

Synthesis and Clay Stabilization of a Water-Soluble Copolymer Based on Acrylamide, Modular β -Cyclodextrin, and AMPS

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ABSTRACT: A modular β -cyclodextrin copolymer for clay stabilization was prepared from 2-O-(allyloxy-2-hydroxyl-propyl)- β -cyclodextrin (XBH), acrylamide (AM), 2-acrylamido-2-methyl propane sulfonic acid (AMPS), and sodium acrylate (NaAA) via redox free-radical copolymerization. The effects of reactive conditions (such as initiator concentration, monomer ratio, reaction temperature, and pH) on the apparent viscosity of the copolymer were investigated and the optimal conditions for the copolymerization were established. The copolymer obtained was characterized by infrared spectroscopy, scanning electron microscope, viscosity measurements, rheological measurement, core stress test, and X-ray diffractometry. The crystalline interspace of MMT could be reduced from 18.95323 Å to 15.21484 Å by copolymer AM/NaAA/AMPS/XBH. And this water-soluble copolymer also showed remarkable anti-shear ability, temperature resistance, and salt tolerance (1000 s⁻¹, viscosity retention rate: 35%; 120°C, viscosity retention rate: 75%; 10,000 mg/L NaCl, viscosity retention rate: 50.2%; 2000 mg/L CaCl₂, viscosity retention rate: 48.5%; 2000 mg/L MgCl₂, viscosity retention rate: 42.9%). © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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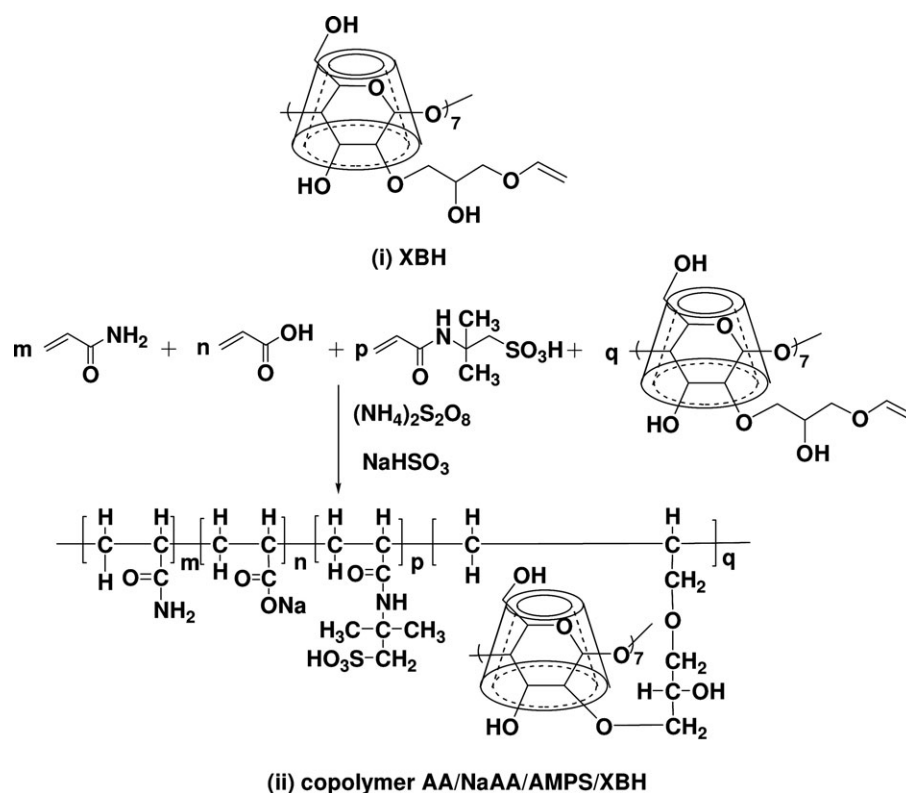
INTRODUCTION

The problems caused by shales in petroleum activities are not rare. It is important to maintain wellbore stability during drilling, especially in water-sensitive shale and clay formations. The rocks within these types of formations absorb the fluid used in drilling and this absorption causes the rock to swell and may lead to a wellbore collapse.¹ What's more, clay minerals in hydrocarbon reservoirs often cause formation damage. The well known examples are clay swelling, fines migration, and hydrothermal mineral reactions.^{2–10} Since the potential resource about shale gas/oil has been confirmed by many researches, the swell of shale has already been a key problem in this field. In the last few decades, the petroleum chemists have made a great effort to control/minimize the formation damage related to clay minerals.^{11–14} The studies of clay stabilizer become much more important as never before.

