Waste Polystyrene Foam-*graft*-Acrylic Acid/Montmorillonite Superabsorbent Nanocomposite

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ABSTRACT: A novel superabsorbent nanocomposite based on partially neutralized acrylic acid, waste polystyrene foam, and sodium type montmorillonite (Na-MMT) powder was synthesized through emulsion polymerization using N, N'-methylenebisacrylamide as a crosslinker, 2,2'-azo-bisiso-butyronitrile, ammonium persulfate, and sodium sulfite as mixed redox initiators. The effects of such factors as amount of Na-MMT, crosslinker, initiator, and neutralization degree on water absorbency of the superabsorbent were investigated. The composites were characterized by Fourier transform infrared spectroscope, X-ray diffraction, thermo gravimetric analysis, and scanning electron microscope. The results show that acrylic acid monomer successfully grafted onto the polystyrene chain, the layers of Na-MMT were exfoliated and dispersed in the composite at nano size after copolymerization. The introduction of waste polystyrene foam in the composite increased the water absorbency rate. The addition of Na-MMT not only enhanced the thermal stability of the composites but also increased its water absorbency, and the optimal water absorbencies of distilled water and saline water ($w_{NaCl} = 0.9\%$) of the nanocomposites were more than 1180 g H_2O/g and 72.6 g H_2O/g, respectively. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 2341–2349, 2007

Key words: superabsorbent nanocomposites; acrylic acid; waste polystyrene foam; montmorillonite

INTRODUCTION

Superabsorbents are lightly crosslinked functional polymers that can absorb, swell, and retain aqueous solutions up to thousands of times of their own weight in relatively short period of time. Because of their outstanding merits, superabsorbents have stimulated considerable interests and have been widely used in specialized areas, such as matrices for enzyme immobilization, and controlled release, as well as ordinary personal care products including infant diapers, feminine hygiene products, and incontinence products.[1–4]

Crosslinked poly(acrylic acid) is one of the superabsorbents with high water absorbency, but the high production cost largely restricts its applications. Montmorillonite (MMT) is a low-cost layered aluminum silicate with a reactive hydrophilic group –OH on its surface. It has been widely used in enhancing various abilities of polymers because of the specialties that can dilate in polar-media and disperse in composites at nano scale, which can bring about the nano size effect and the strong interface effect.[5,6] However, little application of MMT has been recorded in this field.[7,8] Waste polystyrene foam (WPSF) is one of the main sources of which was so called ''white pollution.'' The reclamation and recycling of WPSF is a very important objective, to which a great deal of attention has been paid in recent years, especially in the aspect of finding a way to convert WPSF into functional polymer, which is more valuable than other means.[9–15] To enhance the properties, as well as to reduce the production cost of the PAA superabsorbent and convert waste resources into ''treasures,'' a novel superabsorbent nanocomposite consisting of WPSF, partially neutralized acrylic acid and Na-MMT was synthesized in this article, and its properties, together with the major influencing factors on its absorbency were discussed.

EXPERIMENTAL

Materials

Acrylic acid (AA, chemically pure) was obtained from Lingfeng Chemical Reagent of Shanghai, China. N, N'-methylenebisacrylamide (MBA, chemically pure) and sodium hydroxide (analytical grade) were purchased from Chemical Reagent of Chinese Medicine Group, Shanghai. Ammonium persulfate (APS, analytical grade) and sodium sulfite (SS, analytical grade) were supported by The Second Chemical Reagent Factory of Yixing, Jiangsu, China. 2, 2'-Azo-bisiso-butyronitrile (AIBN, analytical grade) and hexamethylene (analytical grade) were purchased from The Forth Reagent Factory of Shanghai, China. Toluene (analytical grade) and ethyl acetate (analytical grade) were supported by The First Reagent Factory of Shanghai, China. Span-60 (chemically pure) was purchased from Shanghai Chemical Reagent Factory of China. Sodium type montmorillonite (Na-MMT) was made of original MMT (Fenghong Chemical Clay Factory of Zhejiang, China) in our lab. Waste Polystyrene Foam (WPSF), with the $M_W = 170,000$ (tested by GPC), was selected from the package of household electric appliances, and was washed with distilled water and dried at room temperature before use.

Preparation of Na-MMT

In a flask, original MMT and distilled water, with the mass ratio of 1 : 4, were stirred at room temperature for 30 min initially, and then deposited for at least 4 h to remove impurities. Na-MMT powder was obtained after the upper suspending solution was modified with sodium carbonate powder under stirring for 30 min, dried at 80°C over night, milled through a 300-mesh screen. The amount of sodium carbonate powder is 5 wt %, which is the mass ratio of sodium carbonate powder and MMT that existed in the upper suspending solution.

Preparation of PS-*graft*-AA-MMT superabsorbent nanocomposites

There are three steps in the preparation process of PS-*graft*-AA/MMT superabsorbent. The first step is the preparation of oil-soluble solution: a proper amount of WPSF was dissolved in a mixed solvent under room temperature for several minutes, and the solvent consisting of hexamethylene, toluene, and ethyl acetate with the ratio of 3 : 7.6 : 6. And certain amount of oil-soluble initiator 2, 2'-Azo-bisiso-butyronitrile (AIBN), as well as appropriate amount of dispersant Span-60, was put in the solution under stirring.

