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Preparation and properties of organic–inorganic composite superabsorbent based on xanthan gum and loess

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ABSTRACT

A new, low-cost, and eco-friendly organic–inorganic composite superabsorbent was successfully synthesized in aqueous solution by polymerization xanthan gum (XG), neutralized acrylic acid (AA) and loess using ammonium persulfate (APS) as initiator and N,N-methylenebisacrylamide (MBA) as crosslinker. Structure and morphological characterizations of the composite superabsorbent were investigated by Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The loess content, pH values, surfactants, salts and temperature which could affect the swelling and water-retention capabilities of the composite superabsorbent were investigated. The composite superabsorbent exhibits excellent water absorbency (610 g/g in distilled water), pH-stability (pH 5–10), and higher swelling capacity in anionic surfactant solution; on the other hand, the composite superabsorbent can be used for removing multivalent metal ions.

1. Introduction

Superabsorbent polymers are high-performance water absorbent and retention materials with three-dimensional network structures. They can absorb water and other liquids from tens to thousands times of their own weight in a relatively short time, and 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). Because of their excellent characteristics, superabsorbents are widely used in many fields such as agriculture (Puoci, Iemma, & Spizzirri, 2008), hygienic products (Kosemund, Schlatter, Ochsenhirt, Krause, Marsman, & Erasala, 2009), wastewater treatment (Kasgoz & Durmus, 2008) and drug-delivery (Wang, Zhang, & Wang, 2009a, 2009b). 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; Wu, Liu, & Liang, 2008; Wu, Zhang, Liu, & Yao, 2012). However, most of the superabsorbents are based on fully petroleum-based polymers, which are costly, poorly degradable, and environmentally unfriendly (Kiatkamjornwong, Mongkolsawat, & Sonsuk, 2002). Given the gradual depletion of petroleum resources and the growing environmental pollution crisis from polymer syntheses, the utilization of low-cost and biodegradable resources for preparing superabsorbents, or the design and synthesis of organic–inorganic composite superabsorbents based on natural materials has become the focus of current studies (Dong et al., 2008).

In recent years, many kinds of materials have been used for preparing superabsorbents, among them the naturally available resources such as polysaccharides (starch, cellulose, chitosan, alginate, gelatin etc.) and inorganic clay minerals (palygorskite) or their modified products have shown particular advantages and drawn considerable attention (Bao, Ma, & Li, 2011; Wang, Zhang, & Wang, 2009a, 2009b; Yadav & Rhee, 2012). The use of polysaccharides and inorganic clay minerals or their modified products can not only improve the performance of superabsorbents, but also reduce the superabsorbents cost. These eco-friendly superabsorbents show potentials as substitutes for existing petroleum-based superabsorbent materials.

Xanthan gum is a natural polysaccharide with branched chains and acidic characteristic produced predominantly by *Xanthomonas campestris* in aerobic conditions from sugar cane, corn or their derivatives (Dumitriu, 2005). It consists of D-glucosyl, D-mannosyl, and D-glucuronyl acid residues in a 2:2:1 molar ratio and variable proportions of O-acetyl and pyruvyl residues. Trisaccharide sidechains are composed of mannose (β-1,4) glucuronic acid

