

Research Article

Preparation and Properties of Polymer/Vermiculite Hybrid Superabsorbent Reinforced by Fiber for Enhanced Oil Recovery

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Received 20 August 2014; Accepted 21 October 2014; Published 12 November 2014

Academic Editor: Nurettin Sahiner

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A series of polymer/clay hybrid superabsorbent composites (SACFs) comprising acrylamide, acrylic acid, sodium 2-acrylamido-tetradecyl sulfonate, fiber, and vermiculite by *in situ* intercalation and exfoliated method was successfully synthesized. The structure of SACFs was characterized by IR, SXRD, and SEM measurements. Much notable absorbency for SACF-2 was observed compared to that for SACF-1 in the absence of hydrophobic group in the high cationic solution due to the alkyl carbon chain and sulfonic acid group of hydrophobic moieties protecting the cations from attacking the carboxylate groups. What is more, high temperature fiber which acts as bridge connection for the polymeric network structure enhanced both toughness and strength for SACF-4 in the harsh conditions. At the total dissolved substance of 212000 mg/L for Tarim Basin injected water and the temperature of 120°C, desired absorbency as well as water retaining property for SACF-4 was observed during the long period of thermal ageing. Core flooding experiments demonstrated that SACFs could migrate as amoeba in the porous medium and accumulated in the narrow channel to adjust injection profile, promoting the subsequent water diverting into the unswept zones. Finally, characteristic parameters for SACFs calculated from flooding experiment further confirmed these polymer/clay hybrid composites reinforced by fiber would have robust application in the mature oilfield for profile control.

1. Introduction

As for the mature oilfield, oil production rapidly decreased with water cut from oil well increasing because of years of water flooding making reservoirs much more heterogeneous [1]. Polymeric matrix hybrid clay superabsorbent attracted considerably industrial interests due to their swelling and water retention in the porous medium. Moreover, such granular superabsorbent can migrate like amoeba in the reservoir, adjust injection profile, and prompt the subsequent water flowing into the fresh formation, implementing deep profile control [2, 3].

However, conventional polymeric networks such as polyacrylamide/montmorillonite (MMT), epoxy resin/MMT, and poly(acrylamide/acrylate)/kaoline composites suffer great deal in the harsh conditions with reservoir temperature

higher than 100°C and total salt concentration higher than 100000 mg/L being rich in divalent ions [4]. Such superabsorbent containing flexible groups, for instance, carboxyl group and amide pendent, displays uncontrolled swelling, poor toughness and quick water release. Such drawbacks limit particle gels' wide application in mature oilfields [5]. It is noted that hydrophobic groups modified polyacrylamide hybrid clay exhibits reasonable thermal endurance and salt resistance [6, 7]. What is more, interpenetrating polymeric network enhanced by nature materials, such as polysaccharide [8], fiber [9, 10], and rubber [6], shows more stability in mature formation, guaranteeing particle gels as good candidates for water shutoff and profile-control operations.

In this paper, alkyl sulfonate hydrophobic monomer modified polymeric vermiculite superabsorbent gel reinforced by fiber is preferentially synthesized. The structure of

TABLE 1: The basic compositional parameter (wt.%) for preparation of SACF.

Sample	Monomer composition			HTF (g)	CVMT (g)	Deionized water (g)	pH
	AM	AA	NaAMC ₁₄ S				
SACF-1	8.256	1.748	/	0.508	4.996	49.984	7.02
SACF-2	8.253	1.749	0.448	/	4.983	50.104	7.18
SACF-3	8.248	1.752	0.248	0.506	5.008	50.126	7.08
SACF-4	8.251	1.752	0.451	0.495	4.987	50.086	7.15

gels was characterized by Fourier transform infrared (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The effect of temperature and salt concentration on the water swelling and aged stable properties for particle gels has been investigated. Finally, three groups of core displacement experiments have been conducted to measure their shut-off capacity and profile control ability. To our knowledge, it is the first to report superabsorbent filled by fiber used as profile control agent for enhanced oil recovery.

2. Experimental Section

2.1. Materials. Unexpanded Vermiculite micropowder (UVMT, WeiFang Hongyi Colloidal Co., Ltd, China) milled through a 325-mesh screen was used, with a chemical composition of analytical SiO₂ 44.5 wt%, Al₂O₃ 15.2 wt%, Fe₂O₃ 14.3 wt%, MgO 16.7 wt%, CaO 4.02 wt%, TiO₂ 0.92 wt%, MnO 0.15 wt%, K₂O 3.65 wt%, Na₂O 0.33 wt%, and P₂O₅ 0.05 wt%. Acrylamide (AM) purchased from Chengdu KeLong Chemical Reagent Co., Ltd (China), was recrystallized twice from acetone and vacuum dried at room temperature prior to use. Acrylic acid (AA), analytical reagent (AR), and cetyltrimethylammonium chloride (CTAC, AR) obtained from Aladdin chemistry (Shanghai, China) Co., Ltd, were used without further purification. *N,N'*-Methylenebisacrylamide (NMBA, analytical reagent, Shanghai Fusheng Chemical Reagent Factory, Shanghai, China) was used. High temperature resistance fiber purchased from Aladdin Regent Co., Ltd (Shanghai, China) was used directly in the experiment. Azoinitiator V-50 (2,2'-azobis (2-amidino-propane) dihydrochloride) (UA, Alfa Aesar 99%) was used without further purification. Water was double deionized with a Millipore Milli-Q system. Other reagents, purchased from Chengdu Kelong Chemical Reagent Co., Ltd (China), were analytically pure and were used as received.

