

Study of action mechanisms and properties of Cr³⁺ cross-linked polymer solution with high salinity

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Abstract: Performance characteristics of partially hydrolyzed polyacrylamide (HPAM) and cross-linked polymer (CLP, Cr³⁺ as the cross linker) solutions have been investigated. A Brookfield viscometer, rheometer, dynamic light scattering system, and core flow device have been used to measure the viscosity, viscoelasticity, polymer coil dimensions, molecular configuration, flow characteristics, and profile modification. The results show that, under conditions of high salinity and low HPAM and Cr³⁺ concentrations, cross-linking mainly occurred between different chains of the same HPAM molecule in the presence of Cr³⁺, and a cross-linked polymer (CLP) system with a local network structure was formed. Compared with an HPAM solution of the same concentration, the apparent viscosity of the CLP solution increased slightly or remained almost unchanged, but its viscoelasticity (namely storage modulus, loss modulus, and first normal stress difference) increased, and the resistance coefficient and residual resistance coefficient increased significantly. This indicates that the CLP solution exhibits a strong capability to divert the sequentially injected polymer flood from high-permeability zones to low-permeability zones in a reservoir. Under the same HPAM concentration conditions, the dimensions of polymer coils in the CLP solution increased slightly compared with the dimensions of polymer coils in HPAM solution, which were smaller than the rock pores, indicating that the cross-linked polymer solution was well adapted to reservoir rocks. Core flood experiments show that at the same cost of reagent, the oil recovery by CLP injection (HPAM-1, Cr³⁺ as the cross linker) is 3.1% to 5.2% higher than that by HPAM-2 injection.

Key words: Cross-linked polymer solution, apparent viscosity, viscoelasticity, polymer coil dimension, molecular configuration, flow characteristics, profile modification

1 Introduction

In recent years, polymer flooding has been successfully applied in many oilfields in China, such as the Daqing, Shengli, and Henan oilfields, reducing water production and increasing crude oil production (Wang et al, 2005; Sun, 2006; Liu et al, 2008). Owing to long-term water flooding, heterogeneity of oil reservoirs and intra-bed and inter-bed complications became more serious, which cause part of injected polymer solutions to inefficiently circulate mainly in the high permeability formations leading to poor oil recovery in the polymer flooding and water flooding operations. Furthermore, commonly used polymer flooding hardly meets the practical needs in high-temperature and high salinity reservoirs (Wang et al, 2009; Zhang et al, 2006). Thus, it is necessary to develop a low-cost displacing agent, which has good ability to control fluid mobility and good compatibility

with reservoir rocks.

Plenty of research has been done on the viscosity, polymer coil dimensions, and configuration of Al³⁺ cross-linked polymers (Smith and Mack, 1997; Han et al, 2006; Lu and Wang, 2006; Wang et al, 2008; Al-Assi et al, 2009; Lu et al, 2009; 2010; Spildo et al, 2010; Shi et al, 2010). According to geological characteristics of offshore heavy oil reservoirs and fluid properties, the authors studied the viscosity, viscoelasticity, polymer coil dimensions, molecular configuration, flow characteristics, and profile modification of Cr³⁺ cross-linked polymer of high salinity and low Cr³⁺ and polymer concentrations. At the same time, the performance of Cr³⁺ cross-linked polymer solution was compared with the commonly used polymer solution and the action mechanism was analyzed.

2 Experimental

2.1 Materials

Water The water used in all experiments was injection

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water taken from the LD10-1 Oil Field, Bohai Sea, China and its composition is listed in Table 1.

Polymer Two partially hydrolyzed polyacrylamides (HPAMs), provided by the PetroChina Daqing Refining & Chemical Company, were used as the polymer. HPAM-1 has a relative molecular weight of 1.9×10^7 , active ingredient of 88.0%, and degree of hydrolysis of 25.6mol%; HPAM-2 has a relative molecular weight of 3.5×10^7 , active ingredient of 90.1%, and degree of hydrolysis of 26.0mol%.

Cross-linker Chromium acetate solution was used as the cross-linker, with an active content of Cr^{3+} of 2.7%.