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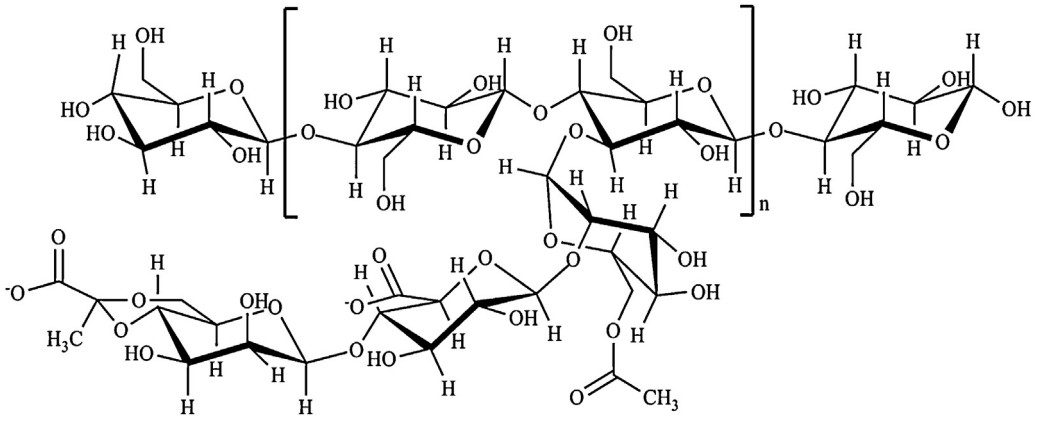


Scheme 1. Structure of natural xanthan gum.

(β-1,2) mannose attached to alternate glucose residues in the backbone by α-1,3 linkages (Bueno, Bentini, Catalani, & Petri, 2012) (Scheme 1).

Loess clay is a type of hydrous magnesium aluminum silicate with reactive hydroxyl groups on its surface. Because of its hydrophilic property, abundant reserves and extremely low prices, so loess is an ideal inorganic component for added superabsorbent network to improve the swelling properties. By the incorporation of loess into polysaccharide based superabsorbent, the stiffness loess could weak the strong hydrogen-bonding interaction and reduces some physical intertwining between the polymeric chains.

In the present work, the eco-friendly materials such as loess, xanthan gum and acrylic acid were used as raw materials to synthesis low-cost, eco-friendly organic–inorganic composite superabsorbent xanthan gum-g-poly(acrylic acid)/loess (XG-g-PAA/loess) in aqueous solution. The structure and morphology were investigated. Meanwhile, the factors such as the amount of loess, pH value, surfactants, salts and temperature that could affect the characterization of synthesized superabsorbent were also discussed (Scheme 2).

2. Experimental

2.1. Materials

Loess was collected from WuQuan mountain in Lanzhou city, Gansu province, China, Xanthan gum (XG, analytical grade) from Tianjin Guangfu Institute of Fine Chemicals, China, acrylic acid (AA, analytical grade) was from Tianjin Kaixin Chemical Industrial Co., China, ammonium persulfate (APS, analytical grade) was from Tianjin Damao Chemical Reagent Factory, China, N,N-methylenebisacrylamide (MBA, chemically pure) was from Sinopharm Chemical Reagent Co., China, sodium dodecyl benzene sulfonate (SDBS, analytical grade) was from Sinopharm Chemical Reagent Co., China, and dodecyl trimethyl ammonium bromide (DTAB, analytical grade) was from Shanghai Zhongqin Chemical Reagent Co., China. All other reagents used were of analytical grade and all solutions were prepared with distilled water.

2.2. Loess purification

Natural loess was found to contain small amounts of mineral impurities and the loess was purified by suspension method. A purification process that was similar to Xue, Reinholdt, and Pinnavaia (2006) was carried out by preparing a 10 wt% aqueous suspension of the loess and allowing the more dense impurities quartz and carbonates to sediment out. After a certain sedimentation time, the supernatant suspension that is pure loess was decanted off, and then the pure loess was collected by filtrated, dried, grinded and sieved. After that, the pure loess with particle sizes about 0.074 mm (200 mesh) was obtained.

