Influence of Potassium Humate on the Swelling Properties of a Poly(acrylic acid-*co*-acrylamide)/ Potassium Humate Superabsorbent Composite

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ABSTRACT: A novel superabsorbent composite, poly (acrylic acid-*co*-acrylamide)/potassium humate (PAA-AM/KHA), was prepared by aqueous solution polymerization from acrylic acid, acrylamide, and potassium humate (KHA) with N,N'-methylenebisacrylamide as a crosslinker and potassium peroxydisulfate as an initiator. The effects of incorporated KHA on the water absorbency, swelling rate, and reswelling capability were investigated. The swelling property of PAA-AM/KHA in various saline solutions was studied systematically. The results show that the comprehensive properties and especially salt-resistant ability of PAA-AM/KHA were enhanced. There was a linear relationship between the saturated water absorbency and the minus square root of the ionic strength of the

INTRODUCTION

Superabsorbents are hydrophilic crosslinked polymers that can imbibe huge amounts of water. Since the U.S. Department of Agriculture reported the first superabsorbent polymer,¹ superabsorbents have been used widely in many fields and applications, such as feminine napkins, disposable diapers, and agriculture,²⁻⁵ for which the water absorbency of superabsorbents in saline solutions is critically important. Recently, research on the use of superabsorbents as water-managing materials for the renewal of arid and desert environments has attracted great attention. However, their application in this field has met some problems because most of these superabsorbents are mainly based on pure poly (sodium acrylate) and so are too expensive and not suitable for saline-containing water and soils.⁶ Consequently, the preparation of cost-efficient and saltresistant superabsorbents has been the focus of much research. There have been many reports on introducing inorganic clays, such as kaolin,7 montmorillonite,8 and attapulgite,9 into pure polymeric superabsorbents to improve the swelling properties

WVILEY InterScience® external medium, and the water absorbency of PAA-AM/ KHA in various salt solutions had the following order: $NH_4Cl_{(aq)} = KCl_{(aq)} = NaCl_{(aq)} > MgCl_{2(aq)} > CaCl_{2(aq)} >$ $AlCl_{3(aq)} > FeCl_{3(aq)}$. Moreover, the polymeric net structure of PAA-AM/KHA was examined with respect to that of poly(acrylic acid-*co*-acrylamide). The results indicate that the polymeric net of PAA-AM/KHA was improved by the introduction of a moderate amount of KHA into the superabsorbent composite and made more suitable for agriculture and horticulture applications. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 3727–3733, 2008

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and reduce the production costs. In our previous studies,^{15–17} the synthesis and swelling behaviors of superabsorbent composites based on poly(acrylic acid) (PAA) and sodium humate in distilled water have been reported. Almost all these investigations were focused on the improvement of the water absorbency and swelling rate of superabsorbents in distilled water; only a small effort has been made to study the swelling properties of superabsorbents in various saline solutions.^{10–14} Therefore, it is essential and practical to investigate the swelling properties of superabsorbents in various saline solutions. In our continuation of this research in the field of superabsorbent composites, attention is directed toward the preparation of a novel superabsorbent composite based on poly(acrylic acid-co-acrylamide) (PAA-AM) with potassium humate (KHA) as a functional filler. KHA is a low-cost raw material that can be extracted from leonardite and contains large numbers of functional hydrophilic groups (e.g., carboxylates and phenolic hydroxyls).^{15,16} Also, KHA is a kind of effective environmentally friendly fertilizer that can regulate plant growth, accelerate root development, improve soil cluster structures, and benefit the absorption of nutrient elements.^{15,16} Moreover, PAA-AM has a better salt-resistant ability than PAA.9,15,18 Therefore, the chemical blending of PAA-AM and KHA and fabrication of a superabsorbent composite

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TABLE I Typical Recipe for PAA-AM/KHA and Preparation Conditions

MBA/AA (mol/mol)	KPS/AA (mol/mol)	KOH/AA (mol/mol)	AM/AA (mol/mol)	KHA/(AA + AM) (wt %)
0.0005	0.01	0.6	0.05	6.6

Reaction temperature = 90° C; neutralization degree = 60% (mol/mol); initial concentration of AA and AM = 35 wt %.

can significantly reduce the production costs and improve the comprehensive properties, especially the salt-resistant ability, of superabsorbent composites.

