Synthesis and characterization of a novel cellulose-g-poly(acrylic acid-co-acrylamide) superabsorbent composite based on flax yarn waste

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ABSTRACT

A new, low-cost, and eco-friendly cellulose-based superabsorbent was successfully prepared from flax yarn waste. The method used was a free-radical graft copolymerization of AA and AM onto a cellulose backbone in a homogeneous aqueous solution. APS was used as the initiator in the presence of a crosslinker, MBA. The effects of various factors on water absorbency were discussed. The factors included reaction temperature, initiator amount, monomer amount, salt solution type, and solution pH. Under the optimized conditions, the water absorbencies of the obtained superabsorbent composite were 875 g/g distilled water, 490 g/g natural rainwater, and 90 g/g 0.9 wt% aqueous NaCl solution. The product also had excellent water retention and salt resistance properties. Fourier-transform infrared spectroscopy and scanning electron microscopy were employed to examine the structure of the prepared superabsorbent.

1. Introduction

Superabsorbent polymers are high-performance water absorbent and retention materials with three-dimensional network structures. They are able to absorb water and other liquids tens to thousands times their own weight in a relatively short time. They can also retain a swollen state even under some pressure (Li, Zhang, & Wang, 2007; Liu, Miao, Wang, & Yin, 2009; Ramazani-Harandi, Zohuriaan-Mehr, Yousefi, Ershad-Langroudi, & Kabiri, 2006). They have superabsorbent properties conferred by their hydrophilic groups or domains. Given all these advantages, superabsorbent polymers are widely applied in various fields, such as in hygiene, medicine, nutrition, petrochemistry, agriculture, and horticulture (Chen & Tan, 2006; Pourjavadi, Farhadpour, & Seidi, 2008). Several studies on the agricultural applications of superabsorbent polymers have been done. The results revealed significantly reduced costs of irrigation, greatly improved plant survival rates, and obviously enhanced fertilizer retention in soil (El-Rehim, El-Sayed, & El-Mohdy, 2004; Tomaszewska & Jarosiewicz, 2002; Wu, Liu, & Liang, 2008). However, most of these superabsorbents are synthetic polymers based on acrylic acid (AA) or acrylamide (AM), which are costly, poorly degradable, and environmentally unfriendly (Wang & Wang, 2010; Zohuriaan-Mehr &

Kabiri, 2008). Given the gradual depletion of petroleum resources and the growing environmental pollution crisis from polymer syntheses, material biodegradability has become the focus of current studies (Dond, Xu, Li, & Mo, 2008; Yan et al., 2009).

The mass utilization of crop residues, such as wheat straw, corn stem, pouce, and flax yarn waste, has attracted considerable research interest (Liang, Yuan, Xi, & Zhou, 2009; Liu et al., 2009; Lokhande & Gotmare, 1999). Most of these crop residues are still burned or landfilled, causing both resource waste and environmental pollution (Aloulou, Boufi, & Labidi, 2006; Ren & Liu, 2008). In this regard, the current study aims to provide a novel method for the utilization of flax yarn waste. The major component (70%) of flax yarn waste is cellulose, which has been introduced as a superabsorbent polymer in previous studies. Compared to the cellulose from other sources, the flax cellulose has the longer molecular chain which means that it has more active groups on single molecular chain and has the better hydrophilicity, and is easier to modify. On the other hand, the modified cellulose, like carboxymethyl cellulose (Suo, Qian, Yao, & Zhang, 2007), has the better hydrophilicity and is easier to graft monomer than flax cellulose. Yet an extra modifying process is needed. Meanwhile, cellulose is abundant, renewable, low cost, and biodegradable (Jane, Richard, John, Spear, & Metlen, 2007; Klemm, Heublein, Fink, & Bohn, 2005; Nogi & Yano, 2008).

In the present work, flax cellulose was separated from flax yarn waste by a high-temperature alkali-cooking method. A new cellulose-based superabsorbent composite was homogeneously synthesized by simultaneously grafting the hydrophilic monomers AA and AM onto a cellulose backbone in low-cost NaOH/urea solution (Zhang et al., 2003). Ammonium persulfate (APS) and

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N, N′-methylenebisacrylamide (MBA) were used as the initiator and crosslinker, respectively. The effects of various factors on the water absorbency of the synthesized superabsorbent were discussed. The factors included reaction temperature, initiator amount, monomer/cellulose mass ratio, saline solution type, and solution pH. The water retention capacity of the superabsorbent was also investigated.