Cross-linked polymer A measured amount of HPAM was slowly added to stirred water, and then the sample was stirred for 2-3 h until the HPAM dissolved in water. Under stirring, a specified amount of chromium acetate was added and the resulting solution was named CLP.

Oil The oil was prepared by blending kerosene with degassed crude oil from the LD10-1 Oil Field, with a viscosity of 16.8 mPa·s at 65 °C.

Cores Artificial core samples were made of clay-free quartz sand and epoxy resin (as cementing agent).

The homogenous cores (10 cm long, 2.5 cm diameter), with permeability of 2.4 Darcy, were used to evaluate the flow properties of polymer solutions.

The heterogeneous cores (30 cm long, 4.5 cm width, 4.5 cm height) were composed of high, medium, and low permeability layers. The three layers had thicknesses of 1, 2, and 1.5 cm, respectively, and gas permeability of 6.2, 2.5, and 1.0 Darcy, respectively. The cores were used to evaluate the profile modification by polymer solutions.

Table 1 The ion composition of injection water

Ion composition and content, mg/L							Total salinity
$\text{K}^+\text{+Na}^+$	Ca^{2+}	Mg^{2+}	Cl^-	SO_4^{2-}	CO_3^{2-}	HCO_3^-	mg/L
2968.8	826.7	60.8	6051.6	60.0	0.0	208.7	10176.6

2.2 Measurement methods

2.2.1 Viscosity

The viscosity of each sample was measured systemically with an RV2 Brookfield viscometer (made by Brookfield Engineering Laboratories, Inc, USA) at 65 °C at a rotational speed of 6 r/min.

2.2.2 The dimensions of polymer coils in water

The dimensions (D_h) of polymer coils in water were measured with a Brookhaven dynamic/static light scattering system (model BI-200MS, Brookhaven Instruments Cop, USA) at 65 °C, which included a BI-9000AT laser correlator, a signal conditioner, and an argon ion laser (200 mW, laser wavelength 532.0 nm) at a scatter angle of 90°. Before measurement, all test samples were filtered through a microporous membrane (3.0 μm) and all sample bottles were cleaned thoroughly using a KQ3200DE NC ultrasonic cleaner.

2.2.3 Viscoelasticity

The viscoelasticity of displacing agents was measured with a Haake100 rheometer at 65 °C. The frequency sweeps were performed on polymer solutions at a constant stress of 0.1

Pa and the scanning range was 10^{-5} -1 Hz. The stress sweeps were conducted at a fixed frequency of 1 Hz and the scanning range was 0.01-50 Pa, with 0.002 Pa accuracy. The data were automatically recorded and stored on a computer.

2.2.4 Flow properties and profile modification

Flow properties of the HPAM or CLP solutions are characterized by injection pressure, resistance coefficient, and residual resistance coefficient. In this paper, the resistance coefficient, F_R , and the residual resistance coefficient, F_{RR} , were used to describe the remnant amount of the HPAM or CLP solution within porous media, which can be calculated as follows:

$$F_R = \frac{\delta P_1}{\delta P_0}$$

$$F_{RR} = \frac{\delta P_2}{\delta P_0}$$

where δP_0 is the stable pressure difference between the two ends of the sample when the water is injected into the core sample, MPa; δP_1 is the stable pressure difference when the slug of HPAM or CLP solution is injected, MPa; and δP_2 is the stable pressure difference when the water is subsequently injected, MPa. In the test, the displacing fluids are injected at a constant rate and the injection volume is 3-4 PV.

The flow tests and oil displacement tests were performed at 65 °C and the experimental apparatus and procedures are described elsewhere (Zhang et al, 2011).

3 Results and discussion

3.1 Viscosity and flow properties

When the HPAM concentration, c_{HPAM} was 1,200 mg/L, the viscosities of different displacing agents are shown in Table 2.