In this investigation, a novel superabsorbent composite, poly(acrylic acid-*co*-acrylamide)/potassium humate (PAA-AM/KHA), was prepared, and its water absorbency, swelling rate, and reswelling capability were investigated. The swelling properties of PAA-AM/KHA in various salt solutions were also studied systematically.

EXPERIMENTAL

Materials

Acrylic acid (AA; chemically pure, Beijing Yili Chemical Factory, Beijing, China) was distilled under reduced pressure before use. The initiator, potassium analytical-grade; (KPS; peroxydisulfate Beijing Chemical Factory, Beijing, China), was recrystallized from water. *N*,*N*'-Methylenebisacrylamide (MBA; chemically pure; Shanghai Chemical Reagent Corp., Shanghai, China) was used as purchased. KHA (extracted from leonardite of the Huolinhe Innermongolia Autonomic Region, China) was purified in 0.2 mol/L potassium hydroxide and dried in an oven at 70°C. The other agents used were all analytical-grade, and all solutions were prepared with distilled water.

Preparation of PAA-AM/KHA

A series of PAA-AM/KHA superabsorbent composites were synthesized. The reactions were conducted in a flask equipped with a mechanical stirrer and condenser. Weighed quantities of AA and acrylamide (AM) were dissolved in distilled water. AA was neutralized with a potassium hydroxide solution (30 wt %), and an AM solution was added to the partially neutralized AA solution. KHA powder was dispersed in this mixture, and then the crosslinker MBA was added. The water bath was heated slowly with vigorous stirring after the radical initiator KPS was charged to the reaction mixture. After 5 h of the reaction, the resulting product was washed several times with distilled water and then dried in a vacuum oven at 60°C to a constant weight. The superabsorbent composite was

prepared after the dried product was milled and screened. All samples had a particle size in the range of 40–60 mesh. A typical recipe for the superabsorbent composite and the preparation conditions are listed in Table I.

Preparation of PAA-AM

The preparation procedure for PAA-AM was similar to that for the PAA-AM/KHA superabsorbent composite, except that KHA was not used.

Measurement of the water absorbency and swelling rate

A weighed quantity $(0.05 \pm 0.001 \text{ g})$ of the dry superabsorbent was immersed in distilled water at room temperature to reach swelling equilibrium. Swollen samples were then separated from unabsorbed water by filtration through a 100-mesh screen. The saturated absorbency of the superabsorbents (*Q*) was calculated with the following equation:

$$Q \text{ (g of H}_2\text{O}/\text{g of sample}) = (m_2 - m_1)/m_1$$
 (1)

where m_1 is the weight of the dry sample and m_2 is the weight of the water-swollen sample. Q was calculated as grams of water per gram of the dry sample. Three different samples were examined for each time of swelling equilibrium and averaged. The standard deviation of Q was found to be ± 2.0 g. The sampling procedure was repeated until the degree of absorption became constant.

The water absorbency of the dry sample in various saline solutions (NaCl_(aq), KCl_(aq), NH₄Cl_(aq), CaCl_{2(aq)}, MgCl_{2(aq)}, AlCl_{3(aq)}, and FeCl_{3(aq)}) with different concentrations was tested according to the same procedure, except that the standard deviation was found to be ± 1.0 g.

The swelling rate of the superabsorbent composite was measured according to a previously reported method.¹⁹

Measurement of the reswelling ability

The initial dry sample $(0.05 \pm 0.001 \text{ g})$ was immersed in one milliliter of distilled water to ensure that the swelling equilibrium was achieved. The swollen gel was placed in an oven at 60°C until the



Figure 1 Effect of the KHA content on the water absorbency of PAA-AM/KHA in distilled water (MBA/AA = 0.0005 mol/mol, KPS/AA = 0.01 mol/mol, KOH/AA = 0.6 mol/mol, AM/AA = 0.05 mol/mol).

gel was dried thoroughly. Equal milliliter of water was added to the dried gel, and the swollen gel was placed in the oven again. A similar procedure was repeated, and then the saturated absorbency of the sample after several periods of reswelling was obtained with eq. (1).

