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Synthesis of Ionic Liquids, Solubility for Wood and Its Application for Graft Copolymer with Acrylamide

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1. Introduction

With the increasing awareness in sustainable development of environmental protection, there is a keen aspiration in exploring natural recoverable and environment-friendly materials [1]. Wood is a typical abundant natural material which is environment-friendly and easily-obtained. Cellulose, hemi-cellulose and lignin are the main components of wood powder which build the spongy three-dimension structures [2]. The poor solubility of wood powder in normal solvents strongly limits its application, hence it is important to find out environment-friendly solvents for wood powder.

The conditions of using conventional method to dissolve wood in solvent such as phenol or polyhydric alcohols were very strict and the original structures of wood were destroyed which results in the loss of mechanical properties [3, 4]. In contrast, ionic liquids which were known as green solvents for cellulose [5-9], can partly dissolve wood powder, and improve the reactivity due to the structural laxity [10-12]. The applications of ionic liquids ranged from chemical synthesis, biological catalysis, preparation of functional materials, etc. [13-19]. In addition, as a newly-developed technique which was widely used in various chemical areas, microwave can be applied in dissolving wood powder as a new and high efficient method.

Regarding the importance in preparing environment-friendly polymer materials from natural resources, studies of lignin were reported from several countries such as U.S., Japan, etc for decades. Polyacrylamide, enjoying the reputation of “hundred industry assistants” is used in various fields. But production cost is very high for its application. It has been of inevitable trend and broad prospects to develop multi-functional lignin flocculant using inexpensive lumber [20, 21]. Meanwhile, copolymerizations of lignin or cellulose with vinyl monomers in aqueous medium were reported [22-25], which have the shortages of low conversion of the polymerization, instable final product and difficulty in controlling the reactions. To overcome the disadvantages stated above, here, we report the synthesis of a novel flocculant with wood and acrylamide (AM) by graft copolymerization (AM-g-wood) in ionic liquids in which wood powder was used as alternative to lignin, which can introduce the new concepts of utilizing natural recoverable resources. Besides, graft copolymer of AM-g-wood as a novel modified flocculant, whose production cost was decreased greatly in this research, also could be widely applied in areas such as water treatment, petrochemical industry, mineral processing, papermaking and so on. The related work is being researched.

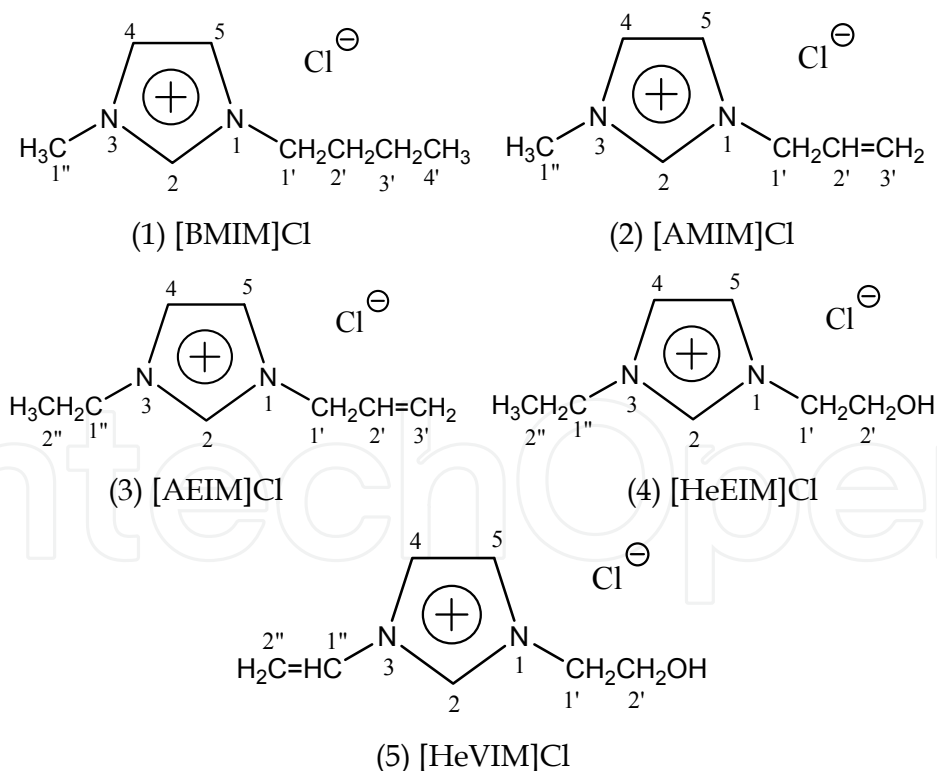
2. Experimental

2.1 Materials

Materials: N-methylimidazole, N-ethylimidazole, and 1-vinylimidazole (Purity \geq 99%) were obtained from Linhai Kaile Chemical Plant; Allyl Chloride and Chloroethanol were from Sinopharm Chemical Reagent Co. Ltd. and Tianjin Guangfu Fine Chemical Research Institute separately; Potassium Persulfate ($K_2S_2O_8$ or KPS) and acrylamide were received from Sinopharm Chemical Reagent Co. Ltd.; Benzene, Methanol, Acetone, Sodium hydroxide (NaOH), and Diethyl Ether were provided by Shanghai Suyi Chemical Reagent Co. Ltd.; Fir powder was donated by Huangshan Wood Factory. All chemicals and materials were used as received without further purification.

