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Synthesis and characterization of poly(sodium acrylate)/bentonite superabsorbent composite

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Abstract. Poly(sodium acrylate)/bentonite superabsorbent composite with water absorbency $1562 \text{ g}\cdot\text{g}^{-1}$ was synthesized by inverse suspension polymerization. The introduction of bentonite improves the water absorbency and facilitates the particle size even distribution of the composite. The network structure in the superabsorbent hydrogel is confirmed.

1. Introduction

Superabsorbent polymer is a special polymeric material with hydrophilic groups and loosely crosslinked networks, it is able to absorb hundreds to thousands times its own mass of water, and the absorbed water is hardly removed under some pressure. Because of its excellent properties, superabsorbent polymer has aroused considerable research and applied in the field of medicine, healthcare, agriculture, artificial intelligence materials, and so on [1-4], since the first superabsorbent polymer was reported by the Department of Agriculture U. S. in 1976 [5].

Clay is a layered aluminium silicate with exchangeable cations in its interlayer and active OH groups on its surface. It is expected that the network structure and hydrophilic groups of the superabsorbents are improved by polymerizing polymer with clay. Consequently, comprehensive absorbent property of the superabsorbent is improved and the production cost is reduced [6-9]. In previous research [10, 11], we reported the synthesis and swelling behaviours of polymer/clay superabsorbent composites prepared by aqueous solution methods. But, due to high water absorbency for superabsorbent composite, the abundant water absorbed in the composite is hardly removed from the products in the production process of superabsorbent composite by aqueous polymerization. In order to overcome the problem, poly(sodium acrylate)/bentonite superabsorbent composite is synthesized by inverse-suspend polymerization method, the superabsorbent composite shows a water absorbency of $1562 \text{ g}\cdot\text{g}^{-1}$.

2. Experimental part

2.1. Materials

Bentonite micropowder (Linan Bentonite Factory, Zhejian, China) milled through 320-mesh screen, then, dried at 105°C for 24 h prior to use. Other agents were purchased from Shanghai Chemical Reagent Factory, Shanghai, China. Acrylic acid monomer was purified by freezing before use.

Initiator potassium persulfate, crosslinker N, N'-methylenebis acrylamide, solvent cyclohexane, surfactant span 60 (sorbitan monostearate) and sodium hydroxide were used without further purification. All solutions were prepared by using distilled water as solvent.

2.2. Preparation of poly(sodium acrylate)/bentonite superabsorbent composite

Poly(sodium acrylate)/bentonite superabsorbent composite was prepared by inverse suspension polymerization according to following steps. A sodium acrylate solution was prepared by slowly dropping acrylic acid monomer into a sodium hydroxide solution to achieve a desired neutralization degrees at 0 °C (cooled by ice-water bath) under stirring. A predetermined amount of bentonite micropowder was added to the sodium acrylate solution. Then, solvent cyclohexane and surfactant span 60 were added. Under a nitrogen atmosphere, the crosslinker N, N-methylenebisacrylamide and the initiator potassium persulphate were added to the above mixture solution. The mixed solution was stirred at room temperature for 30 min, and heated slowly to 65 °C with a water bath under stirring. After the polymerization reaction was carried on for 90 min, a jell-like intermediate product was filtered out, washed with methanol three times and dried in an oven at 70 °C more than 24 h to a constant weight, then milled and screened, a powdered poly(sodium acrylate)/bentonite superabsorbent composite product thus was obtained.

2.3. Characterization and measurement

The morphology of sample was observed with an S-3500N SEM instrument (Hitachi, Japan). The water absorbency of the sample was measured by immersing the dried powdered superabsorbent composite of 3 g in distilled water of 500 ml at room temperature for at least 3 h to reach swelling equilibrium, which resulted in the absorption of water inside the superabsorbent composite and the formation of a swollen hydrogel. Unabsorbed water was separated from the swollen sample by filtered over a 100-mesh screen. The water absorbency (Q_{H_2O}) of the sample was determined according to the following equation:

$$Q_{H_2O} = \frac{(m_2 - m_0) - (m_1 - m_0)}{(m_1 - m_0)} = \frac{m_2 - m_1}{m_1 - m_0} \quad (1)$$

Where m_0 , m_1 and m_2 are the weights (g) of the clay in the sample, the dry sample and the swollen sample, respectively.

