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Synthesis and urea sustained-release behavior of an eco-friendly superabsorbent based on flax yarn wastes

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ABSTRACT

In order to develop an eco-friendly superabsorbent composite, flax yarn waste (FYW) was used as raw material to synthesize a novel flax yarn waste-g-poly(acrylic acid-co-acrylamide) (FYW/PAA) superabsorbent composite. Acrylic acid (AA) and acrylamide (AM) were grafted onto the pretreated flax yarn waste (PFYW) by free-radical graft copolymerization in homogeneous aqueous solution. The properties and synthesis conditions of the FYW/PAA superabsorbent composite were investigated. As a result, the prepared FYW/PAA attained the best water absorbency of 875 g/g in distilled water, 490 g/g in rainwater and 90 g/g in 0.9 wt% NaCl solution. The urea loading percentage of FYW/PAA could be modulated by the concentration of urea. The release of urea from FYW/PAA in water showed a typical three-stages sustained release behavior. Meanwhile, a weight residue of 53.6 wt% was attained after being buried in soil for 90 d.

1. Introduction

The growth of plants and their quality are mainly affected by the quantity of water and fertilizer. So it is very important to improve the utilization of water resources and fertilizer nutrients (Wu, Hao, & Guo, 2008). One method of reducing water and fertilizer nutrient loss is the adoption of superabsorbent composite. Recently, researches on the use of superabsorbent composite as water and fertilizer managing materials have attracted great attention. Encouraging results have been observed as they can reduce irrigation water consumption, improve fertilizer retention in soil, lower the death rate of plants, and promote plants' growth (Lokhande & Varadara, 1992; Nge, Hori, Takemura, & Ono, 2004).

Superabsorbent composite is three-dimensionally crosslinked hydrophilic resin gel with excellent swelling and retaining capabilities. It could absorb liquids tens to thousands times its own weight and retain the liquids even under some pressure (Ma et al., 2011). Given all these advantages, superabsorbent composite has exhibited potential for application in many fields, such as hygiene, medicine, wastewater treatment, agriculture and horticulture (Chen & Tan, 2006; Kamat & Malkani, 2003; Pourjavadi, Farhadpour, & Seidi, 2008; Sadeghi & Hosseinzadeh, 2008; Yi & Zhang, 2008).

Many kinds of materials have been used for preparing superabsorbent composite. However, most of the traditional water absorbing materials are acrylic acid- or acrylamide-based products which have poor degradability. About 90% of superabsorbent composites are used in disposable products, landfilled or incinerated after use (Kiatkamjornwong, Mongkolsawat, & Sonsuk, 2002). This will cause serious environmental problems (Zhang, Li, & Wang, 2006; Zhang, Wang, & Wang, 2007). Meanwhile undesirable water-keeping capacity and high cost largely limit its practical application.

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). Recently, a series of new superabsorbent composites characterized by eco-friendliness and biodegradability were made from some natural materials, such as starch, cellulose and chitosan (Wu, Liu, & Liang, 2008). However, there are few reports which focus on the application of waste cellulose materials in this field.

Till now, most of the waste cellulose materials from textile industry, such as cotton linter, cotton yarn waste and flax yarn waste, are still burned or landfilled, causing both resource waste and environmental pollution (Aloulou, Boufi, & Labidi, 2006). Reutilization of these waste materials could not only reduce the consumption of cotton or flax cellulose, but also decrease the treatment of industrial waste. The major component (70%) of flax yarn waste is cellulose, which has been introduced as a basic skeleton of