2.2. Preparation of Sodium 2-Acrylamido-tetradecyl Sulfonate (NaAMC₁₄S). Hydrophobic monomer was synthesized according to the literature [11] with some improvement. The 200 mL three-necked flask was equipped with a magnetic stirrer, condenser, and an iced bath. 19.5 g (0.3 mole) of 1-tetradecyl and 23.6 g (0.45 mole) of acrylonitrile were added in the flask by turn. The mixture is cooled to 0°C. And then 44.5 g of 98% sulfuric acid (0.45 mole) was slowly dropped into the mixture with mildly stirring such that the temperature cannot exceed 20°C. Subsequently, the mixture is allowed to warm freely. After 12 h, 100 g ice was poured into the flask to terminated reaction and extracted with

200 mL portion diethyl ether. The blend extracts were washed with 200 mL of deionized water, 200 mL of 10% aqueous sodium carbonate solution, and 200 mL of deionized water; the solution was dried over with vacuum rotary evaporation and then filtered and dried in the vacuum oven to gain 61 g (yield 67%) of sodium 2-acrylamido-tetradecyl sulfonate (NaAMC₁₄S).

2.3. UVMT Was First Pretreated with HCl and CTAC. 20 g 325-mesh UVMT was added into 600 mL 2M HCl solution and the resulting slurry was kept stirring for 6 h at the temperature of 80°C to obtain HCl treated UVMT. Then the slurry was filtrated and washed adequately with multiple portions of deionized water until the pH of filtered fluid was 7.0 ± 0.5. The resulting material was then dried at 120°C for 48 h to obtain refined UVMT powder. The 10 g HCl treated UVMT was added to a 200 mL three-neck flask; then 100 mL of cetyltrimethylammonium chloride (CTAC) solution at the concentration of 2 wt% was dropped into the flask with vigorously stirring. Then the mixture was elevated to 85°C and slowly stirred for 3 days. The resulting mixture was washed with multiple portions of deionized water until no precipitate was observed upon titrating the filtrate with 0.1 mol/L AgNO₃. The final UVMT (CVMT) was dried in the vacuum oven and then kept in a dryer for use.

2.4. Preparation of Poly(AM/AA/NaAMC₁₄S)/CVMT Superabsorbent Composites Reinforced by Fiber (SACF). The total concentration of AM and AA in the experiment is constant. A series of superabsorbent composites composed of AM, AA, and high temperature tolerance fiber (HTF) was synthesized by varying the amount of NaAMC₁₄S proportion. 8.25 g AM, 1.75 g AA, and appropriate amount of NaAMC₁₄S were dissolved in desired volume of deionized water. The pH value of the mixture was tuned to 7.0–7.2 using sodium hydroxide solution. Then approximate 20 g CVMT and 0.5 g HTF was added into the mixture solution, and added some volume of deionized water to produce the 20 wt% for total polymerizable monomer (shown in Table 1). The resulting mixture was conducted with a small N₂ stream for half an hour and then kept still for 2 hours to provide monomer intercalation into CVMT. Subsequently, the solution was heated to 45°C. Polymerization *in situ* is initiated by a small amount of V-50 [12] and CVMT exfoliation by polymeric gel is produced by addition of a small proportion of NMBA. The resulting bulk gel was washed twice using acetone and extracted with ethanol to remove any impurities. The product

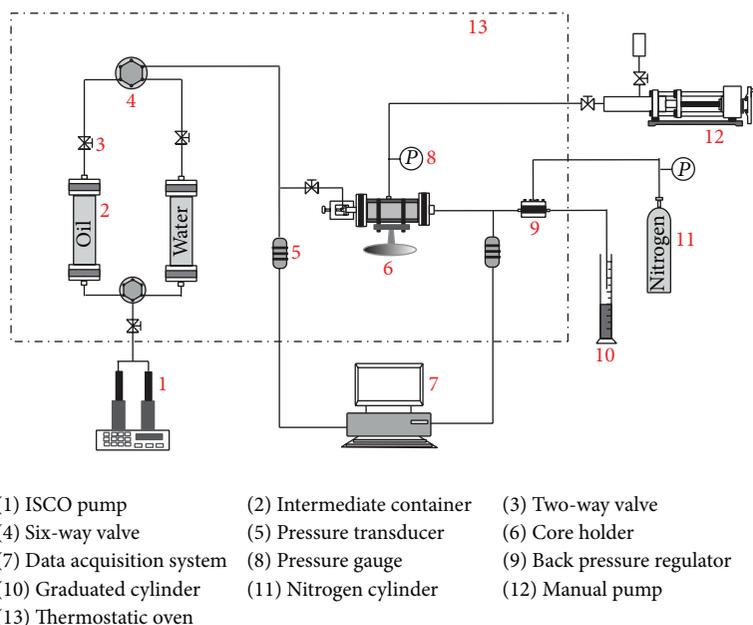


FIGURE 1: Schematic diagram of experimental apparatuses.

was dried under vacuum at 90°C for 6 h to constant weight and mashed bulk particles into 40-mesh powders.

