose, hemicelluloses, and lignin, are interlinked through hydrogen and covalent bonding. During the dissolution process using ionic liquids, the anion part of the ionic liquid disrupts the hydrogen bonding among the biomass components [19]. Although it has been reported that the cationic part of ionic liquid interacts with biomass through the covalent bond [20], however, the anionic part plays a greater role during dissolution process. In this respect, the anionic part of the ionic liquid having high hydrogen bond basicity would be a choice to use for the dissolution of biomass. EmimGly and choline propionate ionic liquids have been reported for their high hydrogen bond basicity [21, 22]; therefore, in this study, it was tested for dissolution of bamboo biomass. Moreover, EmimTFA ionic liquid containing acetate-based anion was also tested due to known capability of acetate-based anion for dissolution of lignocelluloses [11].

In this work, it has been observed that EmimGly ionic liquid is the only effective ionic liquid solvent for dissolving the preconditioned bamboo biomass compared to the other two ionic liquids, i.e., EmimTFA and choline propionate. During the dissolution process, for EmimGly ionic liquid, complete dissolution was observed by the naked eye after 8 h, while for other two ionic liquids, no dissolution was observed even after 24 h. If the molecular structure of EmimGly is compared to EmimTFA ionic liquid (see Fig. 1), it can be easily realized that the cationic part is the same for both, while the difference is only in the anionic part. The glycinate anion in EmimGly has a weak electron withdrawing group ($-NH_2$) on β carbon, which by negative inductive effect will not affect so much the carboxylate part of glycinate anion in making hydrogen bonding with bamboo biomass, thereby disrupt hydrogen bonding among biomass constituents during dissolution process. In contrast, EmimTFA has strong electron withdrawing groups (–F) attached to its carboxylate part which by negative inductive effect will stabilizes the carboxylate group (decrease electron intensity), thereby it would be difficult to form hydrogen bonding with bamboo biomass during dissolution process.

Based on the above facts, it is expected that EmimGly ionic liquid having more hydrogen bond basicity (β=1.19) will have a better dissolution ability as compared to

EmimTFA ionic liquid (β=0.233) [21, 22]. On the other hand, the ionic liquid choline propionate, although having high hydrogen bond basicity (β=0.98) through its anion part (see Fig. 1), was observed to have poor dissolution ability compared to EmimGly [23]. This could be due to two factors: (1) internal interaction of the hydroxyl end group of cationic part with its anionic part which impairs the hydrogen bonding capacity of its anion part and (2) lack of the aromaticity of its cationic part which is expected to enhance the dissolution of the biomass. From these findings, it can be concluded that not only hydrogen bond basicity of ionic liquid is important but its structure will also have significant effect on dissolution process.

After dissolution of the bamboo biomass in ionic liquid, it was regenerated using a reconstitute solvent, namely a mixture of acetone and water in 7: 3 ratios. This ratio of acetone and water in reconstituent solvent was selected due to its high solubility for lignin weight percent as shown in Fig. 3. Figure 3 presents the results obtained from the observation made on the weight percent of dissolved lignin (indulin AT from Sigma Aldrich) at various acetone/water ratios used in the reconstitute solvent. More than 90 wt.% of lignin is observed to solubilize when the volume ratio of acetone to water lies in the range of 8:2 to 5:5

The lignin content measured for preconditioned and regenerated cellulose-rich sample is provided in the Table 2. From Table 2, it is clear that regenerated cellulose-rich materials have a considerably lower lignin content compared to the corresponding preconditioned sample of bamboo. The findings show that EmimGly is quite an effective solvent for delignification of bamboo biomass as indicated by the significant lignin reduction of about 85.3% of total lignin content.

The FTIR spectra of both the preconditioned and regenerated cellulose-rich material from EmimGly ionic liquid represent more or less the same basic structure as indicated in Fig. 4. From Fig. 4, it is clear that the ionic liquid has been completely removed during the regeneration process as the peaks representing the ionic liquids components are not observed in the spectra. The spectrum for regenerated cellulose-rich material as shown in Fig. 4 is found quite similar to that of microcrystalline cellulose (result not shown). A distinctive and broad O–H stretching and C–H stretching absorption bands are observed at around 3,406 and 2,941 cm^{-1}, respectively. In the finger print region between 800 and