The second step is the preparation of water-soluble solution: in an ice bath, AA was neutralized with sodium hydroxide to the desired neutralization degrees. A given amount of sodium type MMT (Na-MMT, having a particle size less than 1.5×10^{-4} m) was added to the neutralized solution and stirred (10,000 rpm) for 5 min.

The last step is the copolymeric reaction: under condition of nitrogen atmosphere and stirring, the above two solutions were mixed and heated to 45°C. A predetermined amount of crosslinker N, N'-methylenebisacrylamide (MBA), water-soluble redox initiator ammonium persulfate (APS), and sodium sulfite (SS), dissolved in 2 mL distilled water, respectively, were added to the polymeric mixture. Afterwards, the polymeric mixture was stirred up to the gel point, when the mixture became sticky, and then transformed to another water bath at 70°C for 3 h. After cut to small pieces and dried at 110°C for 24 h, screened, and milled, nano superabsorbent particles were obtained.

Preparation of PS-*graft*-AA superabsorbent composite

The preparation process of PS-*graft*-AA superabsorbent composite was similar to the preparation procedure of PS-*graft*-AA/MMT superabsorbent composite except without MMT.

Preparation of PAA-MMT superabsorbent nanocomposites

The preparation process of PAA-MMT superabsorbent composite was similar to the later two steps in the preparation process of PS-*graft*-AA/MMT superabsorbent.

Water absorbency measurement

A proper amount of the superabsorbent nanocomposite (0.15 g, particle sizes between 60 and 100 meshes) was immersed in distilled water or saline water at room temperature to reach the swelling equilibrium. The residual water was removed by filtrating with 100-mesh screen. The water absorbency (A_1) and saline water absorbency (A_2) of the composites were calculated according to the following equation:

$$A = (m_2 - m_1)/m_1 \qquad (1)$$

In the above equation, A is the water absorbency or saline water absorbency, m_1 and m_2 are the weight of dry sample and swollen sample, respectively.

Water retention test

A certain amount of swollen superabsorbent samples of PS/AA and PS/AA/MMT (2.5 wt %) together with the comparative amount of pure water were put in three beakers, respectively. Then the beakers were put into an oven, which had been adjusted to 110°C. And their weights were recorded in a regular interval. The weight loss (W) was calculated by the equation below:

$$W = [(w_o - w_i)/w_o] \times 100\% \qquad (2)$$

where w_o is the weight of original swollen sample while w_i is the weight of the heated samples.

Characterization

1. The IR spectra of the dry samples were performed on a 170SX FTIR spectrograph (Thermo–Nicolet, Walthan, MA) in KBr flakes in the frequency range of 400–4000 cm^{-1}.
2. The XRD patterns of the dried samples are obtained by using D/Max-RC X-ray diffract meter (Rigaku, Tokyo, Japan, 40 kV, 100 mA) with copper target at a rate of 2 degree per min, for each interval of 0.02°. The diffracted X-ray intensity is recorded automatically.
3. The thermal stabilities of the dry samples were recorded by a Perkin–Elmer TGA-7 thermo gravimetric analyzer (Perkin–Elmer, Norwal, CT) with the temperature range from 20°C to 750°C at a heating rate of 20°C/min.
4. The morphologies of the nanocomposites were examined by JSM-5610LV (JEOL, Tokyo, Japan) scanning electron microscope instrument after the samples were coated with gold in JFC-1600 Fine Coater (JEOL, Tokyo, Japan) at 20 mA for 200 s.

RESULTS AND DISCUSSION

Effect of mass ratio of AA and WPSF on water absorbency of superabsorbent

The relationship between water absorbency and the mass ratio of AA and WPSF is depicted in Figure 1(a). It can be obviously seen that the water absorbency increases with the increasing of ratio value, especially before the ratio value reaches 8. In other words, the more percentages of AA in the composites, the higher absorbency of the superabsorbent is achieved. The saline water absorbency trend [Fig. 1(b)] is similar to the trend of water absorbency.