Table 2 The viscosity data of displacing fluids

Displacing fluid	Viscosity at different standing times, mPa·s						
	0	2 d	5 d	15 d	25 d	40 d	60 d
HPAM-2 solution	9.0	11.9	11.7	11.2	11.0	10.5	10.6
HPAM-1 solution	7.4	9.7	9.0	8.6	8.4	8.2	8.4
CLP solution	7.4	9.5	8.1	8.7	8.8	8.2	9.2

Notes: In the CLP solution, the mass ratio of HPAM to Cr^{3+} , $m_{\text{HPAM}}:m_{\text{Cr}^{3+}}$ was 270:1.

As shown in Table 2, the viscosity of the CLP solution was essentially the same as that of the HPAM-1 solution, indicating that the cross-linking reaction did not take place, or cross-linking mainly took place in different chains of the same HPAM molecule. That is to say, intramolecular cross-linking of HPAMs mainly occurred supplemented with intermolecular cross-linking, and a "local" network structure of the CLP was formed in the solution.

When the HPAM concentration was 1,200 mg/L and the core permeability was 2.4 Darcy, the flow properties of different displacing fluids were measured and the results are

shown in Table 3 and Fig. 1.

Table 3 The resistance coefficients and residual resistance coefficients of different displacing fluids

Displacing fluid	Resistance coefficient F_R	Residual resistance coefficient F_{RR}
1	1967	4839
CLP 2	4032	6290
3	1065	4516
HPAM-1	8.5	3.2
HPAM-2	9.7	4.0

Notes: In the CLP solution, $m_{HPAM} : m_{Cr^{3+}} = 270:1$.

Tables 2 and 3 indicate that the CLP had a viscosity close to HPAM-1 solution, but its resistance coefficient (F_R) and residual resistance coefficient (F_{RR}) were much higher than that of the HPAM solutions. Moreover, the flow properties of the CLP solution were different from the HPAM solutions. The injection pressure of the subsequent water flooding increased by a large margin instead of decreasing. Table 3 shows that F_R and F_{RR} of CLP-2 was different from that of CLP-1 and CLP-3 because the cross-linked degree (cross-linked temperature: CLP-1, 45 °C; CLP-2, 65 °C; CLP-3, 80 °C) of the CLP-2 was slightly different from that of CLP-1 and CLP-3.

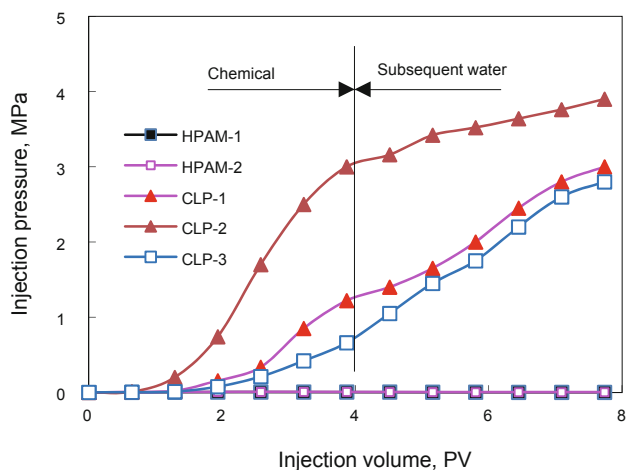


Fig. 1 The relationship between the injection pressure and injection volume of the displacing fluid

3.2 Polymer coil dimensions and the influential factors

The HPAM concentration influenced the dimensions (D_h) of polymer coils in displacing fluids and the results are shown in Table 4. The dimensions of coils in HPAM and CLP solutions increased with an increase in the HPAM concentration. Compared with HPAM-1 solutions, the dimensions of CLP coils almost unchanged. Moreover, the dimensions of polymer coils in the HPAM-2 solution was larger than that in the HPAM-1 and CLP solutions due to higher relative molecular weight.

Table 4 Dimensions of polymer coils in displacing fluids at different HPAM concentrations

HPAM concentration mg/L	D_h of polymer coils, nm		
	HPAM-1	CLP	HPAM-2
50	99.5	100.9	181.4
100	215.2	220.6	290.2
150	242.1	252.6	343.7

Notes: In the CLP solution $m_{HPAM} : m_{Cr^{3+}} = 270:1$.