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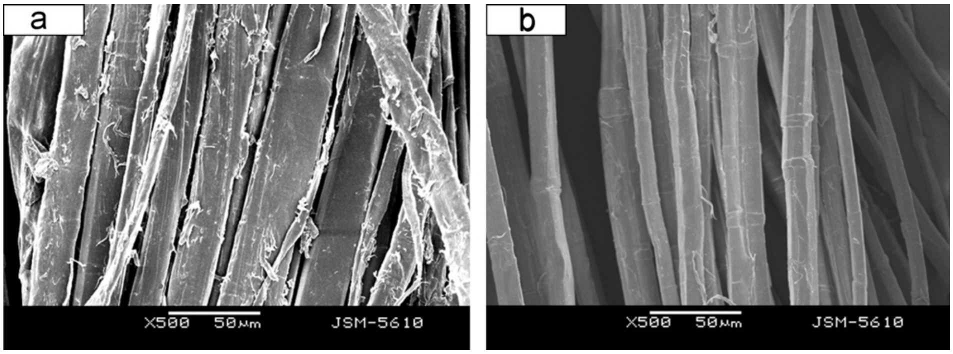

Fig. 1. Micro-morphologies of the used flax yarn waste. (a) Before the pretreatment. (b) After the pretreatment.

superabsorbent composite in previous studies (Jane, Richard, John, Spear, & Metlen, 2007; Klemm, Heublein, Fink, & Bohn, 2005; Nogi & Yano, 2008). So in a continuation of the research in the field of organic–inorganic superabsorbent composite (Wu, Zhang, Liu, & Yao, 2012), our attention was directed towards providing a novel method for the management of flax yarn waste, i.e. the incorporation of it into superabsorbent composite.

In this study, a new, eco-friendly and flax cellulose-based superabsorbent composite was synthesized. It was based on the pretreated flax yarn waste (PFYW), acrylic acid (AA) and acrylic amide (AM), with ammonium persulfate (APS) as initiator and N,N′-methylenebisacrylamide (MBA) as cross-linker. The properties and synthesis conditions of the flax cellulose-g-P(AA-co-AM) (FYW/PAA) superabsorbent composite were investigated.

In this study, the synthetic process of the superabsorbent composite is eco-friendly mainly. All the raw materials added are transformed into product. And no waste or pollutant is discharged to the environment. The product does not produce secondary pollution in use. The basic skeleton of the synthetic product is natural cellulose which can be used as organic matter of soil. Copolymers of AA and AM have been widely used for soil improvement. They are prone to decompose in soil by natural method. For these reasons, the new superabsorbent composite is biodegradable in soil under normal conditions.

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 Biotechology Co., Ltd., China) was used as received. APS (analytical grade; TianjinYongda 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. Preparation of FYW/PAA superabsorbent composite

2.2.1. Pretreatment of flax yarn waste

The flax yarn waste was cut into fragments with the length of 5–10 mm, washed and dried in a 105 °C drying oven for 12 h. Then 20 g fragments was weighed and dipped in a 1000 mL beaker with 1 wt% NaOH at a mass ratio of 1:30, heated at 100 °C for 1 h, and the mixture was filtrated and washed. After that, the filtrated residue was transferred into a new beaker with 1.5 wt% NaOH, 0.1 wt% Na_2SiO_3 and 0.2 wt% $Na_5P_3O_{10}$ at the same mass ratio and heated at 100 °C for 2 h. Finally, the mixture was neutralized by 0.2 wt% H_2SO_4 for 5 min and filtrated, washed by distilled water, and then dried at 105 °C for 24 h to obtain the PFYW (Li et al., 2009). The purity of flax cellulose in PFYW was determined by measuring α-cellulose content, which was 97.6%. Fig. 1 presents the micro-morphologies of the used flax yarn waste before and after the pretreatment.

2.2.2. Grafting of PFYW with AA + AM in homogeneous condition

Fig. 2 shows the synthetic principle of the target FYW/PAA superabsorbent composite product. The dissolution process of cellulose was carried out according to literature (Cai et al., 2008). 1 g PFYW (cellulose particles could pass through 200 mesh screen) was added into 25 g 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. Then 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 70 °C. Oxygen-free nitrogen gas was bubbled into the solution for 30 min before adding the monomer. A distilled water solution containing a certain amount of APS was added. After 15 min of stirring, AA + AM with a predetermined weight ratio (3:1) (Wu et al., 2012) containing a certain amount of MBA and 5 mL distilled water were added to the reaction mixture. The water bath was kept at a certain reaction temperature and for the prescriptive reaction time to complete the polymerization process. The obtained gel was washed with distilled water several times, and poured into excess non-solvent ethanol 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)) (PAA) was prepared in the same procedure as mentioned above, but without the addition of PFYW.