Here m_1 refers to the weight of the initial dry sample (0.05 g \pm 0.001 g); m_2 refers to the weight of the water-swollen sample after the reswelling procedures.⁹

RESULTS AND DISCUSSION

Effect of the KHA content on the water absorbency of PAA-AM/KHA

KHA is an important factor influencing the water absorbency of the PAA-AM/KHA composite because it contains large numbers of functional nonpolar and polar hydrophilic groups (e.g., carboxylates and phenolic hydroxyls), which are mostly located on its aromatic rings.^{15,16,20} The water absorbency of the PAA-AM/KHA superabsorbent composites incorporated with different amounts of KHA in distilled water is shown in Figure 1. The water absorbency increases from 620 to 769 g/g with an increasing amount of KHA in a weight range of 0-6.6% in the feed. When the amount of KHA in the feed is above 6.6%, the water absorbency decreases with an increase in the amount of KHA. In comparison with PAA-AM, the water absorbency of PAA-AM/KHA is improved by the introduction of KHA into the PAA-AM polymer network. This increase in the water absorbency of PAA-AM/KHA may be due to the fact that the effective crosslinking density decreases with the introduction of KHA into the PAA-AM network. Schematic illustrations of the

structures of PAA-AM and PAA-AM/KHA in water are shown in Scheme 1(a,b). In the PAA-AM network, the -COOH (or -CONH₂) groups bonded onto PAA-AM chains can interact with each other by hydrogen bonding,^{9,21} which in turn causes an increase in the crosslinking density and results in a tighter network, which prevents the expansion of the polymeric network in water.21,22 In the PAA-AM/ KHA network, there are large numbers of KHA particles, which can weaken the formation of hydrogen bonds between -COOH (or -CONH₂) groups, thus improving the polymeric network. At the same crosslinker content, the effective crosslinking density of PAA-AM is higher than that of PAA-AM/KHA. According to Flory's network theory,²³ a low crosslinking density of a hydrogel would lead to a high water absorbency. This phenomenon is in agreement with previous studies.^{15–18,21} However, further increasing the amount of KHA results in a remarkable decrease in the water absorbency. This may be attributed to the fact that KHA contains condensed aromatic ring structures,16 too much Equal KHA in



- - - Hydrogen bond # Humate acid ====== Complexating bond

Scheme 1 Illustration of structures of PAA-AM and PAA-AM/KHA: (a) hydrogen bond in the polymeric network of PAA-AM in water, (b) KHA particles in the polymeric network of PAA-AM/KHA, and (c) complexing bond between the carboxylate groups and metal ions in the polymeric network of PAA-AM in a saline solution.

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Figure 2 Swelling rate in distilled water for PAA-AM/KHA and PAA-AM [MBA/AA = 0.0005 mol/mol, KPS/AA = 0.01 mol/mol, KOH/AA = 0.6 mol/mol, AM/AA = 0.05 mol/mol, KHA/(AA + AM) = 6.6 wt %].

the composite leads to too many hydrophobic regions, and the hydrophilicity of the PAA-AM/ KHA composite decreases; then, the corresponding superabsorbent composite shrinks.^{16,18,21}

The variation of the water absorbency with an increasing amount of KHA indicates that a suitable content is needed for the PAA-AM/KHA composite to acquire the maximum water absorbency. The composite incorporated with 6.6 wt % KHA has the highest water absorbency of 769 g/g in distilled water under the experimental conditions in this study. The water absorbency of the composite incorporated with 14 wt % KHA is still higher than that of PAA-AM, and this may reduce the costs of super-absorbents and find practical use in agriculture.^{17,22}

