The Investigation of a New Moderate Water Shutoff Agent: Cationic Polymer and Anionic Polymer

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ABSTRACT: Cationic polymers and anionic polymers were selected as a moderate water shutoff agent for water production control. Due to the adsorption of polymers on the sand surface, the adsorption capacity under static condition, in porous media, and adsorption morphology on mica were investigated through starch–cadmium iodide method, core flow test, and atomic force microscopy measurement. The adsorption quantity on the sand surface increased with the high polymer concentrations and long adsorption time. With the increase of temperature and shearing time, the adsorption capacity slightly decreased. In addition, the adsorption capacity under water wettability condition was significantly larger than that under oil wettability condition. Alternate injection of cationic polymer and anionic polymer caused larger adsorption capacity in the core test. An adsorption multilayer was formed through alternate adsorption of cationic polymer and anionic polymer confirmed by atomic force microscopy. The visual simulation experiment was also conducted to illustrate adsorption and enhanced oil recovery mechanism. The polymers preferentially entered the high permeability zone and adsorbed on the sand surface, thus enhanced oil recovery. Furthermore, alternate injection of cationic and anionic polymers as a moderate water shutoff agent was successfully applied for water production control in oilfield test.
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INTRODUCTION

Reduction in water production of oil wells becomes an increasingly important objective for mature oilfields. Profile control and water shutoff treatments are widely practiced to reduce water production and improve oil production.[1–3] Mechanical and chemical methods are available for profile control and water shutoff treatments in high water cut oil wells. Mechanical methods, such as drilling multilateral wells and placing a bulkhead, are generally expensive and not affordable. So various chemicals methods, including injection of polymers, gelling systems, particle systems, resin systems, foams, and so on, have been widely used for water shutoff treatments in worldwide field trials.[4–7] Among them, the most commonly used chemical methods for controlling water production treatments are to inject polymers for its simple and economical properties. Polymer flooding usually involves injection of polymers into fractures or high-permeability zones from injection wells. The injected polymer changes the flow properties of following water and improves volumetric sweep efficiency of water flooding. Generally, partially hydrolyzed polyacrylamide (HPAM) is used

in the oil industry. The carboxylate groups in HPAM molecules cause chain expansion due to the repulsion of ionic groups, which leads to higher solution viscosity and increases water flow resistance, thus reduces the water permeability in the formation.[8] However, the adsorption of polymer is inevitable due to electrostatic and hydrogen bonding interactions in the polymer flooding. If the adsorption quantity of polymer is excessive on the rock surface, the solution viscosity decreases, and affects the oil displacement efficiency.[9] So, the anionic polymer is mainly used for polymer flooding, but excessive adsorption also affects volumetric sweep efficiency. Since the adsorption cannot be inevitable, in order to take fully advantage of this adsorption property for water shutoff treatments, the cationic and anionic polymers as a moderate water shutoff agent for oil well treatment are studied. When the polymers are injected into the formation, they can adsorb on the sand surface and form multilayer, in which leads to the following water turn to the low permeability zone and improve oil recovery.

However, injecting a polymer solution into a production well is risky, especially if there is no possibility of zone isolation. In

addition, few tests using cationic polymer and anionic polymer for water shutoff treatment have been conducted in the oilfield. That brings us difficulties for water shutoff treatment. So, in this study, we primarily studied the adsorption capacity of cationic polymer and anionic polymer in static condition and porous media. The oilfield test is also conducted to confirm the efficiency of polymers for water production control. Through the laboratory experiments and oilfield test, we hope this study can be further promotion and application for water production control in mature oilfield.

MATERIALS AND METHODS

Materials

Anionic polymer with the hydrolysis of hydrolysis 24.38% and average molecular weight of 12,000,000 was provided by Yuguang Co., China. Cationic polymer with the degree of cationic degree 33.48% and average molecular weight of 10,000,000 was provided by Zhengli Co., China. The salinity of brine was 5022 mg/L.

Determination of Adsorption

Determination Principle and Procedure. The adsorption quantity is determined by the starch–cadmium iodide method.[10,11] The method is based on the bromine oxidation of the amide functional group in anionic polymer and cationic polymer. Under a pH 5.5, the amide group in polymer reacts with bromine to form N-bromoamide oxidation. Then the N-bromoamide oxidation reacts with iodide ion to form iodine. Finally, the iodine was measured as the starch–triiodide complex.

The steps for determining adsorption were as follows: (1) adding 5 mL buffer solution and up to 30 mL aqueous sample containing from 15 to 300 μg polymer into a 50 mL volumetric flask; (2) diluting the solution to about 35 mL and adding 1 mL bromine water, then allowing the mixture to react for 10 min; (3) sequentially adding 5 mL of 1% sodium formate and reacting for 5 min; (4) adding 5 mL starch–cadmium iodide reagent and diluting the solution to 50 mL with distilled water, then allowing the mixture to react for 10 min and using spectrophotometric to determine the adsorption at 610 nm versus a reagent blank.

Determination of Static Adsorption Quantity. The tests were performed with spectrophotometer system (722 type, Shanghai Third Analysis Instrument Factory, China). The polymer solution (20 g) and the sand (5 g) were stirred to produce uniform solution and added to an Erlenmeyer flask. Then, the solution was put into the shaking water bath for some time at 75°C. Finally, the supernatant liquid was used to determine the adsorption capacity.