The reason why these happen is that the absorbency of the superabsorbent attributes to the existence of crosslinked hydrophilic network that grafted on the PS chain. The hydrophilic groups in the network can form hydrogen bond when contracting with
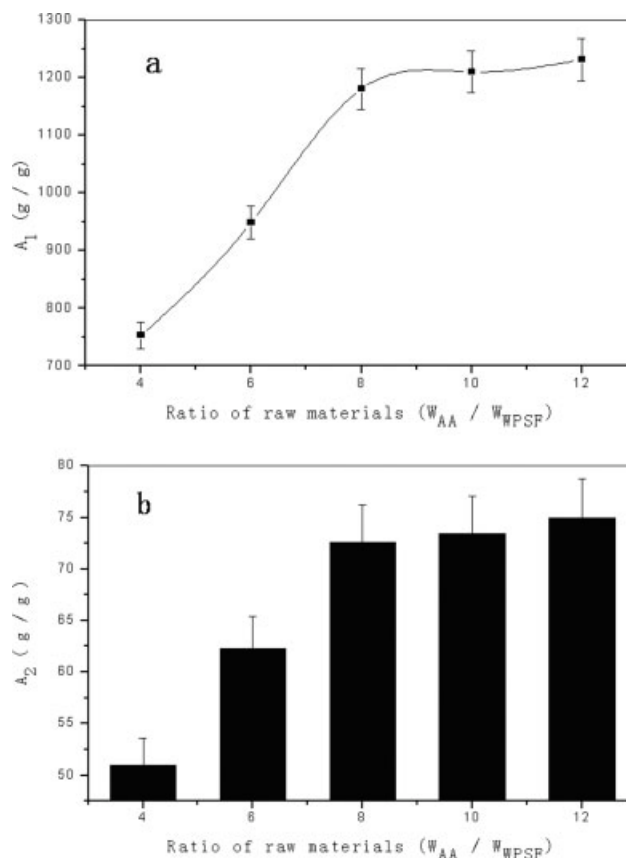
Figure 1 (a) Water absorbency (b) and saline water absorbency of the nanocomposites with various ratios of mass ratio of AA and WPSF (2.5 wt % Na-MMT; 0.06 wt % crosslinker; 0.92 wt % initiator; 85% neutralization degree; 3 wt % dispersant; react at 70°C for 3 h).

water. However, PS chain is hydrophobic, and its amount directly affects the absorbency of the superabsorbents. The reaction between PS chain and AA may be described as Scheme 1.

It means a brand-new network that is synchronous with hydrophilic ability and hydrophobic ability was formed. Therefore, the increasing of the WPSF amount decreases the absorbency consequentially.

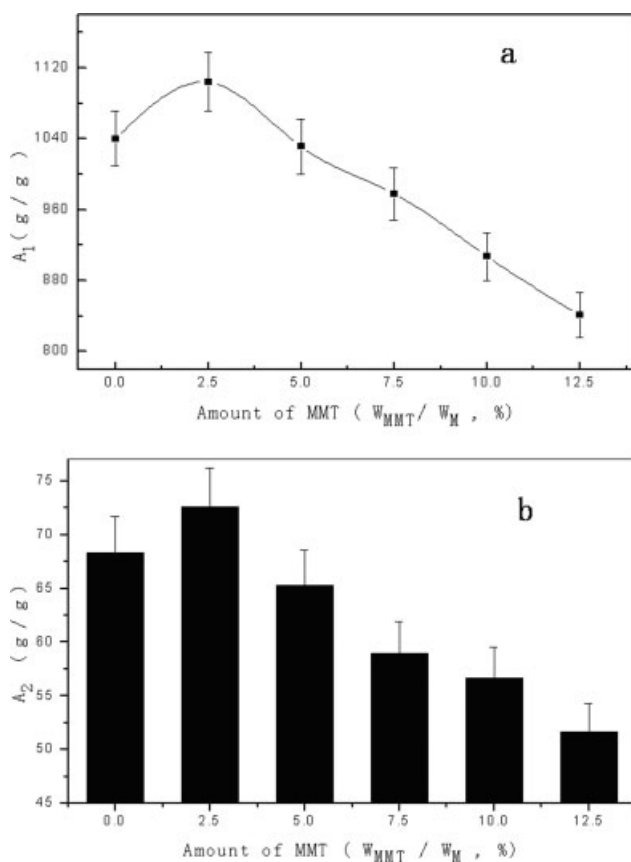### Effect of Na-MMT amount on water absorbency of superabsorbent

It can be evidently seen from Figure 2 that the water absorbency and saline water absorbency of the nanocomposite increases with the increasing of Na-MMT amount before its percentage reaches 2.5 wt %, and then decreases with the further increasing of Na-MMT amount.

This is because the reactive group –OH that exists on the surface of exfoliated MMT layers reacted with carboxylic acid group that exists on the network (shown in Scheme 2),[16] and that deduce has been
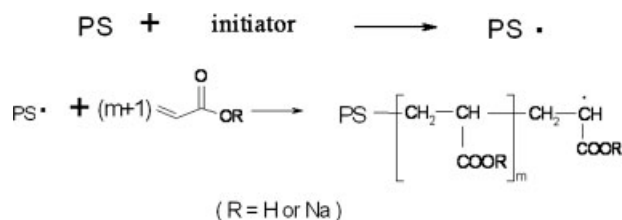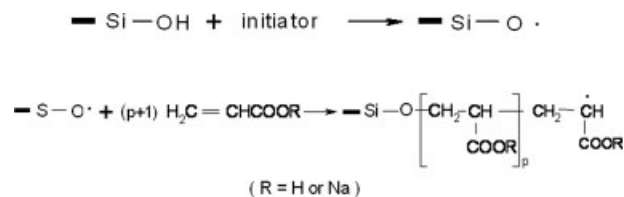
Scheme 1 Schematic reaction between PS foam and vinyl monomer.