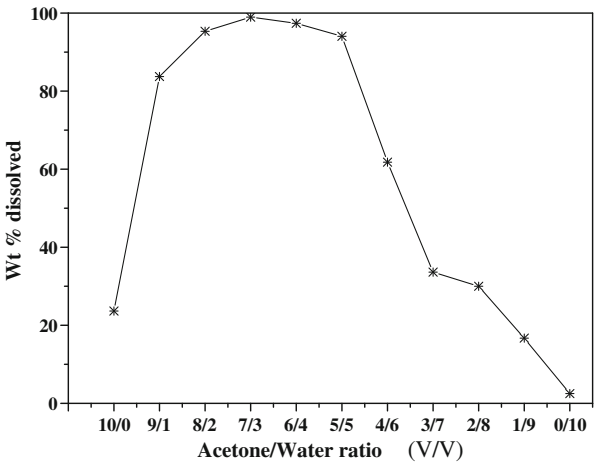

Fig. 3 Plot of dissolved lignin (weight percent) vs. acetone/water ratio (volume/volume)

Table 2 Lignin content of preconditioned sample and regenerated cellulose-rich material as determined by NREL [16]

Sample	Acid-insoluble lignin (%)	Acid-soluble lignin (%)	Total lignin (%)
Preconditioned bamboo sample	24.1	2.44	26.45
Regenerated cellulose-rich material	3.6	0.3	3.9

1,800 cm^{-1}, the non-conjugated C=O stretch (in hemicellulose) is observed at 1,739 cm^{-1} in spectra A (preconditioned bamboo), which is absent in spectra B (regenerated bamboo from ionic liquid) [24]. It has been argued earlier by Sun et al. [11] that this is due to the loss of hemicellulose during dissolution and regeneration from the ionic liquid. Comparing spectra A and B, lignin characteristic peaks at 1,604, 1,510, and 1,465 cm^{-1} are not observed in spectra B. This absence of lignin characteristic peaks confirmed the delignification of the regenerated biomass. The peaks which appear at around 1,328, 1,159, 1,037, 1,056, and 896 cm^{-1} are mainly attributed to the carbohydrates which are present in both spectra [24, 25].

The FTIR spectra of the precipitated lignin and indulin AT also show similar pattern of peaks as shown in Fig. 5. The lignin molecule contains various functional groups such as O–H, C–H, C–O, etc., for which bond vibrations are shown in Table 3 [25]. Bamboo lignin contains high proportion of syringyl residues which can be observed by an intense single peak at 832 cm^{-1} and more intense peaks at 1,128 and 1,320 cm^{-1} in the FTIR spectrum B [26]. The dot line shows the extra peak which was not observed in spectra A, which represents indulin AT, which might be the difference in lignin source.

The X-ray diffraction patterns for the preconditioned bamboo biomass (A) and the regenerated materials one from EmimTFA (B), choline propionate (C), and EmimGly (D) ionic liquids are shown in Fig. 6. In spectra A–C, the typical diffraction peaks at 2-theta 15.8° and 22.2° of cellulose I as discovered earlier by Takahashi et al. [27] are observed, while in spectra D low intensity diffraction peaks are observed at 2-theta 20.3° which are attributed to cellulose II. From this shifting of peaks, it is clear that a change in crystallinity

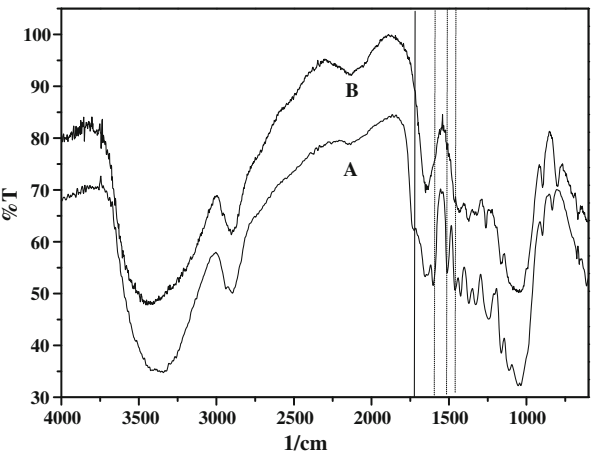

Fig. 4 FTIR spectra of *A* preconditioned bamboo and *B* regenerated bamboo from the ionic liquids EmimGly (*solid line* and *dotted line* represent hemicelluloses and lignin peaks, respectively)