Determination of Dynamic Adsorption. The core flow test was carried out to determine the dynamic adsorption capacity. The experimental flow chart is shown in Figure 1. The steps for determining dynamic adsorption capacity are as follows: (1) filling the pack with sand and weighing the dry core; (2) saturating the core with brine and weighing the wet core, then calculating the pore volume (PV); (3) injecting polymer solution into the core and placing it into an oven at 75°C for a certain time; (4) water flooding and calculating the dynamic adsorption
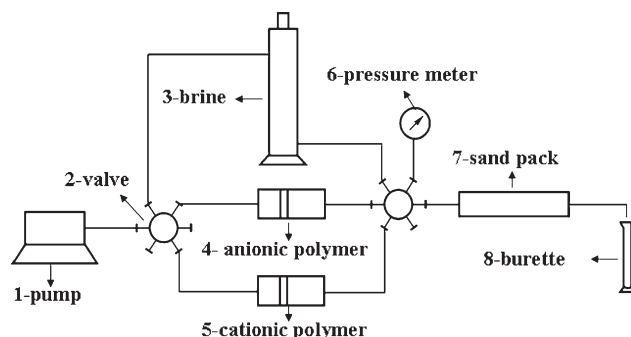

Figure 1. The experimental flow chart.

capacity. All injection of polymer solution was performed in accordance with the following modes:

AA injection mode: alternate injection of anionic polymer for three rounds;

CC injection mode: alternate injection of cationic polymer for three rounds;

AC injection mode: alternate injection of anionic polymer and cationic polymer for three rounds;

CA injection mode: alternate injection of cationic polymer and anionic polymer for three rounds.

Visual Simulation Experiment

The experimental flow chart is shown in Figure 2. The size of the visual model in the experiment was 25 cm × 25 cm × 1 cm. The space in visual model was filled with sand. The edges were sealed except two pores on the diagonal of the model, which were used to simulate as an injection well and an oil well. There was also a high permeability zone between injection well and production well in the model. The steps were as follows: (1) saturating brine in the model; (2) saturating oil in the model; (3) water flooding until the effluent water cut up to 98%; (4) alternatively injecting five rounds of cationic polymer and anionic polymer into visual model; (5) water flooding until the effluent water cut up to 98% again, and calculating the oil recovery during the process.

Atomic Force Microscopy (AFM) Measurements

The microscopy of the polymers adsorption on mica was analyzed with a Nanoscope IVa MultiMode AFM (Digital
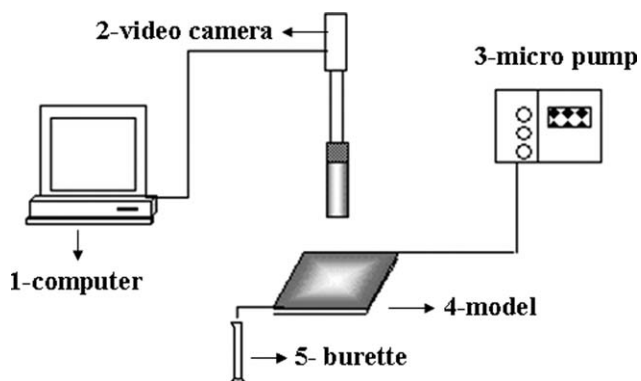

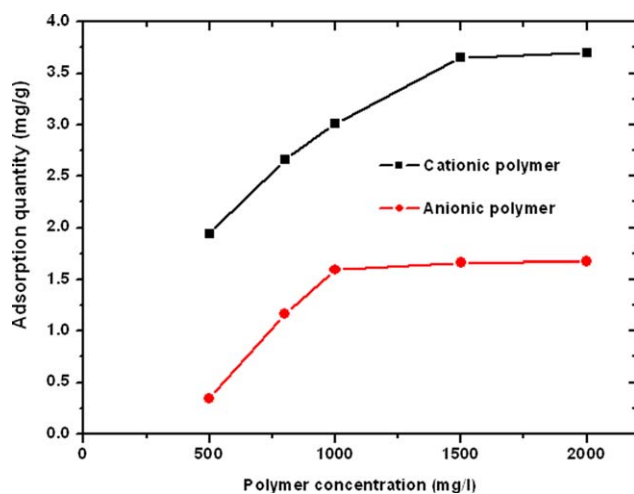**Figure 2.** The visual simulation experimental flow chart.

Figure 3. Effect of polymer concentration on adsorption capacity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
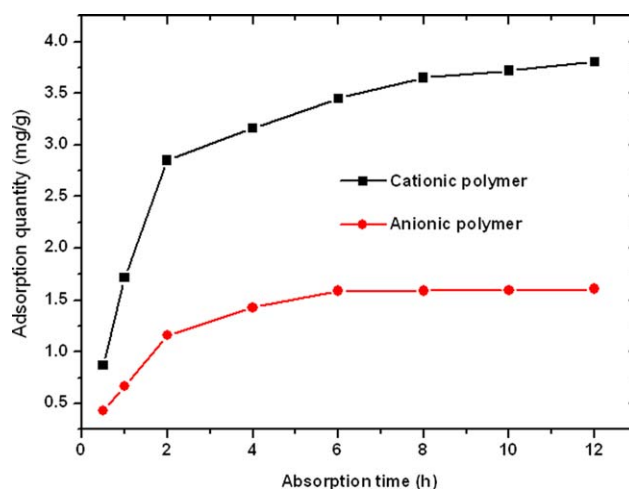

Figure 4. Effect of time on adsorption capacity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Instruments, Santa Barbara, CA) in tapping mode and operated at a scanning speed of 1 Hz. Samples were prepared for AFM measurements by dropping 10 μl of polymer solution onto freshly cleaved mica surface for adsorption equilibrium time. Then, the mica surface was dried gently with ultrapure nitrogen gas. Topographic and phase images were concurrently recorded under ambient conditions, at 512 $\times$ 512 pixel resolution, integral and proportional gains 0.1–0.2 and 0.2–0.3, respectively.