2.3. Synthesis of XG-g-PAA/loess superabsorbent

The XG-g-PAA/loess superabsorbent was synthesized as follows: firstly, XG (1.20 g) was dispersed in 40 mL NaOH solution (0.067 mol/L) in a 250 mL four-necked reaction flask with a mechanical stirrer, a reflux condenser, a constant pressure dropping funnel, and a nitrogen line. The obtained solution was heated to 70 °C in an oil bath for 1 h to form colloidal slurry. Then, 4 mL APS (0.10 g) aqueous solution was added to the slurry and continuously stirred at 70 °C for 20 min to generate radicals. Secondly, 7.20 g of acrylic acid was added into 8.5 mL NaOH solution (8 mol/L) to obtain neutralized acrylic acid with neutralization degree of 70%, then 21.60 mg crosslinker MBA and certain loess were added into the neutralized acrylic acid under magnetic stirring to form a uniform mixture solution. After the solution was cooled to 50 °C, it was added into the reaction flask. The reaction temperature was slowly heated to 70 °C and kept for 3 h to finish polymerization. A nitrogen atmosphere was maintained throughout the reaction period. The obtained gel products were dried to constant weight in an oven at 70 °C, the dried gels were ground and all the samples used for tests had a particle size about 50 mesh. This was similar to Bao et al. (2011) and Shi, Wang, Kang, and Wang (2012).

For comparison purpose, XG-g-PAA composite was synthesized under same conditions.

2.4. Method of characterization

2.4.1. FTIR

The samples were vacuum-dried and dispersed in KBr. The spectra of the samples were taken at 4000–400 cm^{-1} wavelength with FTIR-FTS3000.

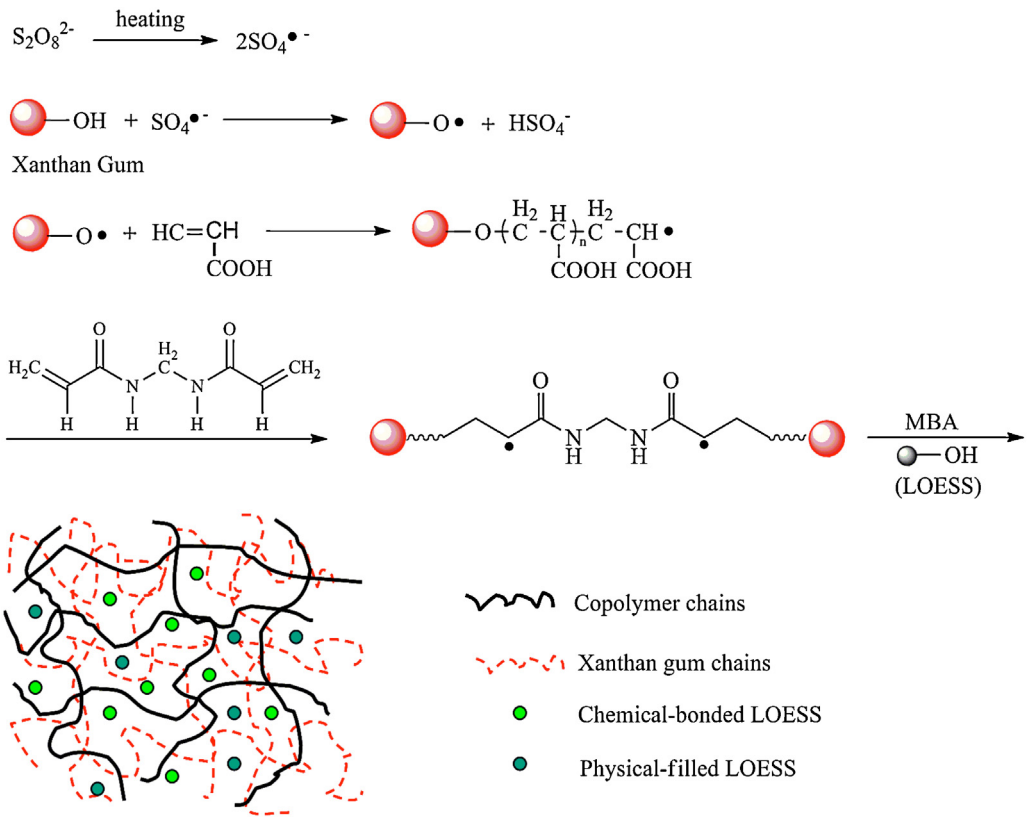
2.4.2. SEM

The morphologies of the superabsorbent composites were examined using a JSM-5600LV SEM instrument (JEOL) after coating the sample with gold film.