Clay stabilizers act in a chemical manner rather than in a mechanical manner. They change the ionic strength and the transport behavior of the fluids into the clays. Clay stabilizers retain the clay platelets in position by controlling the charge and electrolytic characteristics of the treatment fluid.^{15,16} At present, the

clay stabilizers based on chemical components can be divided into four categories: inorganic salts and alkali, inorganic polymer, cationic surfactant, and organic cationic polymers. As we know, these anti-clay swelling agents are based on the charge neutralization. To stabilize the clay, we could also explore a new way that building separation layers by adsorption on the surface of the clay which might prevent clay from water.¹⁴

In the petroleum industry, polyacrylamide (PAM) has been applied almost every aspect,^{17–25} and the application of 2-acrylamido-2-methyl propane sulfonic acid (AMPS) is pretty widespread.²⁶ And β -cyclodextrin (β -CD) is cyclic oligosaccharides consisting of seven glucose units, which can be obtained on a large scale from starch. The cavity of β -CD is hydrophobic nature and its rim is hydrophilic leading to the inclusion compound and molecular assembly system of β -CD with a plenty of organic or inorganic molecules by Van der Waals force, hydrophobic forces, and matching function between subject and object molecules.^{27–30} β -CD can be chemically modified for many different purposes: polymerized cyclodextrin or cyclodextrin bound to a polymer carrier have already been employed in gel inclusion and affinity chromatography.^{31,32} The hydrophobic



Scheme 1. (i) The structure of monomer XBH; (ii) The synthetic process of copolymer AM/NaAA/AMPS/XBH.

interactions of β -CD derivatives are so important for oilfield chemical additives.

Keeping in mind of these fundamental conditions, herein, modular β -CD named 2-O-(allyloxy-2-hydroxypropyl)- β -cyclodextrin (XBH) [see Scheme 1(i)] was synthesized. And a novel copolymer was prepared from acrylamide (AM), sodium acrylate (NaAA), AMPS, and XBH for clay stabilizer.^{33–37}

EXPERIMENTAL

Materials

1-Allyloxy-2, 3-epoxy propane (AGE) was purchased from Nanjing Forward Chemical (Nanjing/China). β -CD, AM, AMPS, acrylate (AA), sodium sulfite (NaHSO_3), and ammonium persulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) were purchased from Chengdu Kelon Chemical Reagent Factory Co., (Sichuan, China). XBH was synthesized according to the reported method.³⁸ Other chemicals and sodium montmorillonite (Xinjiang Xiazijie Bentonite, Xinjiang/China) were commercially available and used directly without further purification.

Synthesis of XBH

NaOH (1.5000 g) was dissolved in 100 mL deionized water. After the alkali liquor cooled to room temperature in the flask, 34.0500 g β -CD was added under constant stirring until the paste turning to clear. Drip 3.4242 g AGE slowly into the flask in 5 min. After the AGE was added, the mixture system changed into white paste again. Then the reaction was allowed to occur at 10–15°C about 5–8 h until the mixture is clear. Next, the solution was neutralized by 85% phosphoric acid, the system

changed into paste because of the β -CD remaining after reaction which would be filtrated later. After the filtrate was dissolved by ethanol, certain salt could be observed at the bottom which would also be filtrated. Finally, the filtrated liquid was distilled under reduced pressure, and a faint yellow paste liquid was obtained.³⁸

Preparation of Quaternary Copolymer (AM/NaAA/AMPS/XBH)

AM, AA, AMPS, and XBH were dissolved in degassed and distilled water in the indicated ratio to obtain an aqueous solution. Then NaOH was added into the solution to adjust the pH value and the solution was kept in water bath heater at indicated temperature. NaHSO_3 – $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (indicated loading, and 1/1 mol ratio) solutions were added slowly. The copolymerization which was shown in Scheme 1(ii) was then proceeded at indicated time under indicated conditions. The final solution was clear and highly viscous. The polymer was then isolated by precipitation with ethanol and dried in vacuum for 6–10 h to yield the corresponding quaternary copolymer AM/NaAA/AMPS/XBH. The compositions of the final copolymer based on monomer units were recorded using “m, n, p and q” in Scheme 1(ii) which were about 63.616%, 28.520%, 7.851% and 0.013% by means of mole ratio range, respectively.