2.2 Synthesis of ionic liquids

Ionic liquids were synthesized from imidazole and allyl chloride (or chloroethanol), and their chemical structures were confirmed by FTIR and 1H NMR. Typical procedure is: into a round bottom flask, imidazole materials and allyl chloride (or chloroethanol) were added. The reaction was conducted under refluxing for 8-48 h. After reaction, the mixture was extracted with ether for several times to remove the residual imidazole. The ionic liquids can be obtained after being dried at $80^\circ C$ (vacuum: 0.08MPa) for 24h (yield: $>90\%$). The chemical structures of the ionic liquids were shown in Scheme 1.



Scheme 1. Chemical structures and abbreviation of the examined ionic liquids

1. 1-butyl-3-methyl imidazolium chloride ([BMIM]Cl) was synthesized according to the general procedure provided by Lu et al.[26] with a slight of modification. FTIR (film): $\nu=3422$ (ν O-H), 2918 and 2843 (ν C-H), 1632 (ν C=C), 1575 (ν C=N), 1472 (β C-H), 1169

- (δ C-H), 718 (δ imidazole ring); ^1H NMR (DMSO, 25°C): δ =0.84 (3H, t, H-4'), 1.26 (2H, m, H-3'), 1.76 (2H, m, H-2'), 4.00 (3H, s, H-1''), 4.23 (2H, t, H-1'), 7.42 (1H, d, H-4), 7.58 (1H, d, H-5), 10.26 (1H, s, H-2).
- 1-allyl-3-methyl imidazolium chloride ([AMIM]Cl) was synthesized according to the general procedure provided by Zhang et al.[9] with a slight of modification. FTIR (film): ν =3409 (ν O-H), 3083 and 2857 (ν C-H), 1645 (ν C=C), 1573 (ν C=N), 1424 (β C-H), 1167 (δ C-H), 998 (ρ C-H), 950 (ρ C-C), 763 (δ imidazole ring); ^1H NMR (DMSO, 25°C): δ =3.88 (3H, s, H-1''), 4.80 (2H, dd, H-1'), 5.22 (2H, m, H-3'), 5.81 (1H, m, H-2'), 7.26 (1H, d, H-4), 7.59 (1H, d, H-5), 10.08 (1H, s, H-2).
 - 1-allyl-3-ethyl-imidazolium chloride ([AEIM]Cl) was synthesized from allyl chloride 25.3g (0.33mol) and N-ethylimidazole 28.8g (0.3mol). The reaction was conducted under refluxing for 8h at 70°C in water bath with stirring under the protection of N_2 . FTIR (film): ν =3432 (ν O-H), 3082 and 2980 (ν C-H), 1645 (ν C=C), 1563 (ν C=N), 1449 (β C-H), 1163 (δ C-H), 997 (ρ C-H), 943 (ρ C-C), 761 (δ imidazole ring); ^1H NMR (DMSO, 25°C): δ =1.45 (3H, t, H-2''), 4.20 (2H, q, H-1''), 4.77 (2H, dd, H-1'), 5.34 (2H, m, H-3'), 6.00 (1H, m, H-2'), 7.43 (1H, d, H-4), 7.47 (1H, d, H-5), 8.74 (1H, s, H-2).
 - 1-(2-hydroxyethyl)-3-ethyl imidazolium chloride ([HeEIM]Cl) was synthesized from chloroethanol 16.7mL (0.25mol) and N-ethylimidazole 28.8g (0.3mol). The reaction was conducted under refluxing for 48h at 80°C in water bath with stirring. FTIR (film): ν =3392 (ν O-H), 3145 and 2994 (ν C-H), 1632 (ν C=C), 1566 (ν C=N), 1449 (β C-H), 1165 (δ C-H), 1072 (ν C-O), 750 (δ imidazole ring); ^1H NMR (DMSO, 25°C): δ =1.43 (3H, t, H-2''), 3.73 (2H, m, H-2'), 4.22 (2H, q, H-1''), 4.26 (2H, t, H-1'), 5.48 (1H, br, H-O-H), 7.81 (1H, d, H-4), 7.86 (1H, d, H-5), 9.39 (1H, s, H-2).
 - 1-(2-hydroxyethyl)-3-ethylene imidazolium chloride ([HeVIM]Cl) was synthesized from chloroethanol 16.7mL (0.25mol) and N-ethylene-imidazole 26.3g (0.28mol). The reaction was conducted under refluxing for 10h at 60°C in water bath with stirring under the protection of N_2 . FTIR (film): ν =3406 (ν O-H), 2994 and 2878 (ν C-H), 1651 (ν C=C), 1572 (ν C=N), 1495 (ν_{as} C-H), 1372 (β C-H), 1078 (ν C-O), 962 (ρ C-H), 916 (ω O-H), 750 (δ imidazole ring); ^1H NMR (DMSO, 25°C): δ =3.60 (2H, m, H-2''), 3.77 (2H, m, H-2'), 4.30 (2H, t, H-1'), 5.47 (1H, br, H-O-H), 7.40 (1H, m, H-1''), 7.97 (1H, d, H-5), 8.32 (1H, d, H-4), 9.80 (1H, s, H-2).