Swelling rate

The swelling rate for PAA-AM/KHA and PAA-AM in distilled water is shown in Figure 2. For PAA-AM/KHA, the swelling rate is much higher in 0–10 min, and more than 89.6% of the maximum swelling capacity is achieved within 20 min. After 20 min, the swelling rate becomes lower, and the equilibrium water absorbency is reached approximately at 40 min. However, for PAA-AM, 60 min is needed to achieve 90.1% of the maximum swelling capacity, and about 110 min is needed to reach the equilibrium water absorbency. It has been reported that the swelling rate of a superabsorbent is primarily due to the penetration of water into the polymeric network through diffusion and capillarity.¹⁰ The higher swelling rate for PAA-AM/KHA may be attributed to the fact that the network of PAA-AM/KHA is looser and the capillarity is more evident because of the KHA particles; this accelerates the penetration of water molecules into the polymeric network.

Reswelling capability

It has been reported that introduced functional components (sodium humate) can improve the reswelling capability of PAA superabsorbents.^{16,18,21} Here the reswelling capabilities of PAA-AM/KHA and PAA-AM in distilled water are compared. The results are shown in Figure 3 as a function of the reswelling times. The water absorbency for PAA-AM/KHA and PAA-AM decreases with an increase in the reswelling times, and the resulting dry samples after several periods of reswelling still retain a certain degree of water-absorbing capability. The water absorbency of PAA-AM/KHA decreases from 769 to 311 g/g, and 40.4% of its initial water absorbency is retained after it reswells five times. For PAA-AM, the water absorbency decreases from 620 to 160 g/g, and only 25.8% of its initial water absorbency is retained. The influence of the reswelling times on the saturated absorbency is less evident for PAA-AM/KHA and becomes more observable for PAA-AM. This result indicates that a moderate content of KHA can improve the reswelling capability as it enhances the equilibrium water absorbency and swelling rate of the corresponding PAA-AM/KHA composite.

Water absorbency in various saline solutions

Considering the great impact of saline solutions on the water absorbency of superabsorbents and applications especially for agriculture and horticulture, we investigated the swelling property of PAA-AM/ KHA in various salt solutions. Table II shows the water absorbencies of PAA-AM/KHA and PAA-AM in univalent salt solutions (KCl_(aq), NaCl_(aq), and NH₄Cl_(aq)), bivalent salt solutions (CaCl_{2(aq)}, and



Figure 3 Reswelling capabilities of PAA-AM/KHA and PAA-AM in distilled water [MBA/AA = 0.0005 mol/mol, KPS/AA = 0.01 mol/mol, KOH/AA = 0.6 mol/mol, AM/AA = 0.05 mol/mol, KHA/(AA + AM) = 6.6 wt %].

Saline solution	Concentration (mol/L)	Q (g/g)			
		PAA-AM	PAA-AM/HA		
Na ⁺	0.005	358	486		
	0.01	305	427		
	0.02	284	401		
	0.05	246	372		
	0.1	220	350		
K^+	0.005	360	486		
	0.01	319	429		
	0.02	291	401		
	0.05	252	372		
	0.1	220	351		
$\rm NH_4^+$	0.005	358	486		
	0.01	306	427		
	0.02	285	400		
	0.05	248	372		
	0.1	220	350		
Mg^{2+}	0.005	407	271		
	0.01	351	234		
	0.02	321	177		
	0.05	300	173		
	0.1	286	173		
Ca^{2+}	0.005	396	264		
	0.01	348	191		
	0.02	316	179		
	0.05	280	172		
	0.1	260	168		
Al^{3+}	0.005	128	100		
	0.01	63.0	26.0		
	0.02	28.0	8.80		
	0.05	6.10	2.05		
	0.1	0.70	0		
Fe ³⁺	0.005	120	98.0		
	0.01	50.0	20.0		
	0.02	24.0	5.00		
	0.05	2.10	1.05		
	0.1	0.50	0		

TABLE II Water Absorbencies of PAA-AM/KHA and PAA-AM in Various Saline Solutions

MgCl_{2(aq)}), and trivalent salt solutions (AlCl_{3(aq)} and FeCl_{3(aq)}) with various concentrations.