2. Experimental

2.1. Materials

Flax yarn waste was obtained from the Zhejiang Mingrui Linen Textile Co., Ltd. (Jiaxing, China). AA (analytical grade; Aladdin Chemistry Co., Ltd., China) was distilled under reduced pressure before use. AM (analytical grade; Yingguangtai Biotechnology Co., Ltd., China) was used as received. APS (analytical grade; Tianjin Yongda Chemical Co., Ltd., China) was recrystallized from water before use. MBA (analytical grade; Guoyao Chemical Reagent Co., Ltd., China) and urea (analytical grade; Beijing Solarbio Science Technology Co., Ltd., China) were used as purchased. All other agents used were analytical grade, and all solutions were prepared with distilled water.

2.2. Dissolution of cellulose

Flax cellulose was separated from flax yarn waste by a high-temperature alkali-cooking method (Li et al., 2009). The purity of flax cellulose was determined by measuring α-cellulose content, which was 97.6%. The dissolution process of cellulose was carried out according to the literature (Cai et al., 2008). 1 g milled flax cellulose (cellulose particles could pass through 200 mesh sieve) was added into 25 g aqueous 7 wt% NaOH/12 wt% urea solution. Then the solution was pre-cooled to −12 °C and stirred for 5 min to obtain a transparent and ropy cellulose solution.

2.3. Grafting of cellulose with AA–AM in homogeneous conditions

The cellulose solution was transferred to a 250 ml four-neck flask equipped with a magnetic stirrer, reflux condenser, and nitrogen line. The reactor was immersed in a water bath preset at the desired temperature (70 °C). Oxygen-free nitrogen gas was bubbled into the solution for 30 min before adding the monomer. A solution of APS in distilled water was added. After 15 min of stirring, AA/AM with a predetermined weight ratio containing a certain amount of MBA and 5 ml of distilled water was added to the reaction mixture. The water bath was kept at 70 °C for 2 h to complete the polymerization process. The obtained gel was washed with distilled water several times, and poured into excess non-solvent ethanol (anhydrous ethanol) (analytical grade) for dehydration. After extraction with acetone for 24 h at room temperature to dissolve the homopolymer, the gel was cut into small pieces and dried at 70 °C to constant weight. The dried gel was milled, and the sizes of the obtained particles were 40–60 mesh. The control sample (P(AA-co-AM)) was prepared in the same procedure mentioned above, but without the addition of cellulose.

The grafting percentage (GP) and grafting efficiency (GE) were calculated by the following equations:

$$GP = \frac{W_2 - W_0}{W_0} \times 100 \tag{1}$$

$$GE = \frac{W_2 - W_0}{W_1} \times 100 \tag{2}$$

where W_0, W_1, and W_2 are, respectively, the weights of the flax cellulose, the monomer (AA + AM), and the cellulose-g-poly(acrylic acid-co-acrylamide) after the homopolymers are removed.

2.4. Measurement of water absorbency

The tea bags (100 mesh nylon screen) containing the accurately weighed superabsorbent composite powder (0.1 ± 0.0001 g) were immersed in 1 l of deionized water, 500 ml of saline solution, and 500 ml of natural rainwater at room temperature to reach the swelling equilibrium (about 1 h). The tea bag was hung for 10 min to remove the excess solution. The equilibrium swelling Q_{eq} was calculated using the following equation:

$$Q_{eq} = \frac{m_2 - m_1}{m_1} \tag{3}$$

where m_1 and m_2 are the weights of the dry and swollen samples (g), respectively and Q_{eq} is the water absorbency per gram of dried sample (g/g). Data were reported as the means of three measurements.

2.5. Characterization

The Fourier-transform infrared (FT-IR) spectra were recorded on a Thermo Nicolet 5700 FT-IR spectrometer using KBr pellets. The scanning electron microscopy (SEM) images of the grafted cellulose were obtained by a JSM-5610 LV SEM system using an acceleration voltage of 8 kV. The samples were sputter-coated with a gold layer in a vacuum using an Emitech K 550 Sputter Coater, with a current of 10 mA for 40 s.