2.3 The pretreatment of wood powder

2.3.1 Extraction of wood powder with benzene and ethanol

Wood powder with particle size at 0.2mm was extracted with a mixture of benzene/ethanol (volume ratio: 1/1) using soxhlet extractor [27]. Then, the powder was dried at 70°C (vacuum: 0.04MPa) for 12h in Vacuum drying oven.

2.3.2 Treatment of wood powder with 25% NaOH

Wood powder with particle size at 0.2mm was treated with 25% NaOH solution in air for 2h. Alternative conditions are: microwave treatment for 5min at 60°C (360w), or treated MCC in 25% NaOH solution (40mL) at 140°C for 2h in an airtight high pressure pot (60mL), which was placed in Electronic constant temperature oven. The mixture was filtered, neutralized and washed with water several times. Then the fir powder was dried at 70°C (0.04 MPa) for 12h in Vacuum drying oven.

2.4 Dissolution of fir powder by microwave heating

Into a flask, ionic liquid and pretreated fir powder (W_0) above (weight ratio: 25:1) were introduced. The flask was placed in microwave oven (NJL 07-3) and heated at 90°C (400w) for 1h with stirring. The parallel experiment was copied. One of them was shifted to polymerization tube for future use. The other mixture was allowed to cool to room temperature followed by the centrifuge separation (2500r/min, 5min). The final fir powder was dried at 70°C (0.04Mpa) for 12h in order to calculate dissolution rate [11].

2.5 Synthesis and purification of AM-g-Wood

2.5.1 Synthesis of AM-g-Wood

Into a polymerization tube which was placed in water bath at 45°C, the mixture of fir powder/ionic liquid treated by microwave was added. Then air was purged out by N_2 stream before the introduction of initiator $K_2S_2O_8$ (I/M: 1/1000). AM was added dropwise into the mixture, and the dropping rate was controlled to avoid the homo-polymerization. The polymerization was stopped after 3h, and aged for 24h before purification.

2.5.2 Purification of AM-g-Wood

Typical purification procedure of AM-g-Wood is: the copolymer was washed over ethanol several times to remove the ionic liquid and residual monomer, followed by drying at 60°C under vacuum (0.08 MPa) for 24h. Then, the raw product (W_1) was extracted with acetone for 48h using soxhlet extractor to give the refined product, whose mass was W_2 after being dried under the same conditions above. The effect of graft reaction was evaluated by grafting degree (D) and grafting efficiency (E) which can be calculated by the following formulas:

$$\text{Grafting degree (\%)} = (W_2 - W_0) / W_2 \times 100\%$$

$$\text{Grafting efficiency (\%)} = (W_2 - W_0) / (W_1 - W_0) \times 100\%$$

W_0 -----Total mass of wood powder

W_1 -----Mass of raw product of AM-g-Wood

W_2 -----Mass of refined product of AM-g-Wood

2.6 Characterizations

MAGNA-IR750 FT-IR Spectrometer and AVANCE AV-400 Super-conducting Fourier Digital NMR Spectrometer was used to analyze the structures of ionic liquids; MAGNA-IR750 FT-IR Spectrometer, D/Max-rB X-ray diffractometer was used to analyze the structures and the crystallinity of pretreated fir powder and the graft copolymer of AM-g-Wood; ShimadzuTGA-50H Thermo-gravimetric Analyzer and JSM6700F Field Emission Scanning Electron Microscope were applied to analyze the thermal stability and apparent morphology of AM-g-Wood respectively.

3. Results and discussion

3.1 Wood pretreatment and dissolution in ionic liquids

3.1.1 Chemical structures of fir powder pretreated with different methods

Fir powder was extracted with a mixture of benzene/ethanol or activated with 25% (mass fraction) NaOH under normal temperature and pressure, microwave and high pressure in

order to make it dissolve more easily in ionic liquids. Chemical structures of the treated fir power, including the change of wave number, wave form and the interaction of hydrogen bonding, were examined by FTIR and the results were shown in Fig.1.

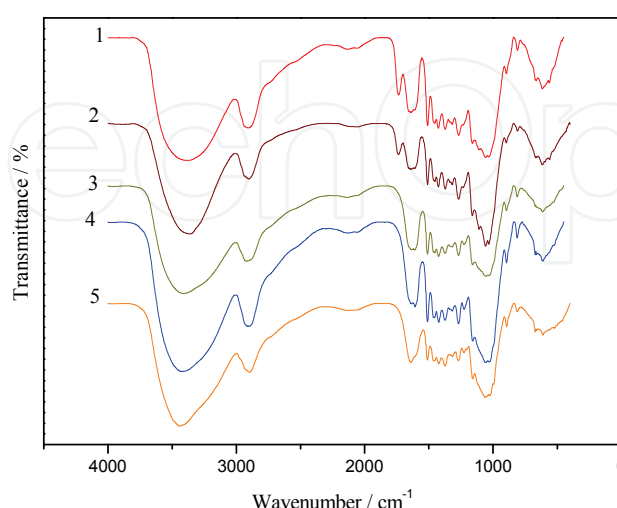


Fig. 1. FTIR spectra of fir powder pretreated with different methods

(1-Original wood; 2-Extracted by benzene/ethanol; 3-Treated with 25% NaOH (mass fraction) at normal temperature and pressure; 4-Treated with 25% NaOH by microwave heating; 5-Treated with 25% NaOH in high pressure pot)