Monomer conversion of AM was determined by high performance liquid chromatography technology (Shimadzu company, Japan) using ODS column at UV detector (210 nm), $\text{H}_2\text{O}/\text{CH}_3\text{OH} = 90/10$ (vol/vol). The conversion of AM was calculated with the following equation:

$$W\% = \frac{W_{AM} - \frac{AC_o \times V}{A_o}}{W_{AM}} \times 100\% \quad (1)$$

Here, $W\%$ is the conversion of AM, W_{AM} is the total weight of AM in the reaction, C_o is the concentration of standard sample of AM, A_o is the chromatographic peak area of standard sample of AM, A is the chromatographic peak area of the unreacted AM, and V is the solution volume of ethanol in which the copolymer was isolated by precipitation.

Infrared Spectroscopy

The IR spectra of XBH, copolymer AM/NaAA/AMPS/XBH were recorded by using a WQF-520 IR spectrophotometer. All samples were recorded as KBr pellets prepared by blending 2 mg of the XBH or copolymer with 200 mg KBr, previously dried at 105°C for 24 h. The infrared spectra were obtained in the range of 4500–500 cm^{-1} .

Scanning Electron Microscope

The 5000 ppm copolymer AM/NaAA/AMPS/XBH solution was scanned by scanning electron microscope at room temperature.

Rheological Characterization

The 5000 ppm copolymer AM/NaAA/AMPS/XBH solution was tested by HAAKE Rheo Stress 6000 rheometer (Thermo Fisher Scientific, Waltham/Massachusetts) to get the fluid thixotropic behavior and its viscosity–temperature characteristic.

Salt Tolerant Test

The salt resistance of copolymer AM/NaAA/AMPS/XBH solution was investigated by NaCl, CaCl_2 , and MgCl_2 with different concentrations, respectively.

Core Stress Test

Since the hydration of clay will lead the formation easier to collapse. The core stress of the formation sand sample which was soaked in the copolymer solution at 80°C for 24 h was researched. And the final data were compared with pure water and inorganic salt (KCl, NaCl, and CaCl_2). Then the copolymer solution was associated with inorganic salt to study the inhibition effect of the compound system.

X-Ray Diffractometry

It is the most important internal cause that clay mineral undergoes hydration expansion when the external water environment have changed leading to reduced strength of shale formation and inducing wellbore instability.^{39,40} To investigate the details of the effect, we choose sodium montmorillonite (MMT) which is the most common clay mineral in the stratum to study its crystalline interspace changes before and after swelling.^{41,42}

After drying, the MMT was prepared as 11 samples and each weighed 2 g. First, the crystalline interspace of dry MMT was measured. Then, the MMT sample was mixed with copolymer AM/NaAA/AMPS/XBH solution of which the concentration was confirmed by core stress test. And different salts (10 wt %; KCl, NaCl, and CaCl_2) were added into the copolymer solution with certain concentration, respectively. And these solutions would be mixed with new MMT samples. All above MMT samples were made into tablet and the crystalline interspace would be measured by X-ray diffraction (X' pert PRO, PANalytical B.V., The Netherlands).

Table I. Effects of the Initiator Concentration, pH, and Temperature

Entry ^a	Initiator (wt %)	pH	Temperature (°C)	Apparent Viscosity (mPa s) ^b	Conversion (%) ^c
1	0.2	7	40	– ^d	– ^d
2	0.3	7	40	– ^e	95
3	0.5	7	40	1068	95
4	0.7	7	40	980	93
5	0.9	7	40	960	92
6	0.5	5	40	940	90
7	0.5	7	40	1100	95
8	0.5	9	40	980	93
9	0.5	10	40	900	92
10	0.5	11	40	900	92
11	0.5	7	30	2729	90
12	0.5	7	35	3089	97
13	0.5	7	40	1070	95
14	0.5	7	45	1700	95
15	0.5	7	50	1560	95

^aConditions: AM/AA/AMPS/XBH = 5.600/2.400/1.980/0.020 (wt), concentration = 20% (wt %).