2.5. Characterization. IR was carried out using Shimadzu-1800S spectrometer on KBr pellets in the range of 4400–400 cm^{-1} . The peak intensities are characterized as follows: vs = very strong, s = strong, w = weak, and ν corresponds to a stretching elongation. Small-angle X-ray diffraction (SAXRD) measurement was recorded on a Rigaku Geiger Flex D/max-RB diffraction meter using Ka radiation (40 kV, 30 mA). The distance between two layers of crystal plate was calculated through Bragg equation: $2d \sin \theta = n\lambda$, where d is the layer distance, θ is the diffraction angle, λ is the length of the diffraction light, 0.154 nm, and n is the diffraction series, in the experiment $n = 1$. The morphology of samples was observed by Environment Scanning Electron Microscope (SEM XL 30). The sample solutions were maintained at -3.5°C and the pressure was controlled below 5 Torr to keep samples solution state during the whole observation.

2.6. Measurement of Water Absorbency of SACF. A certain weight (W_0) of SACF was immersed into excess volume of salt solution and allowed to swell at the set temperature. After a period of time, the swollen SACF was filtrated through an 80-mesh sieve to remove free water and weighed (W_t). The water absorbency was calculated using the following equation:

$$S_f = \frac{(W_t - W_0)}{W_0}, \quad (1)$$

where S_f is the water absorbency per gram of dried SACF powder.

2.7. Core Flooding Experiment. Six Berea sandstone cores were preferentially prepared and the basic parameter of cores

is given in Table 1. Flooding instrument and procedure of the experiment are depicted in Figure 1. The flooding rate for both water flooding and swelled particle gel flooding (40-mesh SACF particle was swelling in the salt solution for 10 days) was set as 0.5 mL/min and kept unchanged. Injected pressure versus particle gel solution slug was automatically scaled by the flooding instrument. The residual resistance factor (f_{rr}) and the shut-off efficiency (η) of particle gel can be calculated through the inference of Darcy Formula [1]:

$$f_{rr} = \frac{K_w}{K_f}, \quad (2)$$

$$\eta = \left(1 - \frac{K_f}{K_w}\right) \times 100\%.$$

Herein, K_w is aqueous phase permeability, mD; K_f is the core permeability after injection of some designed pore volume (PV) of SACF particle gel, mD.

3. Result and Discussion

3.1. Characterization of NaAMC₁₄S and SACF

3.1.1. IR Analysis. The IR spectrograms of NaAMC₁₄S, SACF-1, and SACF-3 are depicted in Figure 2. For NaAMC₁₄S, IR (KBr, ν/cm^{-1}): 3412 (s, $\nu(\text{C}=\text{O})$), 2914 (vs, $\nu_{\text{as}}(\text{C}-\text{H})$), 2830 (s, $\nu_{\text{s}}(\text{C}-\text{H})$), 1665 (w, $\nu(\text{C}=\text{C})$), 1375 (s, $\nu(\text{C}-\text{N})$), 1249 (s, $\nu(\text{S}=\text{O})$), 1008 (s, $\nu(\text{S}-\text{O})$ and w, $\nu(\text{C}-\text{O})$). For SACF, IR (KBr, ν/cm^{-1}): 3412 (vs, $\nu_{\text{s}}(\text{NH}_2)$), 2914 (w, $\nu(\text{C}-\text{H})$), 1675 (s, $\nu(\text{C}=\text{O})$), 1304 (w, $\nu(\text{S}=\text{O})$), 1017 (s, $\nu(\text{C}-\text{O})$ and s, $\nu(\text{S}-\text{O})$).

3.1.2. XRD Characterization. The SAXRD spectrograms of CVMT and SACF-3 are shown in Figure 3. The SAXRD pattern of CVMT displayed a strong peak at $2\theta = 5.52$, which

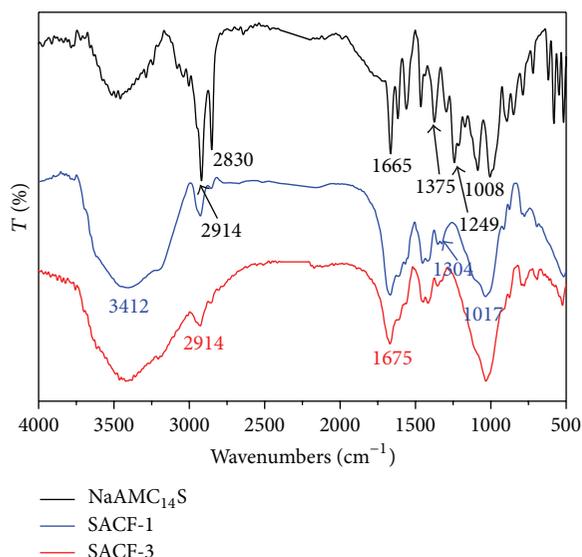


FIGURE 2: IR spectra of NaAMC₁₄S, SACF-1, and SACF-3.