2.3. Water absorbency and retention measurement

2.3.1. Water absorbency measurement

The tea bag (100 mesh nylon screen) containing the accurately weighed FYW/PAA superabsorbent composite powder (0.1 ± 0.0001 g) was immersed in 500 mL of distilled water, saline solution and rainwater at room temperature respectively 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:

$$Q_{eq} = \frac{m_2 - m_1}{m_1} \qquad (1)$$

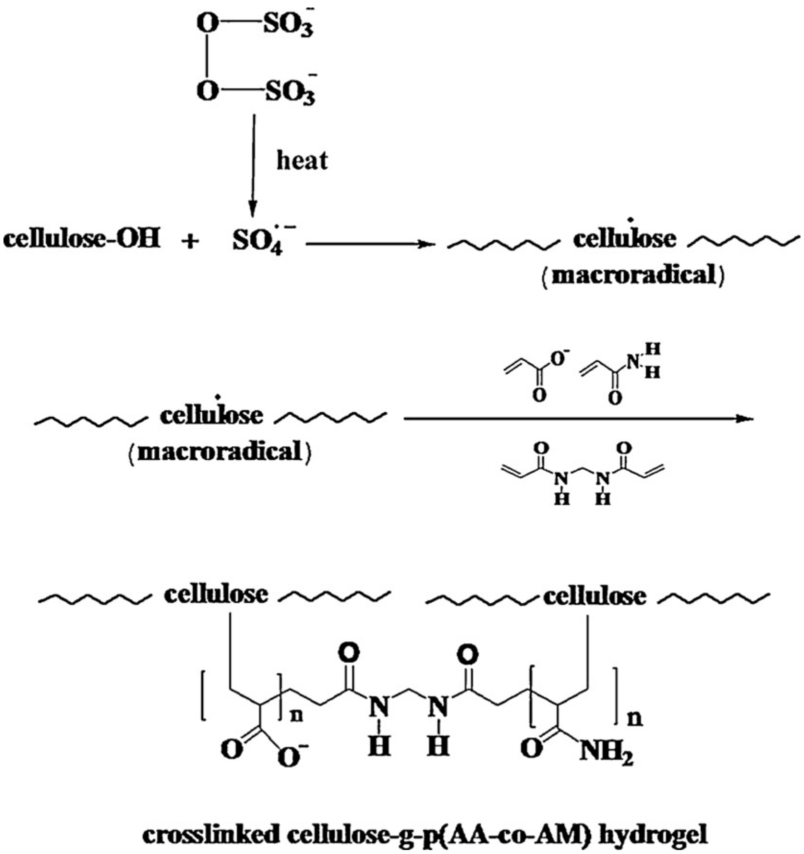

Fig. 2. Synthetic principle of the FYW/PAA superabsorbent composite.

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).

2.3.2. Water retention at various pressures

The sealing tea bag (100 mesh nylon screen) containing the equilibrium swelling FYW/PAA superabsorbent composite was put on a metal mesh. A 100 mm × 100 mm polypropylene plate was press on the superabsorbent with the designed pressures at 25 °C for 30 min. The water retention at various pressures (R_L) was calculated by the following equation:

$$R_L\% = \frac{m_1 - m_2}{m_0} \times 100 \tag{2}$$

where m_0 and m_1 are the weights of the dry FYW/PAA superabsorbent composite and the swollen FYW/PAA superabsorbent composite with tea bag (g), respectively, and m_2 is the weight of the tea bag (g).

2.4. Loading and release of urea

2.4.1. Loading of urea

The loading of urea was carried out by immersing the preweighed dry gel into aqueous solution of urea for 12 h. Thereafter, the swollen gel was dried at 40 °C for 3 d. The loading percentage was calculated by Eq. (3):

$$\text{Loading} \quad \% = \frac{W_1 - W_0}{W_1} \times 100 \tag{3}$$

where W_0 and W_1 are the weights of unloaded and loaded dry gels (g).