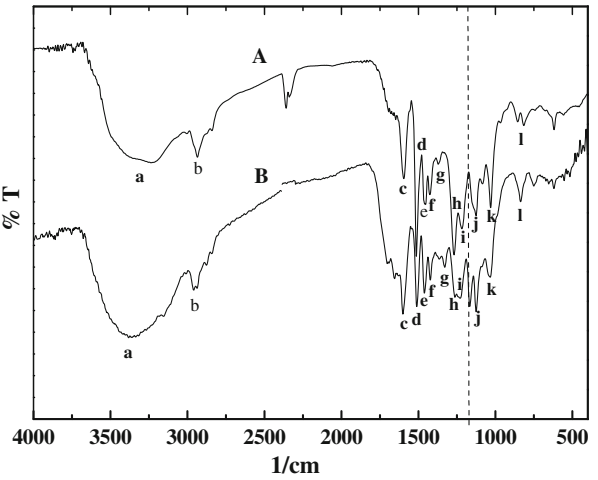

Fig. 5 FTIR spectra of *A* indulin AT and *B* recovered lignin from EmimGly

of the cellulose has taken place during the dissolution and regeneration of the bamboo biomass from ionic liquid. The range of possible structures for cellulose is presented elsewhere [28, 29].

During the dissolution process, the ionic liquids rapidly broke the intermolecular and intramolecular hydrogen bonds within the bamboo biomass structure and destroyed the original crystalline form as indicated by the peak shift in spectra D. While in spectra B and C, the patterns of peaks observed are similar to that in spectra A. This confirmed that the biomass did not dissolve in the other two ionic liquids, i.e., EmimTFA (B) and choline propionate (C).

In order to further confirm the findings, SEM images on the morphology of the preconditioned bamboo (a) and the regenerated material treated from choline propionate (b), EmimTFA (c), and EmimGly (d) are captured and the results are shown in Fig. 7. From

Table 3 Absorption FTIR spectra peaks of indulin AT and recovered lignin

Wave number (cm^{-1})	Remarks
3,378 (a)	O–H stretching
2,935 (b)	C–H stretching
1,595 (c)	Aromatic skeletal vibration in lignin
1,508 (d)	Aromatic skeletal vibration in lignin
1,459 (e)	C–H deformation in lignin and carbohydrates
1,419 (f)	C–H deformation in lignin and carbohydrates
1,320 (g)	C–O vibration in syringyl derivatives
1,261 (h)	Guaiacyl ring breathing with C–O stretch
1,226 (i)	Syringyl ring and C–O stretch in lignin and xylan
1,128 (j)	Syringyl ring
1,027 (k)	C–O stretch
832 (l)	C–H deformation in syringyl unit

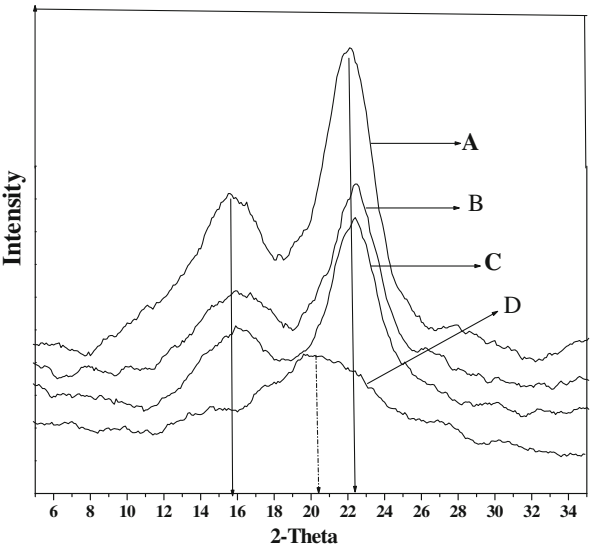

Fig. 6 Diffractograms of *A* preconditioned bamboo, *B* regenerated material from EmimTFA, *C* regenerated material from choline propionate, and *D* regenerated cellulose-rich material from EmimGly

Fig. 7b, c, it is clear that the biomass materials that were treated with choline propionate and EmimTFA, respectively, contain similar fiber and debris in their structures as in Fig. 7a which represents the preconditioned bamboo biomass. The presence of the fiber and debris confirmed that no dissolution of the biomass has taken place in these ionic liquids.