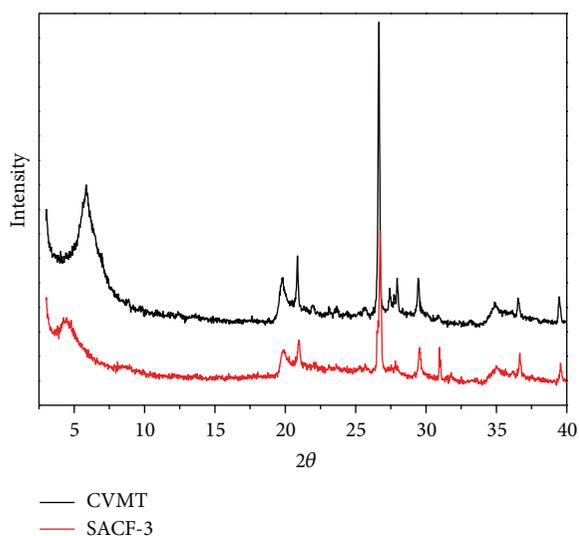


FIGURE 3: SXRD spectrograms of CVMT and SACF-3.

corresponds to a basal spacing of 1.77 nm compared to 1.43 nm pure UVMT [7], indicating that the basal spacing of original UVMT was expanded 0.34 nm after procedure of CTAC treatment. This peak was not observed in the SXRD pattern of the SACF, which indicated the fact that the crystalline plate had been further exfoliated by polymeric gel after the *in situ* copolymerized and crosslink reaction in the crystalline plates of polymeric monomer. The absence of such characteristic diffraction peak in the SXRD pattern of SACF was $2\theta = 4.17$ with 2.35 nm of basal spacing. We could speculate that nanocomposite containing exfoliated UVMT and polymeric matrix was successfully synthesized by successive intercalation of CTAC and polymerizable monomers and *in situ* reaction of monomers.

3.1.3. SEM Morphology. In order to investigate polymer/silicate nanocomposite structure, SXRD analysis should be always accompanied with microstructure observation, such as TEM and SEM. This is because the coexistence of stacked interacted, disordered or exfoliated structure that a SXRD pattern may hide large part of exfoliated layers [6]. Typical SEM morphologies of the nanocomposite of original UVMT and of various modified UVMT are depicted in Figure 4. The unmodified UVMT nanosheets disorderly stacked to form multilayer aggregation due to the Van der Waals' force between adjacent layers (Figure 4(a)). Thus, neither monolayer nor exfoliated layer was existence. The original silicates were loose and porous enough for monomer intercalation and exfoliation. Poly(AM/AA/NaAMC₁₄S)/CVMT morphology is depicted in Figure 4(b). Loose and multi-hole nanostructure with exfoliated silicate layer dispersed in the polymeric matrix was observed. The smooth network for polymeric bulk gel was disrupted when introducing proper silicate, producing the wrinkled and multihole microstructure which facilitated the external water into the network structure and prevented moisture loss. This confirmed that polymer exfoliated the multilayer plate pretreated by CTAC and the dispersion of nanocomposite into the polymeric matrix is well prepared. The microstructure of polymer/silicate composite filled by high temperature resistance fiber (HTF) is shown in Figure 4(c). The white strips in the Figure 4(c) represent the HTF, which randomly disperse in the polymeric matrix. The morphology of nanocomposite was displayed as scales, suggesting that fiber acted as linkage of the network structure in composite, reinforcing the toughness and strength of nanocomposite.

3.2. Swelling Behavior of SACF. The effect of AM, AA, and clay content on the swelling properties of polymer/silicate composite has been extensively reported, and the understanding of these properties has been constructed. Here, we are focused on the hydrophobic group (NaAMC₁₄S) and fiber content upon the composite absorbent properties in the salt solution.

3.2.1. Effect of NaAMC₁₄S Content on Water Absorbency. A typical profile of water absorbency for SACFs as a function of various ionic strengths in the salt (NaCl, MgCl₂, and CaCl₂) solution is depicted in Figure 5. The water absorbency of SACFs was maximum value in the aqueous solution. Such value decreased as the ionic strength of all three cationic salt solutions increased. One observed that hydrophobic moistures play a significant role in controlling absorbency (SACF-2) in the salt solution compared to that of absorbency for polymer/silicate without hydrophobic group. For SACF-2, the curve of water absorbency decreased slowly with increasing ionic strength. This can be explained such that the increasing cationic concentration compressed the loose network by screening the negative charges of carboxylate group, resulting in the release of water in the salt solution. The hydrophobic microdomain formed by alkyl carbon chain of SACF-2 prevented the cations from attacking the carboxylate

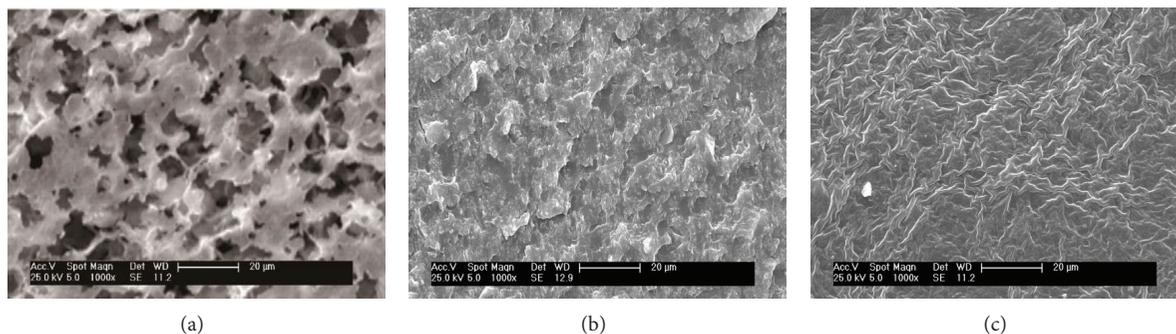


FIGURE 4: SEM images for original UVMT and for SACF-1 and SACF-3.

