

# Synthesis and Analysis of Novel Poly (2-hydroxyethyl methacrylate-acrylamid)/Clay Superabsorbent Nanocomposites

An-Feng Zhu<sup>1,2</sup>, Jun-Ming Dai<sup>1</sup>, Juan Xie<sup>1</sup>, Guang Li<sup>1\*</sup>, Jian-Ming Jiang<sup>1</sup>

<sup>1</sup>State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, Donghua University, Shanghai 201620, China

<sup>2</sup>Key Laboratory for Chemistry of Low-Dimensional Materials, Jiangsu Province, Huaiyin Normal University, Huaian, 223300, China

**Abstract:** A series of novel superabsorbent nanocomposites based on 2-hydroxyethyl methacrylate (HEMA) and acrylamid (AM) were prepared via in situ free radical polymerization, using clay (Laponite XLS) as a crosslinker and ammonium persulfate (APS) as an initiator. The structure and surface morphology of the superabsorbent nanocomposites were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). The water absorbency was found to be 612.5 and 182.6 g H<sub>2</sub>O g<sup>-1</sup> in distilled water and 0.2% NaCl solution, respectively. In addition, the superabsorbent nanocomposites reported here might be a potentially smart material in some range of applications including adsorption materials, superabsorbent fiber and drug-delivery devices.

**Keywords:** Superabsorbent, nanocomposite, clay, poly(HEMA-AM), in-situ, swelling.

## 1. Introduction

Hydrogel is a kind of three-dimensional crosslinked polymeric material and attracts much attention as functional soft materials. It not only absorbs hundreds to thousands times of its own mass of water to form a stable hydrogel, but also the absorbed water is hardly removed even under higher pressure [1]. Some hydrogels are found to reversibly change their volumes and shapes in response to external stimuli [2-5], such as pH, solvent composition, ionic strength, temperature, and electric field. Based on these environmentally responsive hydrogels, many biomedical and technological applications such as artificial implants, contact lens, pharmaceutical, and biosensors have been developed. However, these chemically crosslinked materials have low fracture toughness, which restricts their applications in biomedicine [6], organ engineering, and other fields [7-9]. Many efforts have been made to increase the mechanical strength of hydrogels by the introduction of inorganic nanoclay into organic polymer networks [10-12], in which the studies on mechanical and functional properties of hydrogels were highly significant.

In this study, a series of novel superabsorbent nanocomposites were designed and prepared by introducing a special kind of hectorite (Laponite XLS) into poly (2-hydroxyethyl methacrylate-acrylamide) network, in which the nanosized hectorite (Laponite

XLS) worked as the cross-linker instead of conventional chemical cross-linker. The structure and morphology of the superabsorbent nanocomposites were characterized by scanning electron microscopy, Fourier transform infrared spectrometry and differential scanning calorimetry. The swelling properties of the superabsorbent nanocomposites in both distilled water and 0.2%NaCl were also investigated. In addition, the superabsorbent nanocomposites reported here might be a potentially smart material in some applications, including coatings, cosmetics, superabsorbent fiber and drug delivery devices.

## 2. Experimental

### 2.1. Materials

2-Hydroxyethyl methacrylate (HEMA, TCI Chemical Co. Tokyo, Japan), was purified by vacuum distillation and stored at -5°C before use. Acrylamide (AM, chemically pure, Shanghai Fine Chemical Material Institute ), Synthetic hectorite Laponite XLS (Clay-S) (Rockwood, USA, 92.32 wt % [Mg<sub>5.34</sub>Li<sub>0.66</sub>Si<sub>8</sub>O<sub>20</sub>(OH)<sub>4</sub>]Na<sub>0.66</sub> and 7.68 wt % Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>), NMBA, Ammonium persulfate (APS, analytical grade, Shanghai Chemical Reagent Co.) were recrystallized from methanol and ethanol solvent, and *N,N,N',N'*-tetramethyldiamine (TEMED) (analytical reagent; Shanghai Chemical Reagent) were

\*Corresponding author's email address: lig@dhu.edu.cn  
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used as received. Deionized water used for all experiments was bubbled with N<sub>2</sub> gas for more than 3 h prior to use. All solutions used in the experiments were prepared in deionized water.