Scheme 2 Schematic reaction between vinyl monomer and MMT layer.

proved by IR analysis (Fig. 6). It means that MMT layers act as the additional crosslinking points of the network and branches of the network have been utilized to form a more efficient network (performed in Scheme 3),[17] in which it can bond more water. In addition, the hydrophilic group –OH, existing on the MMT surface, also contributed to the water absorbency of the superabsorbent because it can form hydrogen bond with water. However, overabundant amount of MMT will lead to too dense of crosslinking density and some MMT layers act as physical filler in the network, therefore decreases the absorbency.

Effect of initiator amount on water absorbency of superabsorbent

A compounded redox initiator, consisting of AIBN, APS, and SS, was utilized in the emulsion system. And the effect of the initiator amount on the water absorbency of the superabsorbents is obviously shown in Figure 3. The water absorbency increases with the increasing of the total initiator amount up to 0.92 wt % and decreases with the further increasing of the initiator amount. The water absorbency of the superabsorbent is related to its network structure and the relation between the average kinetic chain lengths (V) and concentration of the initiator in free-radical polymerization is given as the eq. (3):[18]

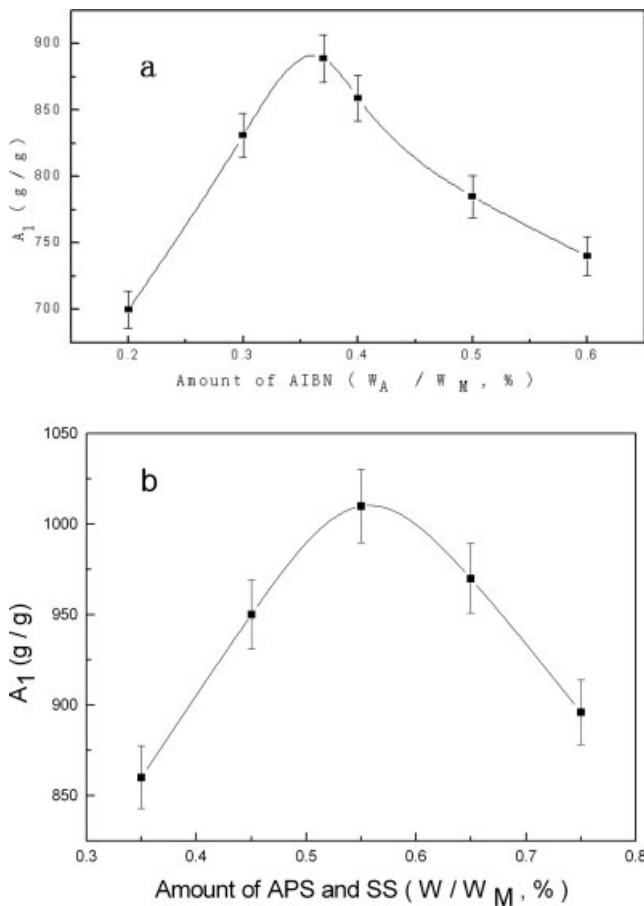$$V = 1/2\, k_P (f\, k_i\, k_t)^{-1/2}[I]^{-1/2}[M] \tag{3}$$

in the equation, k_P, k_i, and k_t are the rate constants for propagation, initiator, and termination, respectively; f is the initiating efficiency of the initiator; and $[I]$ and $[M]$ are the initial concentration of the initiator and reactive monomer. According to the equation, the more initiator concentration, the shorter average kinetic chain length is gained. This is because plentiful initiators produce superfluous free radicals, which lead to a too dense of grafting density (AA grafted onto PS chain) and crosslinking density (between AA and crosslinker); on the other hand, as reported in another study,[19] the polymeric chain ends do not contribute to the water absorbency. So the increases of initiator amount decrease the water absorbency. However, when the initiator concentration is below the optimal value, there are not enough free radicals to form an efficient network, therefore it cannot reach the optimal water absorbency.

Effect of crosslinker amount on water absorbency of superabsorbent

Crosslinking density is an extremely important factor that affects the water absorbency of the superabsorb-

Figure 2 (a) Water absorbency (b) and saline water absorbency of the nanocomposite with various Na-MMT amounts (AA and WPSF mass ratio is 8; 0.075 wt % crosslinker; 1.05 wt % initiator; 90% neutralization degree; 3 wt % dispersant; react at 70°C for 3 h).

Scheme 3 Schematic reaction between MMT and PS chain.