2.5. Measurement of properties

2.5.1. Measurement of equilibrium swelling ratio

Swelling properties of the composite superabsorbent in various media were measured at room temperature which was similar to Zheng and Wang (2008). 0.05 g of dry samples was immersed in 300 mL swollen media for 2 h to reach swelling equilibrium.



Scheme 2. Proposed reaction mechanism for synthesis of XG-g-PAA/loess superabsorbent composites.

The swollen gels were filtered using a 100 mesh sieve, and then drained on the sieve until no free water remained. After weighing the swollen samples, the equilibrium water absorbency of the superabsorbent can be calculated by using the following equation:

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

where m_2 and m_1 are the weight of dry sample and swollen sample, respectively. Q_{eq} is the equilibrium water absorbency calculated as grams of water per gram of the sample, which are the averages of three measurements.

2.5.2. Measurement of water retention capability

Water retention capacity of the composite superabsorbents was measured according to the method of Bhattacharya et al. (2012). First, 50 g of fully swollen superabsorbent samples in distilled water were tiled in the bottom of the 250 mL beaker, and allowed to dry in an oven maintained 25, 45 and 60 °C, respectively. The water retention study was measured as a function of time by gravimetry. The percentage water retention (W_r) of the superabsorbent was calculated as follows:

$$W_r\ (\%) = \frac{W_i}{W_o} \times 100\% \quad (2)$$

where W_o is the initial weight of the fully swollen superabsorbent samples in distilled water and W_i is its weight after loss of water at each time interval.

2.5.3. Measurement of swelling at various pH values

To investigate the swelling behaviors of the composite superabsorbents at various pHs, individual solutions with pH 2.5–13 were prepared by dilution of NaOH (pH = 13.0) or HCl (pH = 1.0) solutions, respectively. The pH values of all the solutions were precisely checked by pH meter. Then, 0.05 g of the dried composite superabsorbent was used for the steady state swelling measurements according to Eq. (1).

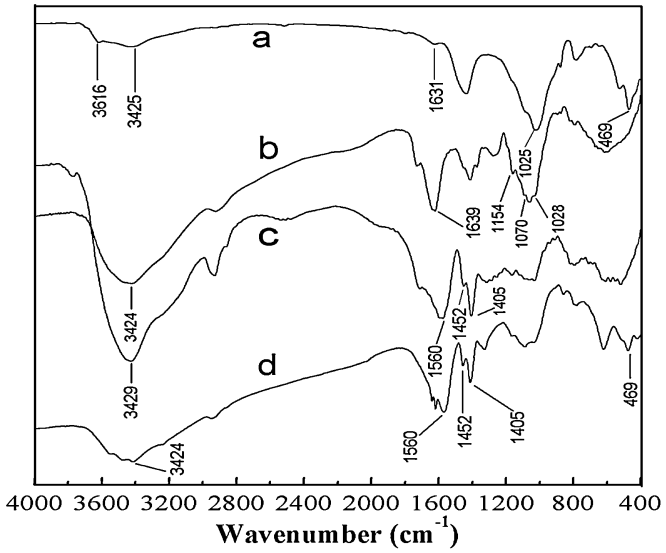


Fig. 1. FTIR spectra of (a) loess, (b) XG, (c) XG-PAA, and (d) XG-g-PAA/loess.