2.6. Absorbency in different salt solutions

The measurement of absorbency was the same as described in Section 2.4, except that different salt solutions (NaCl, KCl, NH₄Cl, CaCl₂, MgCl₂, and FeCl₃) were used.

2.7. Absorbency at various pH values

Test solutions with various acidic and basic pH values were prepared by diluting aqueous NaOH (pH 13.0) and HCl (pH 1.0) solutions to achieve the desired pH. The measurement of absorbency was the same as described in Section 2.4.

2.8. Measurement of water retention in soil

Soil from the school greenhouse was dried in a constant temperature oven at 105 °C for 24 h, and sieved through 10 mesh nylon. Different amounts (0.1, 0.3, and 0.5 g) of the superabsorbent composites were mixed with 100 g soil in 500 ml glass beakers. The soil samples were slowly infiltrated by running water till a little water seeped out from the gaps of soil, but without reaching the soil surface. A control experiment without the superabsorbent was also carried out. The beakers were maintained at room temperature and weighed every day (m_i) for 30 d. The water retention in the soil (R_{is}%) was calculated by the following equation:

$$R_{is} = \frac{m_i}{m_0} \tag{4}$$

where m_0 is the total weight of the saturated water absorbency sample and soil. Data were reported as the means of three measurements.
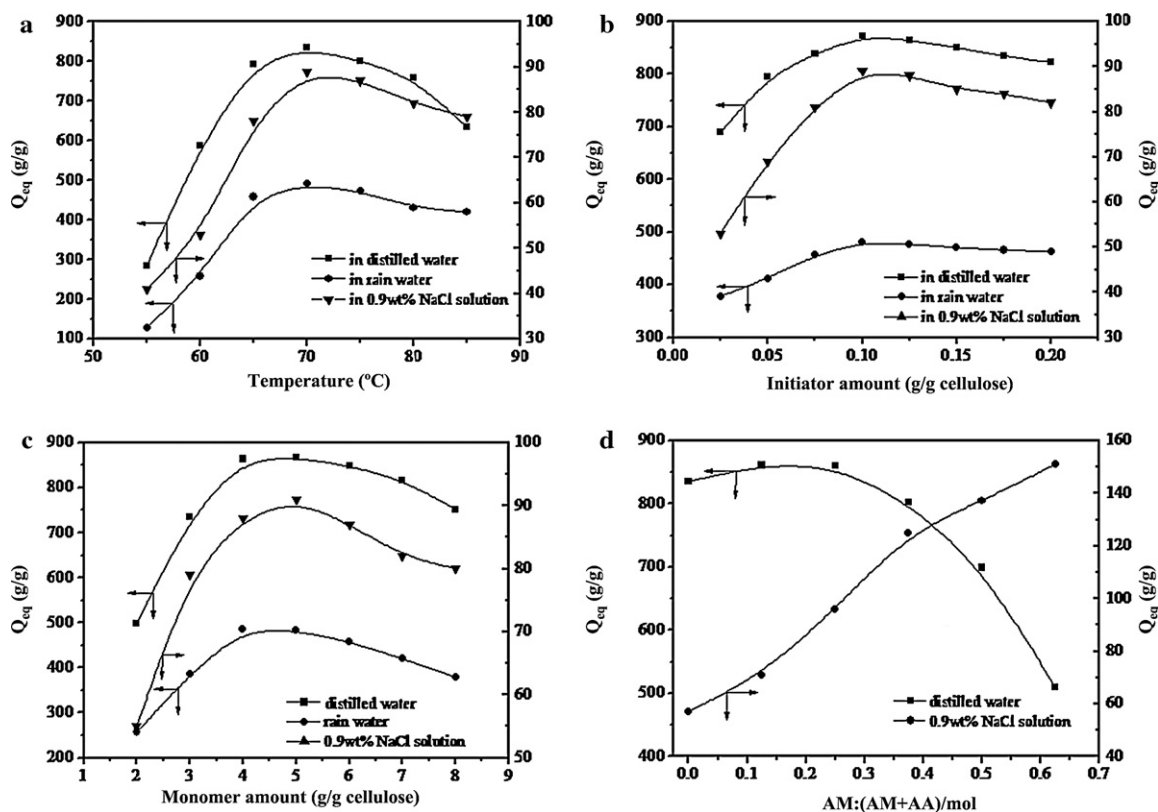
Fig. 1. Effects of synthesis parameters on the water absorbency of superabsorbent composites. (a) Reaction temperature. (b) Initiator amount. (c) Monomer amount. (d) AM to (AM + AA) ratio (the arrows in the figures specify the left or right *y*-axis which the curve follows).