3. Results and Discussion

3.1. Influence of bentonite amount on the water absorbency of the superabsorbent composite

The influence of the amount of bentonite powder in poly(sodium acrylate)/bentonite composite on the water absorbency (Q_{H_2O}) is shown in Figure 1. It can be seen that the Q_{H_2O} increases from 1330 to 1562 g·g⁻¹ when the bentonite percentage in the composite increases from 0 to 20 wt.%. Beyond a bentonite amount of 20 wt.%, the Q_{H_2O} decreases. The composite is formed by polymerizing acrylate onto bentonite. Bentonite powder as a network point plays an important role in the formation of the composite. [9, 10, 12, 13] When the amount of bentonite is suitable, such as amounts of 0–20 wt.%, the crosslinking density of the composite does not change or only changes a little, the superabsorbent composite has enough space to absorb and hold water molecules. However, with the increase of bentonite amount in the composite, such as higher than 20 wt.%, the composite's crosslinking density increases and network space shrinks, which results in the difficult permeation of water molecules into the composite. Thereby, the Q_{H_2O} value decreases with the increase of bentonite percentage (higher than 20 wt.%) in the composite.

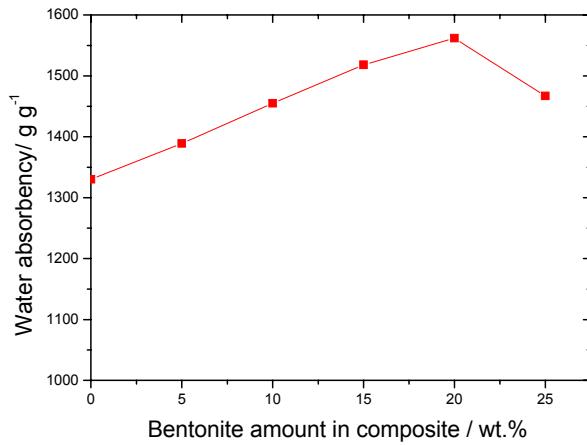


Figure 1. Variation of Q_{H2O} with bentonite amount in poly(acrylate sodium)/bentonite composite. Initiator of 0.2 wt.%, crosslinker of 0.01 wt.%, and polymerization at 60 °C.

3.2. Size and shape of superabsorbent composite

Figure 2 shows the SEM images of poly(sodium acrylate) superabsorbent polymer (a) and poly(sodium acrylate)/bentonite superabsorbent composite (b). The polymer shows [Figure 2(a)] a spherical and ellipsoidal shape with a wide size distribution, the diameter of the small particles is 6-10 µm and the large particles is 100-120 µm. The composite [Figure 2(b)] shows a spherical shape with a narrow size distribution and the diameter in the range of 4-14 µm. The narrower size distribution for the composite is due to that bentonite micropowder acts as a dispersant in the inverse suspension polymerization process. The surface shapes of the particles in Figure 2 (b) are irregular and rough compared with the particles in Figure 2 (a), which maybe result from that bentonite powders are irregular and rough.