3. Results and discussion

3.1. FTIR analysis

The FTIR spectra of loess, XG, XG-PAA and XG-g-PAA/loess were shown in Fig. 1. It can be seen from Fig. 1 b, the weakened absorption bands of XG at 1028, 1076 and 1154 cm^{-1} are ascribed to the stretching vibrations of C—O(H) and the band at

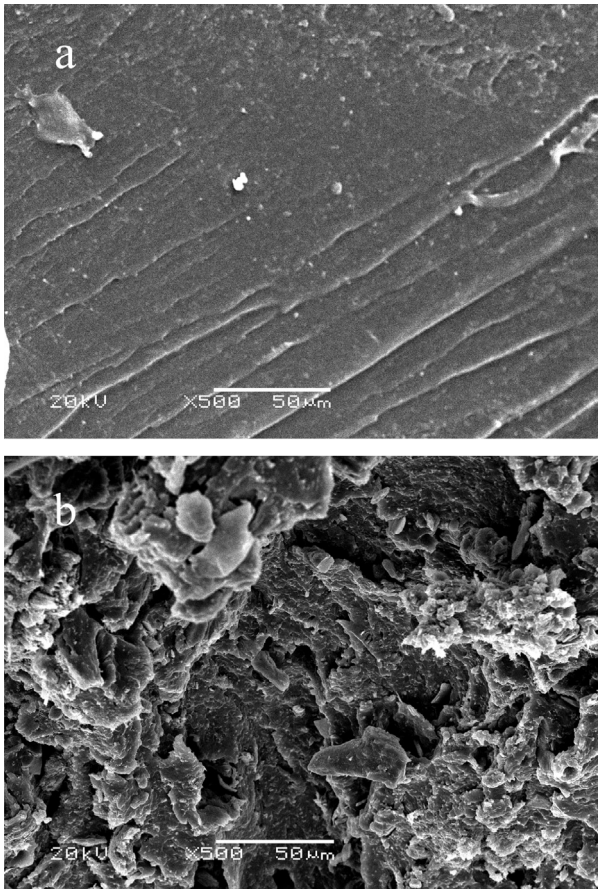


Fig. 2. SEM micrographs of (a) XG-g-PAA and (b) XG-g-PAA/loess (2 wt%).

1639 cm^{-1} is ascribed to bending vibration of —OH groups (Wang, Zhang, & Wang, 2009a, 2009b). However, these absorption peaks of XG almost disappeared and the new peaks at about 1560, 1452 and 1405 cm^{-1} (asymmetric stretching and symmetric stretching in —COO— groups) (Wang & Wang, 2009) appeared in the spectra of XG-g-PAA, XG-g-PAA/loess and after grafting copolymerization with AA (Fig. 1c and d), this indicates that XG had been grafting copolymerization with PAA. In addition, the —OH stretching vibration of loess at 3616 and the —OH bending vibration at 1631 cm^{-1} almost disappeared after reaction, the strong absorption bands at 1025 cm^{-1} ascribed to ═Si—O stretching of loess obviously weaken after reaction, and the Si—O—Si bending vibration of loess at 469 cm^{-1} appears in the spectrum of XG-g-PAA/loess (Fig. 1a and d) (Wang, Zhang, & Wang, 2008). These informations indicate that the loess also participated in the grafting copolymerization reaction.

3.2. SEM studies

Scanning electron micrographs of composite superabsorbent containing 0 wt% and 2 wt% loess are depicted in Fig. 2. Obviously, the fracture surface morphology of the XG-g-PAA/loess composite is different from that of XG-g-PAA. It can be observed that the XG-g-PAA composite (Fig. 2a) displays a smooth and tight fracture surface without any pores. However, the composite containing loess (Fig. 2b) presents a loose and porous fracture surface. This fracture surface is convenient for the penetration of water into the polymeric network, and may be of benefit to water absorbency of corresponding superabsorbent (Liang, Yuan, Xi, & Zhou, 2009).

