

The Shearing Effect on Hydrophobically Associative Water-Soluble Polymer and Partially Hydrolyzed Polyacrylamide Passing Through Wellbore Simulation Device

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ABSTRACT: Hydrolyzed polyacrylamide (HPAM) was the traditional polymer and hydrophobically associative water-soluble polymer (HAWP) was the new polymer with three-dimensional network both used to flood to enhance oil recovery. The wellbore area was the most important part before the polymer solution injected into stratum. In this article, the shearing effects of the two polymers were studied by a wellbore simulation device. The viscosities of HPAM and HAWP solutions were both decreased around perforation of wellbore simulation device. Interestingly, viscosity of HAWP recovered from stratum 0.2 m. Until stratum 1.6 m, its viscosity recovered almost 50% of original. The data of intrinsic viscosity showed that the molecular chains of HAWP and HPAM were both degraded without any recovery. The contradiction was further studied by particle size and its microstructure. The mean particle size and particle size distribution data both showed HAWP recovered but HPAM was not. The microstructures of HAWP by atomic force microscopy images further explain the recovery of viscosity. The disassembled molecular chain was self-assembled into aggregate to newly network by hydrophobes with weaker linking than original solution. While the microstructure of HPAM was thoroughly split up to randomly coil without linking. In addition, the viscoelasticity of HAWP was also recovered to some extent but HPAM was not. All the results proved that HAWP has mobility control ability to displace oil in reservoir even if suffered severely shearing by wellbore.

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INTRODUCTION

Polymer flooding is one of the most attractive methods proposed in 1960s.^{1–8} Requiring little more technology and equipment than conventional water flooding, the process is simple to implement in oil field. When compared with conventional water flooding, great fractions of the reservoir volume may be swept. The plant pathogen *Xanthomonas campestris* and hydrolyzed polyacrylamide (HPAM) were first chosen to be used for flooding. Especially, HPAM was the most widely used mobility control polymer for secondary or tertiary oil recovery.⁹ Only small quantities of HPAM can increase the viscosity of water by two or more orders of magnitude. More literatures^{10–15} showed that HPAM solutions suffer mechanical degradation during the injection process flowing through porous media of wellbore. The mechanical degradation of the polymer solution may severely reduce its ability to provide mobility control of flood-

ing. White et al.¹⁴ was the first to present a figure showing that injection rate limited to minimize polymer degradation at various permeability levels. However, he did not indicate the degree of degradation which would occur if the flow rate limits were exceeded. The other papers^{11,16,17} described experimental studies of polymer degradation: Maerker's^{11,17} articles described an extensive experimental study of mechanical degradation of HPAM solutions. The data described in this article were obtained by a procedure similar to those obtained shown that mechanical shearing degradation of polymer solutions depended on polymer molecular weight, polymer concentration, core permeability, and flow rate. Two papers^{12,18} described field tests designed to determine the extent of mechanical degradation of polymer solutions during injection and the degree to which the mobility control properties of these solutions were affected by flow out of a wellbore into the oil reservoir. Jackson and

Table I. The Composition of Brine Used

Component	Na ⁺	Ca ²⁺	Mg ²⁺	CO ₃ ²⁺	HCO ₃ ⁺	SO ₄ ²⁺	Cl ⁻	Total dissolved inorganic salts
Concentration (mg/L)	3091	276	158	14	311	85	5436	9374

Morris¹⁹ further proved that the degree of shear degradation was found to depend upon the stretching rate and molecular weight of the polymer. HPAM was not the best choice to control mobility to displace oil in reservoir.

Hydrophobically associating water-soluble polymers (HAWP) has attracted increasing attention recently because of its fundamental and practical importance.^{20,21} It is derived from HPAM by incorporating a relatively low amount of hydrophobic groups onto the polymer backbone.²² When compared with HPAM, HAWP exhibits stronger viscosifying capacity at lower concentration, together with improved salinity resistance and mechanical stability in semidilute aqueous solution.^{23,24} In aqueous solutions, the hydrophobes induce molecular chain self-assembly to aggregates comparable with surfactant micelles and the hydrophobes act as transient junctions and connect different aggregates depending on increasing concentration. Above a threshold concentration corresponding to the formation of a three-dimensional reversible network, the solutions behave as physical gels. The viscosity of such gels is higher by several orders of magnitude than that of solutions made with unmodified polymers of same molecular weight and at the same concentration.²⁵ Especially, the three-dimensional reversible network offers HAWP solutions possibility to control mobility even if severely shearing.