Figure 3 Water absorbency of the nanocomposites with various initiator amounts [(a) Various AIBN amount with 0.55% wt water-dissolvable initiator; (b) Various amount of APS and SS with 0.37% oil-dissolvable initiator]. Other conditions: AA and WPSF mass ratio is 8; 0.075 wt % crosslinker; 5 wt % Na-MMT; 90% neutralization degree; 3 wt % dispersant; react at 70°C for 3 h).

ents. The influence of crosslinker amount on the water absorbency is shown in Figure 4. The absorbency increases with the increase of crosslinker amount before it reaches 0.06 wt %. However, when crosslinker amount reaches 0.06 wt %, further increasing of crosslinker content decreases the water absorbency instead. The swelling behavior of the polymers is presented by the eq. (4):[20]

$$Q^{5/3} = \{[i/2 \times V_u \times S^{1/2}]^2 + (1/2 - X_1)/V_1\}/(V_e/V_0) \tag{4}$$

where Q is the absorbency of the superabsorbents; V_e/V_0 is the crosslinking density; $(1/2 - X_1)/V_1$ is the hydrophilic ability of polymeric chain; i/V_u is the concentration of electric charge that bonded in the polymeric chain and S is the ionic intensity of the solution outside. According to the equation, the increase of the crosslinking density will decrease the water absorbency. The reason for this

is that an excessive crosslinking density brings on a too dense polymeric network and leaves smaller space for water to enter. Whereas, low concentration of crosslinker cannot form a fine network, and some uncrosslinked polymer chains may be dissolved in water. So, the decreasing of the crosslinker amount below the optimal amount will decrease the water absorbency of the superabsorbents consequently.

Effect of neutralization degree on water absorbency of superabsorbent

Figure 5 indicates the effect of neutralization degree on absorbency. It is markedly that the absorbency increases with the increase of neutralization degree from 70 to 85%, and sharply decreases with the further increase of the neutralization degree. According to Flory's polymeric network swelling theory,[20] the ionic strength is one of the important factors that affect the swelling ability of ionic polymer. And this behavior may be assuming that the water absorbency of the superabsorbency is attributing to the collaborative absorbable effect of carboxyl and carboxylate in the network. The carboxylate will ionize and produce sodium ions and carboxylate ions, which will contribute to the infiltration pressure and hydrophilic ability, respectively. However, an average neutralization degree will result in repulsion among carboxylate groups for the overfull negative ions bonded on the network. In spite of carboxyl having lower water absorbency than carboxylate groups, it can mitigate the repulsion effect of carboxylate groups.[16,17] Hence, when carboxylate and carboxylic acid groups cooperate in a suitable ratio, the cooperative effect will reach its maximum.
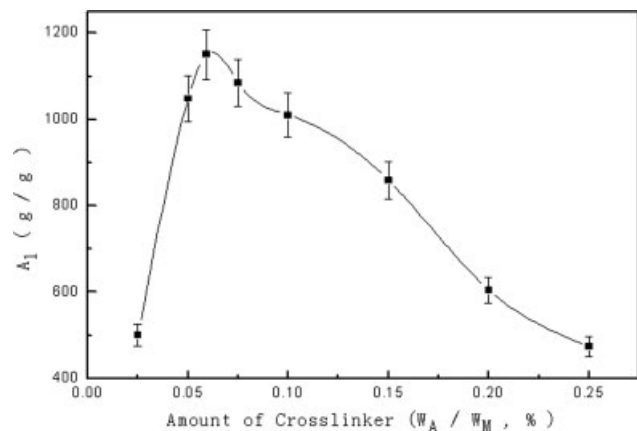

Figure 4 Water absorbency of the nanocomposites with various crosslinker amounts (AA and WPSF mass ratio is 8; 0.92 wt % initiator; 5 wt % Na-MMT; 90% neutralization degree; 3 wt % dispersant; react at 70°C for 3 h).
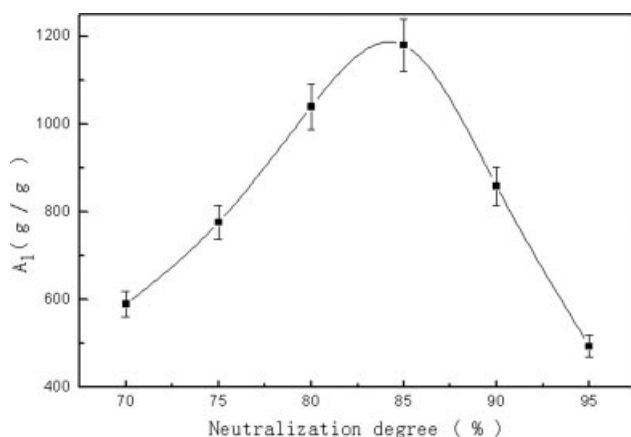
Figure 5 Water absorbency of the nanocomposites with various neutralization degrees of AA (AA and WPSF mass ratio is 8; 2.5 wt % Na-MMT; 0.06 wt % crosslinker; 0.92 wt % initiator; 3 wt % dispersant; react at 70°C for 3 h).