^bApparent viscosity: copolymer solution = 2000 ppm, tested by Brookfield DV-II + Pro Viscometer at 30°C.

^cConversion (wt %): AM, tested by HPLC using ODS column at UV detector (210 nm), $\text{H}_2\text{O}/\text{CH}_3\text{OH} = 90/10$ (vol/vol).

^dNo copolymerization.

^eCopolymer insoluble.

RESULTS AND DISCUSSION

The Optimization of Synthesis Conditions in Copolymerization

We first investigated the effect of initiator concentration, and the result shown in Table I (Entries 1–5) noted that the best loading was 0.5 wt % (Table I, Entry 3). In Entry 1, the condition was non-reactive. And copolymer carried out by Entry 2 could not be dissolved.

And then, the pH effect on copolymerization was studied. The best result could be afforded when the pH changed to 7 with the highest apparent viscosity (Table I, Entry 7). However, as the pH was increased or decreased, the results could not be improved in the quaternary copolymerization (Table I, Entries 6, 8–10).

Temperature is also a great important factor for polymerization. When the temperature is somewhat low, the molecular weight of copolymer would not be satisfied. However, it may lead to explosive polymerization while the temperature is too high. It was found that the best reaction temperature was 35°C in copolymerization (Table I, Entry 12). And other temperature conditions could not afford better results (Table I, Entries 11, 13–15).

The effect of different ratios of AM to AA in the synthesis of copolymer was investigated. The result is shown in Table II (Entries 1–5). The ideal ratio was 5.5/2.5(wt) (Table II, Entry 3).

AMPS is an ideal monomer to improve the salt and temperature tolerance of copolymer. However, over-dose would lead to

Table II. Effects of the Ratio of AM to AA, AMPS Loading, and XBH Loading

Entry ^a	AM (wt)	AA (wt)	AMPS (wt)	XBH (wt)	Apparent Viscosity (mPa s) ^b	Conversion (%) ^c
1	4.500	3.500	1.980	0.020	2569	93
2	5.000	3.000	1.980	0.020	2859.8	93
3	5.500	2.500	1.980	0.020	3759	97
4	6.000	2.000	1.980	0.020	2260	95
5	7.000	1.000	1.980	0.020	1750	95
6	6.310	2.870	0.800	0.020	- ^d	95
7	5.820	2.660	1.500	0.020	- ^d	95
8	5.480	2.500	1.980	0.020	4359	97
9	5.130	2.350	2.500	0.020	3819	95
10	4.800	2.180	3.000	0.020	3759	95
11	5.511	2.504	1.980	0.005	1857	93
12	5.509	2.503	1.980	0.008	2349	95
13	5.507	2.503	1.980	0.010	3782	95
14	5.500	2.500	1.980	0.020	4536	97
15	5.494	2.496	1.980	0.030	3987	85

^aConditions: initiator loading = 0.5 wt %, pH = 7, Temperature = 40°C, concentration = 20% (wt %).

^bApparent viscosity: copolymer solution = 2000 ppm, tested by Brookfield DV-II + Pro Viscometer at 30°C.

^cConversion (wt %): AM, tested by HPLC using ODS column at UV detector (210 nm), H₂O/CH₃OH = 90/10 (vol/vol).

^dCopolymer insoluble.

inhibition of polymerization. So the loading of AMPS is of crucial importance.

The best result could be afforded when the loading changed to 20 (wt %) with highest apparent viscosity (Table II, Entry 8). However, better results could not be obtained by other loadings (Table II, Entries 6, 7, 9, 10). The copolymers produced by conditions of Entries 1 and 2 could not be dissolved.

Functional monomer could drastically improve the property of copolymer. The loading was also a key factor to the copolymerization (Table II, Entries 11–15). In this experiment, the ideal loading was 0.2 wt % (Table II, Entry 13).

Analysis of XBH and Copolymer by IR Spectroscopy

The IR spectra of XBH and copolymer are displayed in Figure 1. The IR spectrum of XBH presented the expected characteristic bands: broad intense band at 3342 cm⁻¹ due to the stretching vibration of —OH, C=C stretching at 1675 cm⁻¹ which indicated that AGE had been reacted with β-CD, and the characteristic stretching of the β-CD ring at 1427 cm⁻¹, 1353 cm⁻¹, and 1282 cm⁻¹, C—O—C stretching vibration at 1035 cm⁻¹.