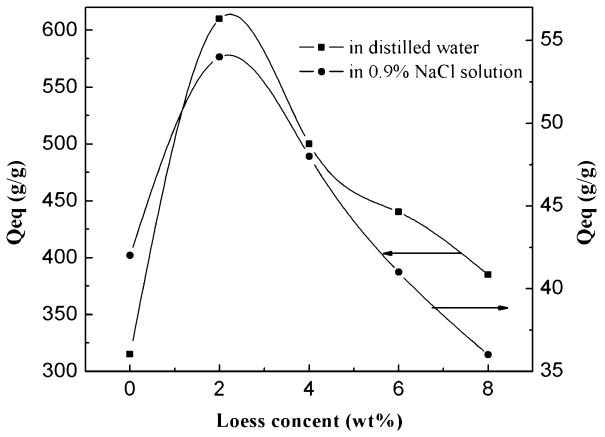


Fig. 3. Effect of loess content on water (salt) absorbency of superabsorbent composites.

3.3. Effect of loess content on swelling ratio

To study the effect of loess content on swelling ratio of the composite superabsorbent, the swelling ratio of the composites with the variable content of loess (0–8 wt%) in distilled water and 0.9 wt% NaCl solution was evaluated. As shown in Fig. 3, when 2 wt% of loess is introduced, the swelling ratio of the composite superabsorbent reaches maximum 610 g/g in distilled water and 54 g/g in NaCl solution respectively. Compared with XG-g-PAA, when 2 wt% of loess is introduced, the swelling ratio increases remarkably by 93.6% and 28.6%. However, the swelling ratio gradually decreases with further increase loess content from 2 to 8 wt%. This may be due to the fact that appropriate rigid loess could weaken the strong hydrogen-bonding interaction and other physical intertwining between the polymeric chains (Shi et al., 2012), which are extremely facilitated to improve the swelling ratio. However, loess can also act as an additional crosslinking point in the polymeric network (Lin, Wu, Yang, & Pu, 2001), increasing loess content will induce a larger crosslink density of the composite. Another reason is the excessive loess particles will plug up some network voids. Therefore, with the loess content increasing, swelling ratio of the composite superabsorbent decreases gradually. But, it is worth pointing out that the water absorbency of the composite superabsorbent containing 8 wt% of loess is still 22% higher than that without it, which is favorable to reduce the production cost.

3.4. Effect of external pH on swelling ratio

Swelling properties of the composite superabsorbent was investigated in various solutions with different pH values ranging from 2.5 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. As shown in Fig. 4, the water absorbency of the XG-g-PAA-loess containing 2% and 4% loess composite superabsorbent increases sharply in the pH range of 2.5–5 and decreases in the pH range of 10–13, but remains almost constant at pH 5–10. In pH < 5 solutions, most of the carboxylate anions were protonated, which restricts the electrostatic repulsion and strengthen the hydrogen-bonding interaction among the polymer chains (Aouada, Moura, de Muniz, Silva, & da Mattoso, 2011). The network space of the superabsorbent polymer is likely to contract and expel water from the gel network and the swelling ratio is lower. When the pH > 10, the dominated electrostatic bonding between —COO— and excess Na^+ of the swelling medium causes a rapid decrease of the osmotic pressure of the polymer network and ultimately reduces the equilibrium swelling capacity of the superabsorbent. However, at a wide pH range of 5–10, the water

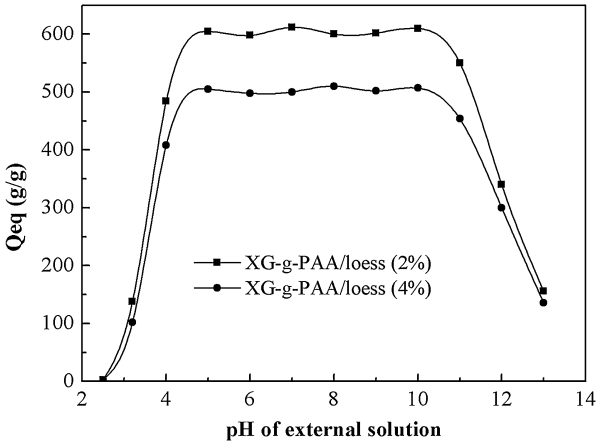


Fig. 4. Effect of external pH values on water absorbency of superabsorbent composites.