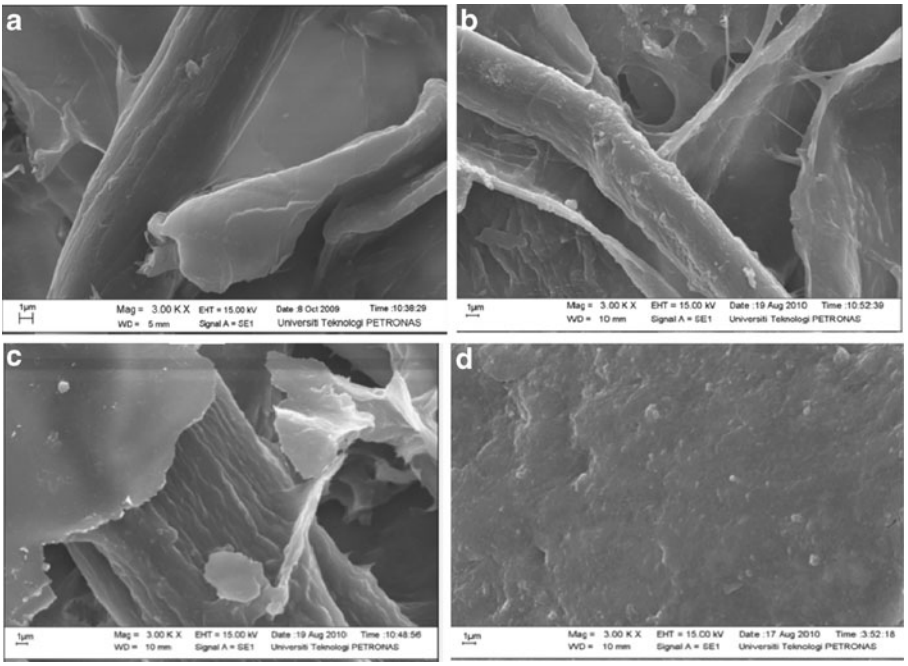

Fig. 7 SEM images of **a** preconditioned bamboo powder, **b** regenerated cellulosic materials from choline propionate, **c** regenerated cellulosic materials EmimTFA, and **d** regenerated cellulosic materials EmimGly

However, the regenerated material from EmimGly ionic liquid (Fig. 7d) shows a different morphology compared to the preconditioned biomass (Fig. 7a) in the sense that the fibers and debris have fused into a more homogeneous macrostructure.

^{1}H NMR spectra were taken for EmimGly (before and after using for dissolution process) in DMSO-d_6 solvent and recorded on a Bruker Avance 300 spectrometer as shown in Fig. 8. Figure 8 shows the corresponded structural peaks for unused (A) and regenerated ionic liquid (B). No extra peaks were noted; therefore, it confirmed the recycling process as well the thermal stability of ionic liquid during pretreatment of preconditioned bamboo sample.

Conclusion

This work has demonstrated that ionic liquids specifically EmimGly amino acid-based has the ability to dissolve the bamboo biomass which could then be regenerated through the use of reconstitute solvent containing a mixture of acetone and water to separate the biomass from the ionic liquids. In line with previous findings, this ability is due to the ionic liquids molecular structure which resulted in more hydrogen bond basicity, a crucial factor for effectively dissolving biomass material. The regenerated bamboo biomass from the ionic liquid treatment containing extracted cellulose-rich material is found to have similar FTIR spectra as pure cellulose (result not shown), and the extracted lignin is also found to have similar FTIR spectra to the commercial one. During dissolution and regeneration from EmimGly ionic liquid, the cellulose crystalline form changed from cellulose I to cellulose II as indicated by shifting of peaks in XRD analysis; this confirms the dissolution process of the bamboo biomass in EmimGly ionic liquid. SEM results presented evidence that the fibrous form of the bamboo biomass structure has changed into a more homogeneous macrostructure. The separation procedure developed for the biomass components in this study presented a possible greener alternative to extract useful biomass components for various applications such as fuel production and others.

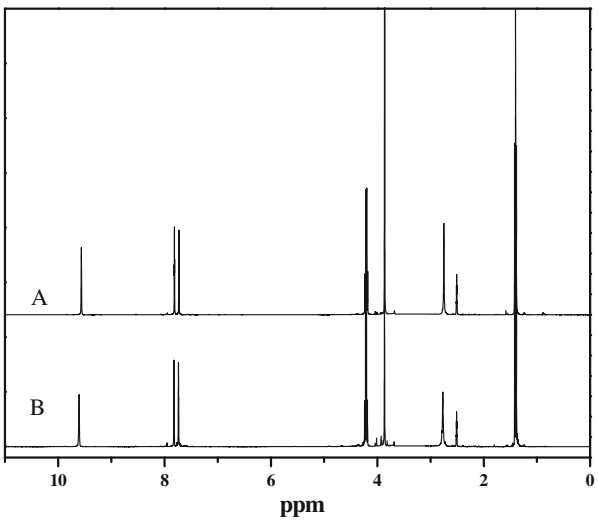

Fig. 8 ^{1}H NMR spectra in DMSO-d_6 of EmimGly (before and after using for dissolution process)

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