The IR spectrum of copolymer AM/NaAA/AMPS/XBH exhibited the following peaks: —OH and —CONH₂ stretching vibration at 3427 cm⁻¹ (compared with the peak at 3342.03 cm⁻¹ in the spectra of XBH, the peak was more wide which indicated that the peaks of —OH and —CONH₂ overlapped) suggesting AM and XBH were copolymerized to the chain of copolymer;

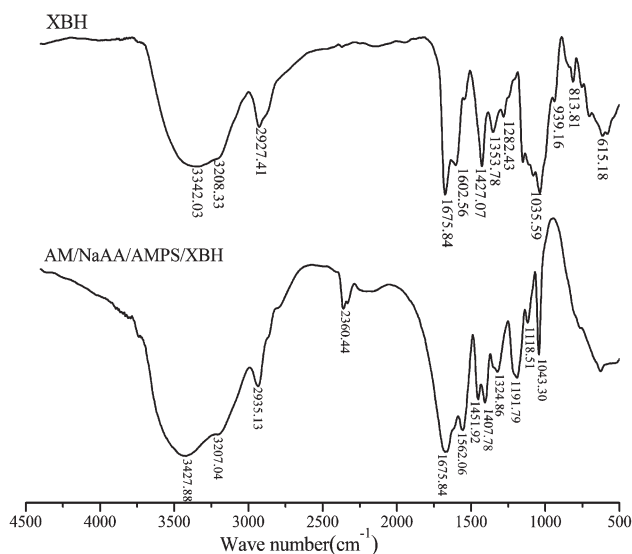


Figure 1. IR spectra of XBH and copolymer AM/NaAA/AMPS/XBH.

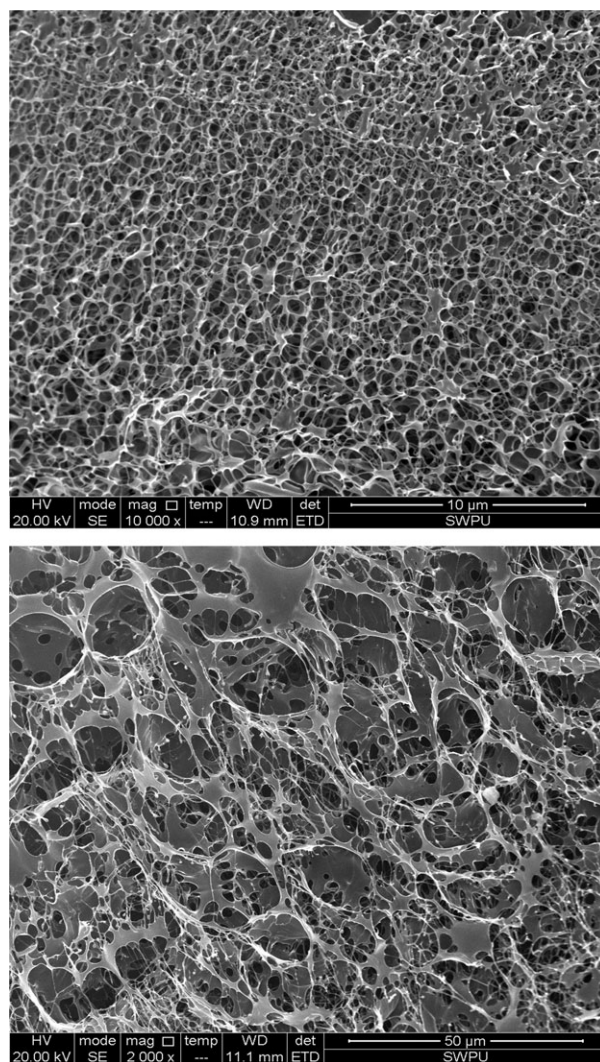


Figure 2. The SEM images of copolymer AM/NaAA/AMPS/XBH.

C=O stretching vibration at 1675 cm^{-1} ; the characteristic stretching vibration of the β -CD ring at 1451 cm^{-1} , 1407 cm^{-1} , and 1324 cm^{-1} , and —SO_3^- stretching vibration at 1191 cm^{-1} revealing the successful copolymerization of AMPS.