Table 5 shows that the standing time influenced significantly the dimensions of polymer coils in the displacing fluids. The dimensions of polymer coils in the HPAM and CLP solutions first increased and then decreased with time. As can be seen from Table 5, the retention ratio of D_h of the CLP solution at 60 days was 85.6%, higher than that of HPAM-1 (31.8%) and HPAM-2 (32.2%), so it can be concluded that the long term stability of the CLP solution was better than HPAM solutions.

Table 5 Variation of polymer coil dimensions with time

Standing time d	D_h of polymer coils, nm		
	HPAM-1	CLP	HPAM-2
1	215.2	220.6	290.2
2	235.3	299.3	535.2
5	231.4	366.8	435.0
15	125.2	335.9	194.8
25	116.2	320.9	104.0
40	75.2	273.6	113.2
60	68.5	188.8	93.6
Retention ratio at 60 days, %	31.8	85.6	32.2

Notes: $c_{HPAM} = 100$ mg/L, $m_{HPAM} : m_{Cr^{3+}} = 270:1$.

Table 6 shows that shearing action has an influence on the dimensions of polymer coils in displacing fluids. The dimensions of polymer coils in HPAM and CLP solutions decreased with an increase in shearing time. Compared with HPAM solutions, the retention ratio of D_h of CLP coils was higher after shearing. This shows that the CLP solution had a relatively high ability to resist shear force (i.e. high shear strength).

Table 6 Variation of polymer coil dimensions before and after shearing

Shear time s	D_h of polymer coils, nm			
	HPAM-1	CLP	HPAM-2	
0	215.2	220.6	290.2	
10	122.3	206.0	165.2	
30	74.1	145.6	106.1	
Retention ratio, %	10 s	56.8	93.4	56.9
	30 s	34.4	66.0	36.6

Notes: $c_{HPAM} = 100$ mg/L, $m_{HPAM} : m_{Cr^{3+}} = 270:1$; the solutions were sheared at a rate of 2000 r/min.

3.3 Viscoelasticity

The viscoelasticity results of HPAM and CLP solutions are shown in Figs. 2 and 3. The viscoelasticity of the CLP solution was different from that of HPAM solutions. Under the same vibration frequency, the CLP solution had higher storage modulus (G'), loss modulus (G'') and the first normal stress difference (N_1) than HPAM solutions. The G' , G'' and N_1 of HPAM solutions tended to increase, but the values of the CLP solution decreased with an increase in the vibration frequency.

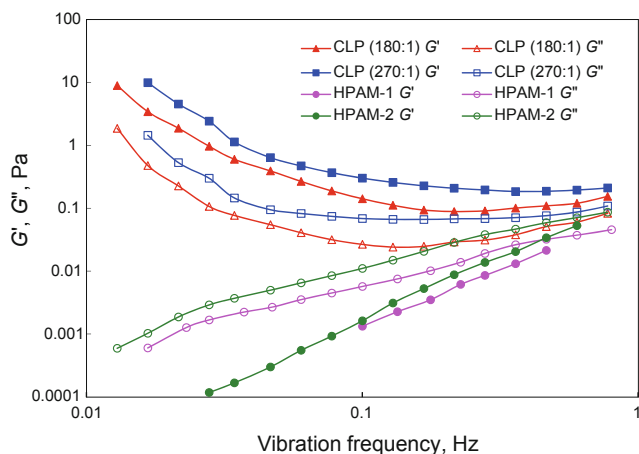


Fig. 2 The relationship of storage modulus (G') and loss modulus (G'') with vibration frequency ($c_{\text{HPAM}}=1200$ mg/L)

The reason for this is that when the vibration frequency was lower, i.e. the action time of force was longer, the polymer coils in water had sufficient time to change their conformation, and to unwind, while the elastic stretch of chains could gradually recover in the flow, so the viscosity dominated in the polymer molecule instead of elasticity. When the vibration frequency was higher, i.e. the action time of the force was shorter, most of elastic deformation energy was stored in the system. The energy of viscous dissipation was relatively small, so the elasticity was dominant (Li et al, 2003). The case of the Cr^{3+} linked polymer solution was

different, because of the coordination bond, the conformation of polymer molecules could not change freely. Only when the weak coordination bonds were destroyed, could the polymer conformation change as polymer molecules in polymer solution. When the vibration frequency was large enough, the coordination bonds of intra-molecular cross-linking were destroyed completely, and G' and G'' of Cr^{3+} -linked polymer solution were consistent with those of the HPAM solution.