2.4.2. Release of urea from FYW/PAA in water

0.5 g urea-loaded dry gel was placed in beakers containing 1000 mL distilled water (release medium) without stirring. At various intervals, 2 mL solution was drawn out from the medium to detect the urea release quantity, which was determined by the ultraviolet spectrophotometer (Watt & Chrisp, 1954).

2.5. Natural soil burial test

The natural soil burial test for polymer degradation was conducted for 90 d. The FYW/PAA superabsorbent composite, shaped to circular disks with a diameter of 10 mm and thickness of 1 mm, was wrapped with 325# stainless steel wire mesh in order to minimize the loss of the polymer fragments during the burial process. A string was tied to the wire mesh, with a part of which left unburied above the soil surface. Each polymer sample was buried approximately 10 cm beneath the surface of the soil sample. The buried specimens were dug out from the soil at 10 d intervals, washed with distilled water and dried in an oven at 60 °C. After drying, the specimens were put into a desiccator to cool before their dry weights were determined, preventing the polymer from reabsorbing moisture from the atmosphere during the cooling process. The control sample (PAA) without the addition of PFYW was treated in the same procedure. The degradation rate was calculated by the following equation:

$$\text{Degradation} \quad \% = \frac{W_1 - W_2}{W_1} \times 100 \tag{4}$$

where W_1 and W_2 are the weights of dry gels before and after the soil burial (g).

Table 1
Factors and levels of the orthogonal experiment (A: APS to cellulose mass ratio; B: AA + AM (AA/AM = 3:1) to cellulose mass ratio; C: MBA to cellulose mass ratio; D: reaction temperature; E: reaction time).

Factor level	A (g/g)	B (g/g)	C (g/g)	D (°C)	E (h)
1	0.05	4	0.02	50	1.5
2	0.10	6	0.03	60	2.0
3	0.15	8	0.04	70	2.5
4	0.20	10	0.05	80	3.0

Table 2
The orthogonal $L_{16}(4)^5$ experiment of APS, AA + AM, MBA, reaction temperature and reaction time (A: APS to cellulose mass ratio; B: AA + AM (AA/AM = 3:1) to cellulose mass ratio; C: MBA to cellulose mass ratio; D: reaction temperature; E: reaction time; Q_d: the water absorbency in the distilled water).

Sample no.	A (g/g)	B (g/g)	C (g/g)	D (°C)	E (h)	Q_d (g/g)
1	1 (0.05)	1 (4)	1 (0.02)	1 (50)	1 (1.5)	785.3
2	1 (0.05)	2 (6)	2 (0.03)	2 (60)	2 (2.0)	847.6
3	1 (0.05)	3 (8)	3 (0.04)	3 (70)	3 (2.5)	774.5
4	1 (0.05)	4 (10)	4 (0.05)	4 (80)	4 (3.0)	821.2
5	2 (0.10)	1 (4)	2 (0.03)	3 (70)	4 (3.0)	873.9
6	2 (0.10)	2 (6)	1 (0.02)	4 (80)	3 (2.5)	842.8
7	2 (0.10)	3 (8)	4 (0.05)	1 (50)	2 (2.0)	610.3
8	2 (0.10)	4 (10)	3 (0.04)	2 (60)	1 (1.5)	738.8
9	3 (0.15)	1 (4)	3 (0.04)	4 (80)	2 (2.0)	827.6
10	3 (0.15)	2 (6)	4 (0.05)	3 (70)	1 (1.5)	738.0
11	3 (0.15)	3 (8)	1 (0.02)	2 (60)	4 (3.0)	799.6
12	3 (0.15)	4 (10)	2 (0.03)	1 (50)	3 (2.5)	675.9
13	4 (0.20)	1 (4)	4 (0.05)	2 (60)	3 (2.5)	762.6
14	4 (0.20)	2 (6)	3 (0.04)	1 (50)	4 (3.0)	706.9
15	4 (0.20)	3 (8)	2 (0.03)	4 (80)	1 (1.5)	778.5
16	4 (0.20)	4 (10)	1 (0.02)	3 (70)	2 (2.0)	724.6