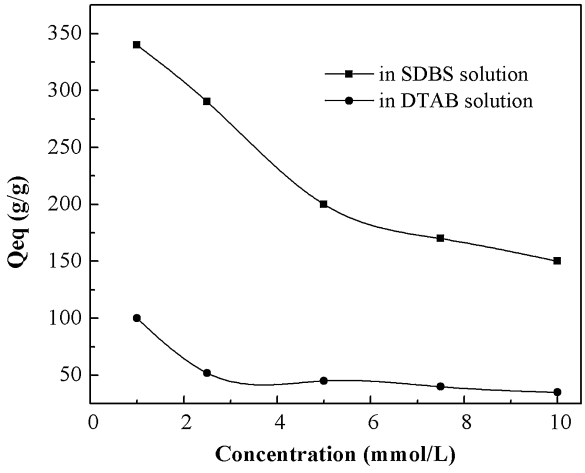


Fig. 5. Effect of different surfactants on water absorbency of superabsorbent composites.

absorbency of the composite superabsorbent is almost equal to its equilibrium water absorbency. This can be attributed to the fact that some of carboxylate groups are ionized and the ionization degree of the carboxylate groups keeps almost constant, which induces a similar osmotic pressure between the hydrogel network and the external solution as well as the electrostatic repulsion among the —COO— groups (Shi, Wang, & Wang, 2011). This behavior of the composite superabsorbent is very advantageous for the use of superabsorbent in various soils for agricultural application.

3.5. Effect of different surfactants on swelling ratio

The swelling capacity of a superabsorbent in different types of surfactant solution has many important practical applications. In this study, the effects of surfactant solution on the swelling behaviors of the composite superabsorbent containing 2% loess were examined by sodium dodecyl benzene sulfonate (SDBS) and dodecyl trimethyl ammonium bromide (DTAB) solution. From Fig. 5, it can be seen that with increasing concentration of the surfactant, the swelling capacity of the sample is significantly decreasing. This may be ascribed to the reduced osmotic pressure difference between the polymer network and the external solution. In addition, it also can be clearly observed that the equilibrium-swelling capacity of the composite superabsorbent in the anionic surfactant solution (SDBS) showed comparatively higher than that of the cationic surfactant solution (DTAB) at the same

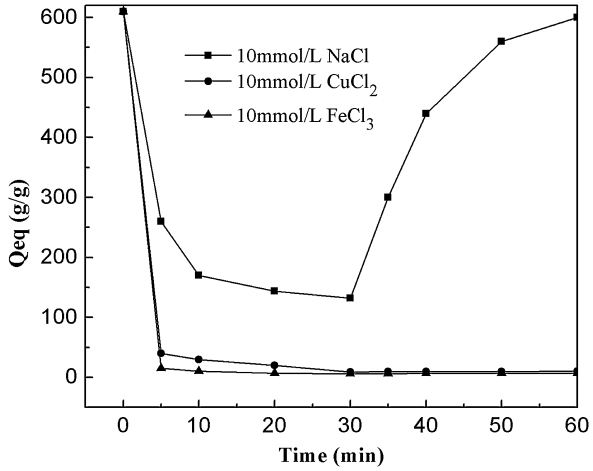


Fig. 6. Swelling–deswelling behavior of superabsorbent composites in different salt solution and distilled water.

concentration. This may be due to the strong association, binding or interaction of cationic surfactant molecules with the counter ions or ionizable groups of anionic superabsorbent (XG-g-PAA/loess) as well as aggregation of the surfactant molecules within or over the networks of superabsorbent (Mohan, Premkumar, Joseph, & Geckeler, 2007).