Infrared spectrum analysis

The infrared spectrums of Na-MMT (a), PS/AA/MMT superabsorbent nanocomposite (2.5 wt % MMT) (b), and PS/AA superabsorbent composite (c) are shown in Figure 6. The characteristic peaks at 3629 and 917 cm^{-1} in Figure 6(a) ascribe to stretching vibration and deformation vibration of –OH on the surfaces of MMT layers, and the latter can be found in the PS/AA/MMT [Fig. 6(b)]. The characteristic peak near 1100 cm^{-1} in Figure 6(a) belongs to the vibration of Si—O in the MMT and it may be covered by other groups' peaks after polymerization. A remarkable characteristic peak at 1716 cm^{-1} in Figure 6(b) arises from the C=O of —COOR, which indicates the grafting reaction between AA and MMT (presented in Scheme 1). Additionally, the characteristic peak at 857 cm^{-1} [Figs. 6(b,c)] corresponding to the contrapuntal replace reaction that occurs on the phenyl of the PS, and this is also clearly shown by Scheme 1.
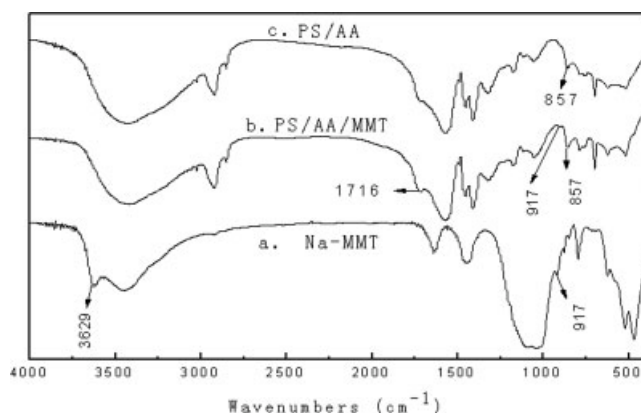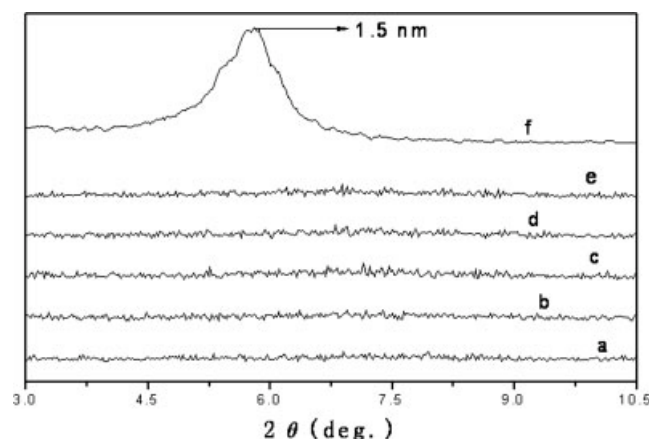
X-ray diffraction analysis

The X-ray diffraction patterns of Na-MMT and PS/AA/MMT with various MMT amounts (from a to e are 2.5, 5, 7.5, 10, 12.5 wt %, respectively.) are shown in Figure 7. It can be clearly seen that a notable peak in Figure 7(f), and its corresponding d-space value is 1.5 nm according to the Bragg Equation. However, there is feckly no obvious peak in other curves. It is indicated that layers of the MMT have almost exfoliated and dispersed in the composite at nano size after copolymerization especially when the amount of MMT is below 5 wt %.

Thermal stability test

Thermo gravimetric analysis curves of PS-*graft*-acrylic acid superabsorbent and PS-*graft*-acrylic acid/montmorillonite nanocomposite are clearly shown in Figure 8. Both thermo grams exhibit a three-step thermal decomposition. The first step, implying a loss of moisture existed in the samples, is the section when temperature below 200°C. The following step is in the range of 217.7–391.7°C. During this period, the weight losses of the two composites start almost at the same temperature, but there is a more complex decomposition in the nanocomposite attributing to the introduction of MMT into the composite. In the last step, the sharp weight loss onset of PS/AA/MMT is 405.1°C, which is slightly higher than that of PS/AA (393.9°C). This behavior is by reason of the fact that MMT layers act as the heat barriers and result in a reverse thermal stability.[6] A significant delay of weight loss (about 12%) has also been performed deriving from the barrier effect.[21]

Scanning electron microscope observation

The SEM micrographs of PS/AA and PS/AA/MMT (MMT amount is 2.5 wt %) superabsorbents are dis-

Figure 6 Infrared spectrums of (a) Na-MMT, (b) PS/AA/MMT nanocomposite (2.5 wt % MMT) and (c) PS/AA composite.

Figure 7 XRD patterns of Na-MMT (f) and PS/AA/MMT nanocomposites (a–e) with various amount of MMT. (The amount of MMT (wt %) in the nanocomposite from a to e are 2.5, 5, 7.5, 10, 12.5, respectively).
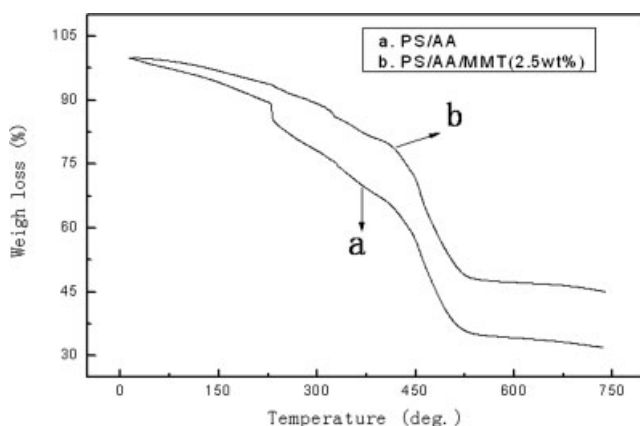
Figure 8 TGA curves of (a) PS/AA and (b) PS/AA/MMT (2.5 wt %) at a heating rate of 20°C/min.