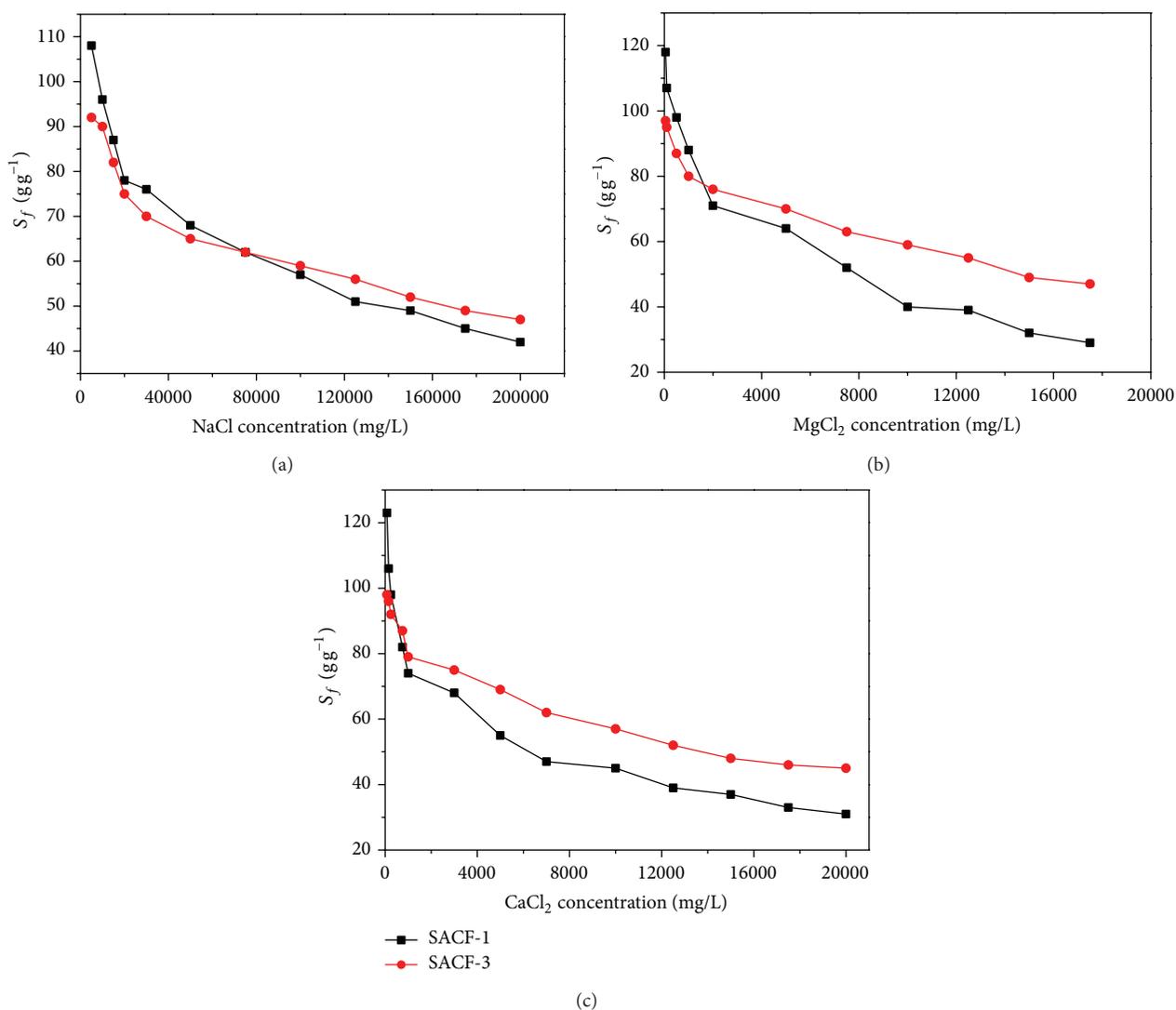


FIGURE 5: Water absorbency for SACFs as a function of cationic concentration at 120°C.

group. On the other hand, sulfonic acid groups brought into chain backbone can partly enhance composite slat resistance. Therefore, more notable absorbency for SACF-2 was observed in comparison to SACF-1. The water absorbency in the monovalent salt solution is higher than these for divalent (calcium and magnesium ions) salt solution. This is because

divalent cationic would interact with carboxylate group to form complex.