The peak around 3400cm^{-1} is stretching vibration of O-H in hydrogen bonding. The peak originally ascribe to the absorption of lignocelluloses weakened which suggested the breaking of hydrogen bonding in the wood system after extraction and NaOH treatments, and this peak shifted to high wave number by the greatest degree and the waveform was the most narrow after high pressure treatment (Fig.1), which indicated the hydrogen bonding of sample 5 was broken most seriously. Similarly, the shrinkage of peak around 1730cm^{-1} indicated that certain amount of fatty acids and acetyl groups or uronic acids of the hemicelluloses have been removed partly by the extraction, and this peak disappeared after NaOH treatment which suggested that the hemicelluloses of wood was degraded and the fatty acids, uronic acids and acetyl groups, etc, were completely dissolved by NaOH solution. However, absorption peaks of wood powder mainly around 1500cm^{-1} , 1268cm^{-1} , 1060cm^{-1} remained unchanged after extraction and NaOH treatments under different conditions [28, 29]. Followed by the change of wave forms and analysis above, the preliminary conclusion could be reached that it is easier to dissolve fir powder treated with 25% NaOH under high pressure.

3.1.2 Crystallinity and dissolution rate of the pretreated fir powder

Crystallinity of fir powder treated under different technologic conditions were measured by Turley method, which scanned and counted in rule times at diffraction angles of 5° , 18.6° , 22.6° and 40° , then wrote down the diffraction peak intensity respectively and calculating the crystallinity. Dissolution rate of pretreated fir powder dissolved in [HeEIM]Cl by microwave heating was studied, and the results were shown in Table 1.

Sample number	Angle and Intensity				Crystallinity (%)	Dissolution rate (%)
	5°	18.6°	22.6°	40°		
1	17177	37523	74608	10498	61.0	6.2
2	16614	21780	38468	4949	60.2	8.4
3	14156	45671	54162	11196	19.6	13.6
4	8171	45386	49822	8773	10.6	16.8
5	8914	42504	43507	10450	2.4	18.3

(1-Original wood; 2-Extracted by benzene/ethanol; 3-Treated with 25% NaOH (mass fraction) at normal temperature and pressure; 4-Treated with 25% NaOH by microwave heating; 5-Treated with 25% NaOH in high pressure pot)

Table 1. Crystallinity and dissolution rate in [HeEIM]Cl of pretreated fir powder

Crystallinity of fir powder, which extracted by mixture of benzene/ethanol (the extract content was 6.1%), had little change while the dissolution rate increased (in Table 1). This is because some impurity and small molecule fattiness of wood powder were extracted by the mixture. When the fir powder was pretreated with 25% NaOH, the crystallinity of sample 5 was decreased and the dissolution rate was increased, especially under high pressure condition, the crystallinity decreased from 61.0% to 2.4%, while the dissolution rate increased from 6.2% to 18.3%. There were two reasons for the result: on the one hand, alkali had a certain effect on decreasing crystallinity and degrading hemicellulose of wood; on the other hand, higher temperature and pressure were helpful to destroy the crystal form of fir powder, causing the crystallinity to decrease and dissolution rate to increase. High pressure method, therefore, was used as the best pretreatment method for wood powder.

3.1.3 Solubility of different ionic liquids

Fir powder pretreated with 25% NaOH under high pressure (Sample 5 in Table 1) was dissolved in different ionic liquids by microwave heating, and the results were shown in Fig.2.

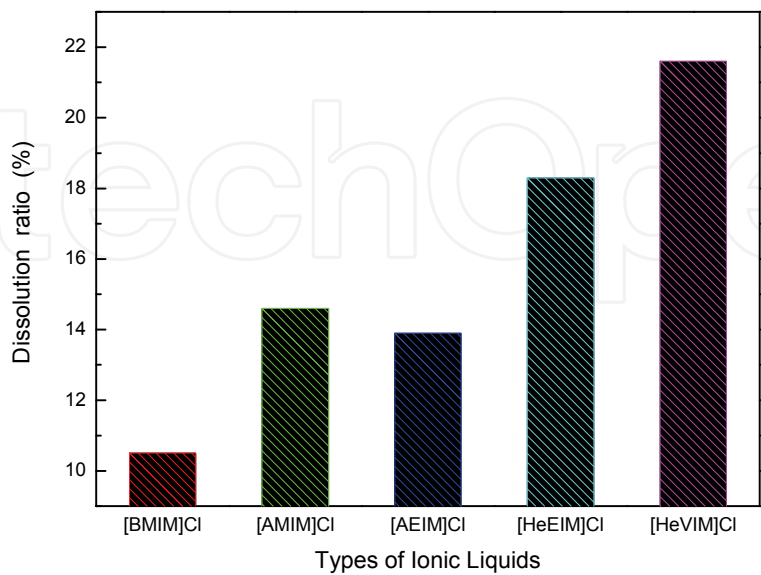
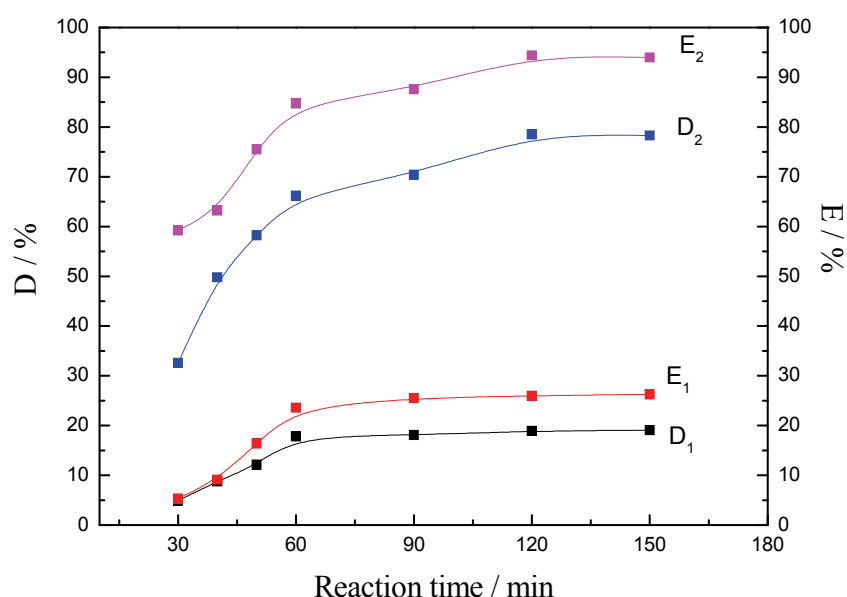