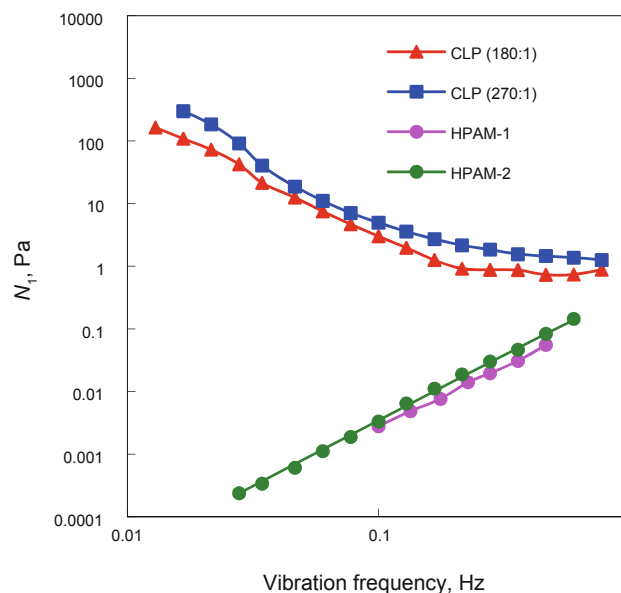


Fig. 3 The relationship between first normal stress difference (N_1) and vibration frequency ($c_{\text{HPAM}}=1200$ mg/L)

3.4 Profile modification

The profile modification tests were performed on artificial core samples using HPAM-2 and CLP solutions and experimental results are listed in Table 7.

As shown in Table 7, the volume of the displacing fluid injected into the core samples (slug size) influenced the profile modification. After 0.05 PV, 0.10 PV, and 0.15 PV of HPAM-2 solution was respectively injected into the core

Table 7 Oil recovery for different core flooding experiments

Displacing fluid	Injection volume PV	Viscosity of the displacing fluid, mPa·s	Oil saturation %	Oil recovery, %		Incremental oil recovery %
				Water flooding	Chemical flooding	
Water flood alone	–	0.6	75.9	41.4	–	–
	0.05	7.5	75.4	28.3	46.1	4.7
	0.10	7.6	76.0	28.6	51.7	10.3
HPAM-2	0.15	7.6	76.5	28.4	54.6	13.2
	0.05	4.3	76.3	28.5	49.2	7.8
CLP	0.10	4.3	75.5	28.4	56.0	14.6
	0.15	4.3	76.2	28.7	59.8	18.4

Notes: 1) In the HPAM-2 solution, $c_{\text{HPAM}}=1600$ mg/L; in the CLP solution, $c_{\text{HPAM}}=1200$ mg/L, $m_{\text{HPAM}}:m_{\text{Cr}^{3+}}=270:1$.

2) Water flood alone: Water injection was continued until the water cut approached 98%.

3) Chemical flooding: Water was injected into the core until the water cut reached 40%, and then a specified volume of the HPAM or CLP solution was injected into the core. After that the core was subsequently flooded with water until the water cut reached 98%.

sample, the incremental oil recovery values were 4.7%, 10.3% and 13.2%, respectively; and as for the CLP solution, the incremental values were 7.8%, 14.6%, and 18.4%, respectively. This shows that the greater the amount of the displacing fluid was injected, the higher the oil recovery increment was. Further analysis shows that the oil recovered by the HPAM solution was lower (3.1% to 5.2%) than that recovered by the CLP solution under the same displacement conditions.

When the injection volume of the chemical slug was 0.1 PV, the relationships of the injection pressure, water cut, oil recovery and the injection volume are shown in Fig. 4.