3. Results and discussion

3.1. Optimization of FYW/PAA synthesis condition

Superabsorbent composites prepared under different synthesis conditions have different water absorbencies due to the different structures. In order to find the best synthesis conditions of FYW/PAA, an orthogonal experiment with five factors and four levels was conducted. "A" stands for APS to cellulose mass ratio, which has four levels of 0.05, 0.1, 0.15 and 0.2 g/g. "B", "C", "D" and "E" stand for AA + AM (AA/AM = 3:1) to cellulose mass ratio, MBA to cellulose mass ratio, reaction temperature and reaction time, respectively. According to the orthogonal array of $L_{16}(4)^5$ presented in Table 1, the following experiments were performed (Table 2) and the result of range analysis was shown in Table 3. The result of the range analysis indicated that the influencing order of each factor on the water absorbency was D > B > A > C > E in distilled water. Reaction temperature is the most important factor, AA + AM followed, and then APS, MBA and reaction time are the last. In the synthetic reaction, hydrophilic groups such as —COO⁻ and —COOH are grafted in the cellulose skeleton from AA and AM, which can greatly improve the water absorbency of FYW/PAA. This factor has a larger effect than other factors. According to the results of the orthogonal experiment, the optimal combination $A_2B_1C_2D_3E_4$ was obtained, which corresponded to the optimal synthesis conditions: 0.1 g/g APS to cellulose mass ratio, 4 g/g AA + AM (AA/AM = 3:1) to cellulose mass ratio, 0.03 g/g MBA to cellulose mass ratio, 70 °C reaction temperature and 3 h reaction time. The prepared FYW/PAA superabsorbent composite here presented a maximum water absorbency of 875 g/g distilled water, 490 g/g rainwater and 90 g/g 0.9 wt% aqueous NaCl solution, respectively. The excellent water absorbency capability of the prepared FYW/PAA preliminarily exhibits its potential applications in modern agriculture and horticulture.

Table 3
Analysis of the orthogonal $L_{16}(4)^5$ experiment (A: APS to cellulose mass ratio; B: AA + AM (AA/AM = 3:1) to cellulose mass ratio; C: MBA to cellulose mass ratio; D: reaction temperature; E: reaction time).

	A	B	C	D	E
K_1 [a]	807.2	812.4	778.1	694.6	760.2
K_2	766.5	783.8	794.0	787.2	751.1
K_3	759.0	740.1	760.4	777.8	764.0
K_4	743.2	740.1	733.0	817.6	800.4
R [b]	64.0	72.3	61.0	123.0	48.9
Influencing order	D>B>A>C>E				
Optimal combination	A_2	B_1	C_2	D_3	E_4
	$A_2B_1C_2D_3E_4$				

[a] $K_1 = (\sum$ the water absorbency in the distilled water of single-factor)/4.
[b] $R = \max K_1 - \min K_1$.

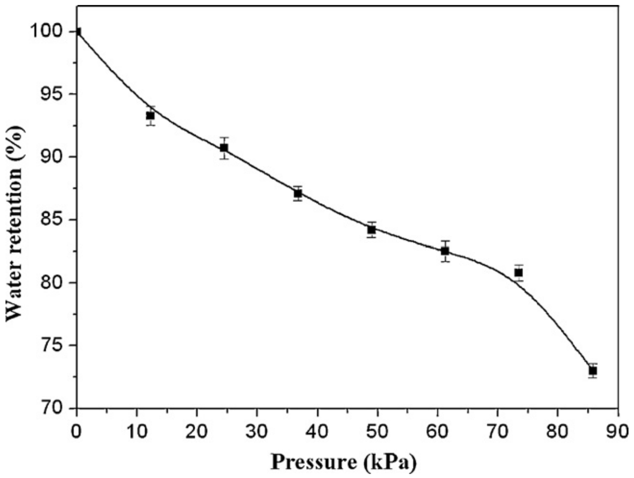

Fig. 3. Water retention capacity of the FYW/PAA superabsorbent composite at various pressures.