2.9. Measurement of water retention at various temperatures

A weighed quantity of the superabsorbent composite was immersed in deionized water at room temperature to reach swelling equilibrium. The swollen gels were hung for 10 min in a tea bag to remove excess water, weighed (W_1), and placed in an oven at 25, 30, 40 and 60 °C. The relative humidity in the oven was constant at approximately 35.0%. The samples were weighed every hour (W_i) for 12 h. The water retention capability at various temperatures was calculated using the following equation:

$$R_{iT} = \frac{W_i - W_0}{W_1 - W_0} \qquad (5)$$

where W_0 is the weight of the dry superabsorbent composite. Data were reported as the means of three measurements.

3. Results and discussion

3.1. Synthesis of superabsorbent composites

3.1.1. Effect of reaction temperature on water absorbency

The effect of reaction temperature on water absorbency was investigated by changing temperature from 55 to 85 °C, and keeping other reaction conditions constant. Fig. 1a shows that the water absorbency significantly increased as the temperature increased from 55 to 70 °C, and then relatively decreased with the further increased temperature. The initial increase in water absorbency can be attributed to the increased reaction temperature, which increased the bimolecular collisions between the molecules of APS and cellulose. These collisions resulted in increased cellulose macroradicals, and thereby speed up the chain initiation and growth reactions. The graft copolymerization process was also enhanced. However, the subsequent decrease after reaching the

maximum absorbency may have been caused by the termination and chain transfer reactions at relatively higher reaction temperatures (Gordon, 2001).

3.1.2. Effect of the initiator amount on water absorbency

Fig. 1b shows the effect of the mass ratio of the initiator to cellulose on water absorbency. The water absorbency initially increased and reached the maximum (872 g/g distilled water, 481 g/g natural rainwater, and 89 g/g 0.9 wt% NaCl solution) at an APS to cellulose ratio of 0.1 g/g. With the further increase of initiator amount, the water absorbency decreased. The increase may be attributed to the increased amount of macroradicals, which was caused by the increase of APS content on the glucose units of cellulose (Lin, Zhan, Liu, Fu, & Lucia, 2009). Consequently, more available sites in the polysaccharide backbone reacted with AA-co-AM. However, the excess APS generated more free radicals, to accelerate the chain termination reaction. Therefore, the increase of the initiator amount was responsible for the decline of the water absorbency of the obtained cellulose-based superabsorbent.

3.1.3. Effect of the monomer (AA + AM) amount on water absorbency

Hydrophilic groups play an extremely important role in water absorbency (Zhang, Li, & Wang, 2006). Fig. 1c shows the effect of the monomer (AA + AM) to cellulose weight ratio on the water absorbency of a superabsorbent. Water absorbency initially increased, and then considerably decreased with the continuously increased monomer (AA + AM) amount. A maximum was reached when the monomer (AA + AM) to cellulose weight ratio was 5 g/g. Apparently, the increased monomer (AA + AM) concentration promoted the monomer (AA + AM) molecules accumulation close to the cellulose backbone. However, water absorbency decreased with the further increased monomer (AA + AM) amount. This result may
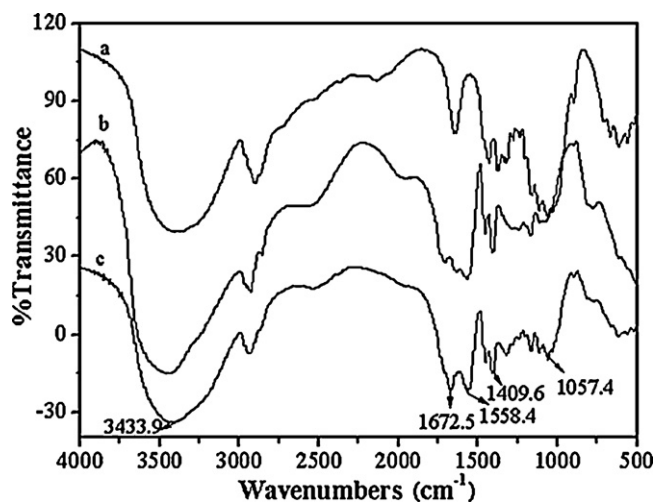
Fig. 2. FT-IR spectra of (a) cellulose, (b) P(AA-co-AM), and (c) cellulose-g-P(AA-co-AM) superabsorbent composites.