As shown in Fig. 4, in the water flooding operation, the injection pressure decreased gradually, but the oil recovery and water cut increased rapidly. In the chemical flooding process, the injection pressure decreased slightly and then increased quickly. In the subsequent water flooding operation, the injection pressure increased rapidly and then decreased, but the water cut first increased rapidly and then decreased rapidly, and at last tended to increase again. The oil recovery increment decreased gradually. Compared with the HPAM-2 solution, the injection pressure and oil recovery increment were higher when the CLP solution was injected into the core. One can conclude that at the same cost of reagents, the oil recovery by the CLP system was 3.1%-5.2% higher than that by the HPAM-2 solution. Therefore, CLP was recommended.

4 Mechanism analysis

4.1 The mechanism of cross-linking reaction

According to fractal growth theory (Zhang, 1995) and the Stern-Grahame model for electric double layers (Shen et al, 2008), the reason for the difference in molecular structure and morphology of the CLP in water is concentration differences of electrolytes, cross-linking agent, and HPAM in water. In the displacing fluid, the ionized carboxyl groups in HPAM chains have the same electric charge, therefore, they repel each other. When the electrolyte concentration in the displacing fluid was lower, but the HPAM and Cr^{3+} concentration was higher, the ionic strength was lower, so the HPAM chains extended, and these stretched polymer chains touched each other or even were intertwined. If Cr^{3+} was introduced into the fluid, the Cr^{3+} was easily linked to the carboxyl groups in different HPAM chains due to its random thermal motion, forming a structural unit. These structural unit would be growing at the mode of "aggregate-aggregate" until another aggregate closed to and excluded it because the growth process would suffer from space constraints, then re-growth would begin from another direction, and finally the CLP system with a "regional" network molecular structure would form in the solution (Fig. 5 (a)). From a macroscopic view, the apparent viscosity would significantly increase. When the electrolyte concentration in the displacing fluid was higher, but the HPAM concentration was lower, the cations would migrate to the Stern layer at the surface of HPAM chains because of high cation concentration, and some of them would enter the Stern layer and neutralize part of negative charges on the molecular chain surface. This would reduce the thickness of the polymer molecular diffusion layer, thus the molecular chains of HPAM would curl and the polymer coil dimensions would decrease, which makes contact between different polymer chains difficult. If Cr^{3+} was introduced into the HPAM solution, intra-molecular cross-linking would occur between different branched-chains of the same HPAM chain, and finally the CLP system was formed with "localized" network molecular structure (Fig. 5 (b)). From a macroscopic view, the apparent viscosity of the system would increase slightly or remain constant.