It can be seen from Table II that the water absorbency of both PAA-AM/HA and PAA-AM decreases with increasing concentrations of various univalent salt solutions. When the concentration increases from 0.005 to 0.1 mol/L, the water absorbency of PAA-AM/KHA decreases from 486 to 350 g/g, and for PAA-AM, the water absorbency decreases from 358 to 220 g/g. PAA-AM/KHA acquires higher water absorbency in univalent salt solutions of the same concentration in comparison with PAA-AM.

The higher water absorbency for PAA-AM/KHA in univalent salt solutions may be due to the fact that there is a lot of K^+ in KHA particles, and the introduction of KHA into PAA-AM will increase the concentration of the charge of the composite

network, as shown in Scheme 1(b). When PAA-AM/ KHA is swelling in water, the additional K⁺ in the PAA-AM/KHA network will increase the osmotic pressure difference between the composite and the external aqueous solution, and this will result in an increase in the water absorbency. A similar observation was previously reported by Li and coworkers.^{9,18,21} It can be seen that univalent cations (K⁺, Na⁺, and NH₄⁺) of the same concentration have a similar effect on the water absorbency for PAA-AM/KHA and PAA-AM and have no relationship with the radius of the cation. This is also in accordance with several previous investigations.^{9,18,24}

Table II also shows the water absorbencies of PAA-AM/KHA and PAA-AM in bivalent solutions (CaCl_{2(aq)} and MgCl_{2(aq)}). The water absorbency of PAA-AM/KHA and PAA-AM decreases with the increase in the concentration, and PAA-AM/KHA acquires higher water absorbency in bivalent salt solutions of the same concentration in comparison with PAA-AM. This behavior is in agreement with that in univalent salt solutions. As can be seen from Table II, for PAA-AM/HA, the water absorbency decreases from 407 to 286 g/g and from 396 to 260 g/g, and for PAA-AM, the water absorbency decreases from 271 to 173 g/g and from 264 to 165 g/g, with the concentration increasing from 0.005 to 0.1 mol/L in MgCl₂ and CaCl₂ solutions, respectively.

This result is due to the complexing ability of the carboxylate group to the cations. KHA, attached to the surface of PAA-AM/KHA, can hinder the formation of a complex between the carboxylate groups and bivalent metal ions (e.g., Mg^{2+} or Ca^{2+}), which produces much more crosslinking density of the polymeric network and decreases the water absorbency of PAA-AM, as shown in Scheme 1(c). The complexing ability of the carboxylate group to Mg^{2+} is larger than that of Ca^{2+} .^{9–21} Therefore, the water absorbency of the superabsorbents in aqueous $MgCl_2$ is higher than that in aqueous $CaCl_2$.

Trivalent salt solutions (AlCl_{3(aq)} and FeCl_{3(aq)}) with various concentrations were also selected to investigate the decrease in the water absorbency for PAA-AM/KHA and PAA-AM. It can be seen from Table II that for PAA-AM/HA, the water absorbency decreases from 128 to 0.7 g/g and from 120 to 0.5 g/g, and for PAA-AM, the water absorbency decreases from 100 to 0 g/g and from 98 to 0 g/g, with the concentration increasing from 0.005 to 0.1 mol/L in AlCl₃ and FeCl₃ solutions, respectively. The water absorbency of PAA-AM/KHA and PAA-AM decreases with the increase in the concentration of trivalent salt solutions, and the behavior is in agreement with that in univalent and bivalent salt solutions.

Table II reveals that the water absorbency of the samples in trivalent cationic solutions is lower than

that in bivalent cationic solutions with an equal concentration. The higher water absorbency of the samples in bivalent cationic solutions (CaCl_{2(aq)} and MgCl_{2(aq)}) versus trivalent cationic solutions $(AlCl_{3(aq)} and FeCl_{3(aq)})$ can also be attributed to their smaller formation constants with carboxylate groups. The logarithm of formation constants of ethylene diamine tetraacetic acid with multivalent cations is 8.7, 10.69, 16.30, and 25.10 for Mg²⁺, Ca²⁺, Al³⁺, and Fe³⁺, respectively.^{6,9} Hence, the higher the formation constant is, the stronger the complexation is, and the lower the water absorbency is. Therefore, it is obvious that the water absorbency of the composite in various cationic solutions is in the order of $NH_4Cl_{(aq)} = NaCl_{(aq)} =$ $KCl_{(aq)} > MgCl_{2(aq)} > CaCl_{(aq)} > AlCl_{3(aq)} >$ $FeCl_{3(aq)}$ at a given concentration.