Fig. 4. Water absorbency of the superabsorbent composites in different salt solutions.

be ascribed to preferential homopolymerization rather than graft copolymerization, and to the enhanced chance of chain transfer to the monomer molecules (Pourjavadi, Soleyman, & Barajee, 2008).

3.1.4. Effect of the ratio of AM to AM + AA on water absorbency

Fig. 1d shows the effect of various molar ratios of AM to AA + AM on water absorbency. The results indicated that water absorbency increased when the molar ratio of AM to (AA + AM) was below 0.25. When this ratio further increased, water absorbency remarkably decreased. With the increased molar ratio of AM to AA + AM, the number of hydrophilic groups also increased. AM is a non-ionic monomer which has a good salt-resistance ability. Meanwhile, AA, as an ionic monomer, holds a better adsorption capability in water. Therefore, when a certain amount of AM and AA was graft copolymerized onto the cellulose backbone, the absorbency in both deionized water and saline solutions increased. This phenomenon may be explained by the collaborative effect of $-CONH_2$ and $-COO^-$ groups. However, the $-COO^-$ group has a better hydrophilic ability than $-CONH_2$, then the water absorbency decreased with the further increased AM (Wu & Liu, 2007). So the appropriate ratio of AA to AM was selected as 3:1.

Consequently, the optimal synthesis parameters for this cellulose-based superabsorbent were summarized as 70 °C reaction temperature, 0.1 g/g initiator to cellulose mass ratio, 5 g/g monomer (AA + AM) to cellulose mass ratio, and 3:1 AA/AM mass ratio. The tested grafting percentage and grafting efficiency under these
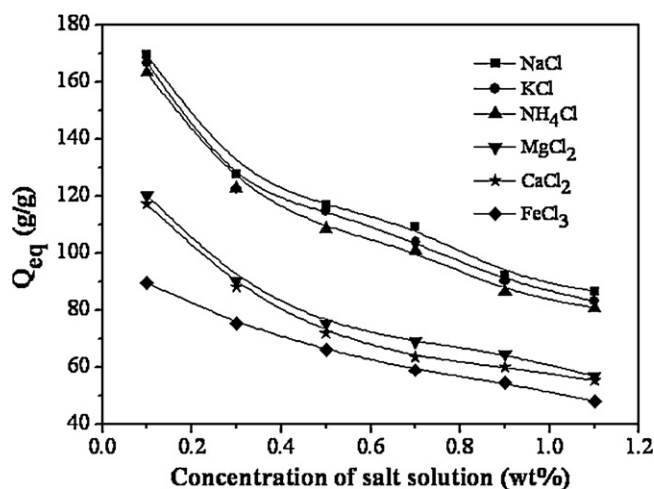
conditions reached 185% and 37.0%, respectively. The percentage of the unreacted monomer (AA + AM) was 6.35%.