3.2. Water retention of FYW/PAA

The water retention ability of the FYW/PAA superabsorbent composite in soil was the most worthwhile to investigate in terms of agricultural and horticultural applications. The FYW/PAA can improve the soil quality, increase seedling survival rates and accelerate plant growth. In our previous work (Wu et al., 2012), we found the water retention of the soil with superabsorbent increased significantly, compared with that of the soil without superabsorbent. Even 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 the FYW/PAA superabsorbent caused effective water retention.

The relationship between pressure and water retention of the prepared FYW/PAA is also shown in Fig. 3. With increased pressure, the absorbed water in the FYW/PAA was gradually released. The water retention of FYW/PAA was still 82.9 wt% at 60 kPa, but decreased significantly after 70 kPa. The outstanding water retention of FYW/PAA at certain pressures was due to the hydroxyl groups on the molecular chains of the superabsorbent, which could bound water molecules within the molecular network of FYW/PAA (Riccardo, 1994). The water retention capacity of the FYW/PAA at various temperatures was also reported by us previously that the samples retained 81.1 wt%, 78.9 wt% and 24.6 wt% of absorbency after being heated for 12 h at 25, 30, 40 and 60 °C, respectively (Wu et al., 2012). Water retention decreased significantly as the temperature and time increased.

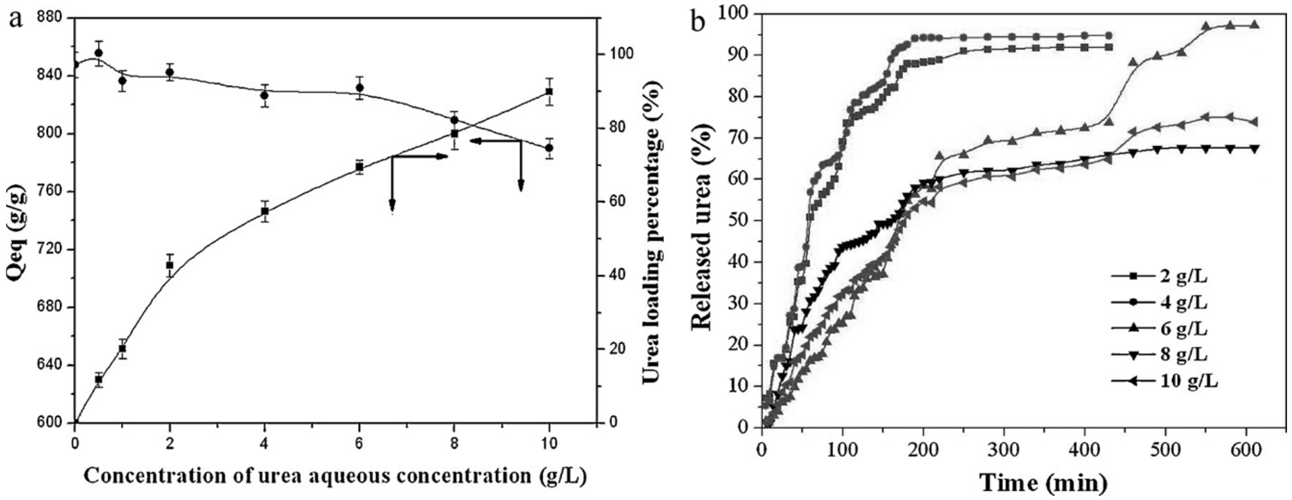

Fig. 4. Urea loading and sustained-release behaviors of the FYW/PAA superabsorbent composite. (a) Equilibrium swelling degree and urea loading percentage of the FYW/PAA swollen in aqueous solutions of urea with different concentrations. (b) Release profiles of urea from the loaded FYW/PAA with different urea loading percentage in distilled water (swollen in aqueous solutions of urea with different concentrations).