3.6. Swelling–deswelling behavior of composite superabsorbent in different salt solution and distilled water

The swelling–deswelling behavior of the composite superabsorbent containing 2% loess in 10 mmol/L NaCl, $CaCl_2$ and $FeCl_3$ solution were tested. As shown in Fig. 6, the swelling capacity of the fully swollen gel samples is significantly decreased in ten minutes, and tends to a constant value at 30 min. In addition, it can be found that water absorbency for the fully swollen composites in the studied solutions is the monovalent > divalent > trivalent cations, and the decreasing tendency of water absorbents with time is more obvious in a multivalent salt solution than in a monovalent salt solution. These may be attributed to charge screening effect of the additional cations causing a non-perfect anion–anion electrostatic repulsion, led to a decreased osmotic pressure difference between the superabsorbent network and the external solution (Wu et al., 2012). In addition, cations can complex with the carboxylate groups in the superabsorbent to form an addition chemical crosslinking, which lead to the deswelling or contraction of the superabsorbent (Shi et al., 2011). But different valent cations complex with carboxyl groups is different. After 30 min, the superabsorbent hydrogel that had been immersed in the multivalent cations solution will not re-swell in water again. This behavior is attributed to multivalent cations strong complexing ability with the carboxylate groups in the superabsorbents. According the swelling–deswelling behavior of the composite superabsorbent, we can confirm that the composite superabsorbent can be used for removal of multivalent metal ions.

3.7. Water-retention properties of composite superabsorbent

Water retention properties at different temperatures have a great influence on superabsorbent application in various fields. In this paper, we used the composite superabsorbent containing 2% loess to investigate the water retention properties at 25, 45 and 60 °C. As shown in Fig. 7, fully swollen composite superabsorbent at 25, 45 and 60 °C for water retention rate decreases with time, and water retention curve at 25 °C is flat than at higher temperature.

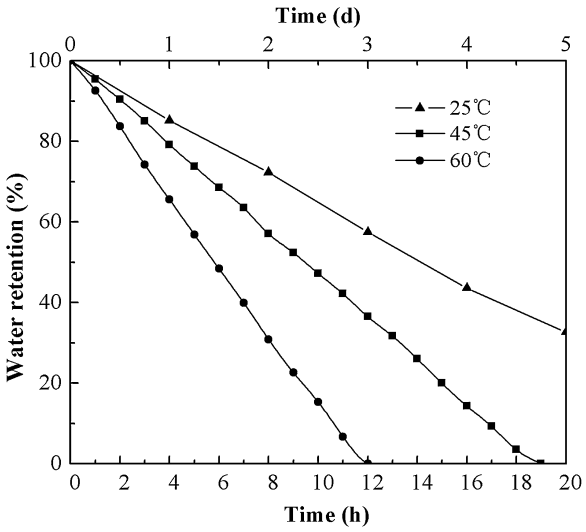


Fig. 7. Water-retention properties of superabsorbent composites at various temperatures.

The water retention for the composite superabsorbent at 25 °C is still more than 32% after 5 d, and at 45 and 60 °C, it can also keep 19 h and 12 h, respectively. This indicated that the composite superabsorbent has an excellent property for water retention.

4. Conclusions

Novel organic–inorganic composite superabsorbent XG-g-PAA/loess was successfully synthesized in aqueous solution. The composite superabsorbent has excellent water absorbency and retention capability. The swelling behaviors studies indicate that the composite superabsorbent exhibit almost constant and relatively higher swelling capacity in the ranges of pH 5–10. In surfactant solution, the equilibrium swelling capacity of the composite superabsorbent depends on the types and concentration of the surfactant. In addition, swelling–deswelling behavior of the composite superabsorbent in 10 mmol/L NaCl, $CaCl_2$ and $FeCl_3$ solution tests indicated the composite superabsorbent can be used for the removal of multivalent metal ions. This novel composite superabsorbent has significant potential to be application in agriculture, horticulture, hygienic products, and wastewater treatment.

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