## 2.2. Preparation of poly (HEMA-AM)/clay superabsorbent nanocomposites

The novel superabsorbent nanocomposites based on 2-hydroxyethyl methacrylate (HEMA) and acrylamid (AM) were prepared via in situ free radical polymerization, using clay (Laponite XLS) as a crosslinker and ammonium persulfate (APS) as an initiator. A series of the samples were prepared by the following procedure:

Typically, powder clay-s (0.3 g) was added to 10 mL of deionized water in a four-neck flask equipped with a stirrer, a condenser, a thermometer and a nitrogen line, and then stirred on a water bath at room temperature for at least 10 h. Subsequently, the 2-hydroxyethyl methacrylate (HEMA, 0.4 g) and acrylamid (AM, 1.0 g) were added to the above mixture solution. Nitrogen was used to remove the dissolved oxygen from the reaction solution. To initiate the polymerization, 10 mL APS solution (5 wt% in deionized water) and 10 μL TEMED were added into the reactive system as an initiator and accelerator, respectively. The water bath was then heated slowly to 70 °C with vigorous stirring after which the radical initiator APS was introduced to the mixed solution. The compositions of hydrogels are listed in Table 1.

When the reaction completed, the product was cut into pieces of same size and immersed in deionized water repeatedly for 72 h to remove any residual monomer, and then dried in a vacuum oven at 70 °C to a constant weight. Thus the superabsorbent nanocomposites were prepared after the dried product was milled and screened. All samples used had a particle size in the range of 40-80 mesh.

Table 1 Feed composition of the superabsorbent nanocomposites

Codes	AM (g)	HEMA (g)	Clay (g)	APS (g)	TEMED (μL)
C3H4	1.0	0.4	0.3	0.015	10
C3H6	1.0	0.6	0.3	0.015	10

## 2.3. Characterizations

### 2.3.1 Measurement of swelling properties

The swelling ratios of superabsorbent nanocomposites

in distilled water and 0.2% NaCl were investigated. As-prepared product 0.05 g was immersed in excess distilled water or 0.2%NaCl solution at room temperature for 24 h to reach the swelling equilibrium. The swollen gels were filtered using a mesh sieve, and then drained on the sieve for 10 min until no free water remained. After weighing the swollen samples, the equilibrium water absorption was derived from the mass changes. The equilibrium swelling ratio (SR) was calculated using the following equation:

$$SR = (W_s - W_d) / W_d \quad (1)$$

where W<sub>s</sub> and W<sub>d</sub> are the weight of the swollen and dry samples, respectively. In all cases an average was made over three parallel samples.

### 2.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectra of samples, which were ground into fine powder after being dried under atmospheric conditions (25 °C) for 72 h and in a vacuum oven at 80 °C for 24 h, was taken as KBr pellets with Nicolet 8700 (Thermo Electron Co., USA) FTIR spectrophotometer in the range of 400-4000 cm<sup>-1</sup>.

### 2.3.3 Thermo Gravimetric Analysis (TGA)

Thermogravimetric analyze was carried out using a STA409PC (Netzsch Co.) instrument, heating samples from 50 to 1000 °C at a heating rate of 10 °C/min in an air flow. The clay content in the dried hydrogel was evaluated from the residual weight at 800 °C.

### 2.3.4 Interior Morphology (SEM)

The SEM analysis of samples was recorded on a JSM-5600 LV scanning electron microscope (Jeol, Japan). The as-prepared samples were quickly frozen in liquid nitrogen and freeze-dried under vacuum at -48 °C for 48 h. Then the freeze-dried samples were fractured carefully and coated with gold before observation.

### 2.3.5 X-ray diffraction (XRD)

XRD patterns of the sample were measured using Rigaku DMAX-2000 X-ray diffractometer with the Cu Kα radiation (k = 1.54056 Å) at a scanning rate of 2°/S in 2θ ranging from 2 to 20°. The sample for XRD was supported on glass substrates.