3.2.2. *Effect of HTF on Water Absorbency.* The curve of water absorbency for SACFs versus cationic concentration is shown in Figure 6. For SACF-2 in the absence of HTF, much

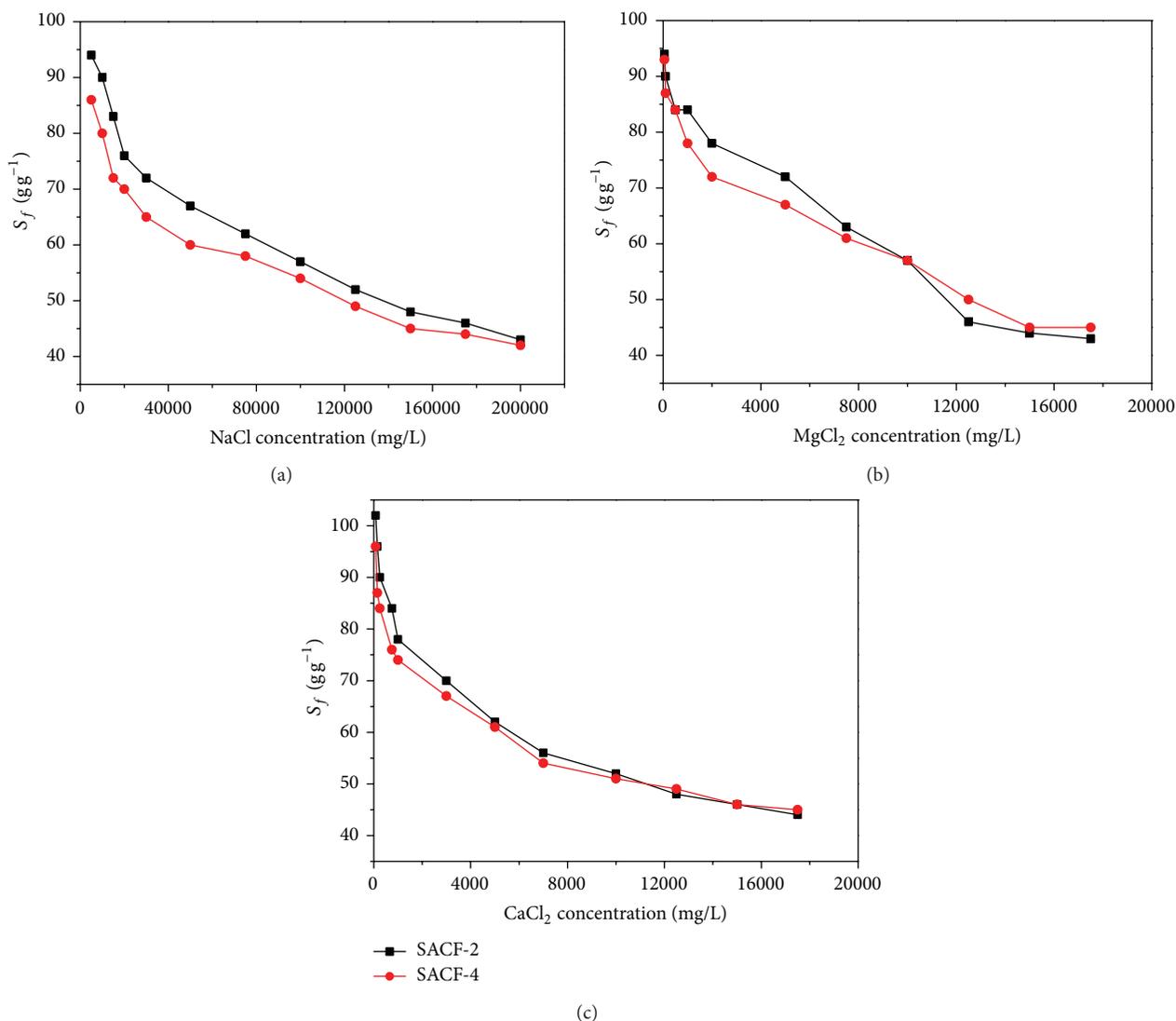


FIGURE 6: Water absorbency for SACFs as a function of cationic concentration at 120°C.

more absorbent capability was observed in comparison to SACF-4 filled by small amount of HTF in the low salinity solution. With further increasing salt concentration, the absorbency for SACF-2 and SACF-4 was decreased. However, the decreased trend of absorbency for SACF-4 is much more flatted than that of SACF-2. This can be interpreted such that the hydrophobicity of fiber would partly hinder the water suction, resulting in the lower water absorbency [13]. On the other hand, ionic strength with high salt concentration is the dominating factor affecting the sucking ability of composite. Elevated cationic concentration screened the carboxyl groups, producing the absorbent gel water release. Hence, similar absorbent values for SACF-2 and SACF-4 at the highest salt concentration are observed.

3.2.3. Salt and Thermal Stability for SACF in the Harsh Condition. The water absorbency for SACFs depended on

aged time in the various salt concentrations at 120°C as shown in Figure 7. At the superhigh temperature of 120°C, salt concentration significantly affected the absorbency and water retention of SACF, which controlled the absorbent profile against aged time. The absorbency of SACFs increased rapidly within 10 days ageing and then remained stable at the total dissolved concentration of 53000 mg/L of diluted Tarim Basin formation water. The dynamic swelling behavior of SACF was consistent with Kelvin-Voigt viscoelastic model reported by Wu and Liu [10]. SACF-4 reinforced by fiber exhibited lower absorbency in comparison to SACF-2 in the absence of fiber. At the ultrahigh salt concentration, the dynamic swelling behavior of SACFs is more complicated. The absorbency rapidly increased in the initial aging stages and the maximum time of SACFs expansion is displayed. Subsequently, the released-water behavior of SACFs shrinks such particle gels. We noted that SACF-4 exhibits slow water-released property, and absorbent index kept stable after