In other words, the water absorbency of the superabsorbent composite decreases with the increasing ionic strength of various external saline solutions. According to Li et al.,¹⁰ the relationship between the swelling ability of a superabsorbent and the ionic strength of the external solution is as follows:

$$Q = A + BI^{-1/2}$$
 (2)

where Q is the saturated absorbency, I is the ionic strength of the external solution, and A and B are empirical parameters. According to this equation, the saturated water absorbency for a superabsorbent (Q) is in direct proportion to the minus square root of the ionic strength of the external solution ($I^{-1/2}$).

The water absorbency for PAA-AM/KHA and PAA-AM against the ionic strength of various salt solutions is shown in Figure 4(a–c). For PAA-AM/ KHA, there are linear relationships between Q and $I^{-1/2}$ all through the range of $I^{-1/2}$ investigated in various salt solutions. For PAA-AM, there are some discrepancies in bivalent and trivalent salt solutions, as shown in Figure 4(b,c). This phenomenon is also attributed to the fact that the carboxylate groups on the PAA-AM network can induce the formation of complexes with bivalent and trivalent cations (e.g., Ca^{2+} and Al^{3+}), which produce much more crosslinking density of the polymeric network and affect the linear relationship between Q and $I^{-1/2}$. The stronger the complexation is, the more discrepancy there is from the linear relationship. It is obvious that the water absorbency of the composite in various cationic solutions is in the order of $NH_4Cl_{(aq)} = NaCl_{(aq)} = KCl_{(aq)} > MgCl_{2(aq)} >$ $CaCl_{(aq)} > AlCl_{3(aq)} > FeCl_{3(aq)}$ at a given concentration. Therefore, it can be concluded from Table II and Figure 4 that PAA-AM/KHA has a better salt-resistant ability than PAA-AM in various salt solutions.



Figure 4 Water absorbency plots for PAA-AM/KHA (6.6 wt % KHA) and PAA-AM against the ionic strength of saline solutions: (a) univalent salt solutions of KCl, NaCl, and NH₄Cl, (b) bivalent salt solutions of MgCl₂ and CaCl₂, and (c) trivalent salt solutions of AlCl₃ and FeCl₃ [MBA/AA = 0.0005 mol/mol, KPS/AA = 0.01 mol/mol, KOH/AA = 0.6 mol/mol, AM/AA = 0.05 mol/mol, KHA/(AA + AM) = 6.6 wt %]. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley. com.]

CONCLUSIONS

The effects of the amount of KHA on the swelling properties of the PAA-AM/KHA superabsorbent composite were investigated. The polymeric network of PAA-AM/KHA is improved by the introduction of a moderate amount of KHA into the PAA-AM polymeric system. The investigation reveals that the PAA-AM/KHA composite incorporated with 6.6% KHA exhibits a higher water absorbency, better swelling rate, and better reswelling ability than PAA-AM. The impact of various saline solutions on the water absorbency of PAA-AM/KHA correlates with the ionic strength and complexing ability of hydrophilic groups with the cations. The water absorbency of PAA-AM/KHA in various cationic solutions is in the order of $NH_4Cl_{(aq)} = NaCl_{(aq)} =$ $KCl_{(aq)} > MgCl_{2(aq)} > CaCl_{(aq)} > AlCl_{3(aq)} >$ FeCl_{3(aq)}, and there is a linear relationship between Q and $I^{-1/2}$. The salt-resistant property of the PAA-AM/KHA composite is enhanced compared with that of PAA-AM. Therefore, it could be more suitable for agriculture applications.

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