Fig. 2. Influence of different ionic liquids on dissolution rate

Due to the presence of hydroxyl and alkene groups in the side chains which contribute to the destroy of hydrogen bond, the solubility of fir powder using [HeVIM]Cl was the highest (Fig.2). The solubility of ionic liquid with hydroxyl side chain ([HeEIM]Cl) was better than alkene side chain ([AEIM]Cl) and alkyl side chain ([BMIM]Cl) (Fig.2). However, solubility of ionic liquids with shorter side chains ([AMIM]Cl) was better than that of [AEIM]Cl, which is ascribe to the reduced efficient chloride concentration [6]. Ionic liquid of [BMIM]Cl, whose side chain was of the longest alkyl groups, therefore, had the worst solubility. [HeEIM]Cl was used as radium of the following graft copolymerizations instead of [HeVIM]Cl, which had the best solubility, but it was more difficult to be purified.

3.2 Effect of reaction medium on graft copolymerization

AM was polymerized with fir powder (Sample 5 in Table 5) at 45 °C using aqueous and [HeEIM]Cl medium respectively. The results were shown in Fig.3.



(1-reaction medium of H₂O; 2- reaction medium of [HeEIM]Cl)

Fig. 3. Influence of reaction medium on grafting degree (D) and grafting efficiency (E)

Graft copolymer of AM-g-Wood could be prepared much more easily in [HeEIM]Cl than in aqueous system (Fig.3). Similarly, grafting degree (D) and grafting efficiency (E) of AM-g-Wood prepared in [HeEIM]Cl were much higher than that in aqueous medium. This is because fir powder can be dissolved in ionic liquid before copolymerization which resulted in the breaking of hydrogen bond and enhancing the reactivity of the powder. In Fig.3, grafting degree and grafting efficiency increased very fast during the beginning of the reaction (≤ 1 h), and became stable in aqueous medium, while increased slowly in [HeEIM]Cl thereafter. This phenomenon can be explained by the typical conventional free radical polymerization mechanism: In the first stage, a great number of free radicals were formed and the reaction was very quick. With the reaction went on, propagation slowed down due to the loss of radicals during the process. The best condition for graft copolymerization, therefore, was used [HeEIM]Cl as the reaction medium with the reaction time of 1h at least.

3.3 Orthogonal test

To optimize the technical conditions, orthogonal test was designed based on three factors and levels (Table 2) separately. The effects of different conditions, such as mass fraction of AM, fluid ratio of [HeEIM]Cl to Wood, and mass proportion of wood to AM on graft copolymerization were carefully studied. Details were shown in Table 2 and 3.

Levels	Factors		
	A (Mass fraction of AM)	B (Fluid ratio of [HeEIM]Cl to wood)	C (Mass proportion of wood to AM)
1	20%	25:1	1:3
2	25%	30:1	1:4
3	30%	35:1	1:5

Table 2. Factors and levels of the orthogonal test

No.	A	B	C	Grafting degree (%)	Grafting efficiency (%)
1	1	1	1	61.2	82.2
2	1	2	2	77.5	89.3
3	1	3	3	70.4	69.5
4	2	1	2	84.1	91.4
5	2	2	3	82.5	75.8
6	2	3	1	78.9	89.5
7	3	1	3	74.8	82.6
8	3	2	1	79.1	94.1
9	3	3	2	74.3	90.5
K1	209.1 / 241.0	220.1 / 256.2	219.2 / 265.8		
K2	245.5 / 256.7	239.1 / 259.2	235.9 / 271.2	—	—
K3	228.2 / 267.2	223.6 / 249.5	227.7 / 227.9		
R	36.4 / 26.2	19.0 / 9.7	16.7 / 43.3		

Table 3. Result of orthogonal test

The data in Table 3 showed that factors A, B and C had different effect on grafting degree and grafting efficiency of the synthesized products. The affecting order on grafting degree was A>B>C, while on grafting efficiency was C>A>B. The best comprehensive effects came from A₂B₂C₂ sample, which means that the mass ratio of AM was 25%, fluid ratio of [HeEIM]Cl to fir powder was 30:1, and mass proportion of wood to AM was 1:4.