3.3. Loading and release of urea

3.3.1. Swelling of FYW/PAA in aqueous solutions of urea

To load urea, the FYW/PAA superabsorbent composite was swollen in aqueous solutions of urea with different concentrations. The equilibrium swelling degrees were all about 800 g/g but the urea loading percentage increased from 0 to 89.9 wt% as presented in Fig. 4a. The results showed that the Q_{eq} of FYW/PAA in aqueous solutions of urea with different concentrations were almost equal. But urea loading percentage was strongly affected by urea concentration. The reason may be that urea is neutral molecule, which could not affect the electrostatic repulsion force of $-COO^-$ on polymer chain. Moreover, urea molecule has hydrophilic sites, such as $-NH_2$. The addition of urea in water would not change the polymer–solvent interaction. Therefore, aqueous solutions of urea with different concentrations can hardly change the swelling

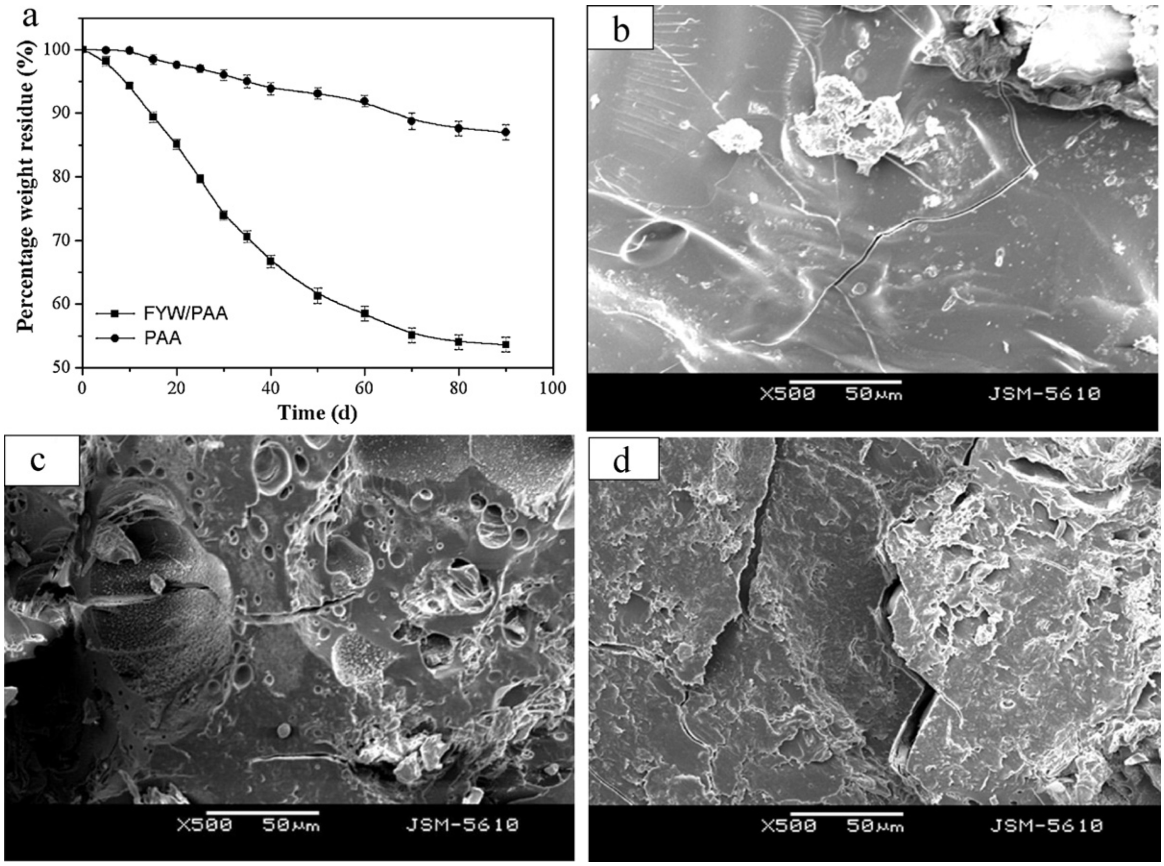

Fig. 5. Weight residue percentages and surface morphologies of the FYW/PAA superabsorbent composite with different weight loss ((a) weight residue percentages of FYW/PAA and PAA; (b–d) surface morphologies of FYW/PAA buried in soil after 10, 50 and 90 d, respectively).

process of FYW/PAA composite. But when FYW/PAA swelled in aqueous solutions of urea with higher concentration, more urea molecules entered into the polymer network. After drying, these urea molecules were left in the three-dimensional network, then the urea loading percentage was high and vice versa. So the urea loading percentage of FYW/PAA could be modulated by the concentration of aqueous solutions of urea (Liang, Yuan, Xi, & Zhou, 2009).

