

Study on superabsorbent composite. IV. Effects of organification degree of attapulgite on swelling behaviors of polyacrylamide/organo-attapulgite composites

Junping Zhang ^{a,b}, Hao Chen ^{a,b}, Aiqin Wang ^{a,*}

^a Center of Ecological and green Chemistry, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou, Gansu 730000, PR China

^b Graduate School of the Chinese Academy of Sciences, Beijing 100049, PR China

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Abstract

A novel kind of superabsorbent composite, polyacrylamide/organo-attapulgite, from acrylamide (AM) and attapulgite (APT) was prepared by aqueous polymerization, using *N,N'*-methylenebisacrylamide (MBA) as a crosslinker and ammonium persulfate (APS) as an initiator. APT was organified with five different degree of hexadecyltrimethyl ammonium bromide (HDTMABr), and the organification degree of APT was proved by FTIR, TGA and XRD. The effects of organification degree of APT on water absorbency and swelling rate of the superabsorbent composite in distilled water and in various saline solutions were investigated in this study. The effect of organification degree on reswelling ability of the superabsorbent composites was also investigated. The results indicate that the organification degree of APT had remarkable influence on swelling behaviors of the superabsorbent composites. The superabsorbent composite acquired its highest water absorbency when the organification degree of APT is 8.02 wt.%.

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1. Introduction

Superabsorbents are loosely crosslinked hydrophilic polymers that can absorb, swell and retain aqueous fluids up to thousands of times their own weight. Due to excellent properties to traditional water absorbing materials (such as sponge, cotton and pulp, etc.)

superabsorbents are widely used in many fields, such as hygienic products, horticulture, gel actuators, drug-delivery systems, as well as water-blocking tapes and coal dewatering [1–6]. Various methods has been tried to improve absorbing properties and to expand application fields of superabsorbents [7,8], since US Department of Agriculture reported the first superabsorbent polymer [9]. Recently, much attention has been paid to inorganic materials for the preparation of superabsorbent composites, such as montmorillonite [10,11], attapulgite [12,13], kaolin [14], mica [15,16], bentonite and sepcite [17]. The incorporation of these clays not only

* Corresponding author. Tel.: +86 9314 968118; fax: +86 9318 2770 88.

E-mail address: aqwang@lzb.ac.cn (A. Wang).

reduce production cost, but also improve the properties (such as swelling ability, gel strength, mechanical and thermal stability) of superabsorbents and accelerate the generation of new materials for special applications [18].

Attapulgite, a kind of hydrated octahedral layered magnesium aluminum silicate absorbent mineral with reactive –OH groups on its surface, is less sensitive to salts comparing with other clays (such as smectite) [19]. As an extension of our previous work on attapulgite based superabsorbent composites [12,13,20,21], the organo-APT with different organification degree was introduced into the PAM polymeric network and the effects of organification degree of APT on the comprehensive swelling behaviors of the superabsorbent composites were investigated in this study.

2. Experimental

2.1. Materials

Acrylamide (analytical grade, supplied by Shanghai Chemical Factory, Shanghai, China) was purified by method as reported [22]. Ammonium persulfate as an initiator was supplied by Xi'an Chemical Reagent Factory (Xi'an, China), and *N,N'*-methylenebisacrylamide as a crosslinker was obtained from Shanghai Chemical Reagent Corp. (Shanghai, China). Hexadecyltrimethyl ammonium bromide, purchased from Beijing Chemical Reagent Factory, was used directly as received. Attapulgite micropowder, supplied by Linze Colloidal Co. (Gansu, China), was milled through a 250-mesh screen. Other agents used were all analytical grade and all solutions were prepared with distilled water.

2.2. Preparation of organo-APT

Organo-APT of different organification degree was prepared as follows: five different amount of HDTMABr were dissolved in 40 ml distilled water, respectively, and then 4.0 g APT was suspended in the above solution. The suspension was stirred vigorously at room temperature for 8 h, and then the organo-APT (HDTMA-APT) was formed. The separated HDTMA-APT was washed with large volume of distilled water to remove excess HDTMABr (until no bromide can be detected by 0.1 N AgNO₃ solution in the filtrate), and then dried in an oven at 70 °C for 6 h until the weight was constant. The organification degree of HDTMA-APT was determined by thermogravimetric analysis (Perkin–Elmer TGA-7 thermogravimetric analyzer (Perkin Elmer Cetus Instruments, Norwalk, CT), with a temperature range of 25–800 °C at a heating rate of 10 °C min^{−1} using a dry nitrogen purge at a flow rate of 50 ml min^{−1}).

2.3. Preparation of the superabsorbent composites

A series of superabsorbent composites from AM and HDTMA-APT micropowder of different organification degree were synthesized according to the following procedure. The crosslinker, MBA (10.3 mg), and AM monomer (7.10 g) were introduced into a 250 ml four-neck flask, equipped with a stirrer, a reflux condenser, a thermometer and a nitrogen line. The mixture was stirred until the MBA was dissolved completely, and then appropriate amount of HDTMA-APT with different organification degree was dispersed in the mixed solution. After being purged with nitrogen for 30 min to remove the oxygen dissolved from the solution, the mixed solution was heated to 40 °C gradually, and then the initiator, APS (40.5 mg), was introduced into the flask. The solution was stirred vigorously and a nitrogen atmosphere was maintained all through the polymerization of 3 h, and then 30.0 ml of sodium hydroxide solution (2 M) was added and the primary product was heated to approximate 95 °C to be saponified for 2 h. After saponification, the product was immersed in excess distilled water and then filtered for several times to remove any unreacted reactants until pH 7 was achieved. The depurative product was dried in an oven at 70 °C until the weight of the product was constant and an ashen polymer was obtained. The product was milled and all samples used for test had a particle size in the range of 40–80 mesh.

2.4. Measurement of water absorbency and swelling rate

Sample (0.05 g) was immersed in excess distilled water (500 ml) at room temperature for 4 h to reach swelling equilibrium. Swollen sample was then separated from unabsorbed water by filtering through a 100-mesh screen. The water absorbency of the superabsorbent composite, Q_{H_2O} , was calculated using the following equation:

$$Q_{H_2O} = \frac{m_2 - m_1}{m_1} \quad (1)$$

where m_1 and m_2 are the weights of the dry sample and the swollen sample, respectively. Q_{H_2O} is calculated as grams of water per gram of sample.

Swelling rate of the superabsorbent composite was measured according to the previously reported method [23].

2.5. Water absorbency in various saline solutions

Superabsorbent (0.10 g) composite was immersed in 250 ml of various saline solutions with different concentration (NaCl_(aq), MgCl_{2(aq)}, CaCl_{2(aq)}, FeCl_{3(aq)}) for 4 h to maintain equilibrium. The swollen samples was filtered through a 100-mesh screen and weighted. The

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