RESULTS AND DISCUSSION

Static Adsorption Capacity

Effect of Polymer Concentration. The polymer concentration is critical for the adsorption capacity of the anionic polymer and cationic polymer. The two polymer concentrations varied from 0.05% to 0.2%. Figure 3 shows the adsorption capacity of two polymers at 75°C.

Figure 3 shows the effect of polymer concentration on the adsorption capacity for anionic polymer and cationic polymer in brine. The adsorption capacity of two polymers increases with the concentration. However, adsorption quantity is almost no longer increased when the polymer concentration is up to a certain concentration, which can be called equilibrium adsorption concentration. The adsorption equilibrium concentration of anionic polymer and cationic polymer are 1000 and 1500 mg/L, respectively. Up to the adsorption equilibrium concentration, the adsorption quantity of cationic polymer is 3.653 mg/L while the adsorption quantity of cationic polymer is only 1.594 mg/L. This difference may be attributed to charge difference between anionic polymer and cationic polymer. The adsorption of positively charged polymers (cationic polymer) occurs through electrostatic (Coulombic) interactions between the cationic groups on the polymer and the negatively charged sites at the sand surface, which increases the adsorption capacity of the cationic polymer. However, little adsorption occurs with negatively charged polymers (anionic polymer) due to initial charge repulsion between the polymer and the sand surface.[12] The increase of adsorption may be mainly attributed to hydrogen

bonding interactions between anionic polymer and sand particle surface.[13,14] The results also confirm that electrostatic interactions play a dominant role in the adsorption on the sand surfaces.

The Effect of Adsorption Time. The effect of adsorption time on equilibrium adsorption is significant. Figure 4 shows the adsorption process of 1000 mg/L anionic polymer and 1500 mg/L cationic polymer at 75°C, respectively.

As expected, the adsorption quantity increased with the time. In general, adsorption reactions of anionic polymer and cationic polymer are extremely rapid at initial stage. During this stage, most of the adsorption reaction took place. However, after a relatively long time, the adsorption quantity became stable and an adsorption balance was achieved and the time was generally called adsorption equilibrium time. Figure 4 shows that the adsorption equilibrium time of anionic polymer and cationic polymer were 6 and 8 h, respectively. Over this time point, the adsorption quantity was almost no longer increased, which may be limited by the number of sites available on the surface of the sand particle. When all the available sites are occupied, no further polymer molecules can be attached to the surface of the sand particle, the adsorption balance is achieved. In addition, the different adsorption equilibrium time between anionic polymer and cationic polymer may be attributed to the different charge in the two polymers.[15] The cationic polymer is adsorbed with a larger segment–surface interaction energy as compared with the anionic polymer, resulting in a long adsorption time.[12]

The Effect of Temperature. The temperature also influences the adsorption capacity of anionic polymer and cationic polymer. In this experiment, the adsorption capacity was investigated from 60°C to 80°C at adsorption equilibrium concentration and adsorption equilibrium time.

Figure 5 shows that the effect of temperature on adsorption capacity of two polymers was not obvious. The adsorption quantity slightly decreases with the increase of temperature. This may be due to the increase of thermal motion of molecules in the solution with the temperature increases, which leads to
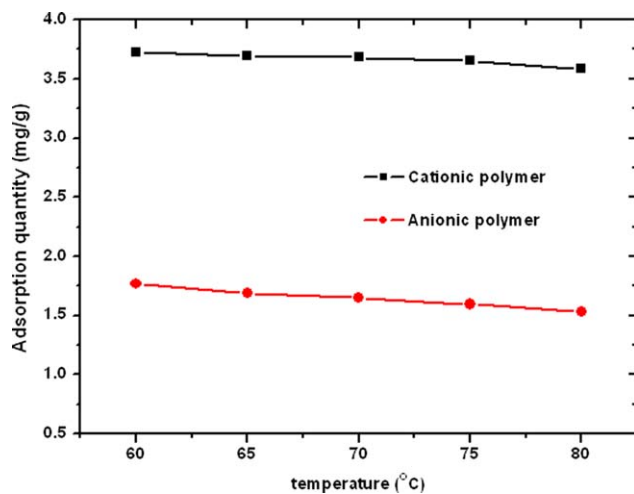
Figure 5. Effect of temperature on adsorption capacity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

accelerate the desorption of the anionic polymer and cationic polymer from the sand particle surface. In addition, the solubility is increased with the temperature, which also causes the decrease of adsorption quantity for the two polymers.

The Effect of Shearing Time. Shearing degradation takes place when a polymer solution flows through injection equipment and formation.[16] Thus, it is important to investigate the effect of shearing on adsorption capacity of polymers. To simulate shearing degradation under field conditions, Waring agitator method was used at a shearing rate of 2000 rpm. Figure 6 shows the effect of shearing on the adsorption capacity of anionic polymer and cationic polymer under adsorption equilibrium concentration and adsorption equilibrium time at 75°C.