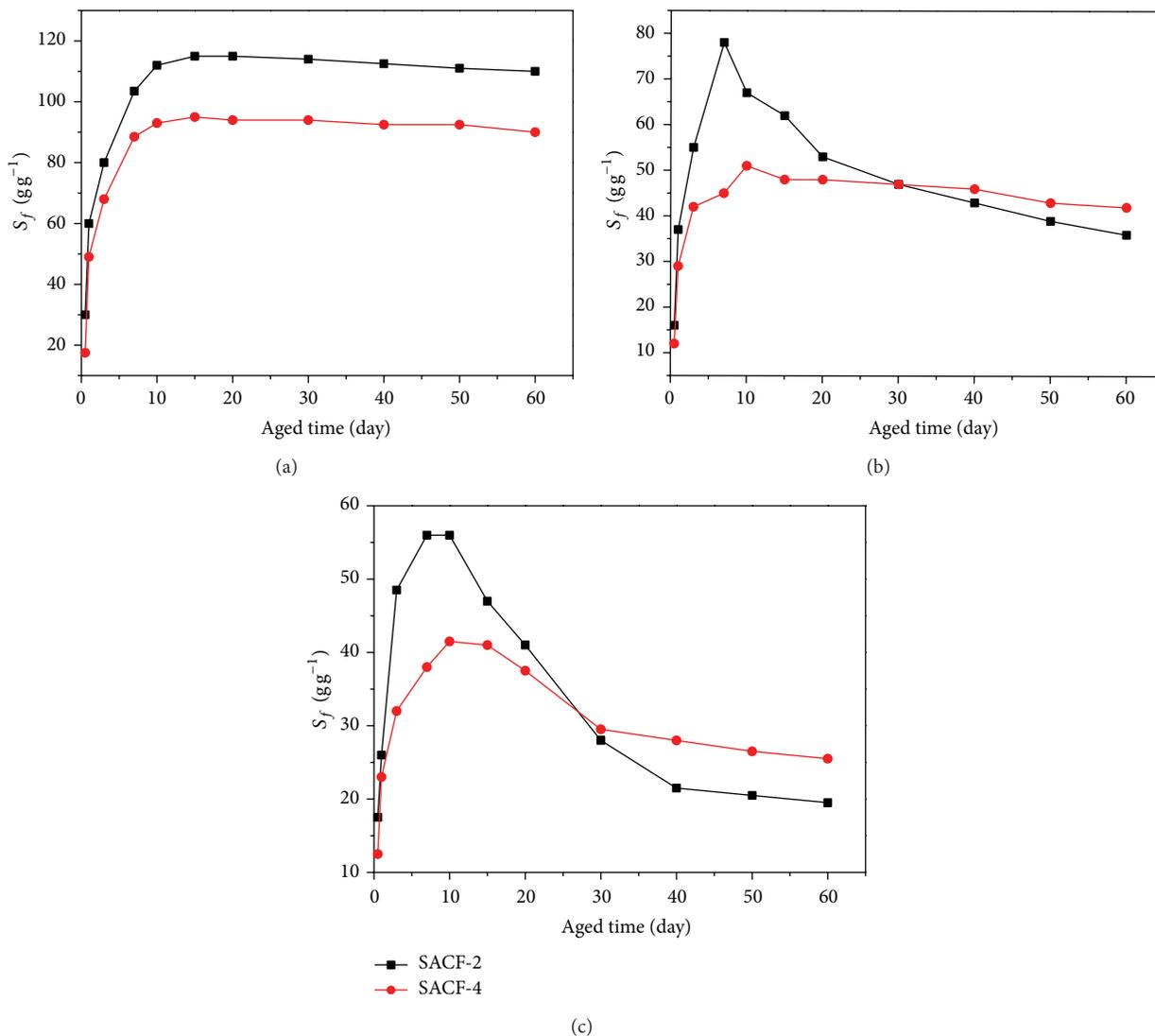


FIGURE 7: Absorbent stability for SACFs at 120°C in the various diluted Tarim Basin formation water (total salt concentrations (a) 53123 mg/L, (b) 106246 mg/L, and (c) 212592 mg/L).

40 days of ageing. With further prolonging ageing period, few swelling gels broke into smashes, indicating that reasonable thermal stability as well as enough toughness for SACF-4 was given by introducing hydrophobic group and fiber into the superabsorbent composite.

For SACF-2, notable released-water behavior shrinks swelling gels during 35 days of thermal ageing. With increasing ageing period, few swelling gels broken into smashes result in absorbent gels' further shrinkage. The different thermal stability for SACF-2 in the salt solution can be interpreted by the following reasons: (1) the partly degraded polymeric matrix defects network structure leading to the undesirable water-retaining capability for absorbent composite; (2) reinforced by fiber, bridge connection was constructed to facilitate network structure to be more tough and intensive. At the highest total dissolved substance of Tarim Basin formation water (212000 mg/L), notable absorbency for SACF-4

was exhibited compared to that of SACF-2 during the 60 days of thermal ageing.