3.4 Chemical structures of original wood and AM-g-Wood

Chemical structures of original wood powder and its graft copolymer of AM-g-Wood, which were prepared in [HeEIM]Cl using the best conditions according to the results of orthogonal test, were confirmed using FTIR. The results were shown in Fig.4.

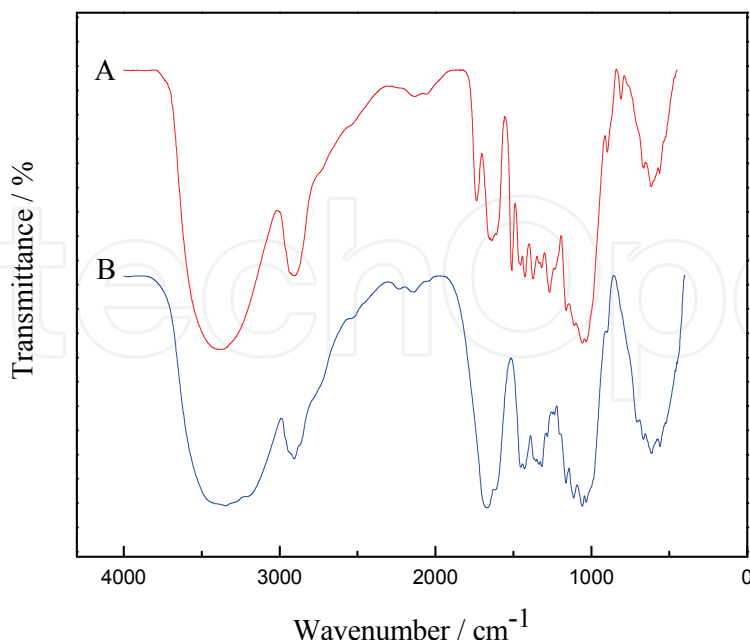


Fig. 4. FTIR spectra of original Wood (A) and graft copolymer AM-g-Wood (B)

For curve A of Fig.4, peak around 1735cm^{-1} was the stretching vibration of $\text{C}=\text{O}$ in fatty molecules and acetyl groups or uronic acids of the hemicelluloses of original wood powder, peak around 1638cm^{-1} was characteristic vibration of conjugated carbonyl in lignin of wood powder, and peaks around 1509cm^{-1} , 1160cm^{-1} , and 1058cm^{-1} were typical absorption of aromatic skeletal vibration, phenol-ether group and aliphatic ether vibration in lignin of wood, peak around 1268cm^{-1} was the stretching vibration of $\text{C}-\text{O}-\text{C}$ in celluloses and hemicelluloses [28-30]. For curve B, the peak around 1735cm^{-1} disappeared after grafted, which suggested that fatty molecules and hemicelluloses have been dissolved by ionic liquids. The new stronger peak in 1671cm^{-1} came from the typical stretching vibration of $-\text{CONH}_2$. There were still existence of peaks around 1160cm^{-1} and 1058cm^{-1} after grafted polymerization which came from the wood. The wave form of peak around 1268cm^{-1} became unnoticeable after grafted, which suggested that the content of cellulose in the wood was decreased. These analysis strongly suggested that AM has been grafted onto the fir powder.

3.5 Crystal form of original wood and AM-g-Wood

Crystal forms of original wood powder and its graft copolymer of AM-g-Wood were measured by X-ray diffractometer and the results were shown in Fig.5.

In Fig 5, diffraction peaks appeared at angles of 16.6° , 22.1° , and 34.8° which demonstrate that original wood powder have the same diffraction results as wood fiber. Crystallinity of the original wood powder was 61% based on the Segal formula calculation [31]. The diffraction peaks from wood powder disappeared after grafted polymerization and the crystal area were completely converted into amorphous region, which suggested that the ordered crystal structures have been destroyed. The change of the crystal type also suggested the graft copolymerization occurred between wood powder and acrylamide (Fig 5).