The results shown in Figure 6 indicate that an increase in shearing time decreases the adsorption capacity of two polymers on the sand particle surface. However, both of the adsorption quantity retention rate remains above 75% after shearing for 60 min, which shows that the two polymers have excellent shearing
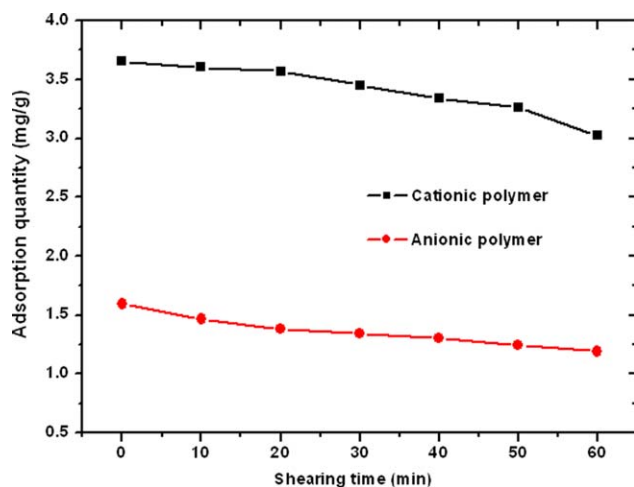
stability as a moderate water shutoff agent. Further study indicates that the adsorption quantity increases as the adsorption time increases after shearing. Based on these results, we can conclude that when the adsorption of polymers occurs on the sand particle surface, some molecular aggregates are formed. The shearing disrupts the molecular aggregates and decreases the adsorption capacity. But there are still many adsorbed segments in the solution which can be adsorbed on the sand particle surface with electrostatic or hydrogen bonding interactions. Moreover, the disrupted molecules reaggregate when shearing is ceased, and the molecules join together again to form polymer coil, which also promotes adsorption capacity on the sand particle surface.[17] In addition, the introduction of acrylate groups to the polymer chains also provides significant chain rigidity, thus allowing the polymers to exhibit better shearing stability.[18]

The Effect of Wettability. As is known to all, there is a large amount of residual oil in the formation after water flooding, especially in low permeability zone. As a result, long-term interaction between the residual oil, water, and rock causes significant changes in the wettability of rock surfaces,[19,20] and affects the adsorption capacity of anionic polymer and cationic polymer on the rock surface. In this case, it is possible that rock wettability have a markable effect on well treatment.[21,22] Four different types of wettability sands were used in the experiment to investigate the adsorption capacity of two polymers. Figure 7 shows the effect of rock wettability on the adsorption capacity of two polymers under adsorption equilibrium concentration and adsorption equilibrium time at 75°C.

Figure 7 shows the adsorption capacity of anionic polymer and cationic polymer under different wettability conditions. Both the adsorption quantity of two polymers under strong water wettability condition is significantly larger than that under oil wettability condition. Moreover, the stronger the oil wettability, the smaller the adsorption capacity. Under oil wettability condition, residual oil covers the sand surface with a wetting film, which makes the sand grains smooth and form pendular structures between adjacent grains. Thus the adsorption sites on the
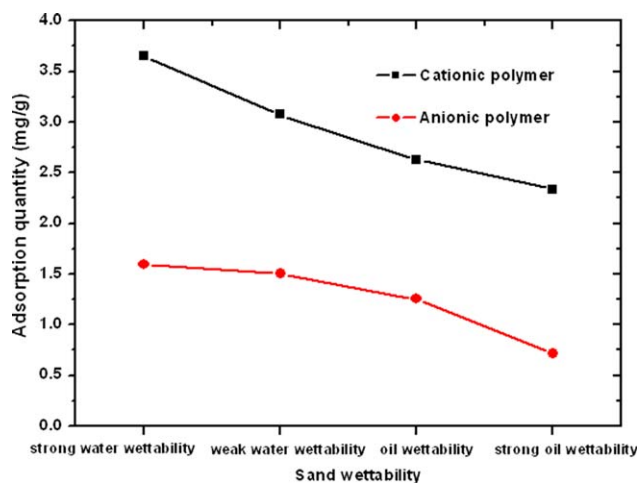

Figure 6. Effect of shearing on adsorption capacity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 7. Effect of wettability on adsorption capacity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
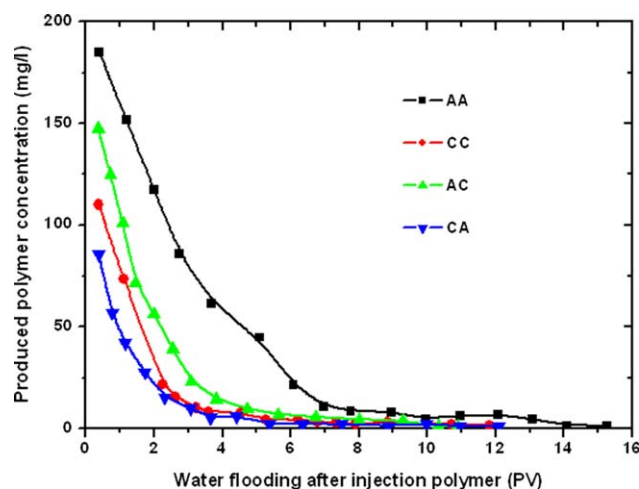
Figure 8. Produced polymer concentration after water flooding. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 9. Dynamic adsorption capacity in porous media. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

sand surface decrease, which can explain the decreased of adsorption quantity in oil wettability systems.[23,24] It is just the selective adsorption properties of two polymers under different wettability conditions that help to form the different adsorption layer in reservoir conditions. As we know, different sizes of channels were distributed in the formation after a long period of water flooding, resulting in the injected water circulating in high permeability channel, thus forming water wettability in high permeability channel, and oil wettability in low permeability zone. When two polymers are injected, they are preferentially adsorbed in high permeability channel and effectively reduce the permeability of porous medium in high permeability channel, resulting in the following injection water into low permeability zones. As a result, both the sweep efficiency and oil recovery are improved.