3.3. Core Flooding Experiment. The injected pressure profile as function of flooding pore volume (PV) for 0.25 wt.% SACFs solution and subsequent injecting water are shown in Figure 8. The climbing pressure for three types of polymer/clay composite was achieved and injected pressure increased accompanied by the increased injected pressure differential during injection of SACFs solution. The injected pressure was further raised at the onset of water flooding after 0.3 PV of SACFs flooding, which decreased rapidly to a platform value. Profile control ability for SACF can be recognized by illustrating the migration and swelling mechanism of particle gels in the porous medium. At the onset of migration, particle gels trend to larger porous channel due to their physical selection. Obviously, resistance of motion is caused with more amount of composite gels

TABLE 2: Characteristic parameters for 0.3 SACF displacement of subsequent water flooding in the core flooding experiment.

Core samples	Core parameter			Particle gels system	Pressure parameter (MPa)			Residual resistance factor	Shut-off efficiency %
	Diameter cm	Length cm	Permeability μm^2		Initial pressure	Breakthrough pressure	Stable pressure		
1#	3.796	6.98	1.83	SACF-1	0.138	1.172	0.745	5.4	81.4
2#	3.798	6.96	1.87	SACF-2	0.126	4.363	1.952	15.5	93.5
3#	3.802	7.01	1.91	SACF-4	0.135	8.432	3.594	26.4	96.2

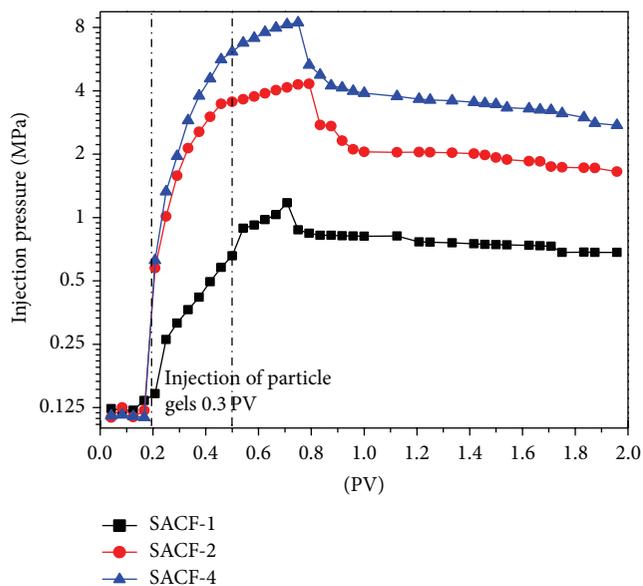


FIGURE 8: Injection pressure as function of particle gels' pore volume and of subsequent water displacement pore volume (Tarim Basin injected water, 120°C).

entrance into porous medium, which is accompanied by the elevated injection pressure and increased injection pressure differential. The swelling particle gels further migrate in the porous medium and begin to accumulate in the narrow throat, leading to the injection pressure increased and to promote the subsequent flooding agent diversion. The accumulated particle gels are swept into scatter at the highest injection pressure. The scattered particle gels move as migrate amoeba in the narrow channel of the porous medium and the dropped injection pressure is measured by the flooding instrument. Finally, relative injection pressure is achieved when the composite stably immigrates in the porous medium.

In the experiment, we observe that SACF-4 containing hydrophobic group and fiber exhibits the most desirable profile control capability and it has the highest stable pressure region in comparison to SACF-1 and SACF-2. Characteristic parameters including initial injection pressure, breakthrough pressure, stable pressure, residual resistance factor, and shut-off efficiency for polymer/clay hybrid composites by core flooding experiment are illustrated in Table 2.

4. Conclusion

Alkyl sulfonate hydrophobic monomer called NaAMC₁₄S was preferentially synthesized in the experiment and its structure was characterized by FTIR. Based on this hydrophobic monomer, novel superabsorbent composite filled with small amount of high temperature fiber (SACFs) through *in situ* intercalation and exfoliated technique was successfully synthesized. FTIR, SXRD, and SEM measurements confirmed that both the notion and the method for preparation of SACFs are scientific and practicable. The incorporation of hydrophobic segments into polymer/clay composite produces the controlled absorbent capacity for SACFs in the salt solution. More notable absorbency for SACF-2 was observed in the higher cationic solution resulting from the alkyl carbon chain and sulfonic acid group hindering the cations from attacking the carboxylate groups. Moreover, both toughness and strength for SACF-4 were reinforced by introducing amount of fiber which acted as bridge connection for the polymeric networks structure. At the total dissolved substance of 212000 mg/L with the temperature of 120°C, notable absorbency as well as water retaining property for SACF-4 was observed during 60 days of thermal ageing. Core flooding experiments demonstrated that SACFs could migrate as amoeba in the porous medium and accumulated in the narrow channel to adjust injection profile and promoted the subsequent water diverting into the unswept formation. Our investigation confirmed that polymer/clay hybrid superabsorbent composite containing hydrophobic group and fiber exhibited the reasonable profile control capability and desired injection pressure region which should have robust potential in the profile controlled application for enhanced oil recovery.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

The authors are grateful to the Major Special Project of China (Grant no. 2011ZX05049-004-004) and the Education Scientific Research Project of Sichuan Province Department to Dr. Jin (Grant no. 14ZB0042) for financial support of this work.

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