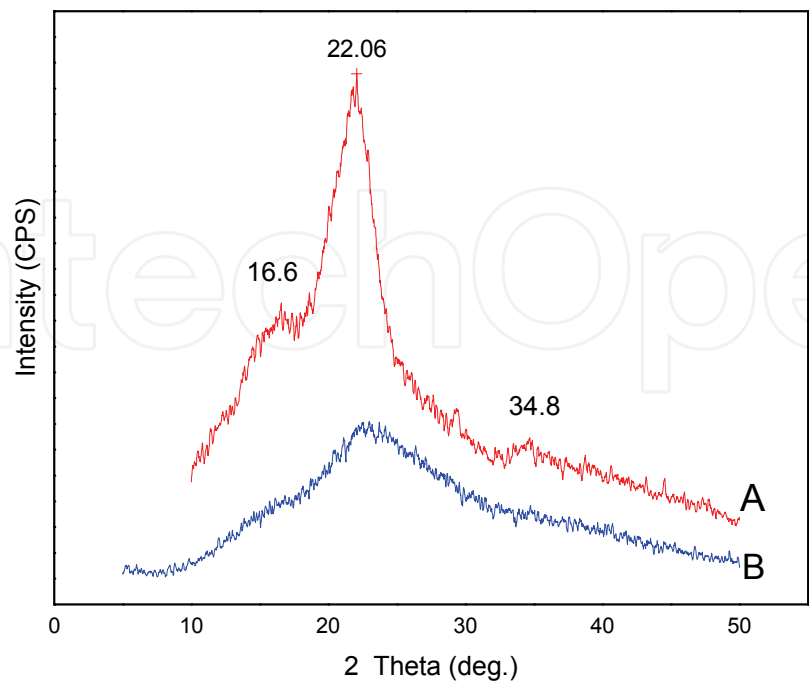


Fig. 5. X-ray spectra of original Wood (A) and graft copolymer AM-g-Wood (B)

3.6 Thermal stability of original wood and AM-g-Wood

Thermal stabilities of original wood powder and its graft copolymer of AM-g-Wood were examined by Thermo-gravimetric analyzer and the results were shown in Fig.6.

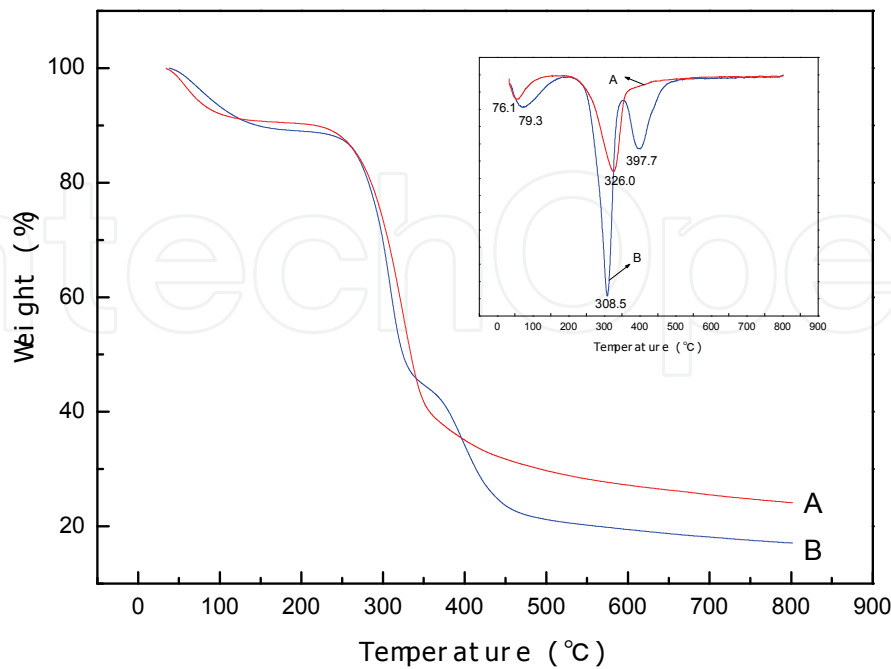


Fig. 6. TGA and DTG curves of original Wood (A) and graft copolymer AM-g-Wood (B)

As shown in Fig. 6, there were two steps for thermal decomposition of original wood: The first one was from 34°C to 175°C, which indicated that wood lost water in this range and the weight loss rate was around 9.5%; The second step was from 175°C to 794°C, responding to the thermal decomposition of wood and the weight loss rate was 66.2%. Actually, the original fir powder began to decompose and the chains of macromolecules were broken at 175°C. The temperature at 326°C refers to the largest weight loss rate. The residual weight rate was 24.4% after the temperature reached 800°C.

However, the thermal decomposition of the graft copolymer AM-g-Wood had three steps: The first step was from 38°C to 189°C, indicating the lost of water from copolymer with weight loss rate around 10.3%. The second step was from 190°C to 343°C, suggesting the weight loss (43.5%) resulted from imino reactions between amide groups of the PAM in the copolymer. The third step was from 343°C to 800°C with weight loss of 28.4%, and this is because the graft chains of PAM were broken during this temperature range. The final residual weight rate was 17.8% after the temperature reached 800°C. As such, the initial thermal decomposition temperature of the grafted wood was 343°C which was much higher than that of the original wood (175°C). Meanwhile, the temperature of the maximum weight loss rate for the grafted wood was 398°C, which was also higher than original wood (326°C). These results demonstrated that AM has been successfully grafted onto fir powder and the thermal stability of the wood after copolymerization was greatly improved.

3.7 Morphology of wood and AM-g-Wood

SEM results of the wood powder before dissolved (A), wood residue after dissolved in [HeEIM]Cl (B) and graft copolymer of AM-g-Wood (C) which was synthesized in [HeEIM]Cl were shown in Fig.7.