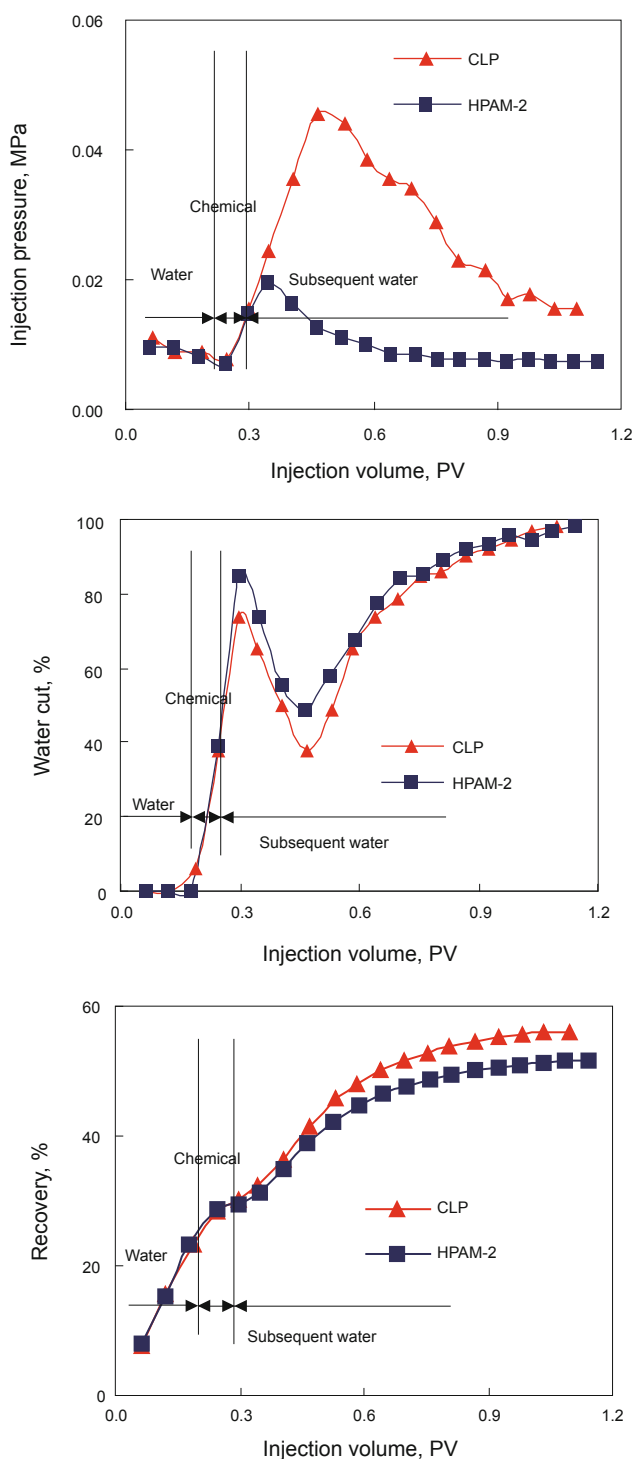
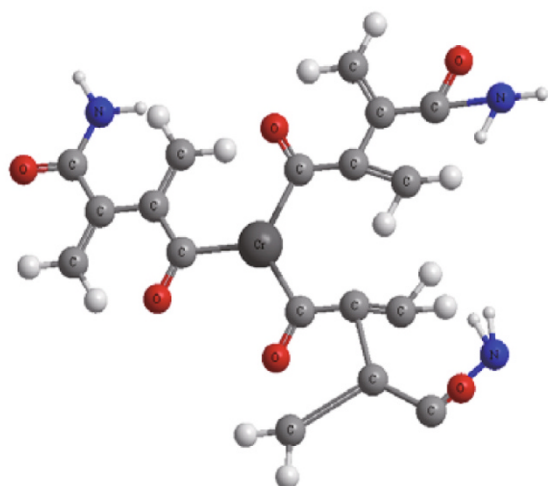
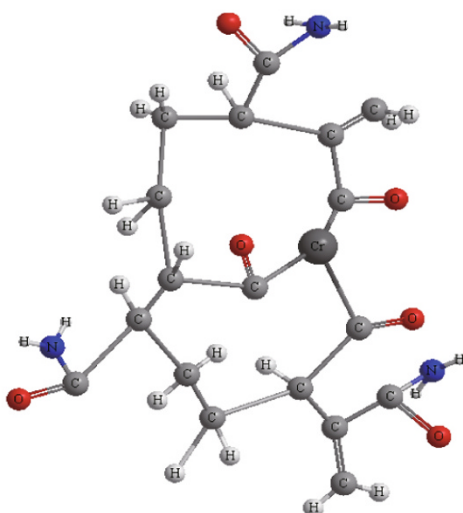


Fig. 4 Dynamic characteristics in the displacement process (In the HPAM-2 solution, $c_{HPAM-2}=1600$ mg/L; in the CLP solution, $c_{HPAM-1}=1200$ mg/L, $m_{HPAM} : m_{Cr^{3+}} = 270:1$)