Adsorption in Porous Media

Although the anionic polymer and cationic polymer show a high adsorption capacity under static condition, the adsorption capacity in porous media and the interaction existed between the polymers and porous media remain unclear. The adsorption capacity in porous media under adsorption equilibrium concentration and adsorption equilibrium time at 75°C is studied. Figures 8 and 9 show the produced polymer concentration and dynamic adsorption capacity of polymers in porous media, respectively.

Figure 8 illustrates the dynamic changes of produced polymer concentration after water flooding in different injection modes. The result shows that the produced polymer under AA injection mode has the largest concentration among the four injection modes. However, the dynamic adsorption capacity is weakest in porous media under AA injection mode (Figure 9). Although negatively charge of reduces the anionic polymer reduces the adsorption on the sand surface under AA injection mode, significant adsorption is still possible for anionic polymer due to the hydrogen bonding interactions. There is a different adsorption behavior between CC and AA injection mode. When injecting cationic polymer in the first round, the adsorption
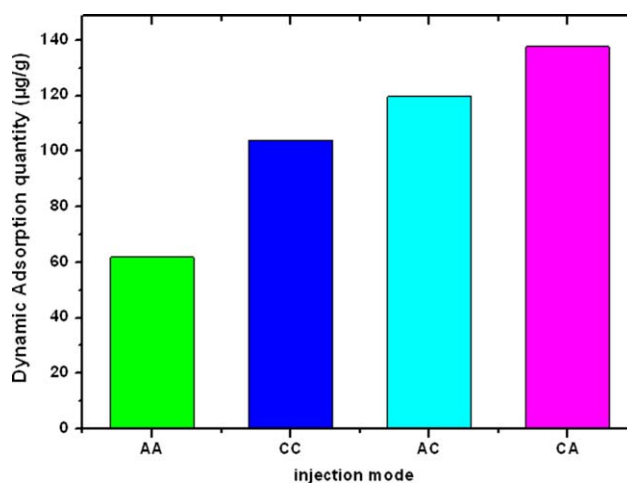
capacity is enhanced for the electrostatic interactions between the polymer and sand surface. However, charge repulsion is predominantly generated after injecting cationic polymer in the second round, resulting in the decrease of the adsorption capacity. If injecting anionic polymer in the second round (CA injection mode), the anionic polymer with negative charge neutralizes the cationic polymer. As a result, a new electrostatic imbalance between the anionic polymer and cationic polymer are created, leading to the formation of a new stable adsorption layer, thus increases adsorption capacity and adhesion capacity in the porous media. Further alternatively injection polymers according to CA injection mode, the adsorption will proceed to increase until adsorption equilibrium. In addition, electrostatic interactions may provide stronger adhesion between the anionic polymer, cationic polymer, and sand surface, resulting in a lower produced polymer concentration and higher dynamic adsorption capacity.

Adsorption Morphology on Mica

To further investigate the adsorption behavior of anionic polymer, cationic polymer, cationic polymer and anionic polymer, AFM was performed to observe the morphology of polymers adsorbed on mica.