3.3.2. Release of urea from loaded FYW/PAA in water

Fig. 4b depicts the release profiles of urea from loaded FYW/PAA with different urea loading percentage in distilled water (swollen in aqueous solutions of urea with different concentrations). As expected, the release of urea showed a typical three-stages sustained release behavior (especially in samples 6, 8 and 10 g/L): an initially high release rate (0–250 min) followed by a relative release equilibrium (250–500 min), and then a rapidly increasing release rate again (after 500 min). Therefore, for the loaded FYW/PAA, the release behavior of urea could be described by the following steps: (1) FYW/PAA was swollen by the water and then transformed to hydrogel, and urea rapidly dissolved; (2) when the urea passed through aqueous solution, there are multiple times of absorbing and deabsorbing processes between urea molecule and distilled water, which contribute to slower the urea release. The urea release and absorption of FYW/PAA reached balance, and the release rate remained stable; (3) the phenomenon of FYW/PAA disintegration was observed in this stage. The urea was released rapidly again with the disintegration of gel (Wu, Hao et al. 2008; Wu, Liu et al., 2008).

3.4. Biodegradability of FYW/PAA

Soil burial test had been established and standardized since last decade to study the behavior of biodegradable polymers when exposed to soil sample (Rizzarelli, Puglisi, & Montaudo, 2004). Fig. 5 compares the weight residue percentages and surface morphologies of the FYW/PAA superabsorbent composite with different weight loss. The weight residue of FYW/PAA buried in soil after 10, 50 and 90 d were 94.3 wt%, 61.3 wt% and 53.6 wt%, respectively. While the corresponding values of PAA were only 99.8 wt%, 93.1 wt% and 87.0 wt%. Based on the flax cellulose skeleton material, the FYW/PAA superabsorbent composite presented an excellent biodegradability compared with PAA. Fig. 5b–d shows the different surface morphologies of FYW/PAA buried in soil after 10, 50 and 90 d, respectively. The surfaces of the soil buried samples were not smooth and dense. And with increased burial time, their surfaces appeared more and more dots and cracks. This phenomenon may be explained by the fracture and degradation of cellulose macromolecular chains in the FYW/PAA superabsorbent composite, which were adhered and catalyzed by microorganisms and enzymes (Kelne & Schacht, 2005).

4. Conclusions

An eco-friendly superabsorbent composite was prepared by free-radical graft copolymerization of AA, AM and PFYW. The properties and synthesis conditions of the FYW/PAA superabsorbent composite were studied. The optimal synthesis conditions were 0.1 g/g APS to cellulose mass ratio, 4 g/g AA + AM (AA/AM = 3:1) to cellulose mass ratio, 0.03 g/g MBA to cellulose mass ratio, 70 °C reaction temperature and 3 h reaction time. The product attained the best water absorbency of 875 g/g in distilled water, 490 g/g in rainwater and 90 g/g in 0.9 wt% NaCl solution. Concentration of aqueous solution of urea had no obvious effect on the swelling behavior of FYW/PAA. The urea loading percentage of FYW/PAA could be modulated by the concentration of urea. The release of urea from FYW/PAA in water showed a typical three-stages sustained release behavior. The weight residue of FYW/PAA buried in soil after 90 d was 53.6 wt%, which presented an excellent biodegradability.

This paper showed a new method in utilizing textile cellulose wastes such as cotton linter, cotton yarn waste and flax yarn waste, in the production of superabsorbent material. The introduction of them into superabsorbent could significantly reduce the production cost and make the technique quite environmental friendly, which is crucial to superabsorbent for agricultural and horticultural use.

Acknowledgment

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