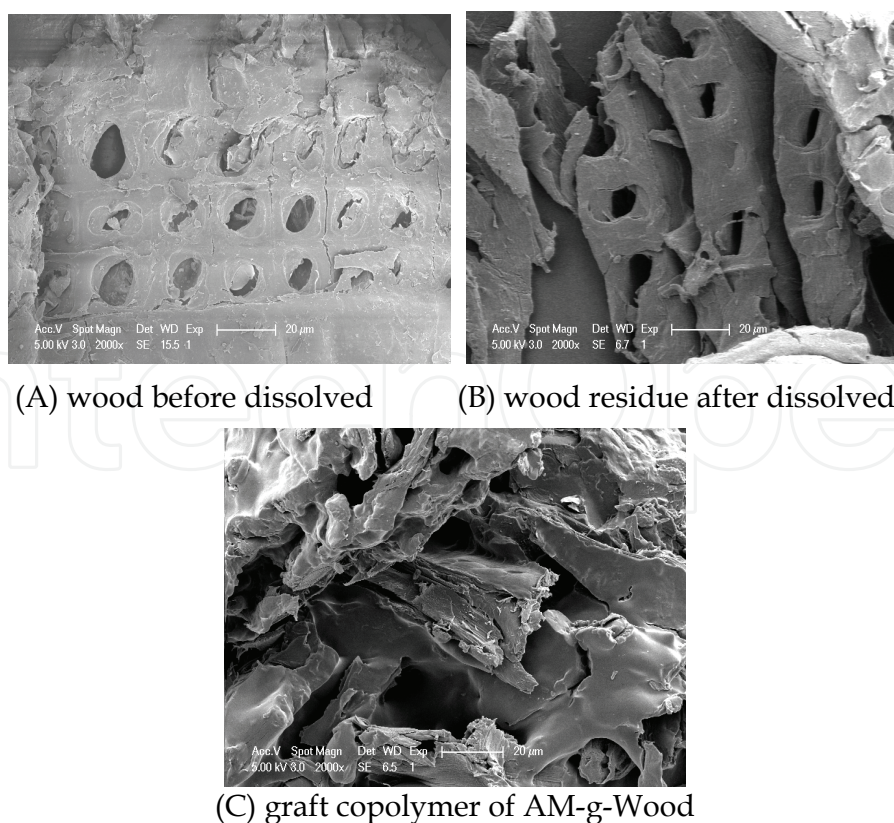


Fig. 7. SEM micrographs of wood and its graft copolymer

Microstructure of the wood powder before dissolved in [HeEIM]Cl was shown in A (Fig.7) which was loose and porous, while looser structure and bigger porous were found in B for wood after dissolved in [HeEIM]Cl. However, the loose and porous structures completely disappeared after polymerization (C) and it was difficult to detect fibril, which is very different from A and B. This difference should be ascribe to the existence of AM-g-wood structure which was synthesized from the graft copolymerization of AM and wood. Followed by the changes of wood appearance, therefore, a graft copolymerization occurred between fir powder and AM, and grafted copolymer of AM-g-Wood should be prepared more easily with wood residue as its looser structure and bigger porous.

4. Conclusions

Various ionic liquids with different functional groups of imidazole side chains have been synthesized, and their solubility for fir powder has been discussed. AM-g-Wood was synthesized in [HeEIM]Cl with $K_2S_2O_8$ as initiator and the conditions of graft copolymerization were optimized by orthogonal test. The specific conclusions were listed as follows:

1. The structures of synthesized ionic liquids have been confirmed by FT-IR and 1H NMR. It was suggested that the nature of side groups greatly affected the solubility of the ionic liquids. The ionic liquid of [HeVIM]Cl which contains hydroxyl and alkene groups in imidazole side chains was the best solvent for fir powder, but it was difficult to be purified.
2. The fir powder which has been pretreated with 25% NaOH solution under high pressure has the lowest crystallinity and the highest dissolution rate.
3. AM-g-Wood could be prepared readily in [HeEIM]Cl. Compared to aqueous medium, AM-g-Wood prepared in [HeEIM]Cl had advantages such as much better grafting degree and grafting efficiency, higher yields and so on.
4. Orthogonal test suggested that the best conditions for graft copolymerization were: mass fraction of AM was 25%, fluid ratio of [HeEIM]Cl to wood was 30:1, and mass proportion of wood to AM was 1:4.
5. Results of FT-IR and SEM demonstrated that AM has been successfully grafted onto the wood powder. The crystal structure of the wood has been completely destroyed after graft copolymerization, and the thermal stability has been greatly improved.

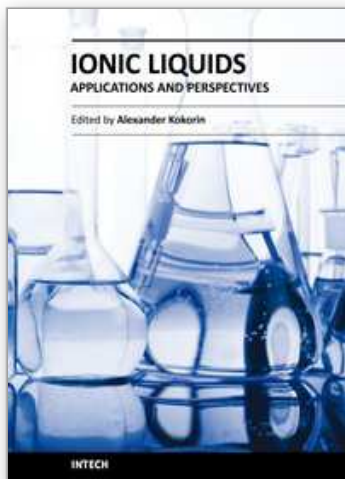
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This book is the second in the series of publications in this field by this publisher, and contains a number of latest research developments on ionic liquids (ILs). This promising new area has received a lot of attention during the last 20 years. Readers will find 30 chapters collected in 6 sections on recent applications of ILs in polymer sciences, material chemistry, catalysis, nanotechnology, biotechnology and electrochemical applications. The authors of each chapter are scientists and technologists from different countries with strong expertise in their respective fields. You will be able to perceive a trend analysis and examine recent developments in different areas of ILs chemistry and technologies. The book should help in systematization of knowledges in ILs science, creation of new approaches in this field and further promotion of ILs technologies for the future.

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