Figure 10 shows the morphology of anionic polymer, cationic polymer, and the aggregates of two polymers. In Figure 10(A) and (B), the adsorbed anionic polymers on mica are almost globular and loose while the cationic polymer is tiled. The alternate adsorption of cationic polymer and anionic polymer leads to form a multilayer on the mica surface. That is directly related to the charge between the polymers and mica surface. As we know, the surface charge density of untreated mica is two negative charges per nm^2,[25] while the anionic polymer is generally negatively charged. When the anionic polymer contacts with the surface of mica, charge repulsion is caused and lead to form loose globular cluster on mica surface [Figure 10(A)]. However, the adsorption of cationic polymer on negatively charged mica can be attributed to electrical interactions between the positively charged
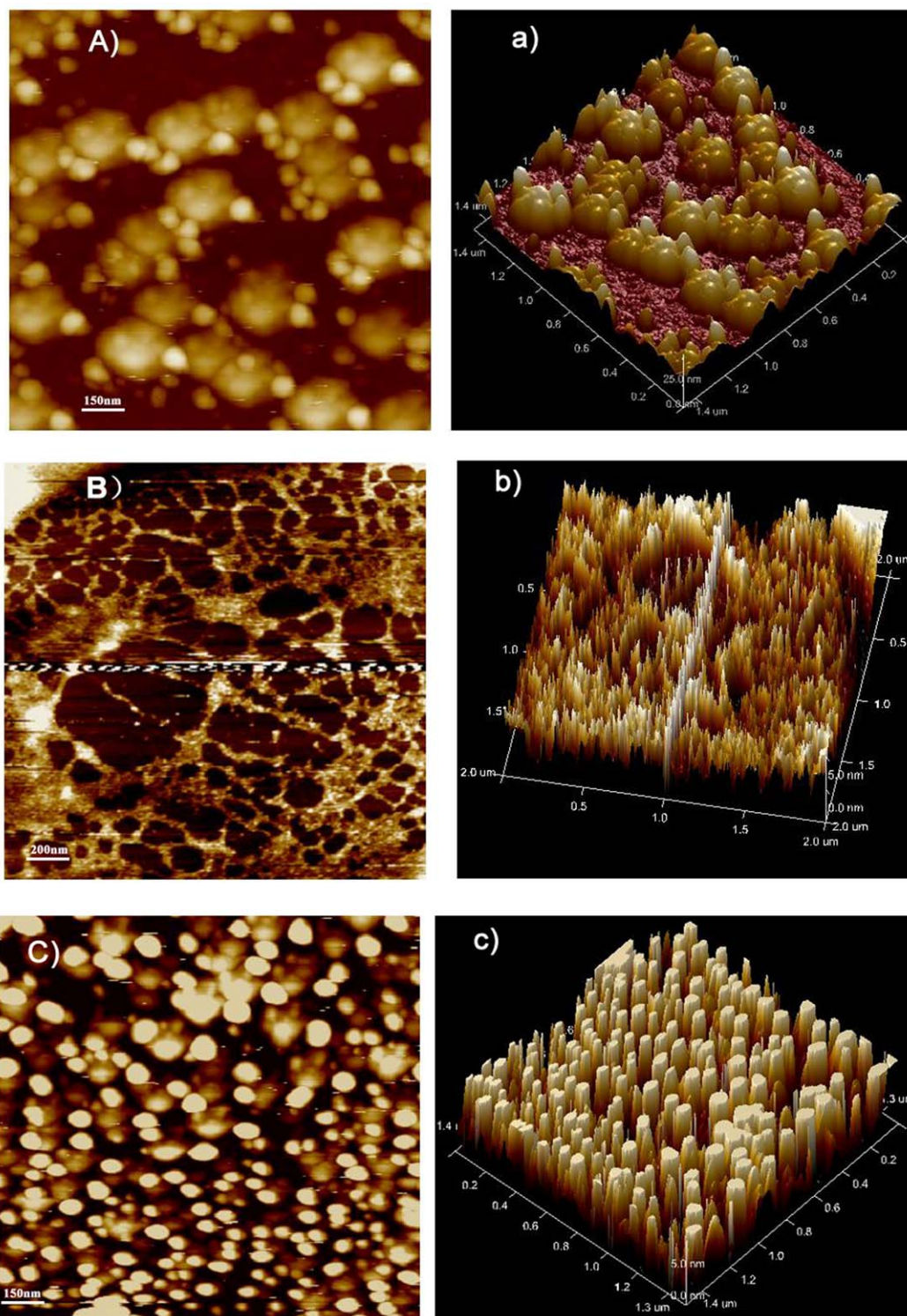
Figure 10. AFM morphologies of anionic polymer (A and a), cationic polymer(B and b), cationic polymer and anionic polymer (C and c) adsorption on mica. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