SEM Images of Copolymer

The scanning electron microscopy images of copolymer were shown in Figure 2, which pointed that β -CD modified copolymer was obvious gel-network structures. The cavity structure could be observed easily. This phenomenon could be related to the XBH branch on the main polymer chain.

Rheology and Temperature Resistance

Since the copolymer solution used in oilfield should employ a good anti-shear ability and temperature tolerance, the fluid characteristic has been measured.⁴³ The results in Figure 3(A) showed that the shear restoration was excellent. The retention rates of apparent viscosity could reach 35% when the shear rate was 1000 s^{-1} .

In Figure 3(B), the copolymer solution exhibited a good thermal stability. About 75% of the solution apparent viscosity was retained at 120°C .

Salt Tolerance

In Figure 4(A, B), with an increasing cations concentration in the polymer solution, the stretched polyelectrolyte chain started

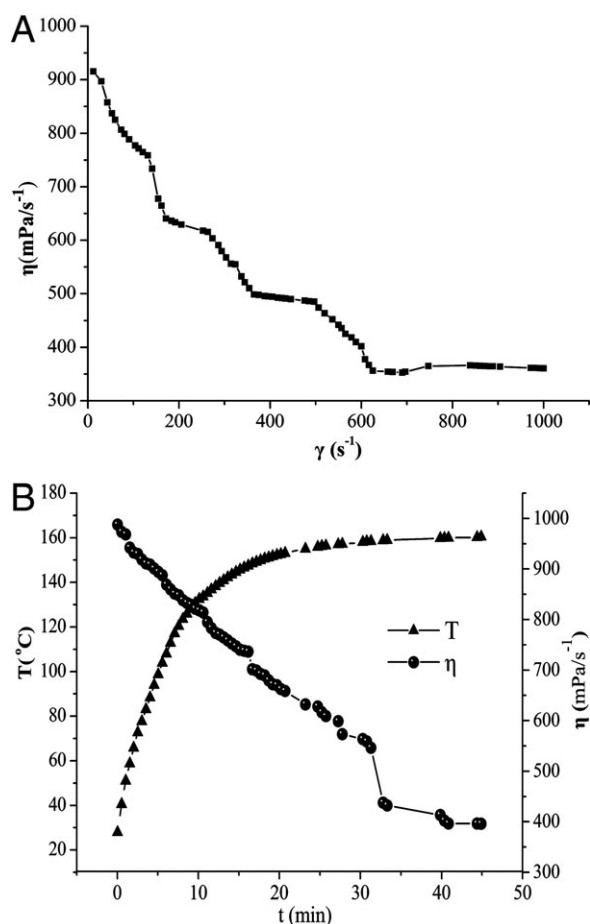


Figure 3. A: The shear-resistance character of copolymer AM/NaAA/AMPS/XBH; (B) The temperature-tolerance character of copolymer AM/NaAA/AMPS/XBH.