tinctly shown in Figure 9. Compared with the micrograph of PS/AA [in Fig. 9(a)], there is a rougher surface in the micrograph of PS/AA/MMT [in Fig. 9(b)], which has better water absorbency and absorbency rate than those of PS/AA. And the nano structure of the nanocomposite can be clearly seen in Figure 9(c,d). These observations are in good agreement with water absorbency rate measurement and XRD analysis.
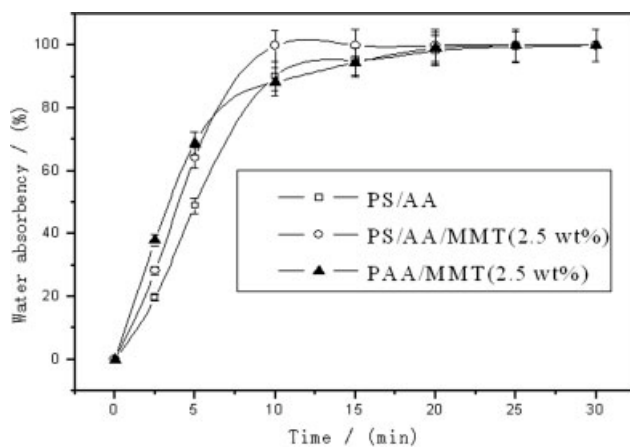
Water absorbency rate of the superabsorbents

Water absorbency rate of the absorbent relates to the swelling ability, surface area, particle size, and cross-linking density of the polymer.[22] Figure 10 obviously illustrates the distilled water absorption rates of the PS/AA, PS/AA/MMT (2.5 wt %), and PAA/MMT (2.5 wt %) superabsorbents (with the particle size between 60 and 100 meshes).

It can be clearly seen that the water absorbency rate of the PS/AA/MMT nanocomposite is faster than that of PS/AA composite from the beginning. And PS/AA/MMT can swell to more than 99% of its maximal absorption while PS/MMT swells to 90% of its maximal absorption in 10 min. This behavior is by reason of that there is a denser density of hydrophilic groups in the PAA/MMT nanocomposite than those of PS-containing superabsorbents, thus there is more serious "flour conglomeration effect" existed in PAA/MMT than those in PS-containing superabsorbents. So, the introduction of
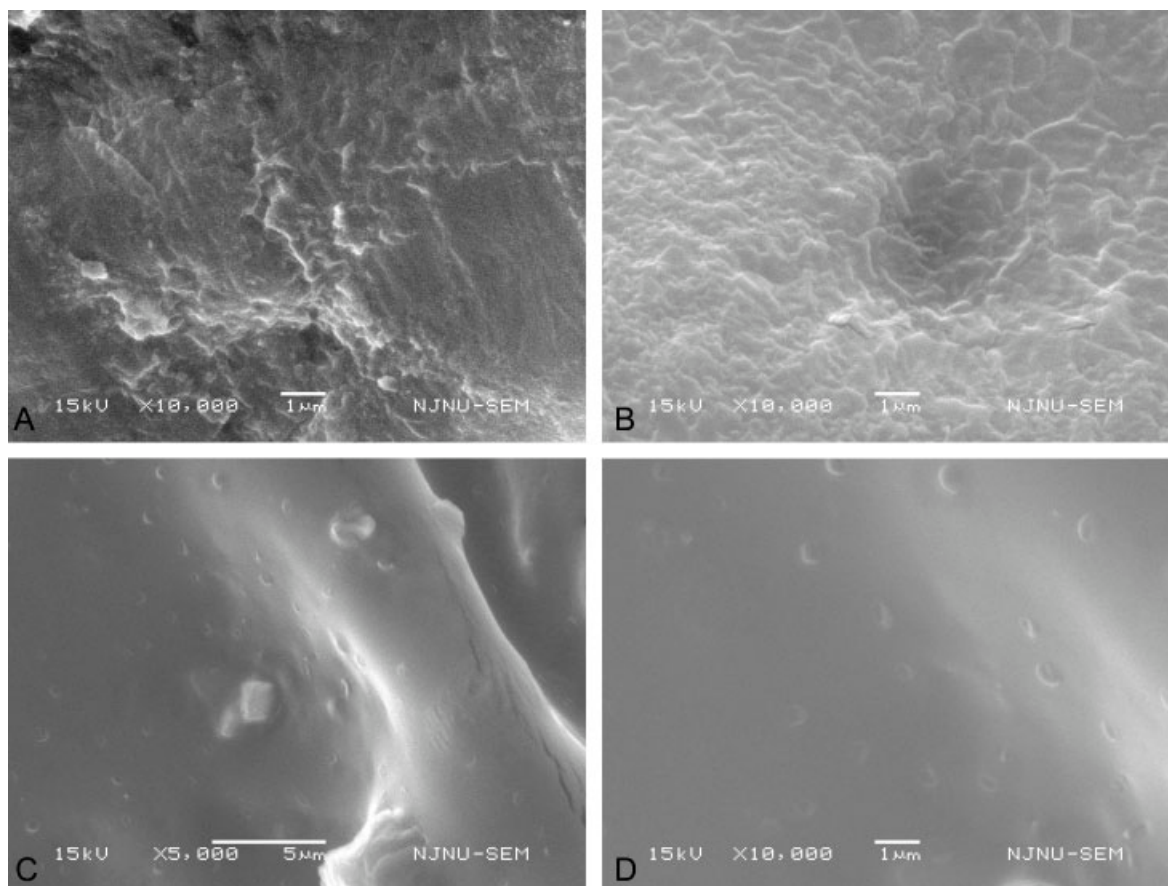
Figure 9 Scanning electron micrographs of PS/AA (a) and PS/AA/MMT (b–d, with 2.5 wt % MMT).