polymer chains and the surface. The positively charged polymer chains neutralize the negative charges, resulting in the cationic polymer fully stretch and adhere on mica,[26,27] which forms a dense and stable adsorption layer on the negatively charged surface [Figure 10(B)]. Further adsorption of anionic polymer on the adsorption layer [Figure 10(B)], the oppositely charged group of the polymers reduces the surface potential and attracts to each other, then a new multilayer adsorption imbalance may be created and adsorption capacity is also promoted [Figure 10(C)].
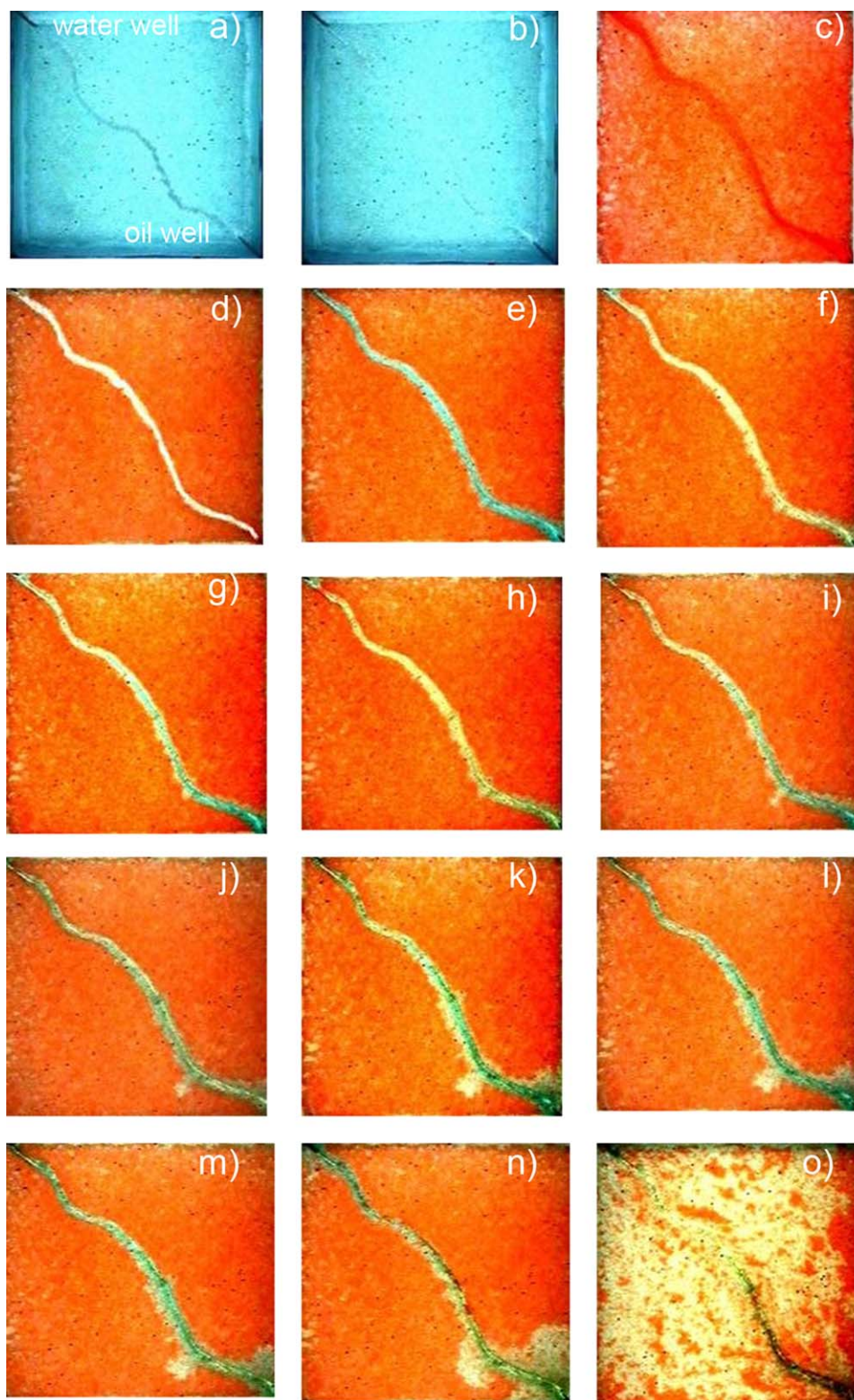
Figure 11. Visual simulation result of alternate injection cationic polymer and anionic polymer. (a): model; (b): saturating water; (c): saturating oil; (d): water flooding until water cut is up to 98%; (e): first round of injecting cationic polymer; (f): first round of injecting anionic polymer; (g): second round of injecting cationic polymer; (h): second round of injecting anionic polymer; (i): third round of injecting cationic polymer; (j): third round of injecting anionic polymer; (k): fourth round of injecting cationic polymer; (l): fourth round of injecting anionic polymer; (m): fifth round of injecting cationic polymer; (n): fifth round of injecting anionic polymer; (o): water flooding until water cut is up to 98% again. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
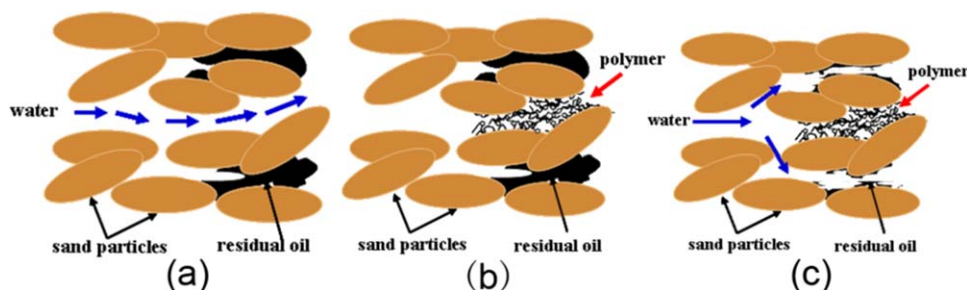
Figure 12. Schematic illustration for the enhanced oil recovery mechanism. (a): a high permeability zone was formed after a long-term water flooding; (b): alternative injection of cationic polymer and anionic polymer from the produced oil well for fully adsorption; (c): water flooding after the treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Adsorption Behavior in Visual Model

Visual simulation experiments were generally used to observe adsorption behavior and illustrate mechanism of enhanced oil recovery. The experiment was performed under CA injection model at 75°C. Figure 11 shows the whole process of using cationic polymer and anionic polymer as water shutoff treatment in visual model.

Figure 11(c) simulated oil distribution in original formation. In this initial stage of development, water flooding was usually used to exploit oil in the formation. However, a high permeability zone [Figure 11(d)] was formed after a long period of water flooding, resulting in invalid circulation of injection water. The oil recovery was only 9% after first water flooding. In order to enhance oil recovery, alternate injection of cationic polymer and anionic polymer were conducted for the moderate water shutoff treatment. The cationic polymer was first injected into the model, which adsorbed on the sand surface for the electrostatic interactions and formed a single adsorption layer, thus plugged pore throats in high permeability zone [Figure 11(e)]. But the plugging capacity was relatively weak, so that anionic polymer was sequentially injected in to the model [Figure 11(f)]. During the process, the anionic polymer neutralized the cationic polymer adsorbed on the sand surface. As a result, a new electrostatic imbalance between the anionic polymer and cationic polymer might be created, leading to the formation of a new stable adsorption layer, which further reduced pore throats. After alternately injecting five rounds of cationic polymer and anionic polymer, an adsorption multilayer was formed and effectively plugged high permeability zones [Figure 11(n)]. When water flooding was employed again after the treatment, the sweep efficiency was improved, and the residual oil was forced out of the small pores into the production well, leading to the enhanced oil recovery up to 60% [Figure 11(o)]. It can be considered as an effective and moderate method for water shutoff treatment.