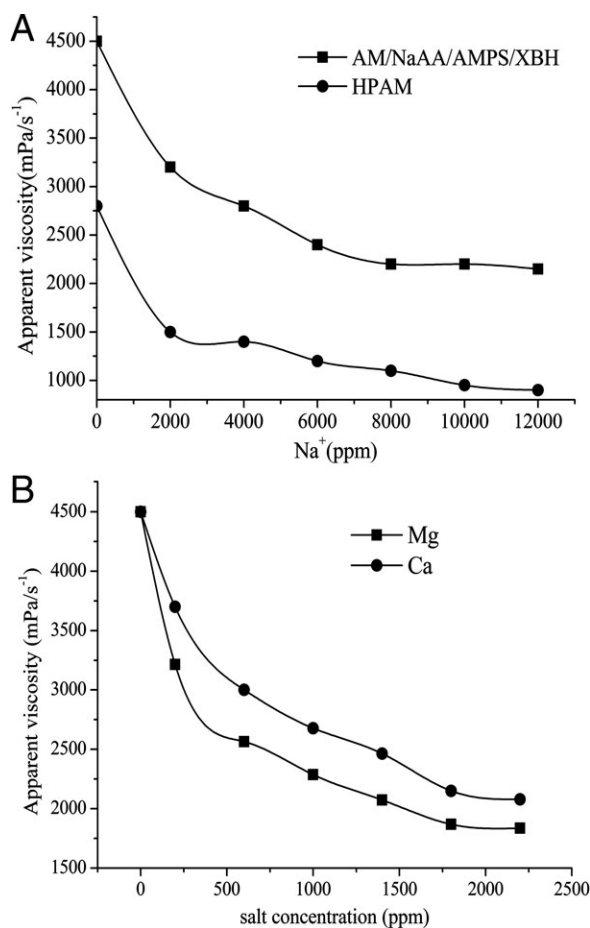


Figure 4. A: The anti-NaCl ability of copolymer AM/NaAA/AMPS/XBH; (B) The anti- CaCl_2 and anti- MgCl_2 abilities of copolymer AM/NaAA/AMPS/XBH.

shrinking because of a reduction in intra-anionic electrostatic repulsion. At a certain salt concentration, the amount of cations present was sufficient to complete the shrinking. Therefore, beyond that certain salt concentration, the addition of salt could not change the corresponding apparent viscosity more, and this is typical of a nonelectrolyte. From the data, good to excellent anti-NaCl ability could be obtained and the resistance of CaCl_2 and MgCl_2 was about up to the average.

Core Stress Analysis

The stress data are shown in Table III (Entries 1–11) and the situations of sand samples after soaking in different solutions are revealed in Figure 5. Comparing these sand samples, Figure 5(B) shows the picture of sand soaking after pure water in which most cracks could be found. Furthermore, Figure 5(C, I) is with the least cracks among the sands which have been soaked in solution. Accordingly, the copolymer's inhibition of clay swelling could be found obviously: 6000 ppm copolymer solution was the optimal concentration and the formulation of 6000 ppm copolymer with 10 wt % KCl was best for stabilizing clay. Thus, the prepared copolymer could be used as clay stabilizer.

X-Ray Diffractometry Analysis

According to Table III (Entries 12–20), the crystalline interspace of MMT could be obviously reduced from 18.95323 \AA to

Table III. The Analysis of Core Stress and the Crystalline Interspace Comparisons of Different MMT Samples

Entry ^a	Condition		Maximum load (KN)	Compaction hardness (MPa)	Crystalline interspace (Å)
	Sample	Formulation			
1	Core	Original (A)	2.1831	490.72	—
2	Core	Pure water (B)	0.5795	130.26	—
3	Core	KCl (10 wt %) (C)	1.8677	419.82	—
4	Core	NaCl (10 wt %) (D)	1.7172	385.99	—
5	Core	CaCl ₂ (10 wt %) (E)	1.2463	280.14	—
6	Core	Copolymer 2000 ppm (F)	0.6637	149.19	—
7	Core	Copolymer 6000 ppm (G)	1.5968	359.92	—
8	Core	Copolymer 10,000 ppm (H)	1.2515	281.32	—
9	Core	Copolymer 6000 ppm-KCl (10 wt %) (I)	1.9932	448.02	—
10	Core	Copolymer 6000 ppm-NaCl (10 wt %) (J)	1.4426	324.27	—
11	Core	Copolymer 6000 ppm-CaCl ₂ (10 wt %) (K)	1.1546	259.54	—
12	MMT	Original	—	—	12.63387
13	MMT	Pure water	—	—	18.95323
14	MMT	KCl (10 wt %)	—	—	15.45829
15	MMT	NaCl (10 wt %)	—	—	15.37453
16	MMT	CaCl ₂ (10 wt %)	—	—	18.15232
17	MMT	Copolymer (6000 ppm)	—	—	18.87169
18	MMT	Copolymer (6000 ppm)-KCl (10 wt %)	—	—	15.21484
19	MMT	Copolymer (6000 ppm)-NaCl (10 wt %)	—	—	15.35696
20	MMT	Copolymer (6000 ppm)-CaCl ₂ (10 wt %)	—	—	18.00884

^aTests: Entries 1-11 belong to core stress test; entries 12-20 belong to XRD analysis.