3.2. Characterization of superabsorbent composites

3.2.1. FT-IR measurements

Fig. 2a–c shows the FT-IR spectra of cellulose, P(AA-co-AM), and cellulose-g-P(AA-co-AM) superabsorbent composite, respectively. The band at 3433.9 cm^{-1} (c) was attributed to the $-NH$ stretching vibration of the AM unit, which overlapped with the $-OH$ groups of the cellulose and acrylate units. The peaks at about 2930 cm^{-1} (a–c) were ascribed to the C–H absorption band that may present in the methyl and methylene groups of P(AA-co-AM) and cellulose. A band (1672.5 cm^{-1}) shift of the C=O stretching vibration, caused by the superposition of amide group absorption (1659 cm^{-1}) and C=O in COOH (1718 cm^{-1}), could be noticed in the spectra. The peak at 1558.4 cm^{-1} corresponded to the asymmetric $-COO^-$ stretching, which indicated the introduction of P(AA-co-AM) into the graft-copolymer composite. The absorption at 1057.4 cm^{-1} was due to the β-1,4-glycosidic bond, and the peak near 1450 cm^{-1} was assigned to the skeletal C=C stretching vibrations in the aromatic rings. Both these peaks manifested the existence of cellulose in the composite (Liang et al., 2009). In addition, the bands at 1457.4 and 1169.6 cm^{-1} were attributed to the $-C-O-$ and OH coupling interactions of the $-COOH$ and C–N stretching vibrations. All these FT-IR
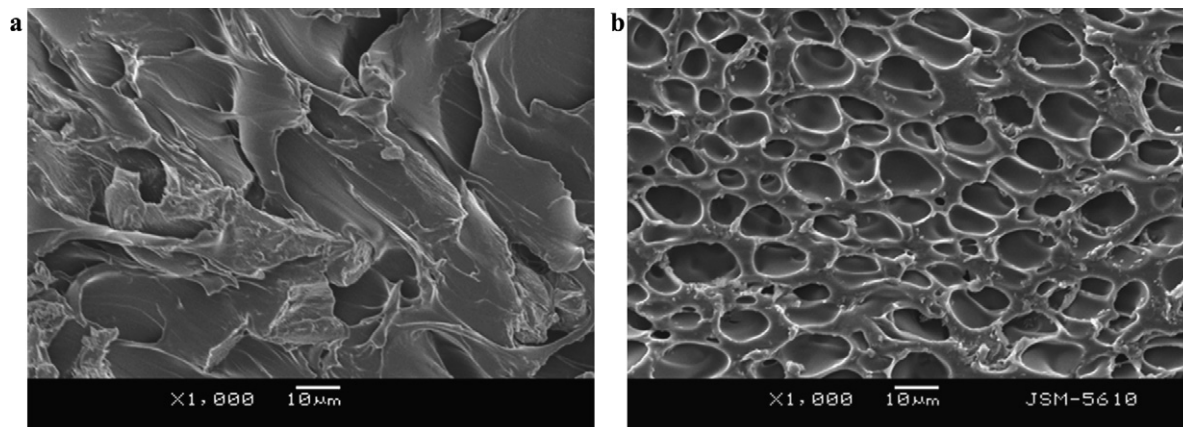

Fig. 3. SEM images of superabsorbent composites. (a) Surface structure. (b) Inner structure.
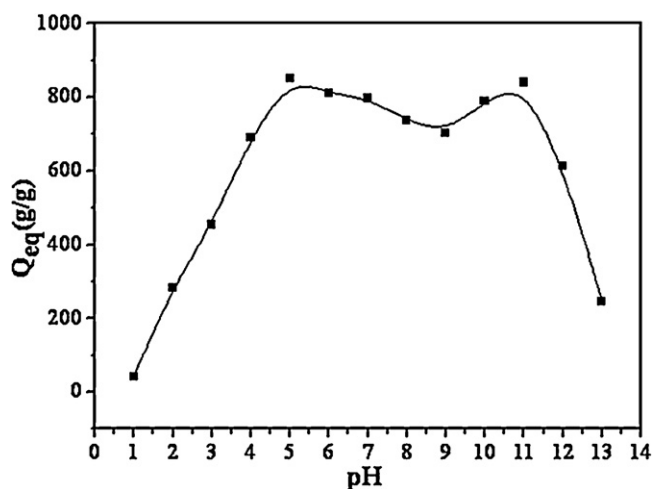
Fig. 5. Effect of pH on the water absorbency of the superabsorbent composites.

information verified that the P(AA-co-AM) chains were successfully grafted onto the macromolecular chains of cellulose.

3.2.2. Morphological analyses

Fig. 3 shows the SEM images of the superabsorbent composite. Based on Fig. 3a, the surface of the superabsorbent composite was coarse and fluffy, which structurally increased the surface area of the superabsorbent composite. Therefore, the composite was able to rapidly absorb water and form a swollen hydrogel when immersed in water. The inner portion of the sample had a porous structure. Many small, uniform, and interconnected pores were observed in Fig. 3b. These pores were the regions of water permeation, where water easily diffused into. The main reason for the formation of pores in the composite was due to the dehydration with ethanol and the drying process. There were also large pores, which caused the high water absorbency and swelling rate of the superabsorbent composite (Kabiri, Mahammad, & Zohuriaan, 2004).