ADSORPTION AND ENHANCED OIL RECOVERY MECHANISM

Based on the above results, a possible adsorption and enhanced oil recovery mechanism of cationic polymer and anionic polymer used for water shutoff treatment has been proposed and shown in Figures 12 and 13. At first, a high permeability zone was formed after a long-term water flooding, resulting in invalid circulation of the injection water, thus a large amount of residual oil was still remained in the formation [Figure 12(a)]. To enhance oil recovery, cationic polymer and anionic polymer were injected for the water shutoff treatment. When alternative injection of cationic polymer and anionic polymer from produced oil well, the polymers preferentially entered the high permeability zone and adsorbed on the sand surface, forming a single adsorption layer. Further injection of cationic polymer and anionic polymer for several rounds led to form an adsorption multilayer [Figure 12(b)] which effectively plugged the high permeability zones, resulting in the following water turned to the low permeability zone [Figure 12(c)]. As a result, the residual oil was driven out and oil recovery was improved.

As discussed above, the adsorption of polymers on the sand surface was critical for the successful water shutoff treatment. Although the hydrogen bonding interactions increased the adsorption capacity, the electrostatic interactions between sand surface, cationic polymer and anionic polymer were the main driving forces for adsorption capacity in the process. The cationic polymer was initially adsorbed on the sand surface [Figure 13(c)], and then the anionic polymer adsorbed on the sites of the cationic polymer for the electrostatic interactions, which formed an adsorption multilayer on the sand surface [Figure 13(e)]. This was in accordance well with the AFM experimental observations.
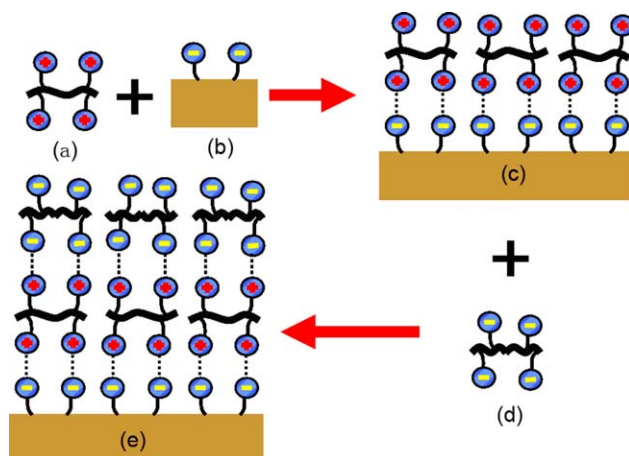

Figure 13. Schematic illustration for the adsorption mechanism. (a) cationic polymer; (b) sand surface; (c) forming a single adsorption layer on the sand surface; (d) anionic polymer; (e) forming an adsorption multilayer on the sand surface. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
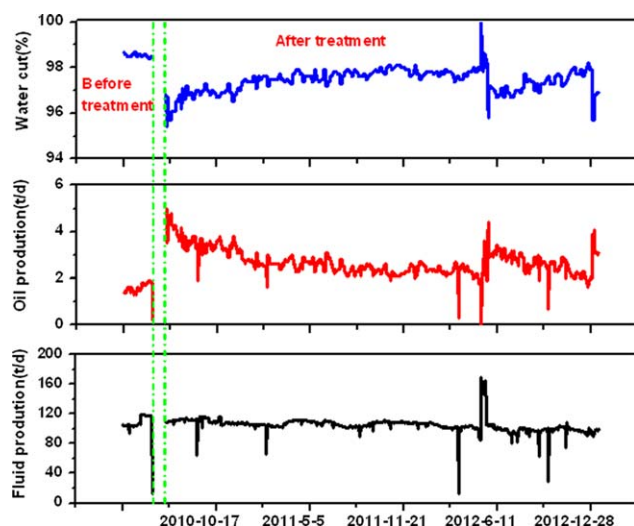
Figure 14. The production curve of Shuangqian 6 well. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

FIELD APPLICATION

Alternative injection of cationic polymer and anionic polymer as a moderate water shutoff treatment has been successfully applied in Henan oilfield of China. Shuangqian 6 well as the production well is located in the south of single sand body in Henan oilfield. Up to June 2010, the total water cut has reached 98.6% and the oil production was only 1.6 ton/day. Further test showed that the wells had a severe areal heterogeneity and a high permeability channel between injectors and producers. So alternative injection with three rounds of cationic polymer (1500 mg/L) and anionic polymer (1000 mg/L) were conducted on Shuangqian 6 well in June 2010. During the treatment process, about 360 m^3 cationic polymer and 360 m^3 anionic polymer were injected into the production well, and then shutted off the oil well for 2 days for the polymers fully adsorbed on the high permeability zone. After water shutoff treatment, the oil production gradually increased from 1.6 ton/day to 2.6 ton/day and the water cut decreased from 98.6% to 96.8% (Figure 14). The results indicate that alternative injection of cationic polymer and anionic polymer as a moderate water shutoff agent was an effective treatment for the high water cut reservoir.

CONCLUSIONS

In this study, cationic polymer and anionic polymer are selected as a moderate water shutoff agent for water production control in mature oilfield. The effect of polymer concentration, adsorption time, temperature, shearing time, wettability, and injection modes on the adsorption behavior between the polymer and sand surface are investigated. The adsorption equilibrium concentration and adsorption equilibrium time of cationic polymer are 1500 mg/L and 8 h, while adsorption equilibrium concentration and adsorption equilibrium time of anionic polymer 1000 mg/L and 6 h. Adsorption multilayer is formed and effectively plugs high permeability zones, which insures a successful treatment for enhanced oil recovery. The treatment have been successfully used in Henan oilfield of China which provides a

reference for the development of other similar high water cut oilfields in worldwide.

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