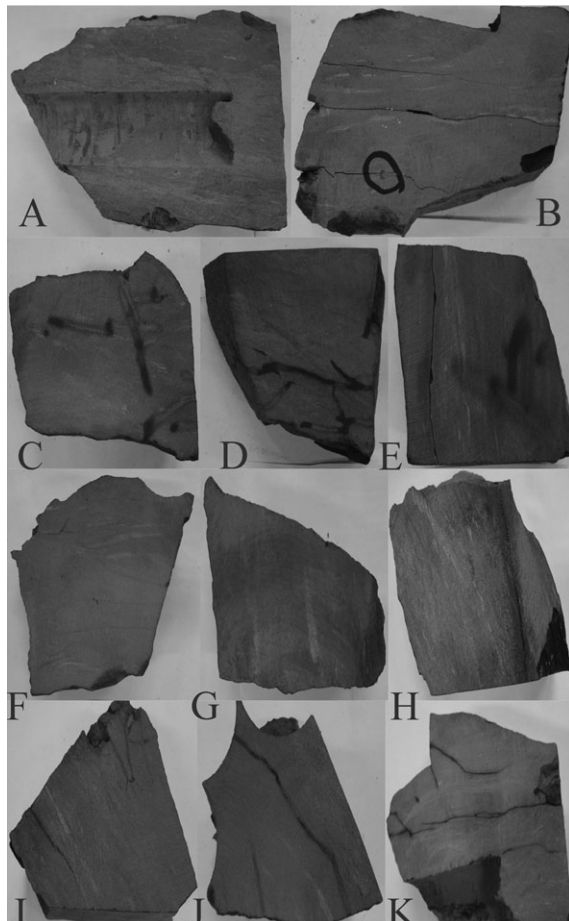


Figure 5. The states of sand samples after soaking in different solutions.

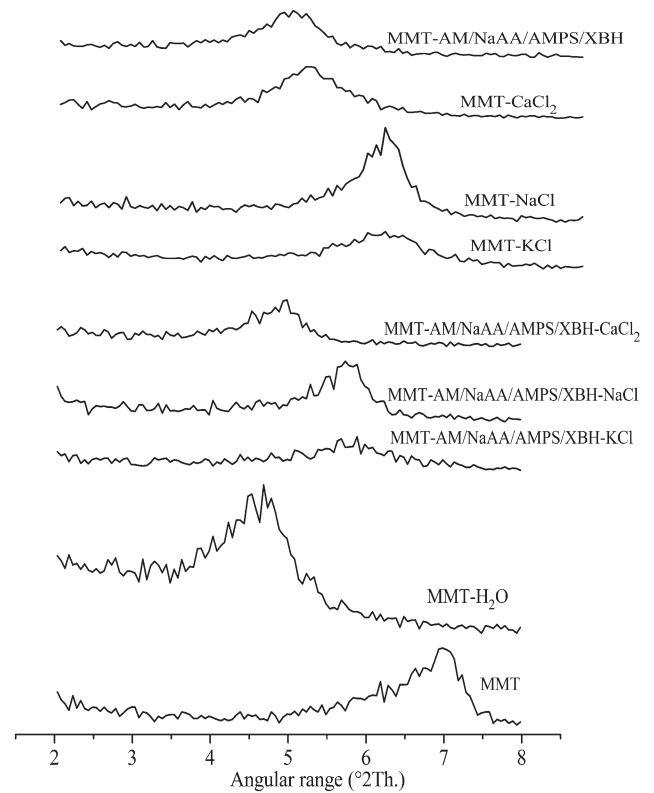


Figure 6. The crystalline interspace comparisons of different MMT samples.

15.21484 Å. When the 10 wt % NaCl, KCl, and CaCl₂ were employed in the copolymer, the crystalline interspace was smaller (15.21484 Å, 15.35696 Å, and 18.00884 Å) than the MMT-copolymer (6000 ppm) (18.87169 Å). In addition, this trend could be found from Figure 6. Considering the above results, the copolymer AM/NaAA/AMPS/XBH solution with salt could help stabilizing the well environment.

CONCLUSIONS

The modular XBH has been successfully copolymerized onto the copolymer chain by using redox system as initiator in aqueous medium. The SEM images reveal evidence of cavity structure which is the typical property of β-CD. Moreover, the viscosity, salt resistance, temperature tolerance, and anti-shear ability of the copolymer solution are improved remarkably. Core stress test and X-ray diffractometry collectively suggest that the synthesized copolymer AM/NaAA/AMPS/XBH could inhibit the swelling of clay to a certain degree and can be applied as clay stabilizer.

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