Figure 10 Water absorption rates of PS/AA, PS/AA/MMT (2.5 wt %), and PAA/MMT (2.5 wt %).

PS in the composite can weaken the "flour conglomeration effect" and increase the absorbency rate.

On the other hand, the water absorbency rate of the PAA/MMT is faster than those of the PS/AA/MMT at the beginning and slower than it after the samples were immersed in distilled water over 6 min. And the PS/AA/MMT can swell to more than 99% of its maximal absorption in 10 min while PAA/MMT swells to 88% of its highest absorption in the same period of time. This is due to the addition of the MMT, which can enhance the affinity ability of the polymeric network toward water molecule[23,24] and effectively reduce the "flour conglomeration effect," thus increase the absorbency rate of the composite.

Water retention ability of the superabsorbents

Figure 11 shows the water retention abilities of PS/AA and PS/AA/MMT (2.5 wt %) superabsorbent
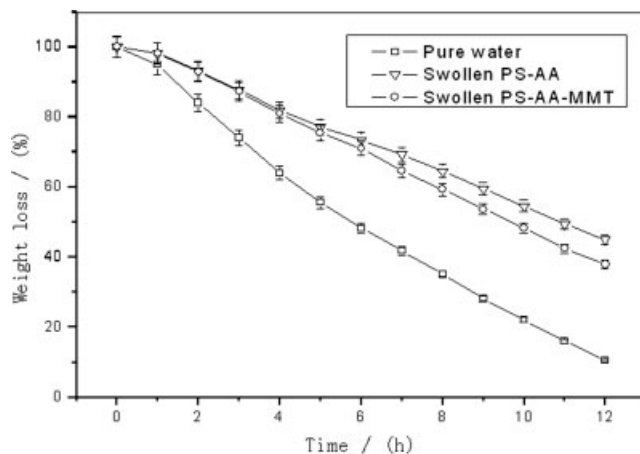
Figure 11 Weight loss of the swollen PS/AA and swollen PS/AA/MMT (2.5 wt %) superabsorbents compared with the weight loss of pure water at 110°C.

composites, which are measured by weight loss of PS/AA and PS/AA/MMT compared with the weight loss of pure water in a oven at 110°C. It is apparent that both the PS/AA and PS/AA/MMT superabsorbent composites have great water retention capacity at 110°C even for 12 h, and the former is slightly better than the latter. According to another study,[25] water exists in swollen superabsorbent composites in three types of forms: free, semibound, and bound. Free water and half-bound water can easily be lost comparing with bound water. In addition, the number of hydrophilic group -COOH in PS/AA/MMT is less than that in PS/AA [eq. (7)], therefore, the introducing of MMT to the superabsorbent will slightly decrease the water retention ability of the superabsorbent composites.

CONCLUSIONS

1. A novel polystyrene-*graft*-acrylic acid/montmorillonite superabsorbent nanocomposite with water absorbency about 1200 times was prepared from partially neutralized AA, WPSF, and Na-MMT powder on the following conditions: AA and WPSF mass ratio is 8, 2.5 wt % Na-MMT, 0.06 wt % crosslinker, 0.92 wt % initiator, 85% neutralization degree, 3 wt % dispersant, react at 70°C for 3 h.

2. By the IR analysis, the grafting copolymer reaction mechanism and the structure of the superabsorbent are supposed; by the XRD and the SEM characterizations, the nano structure of the nanocomposite was proved; by the TGA test, the enhancement of thermal stability of the composite by the addition of MMT was confirmed.

3. It is found that the introduction of PS chain to the composite not only reduced the production cost of the superabsorbent, but also improved the water absorbency rate as a whole, and this trial has provided a new method to convert waste resources into "treasures;" the addition of MMT increased the water absorbency of the superabsorbent, as well as enhanced the water absorbency rate and reduced the production cost.

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