(a) Intermolecular cross-linking



(b) Intramolecular cross-linking

Fig. 5 Molecular configurations of cross-linked polymer systems

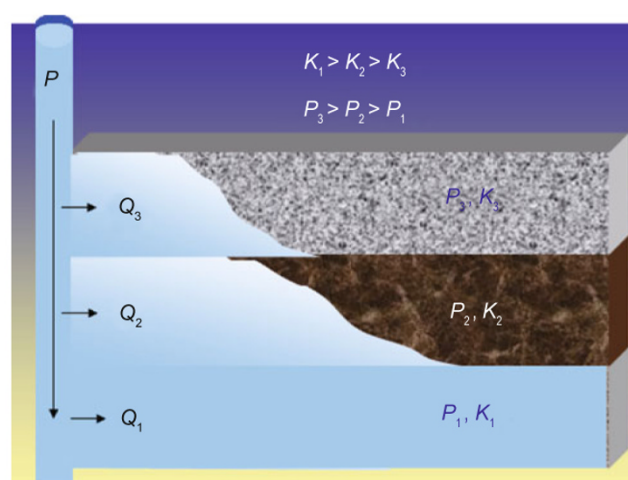
4.2 Mechanism of oil recovery

During the formation of the oil reservoir, due to the impact of environmental changes, the reservoir became badly inhomogeneous in the vertical and horizontal directions, which during production results in the injection water mainly flowing into the high permeability layers, instead of the medium and low permeability ones, combined with the unfavorable mobility ratio of the displacing phase to the displaced phase. Therefore, the key to enhanced oil recovery (EOR) was to expand the macro and micro swept volumes in heterogeneous reservoirs.

To demonstrate the enhanced oil recovery mechanism of chemical flooding, a typical intra-layer heterogeneous reservoir model was established (see Fig. 6). The model was composed of high, medium and low permeability layers, where K_1 , K_2 , and K_3 represent permeabilities of the high, medium, and low permeability layers; P_1 , P_2 , and P_3 represent

the suction threshold pressure respectively; and Q_1 , Q_2 , and Q_3 represent the amount of fluid absorption in each layer. In the water flooding process, because the permeability was higher and the threshold pressure was lower in the high permeability layer, the suction pressure differential (the gap between injection pressure and suction threshold pressure) and the fluid absorption amount were higher, and the fluid absorption amount would increase gradually as the water flooding lasted. In the early stage of the chemical flooding, the displacing agent would enter the high permeability layer first and then adsorbed on the surface of reservoir pores, which would make the cross-section of the pores decrease and the flow resistance and injection pressure (P) increase. As the injection pressure (P) increased, the suction pressure differential of the medium and low permeability layers ($\delta P_3 = P - P_3$, $\delta P_2 = P - P_2$) increased, and the fluid absorption amount improved at the same time, the displacing agent would be retained while entering in the low permeability layer, which caused the flow resistance to increase, and suction pressure differential (δP_3 and δP_2) and the fluid absorption amount to decrease.

Compared with the HPAM solution, the dimensions of polymer coils in CLP solution increased slightly, and both HPAM and CLP solutions had the similar adaptability to reservoir rock. The CLP solution could enter the pores in the reservoir as the HPAM solution did. On the other hand, the viscoelasticity and flow characteristics of the CLP solution were improved significantly, making the injection pressure substantially increase (see Fig. 4), the suction pressure differential (δP_3 and δP_2) of the layer and the fluid absorption amount subsequently increase, and swept volume and oil recovery improve at the same time.

**Fig. 6** Schematic diagram of a typical reservoir model

5 Conclusions

- 1) When CLPs were prepared with high concentration of electrolyte and low concentrations of polymer and Cr^{3+} , intramolecular cross-linking took place between different branched-chains of the same polymer in the presence of Cr^{3+} , and the CLPs were characterized by "localized" cross-linking.
- 2) Compared with the HPAM solution, the apparent

viscosity of the CLP solution increased slightly, but its resistance coefficient and residual resistance coefficient increased substantially.

3) Under the same HPAM concentration, the viscoelasticity of the CLP solution was higher than that of the HPAM-1 and HPAM-2 solutions. Unlike HPAM-1 and HPAM-2 solutions, the elasticity of CLP solution dominated.

4) The CLP was of the similar coil dimensions with the HPAM solution, which was compatible with the reservoir rock.

5) At the same costs of reagents, the oil recovery by the CLP solution was higher 3.1%-5.2% than that by the HPAM-2 solution.

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