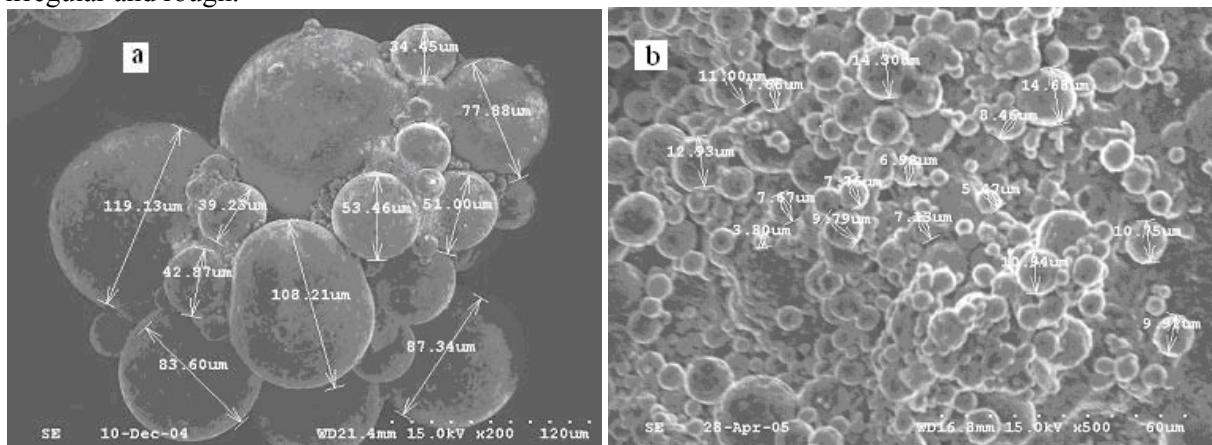


Figure 2. SEM images of (a) poly(sodium acrylate) polymer and (b) poly(sodium acrylate)/bentonite composite

3.3. Microstructure of superabsorbent

The interior morphology of the superabsorbent composite was observed with SEMs. In order to see the interior structure of superabsorbent clearly, two samples were choose. One was the sample just synthesized without drying and swelling, the SEM image is shown in Figure 3 (a); the other was the sample swollen fully at room temperature, keeping at 0 °C, measuring under vacuum at 0 °C, the SEM image is shown in Figure 3 (b). The interior morphology of dried superabsorbent composite is small and irregular pores [Figure 3 (a)]. In contrast, the superabsorbent composites hydrogel presents a

macroporous network structure [Figure 3 (b)]. Due to be not swollen fully, the polymer chain of the sample (a) shows a shranked state and small pores. On the other hand, due to be swollen fully at room temperature, the polymer chains of the sample (b) unfold fully, the space of network is filled with water. Then, the sample (b) is kept at 0 °C, the water is frozen inside of the polymer network and can not evaporate. Under vacuum and 0 °C measuring condition, the water in the polymer network evaporate but the swollen hydrogel's space structure is still kept, superabsorbent hydrogel shows its real structure, which presents a macroporous network structure. From Figure 3, the network structure of the superabsorbent hydrogel is confirmed.

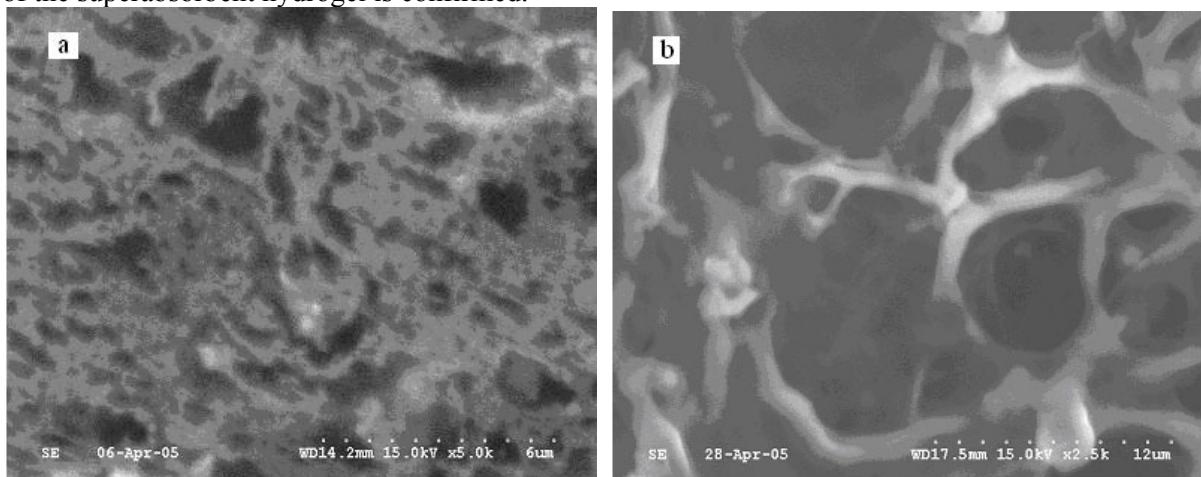


Figure 3. SEM images of dried superabsorbent composite (a) and fully swollen superabsorbent composite hydrogel (b).

4. Conclusion

In summary, poly(sodium acrylate)/bentonite superabsorbent composite with water absorbency of 1562 g·g⁻¹ was synthesized by inverse suspension polymerization. The introduction of bentonite improves the water absorbency and facilitates the particle size even distribution of the composite. The network structure in the superabsorbent hydrogel is confirmed.

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