3.2.3. Water absorbency in different salt solutions

The swelling behavior of a superabsorbent composite depends on the physical and chemical structure of the polymer, as well as on the reaction pH, ionic strength, and temperature. The swelling capacity of a superabsorbent in a salt solution has many important practical applications, especially in agriculture and horticulture. Fig. 4 shows the Q_{eq} of the samples in NaCl, KCl, NH$_4$Cl, MgCl$_2$, CaCl$_2$ and FeCl$_3$, examined in various concentrations. With the increased salt solution concentration, the swelling capacity considerably decreased. This result may be ascribed to the reduced osmotic pressure difference between the polymer network and the external solution. Q_{eq} of the samples decreased with the increased cationic charge (monovalent > divalent > trivalent) at the same salt solution concentration. The absorbency decreased with the increased metal cation activity (from Na$^+$ to Ca^{2+} and Fe^{3+}). This finding may be attributed to the increased cation charge and ionic strength of the medium. On the other hand, the increased degree of crosslinking caused the decreased swelling capacity. Furthermore, a small cationic radius of a same-valence monatomic cation corresponded to a high water absorption capacity (Na$^+$ > K$^+$ and Mg^{2+} > Ca^{2+}). Based on the points above, the swelling ratios in different chloride salt solutions were Na$^+$ > K$^+$ > NH$_4^+$ > Mg^{2+} > Ca^{2+} > Fe^{3+} (Zhao, Su, Fang, & Tan, 2005).

3.2.4. Effect of pH on water absorbency

The adsorption capability of the superabsorbent composites was investigated in various solutions with different pH values ranging from 1.0 to 13.0. The solution pH was adjusted by NaOH (pH 13.0), HCl (pH 1.0), and deionized water to reach the desired value. Fig. 5 shows that the water absorbency of cellulose-g-P(AA-co-AM) superabsorbent considerably increased with the increased pH from 1.0 to 5.0, and decreased within pH 5.0–9.0 and 11.0–13.0. Two sharp swelling behaviors of the superabsorbent were obtained at pH 5.0 and 11.0. The product was synthesized from cellulose-g-P(AA-co-AM), which was an ampholyte comprising both acidic (–COOH) and basic (–CONH$_2$) groups. The two sharp changes of water absorbent can be ascribed to the high repulsion of –NH$_3^+$ groups in acidic media, and –COO$^-$ groups in basic media. At very acidic conditions (pH < 4.0), most –COO$^-$ groups were protonated into –COOH groups. The charge-screening effect of the Cl$^-$ counterions in the swelling medium shielded the ammonium cations, and prevented an efficient repulsion (Flory, 1953; Pourjavadi, Barzegar, & Zeidabadi, 2007). The main anion–anion repulsive forces were eliminated, and as a result, equilibrium water absorbency remarkably decreased. At higher pH (pH > 4.0), the carboxylic acid groups were ionized, and the electrostatic repulsion between –COO$^-$ groups caused an enhanced swelling capacity. However, within pH 5.0–9.0, the majority of the basic and acidic groups were not ionized. Hence, the H-bonding between amine and the carboxylic or carboxamide groups led to a decreased water absorbency. Water absorption then increased with the further increased pH. This result could be associated with the conversion of carboxylic into carboxylate groups, and with the electrostatic repulsive force between the –COO$^-$ groups. In highly alkaline solutions (pH > 11.0), the screening effect of the Na$^+$ counterions in the swelling medium led to decreased water absorption (Lee & Wu, 1996).

3.2.5. Water retention in soil

The water retention ability of the superabsorbent composite in soil was the most worthwhile to investigate in terms of agricultural and horticultural applications. The superabsorbent composite can improve the soil quality, increase seedling survival rates and accelerate plant growth. Fig. 6a shows the water retention capacity of the soil, with or without the superabsorbent, for 30 d. Water retention of the soil with superabsorbent increased significantly, compared with that of the soil without superabsorbent. The water transpiration ratio of the soil without the superabsorbent was 90 wt% after 9 d. For the samples with 0.1, 0.3, 0.5 g superabsorbent per 100 g soil, the water transpiration ratios were 72.9 wt%, 54.1 wt%, and 27.7 wt% after 9 d, as well as 97.6 wt%, 74.2 wt%, and 59.0 wt% after 15 d, respectively. After 30 d, the water retention of the sample with 0.5 g superabsorbent per 100 g soil was still 8.1 wt%, indicating that the addition of superabsorbent caused effective water retention. Compared with similar products (Chen, Zhang, Luo, & Fang, 2004; Ni et al., 2010), the superabsorbent composite prepared in the present work had excellent water retention capacity in soil.

3.2.6. Water retention at various temperatures

Fig. 6b shows the water retention capacity of the superabsorbent composite at various temperatures. Water retention decreased significantly as the temperature and time increased. The samples retained 81.1 wt%, 78.9 wt%, 66.2 wt% and 24.6 wt% of absorbency after being heated for 12 h at 25, 30, 40 and 60 °C, respectively. These results are attributed to the interaction of H-bonding and Van der Waals force between water molecules and the superabsorbent in the swollen gel.
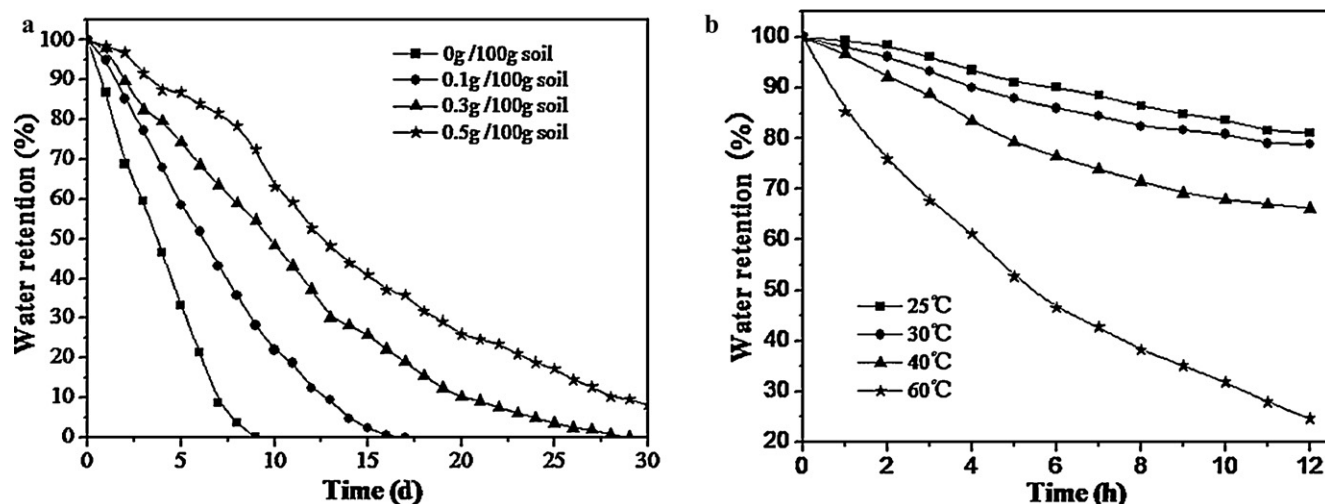
Fig. 6. Effects of superabsorbent mass in soil (a) and temperature (b) on the water retention ability of the superabsorbent composites.

4. Conclusions

A cellulose-based superabsorbent composite was synthesized by grafting AA and AM onto flax yarn waste cellulose. The optimal synthesis parameters were 70 °C reaction temperature, 0.1 g/g initiator to cellulose mass ratio, 5 g/g monomer (AA + AM) to cellulose mass ratio, and 3:1 AA/AM mass ratio. The grafting percentage and grafting efficiency were 185% and 37.0%. The maximum water absorbency was 875 g/g distilled water, 490 g/g natural rainwater, and 90 g/g 0.9 wt% aqueous NaCl solution. The prepared novel superabsorbent composite had excellent water absorbency and retention capability, and hence, had potential applications in modern agriculture and horticulture.

Acknowledgment

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