In this article, we present the results of a detailed study into the shearing effect of the wellbore to the HAWP and HPAM solutions. A shearing simulation model device was designed by oilfield. Rheological behavior at different points of the model device has been investigated. The intrinsic viscosity was investigated to reflect the change of molecular weight. The mean particle size and distribution were studied by dynamic light scattering (DSL). Moreover, the microstructure of the polymer has been studied further by atomic force microscopy (AFM). The viscoelasticity was also investigated through stress relaxation. The observed rheological phenomena are discussed in terms of the microstructure change of the polymer solution, and the intrinsic reason for the change is also deduced. The rheological properties and microstructure difference between the two polymers solutions were also compared and explained.

EXPERIMENTAL

All inorganic salts were purchased from Aldrich (AR grade) and used as received. The brine used in the experiments had similar composition to the water injected into S heavy oil reservoir in Bohai Oilfield. Table I shows the brine composition. All inorganic salts were dissolved in distilled water.

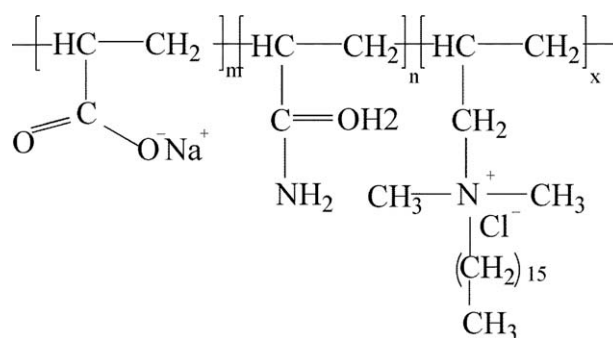
Two types of polymers, HPAM 3830 and HAWP 0312, were used in these experiments. HPAM 3830 was obtained from the laboratory in Daqing Oilfield with molecular weight of 22×10^6 . The HAWP 0312 was obtained from Polymer Guangya

(China) with molecular weight of 12×10^6 (The molecular structure was shown in Figure 1). The hydrophobic monomer is about 0.23% v/v. The two polymers solutions were stirred by mechanical agitation at 45°C for 2 and 4 h in brine, respectively. Then, it was placed for 24 h as the aging time for sample dissolution. To obtain a similar viscosity, the concentration of HPAM 3830 was 6000 mg/L, whereas HAWP 0312 was 1750 mg/L.

Wellbore Simulation Device

The wellbore simulation model was designed based on the condition in oilfield in Bohai of China at the ratio of 1 : 1. The model was composed of pump, intermediate container, wire-wrapped and gravel-pack screen, perforation, and the stratum. The original polymer solution was placed in intermediate container, which was injected into the wire-wrapped and gravel-pack screen by the pump at a certain pressure. The gravel was filled into wire-wrapped and gravel-pack screen, perforation. The stratum was filled with quartz sand and its porosity was 23.1%. The shearing rates of the polymer passing through the different parts of the model were also shown in Figure 2.

The first part is well tubing, through which the original polymer solution was injected into the next part. In the simulation device, the well tubing was designed to be a pot connected with a pump, which could provide appropriate pressure to the solution. The second part was the wire-wrapped and gravel-pack screen, which was the sand control tool. The slot width of the wire-wrapped screen was about 0.1524 mm and was 360° spiral distribution. The gravel-pack screen was 0.04235 m thickness, shown ring-shaped. The porosity of packed layer was 37%, and the average permeability of it was $61.1 \mu\text{m}^2$. The average diameter of the gravel was 0.35 mm. The third part of the model device was perforation, which was the only route way between the well tubing and the stratum. The diameter of it was 0.021 m and the length was 0.18 m. The perforation tunnel was packed with gravel with the size and porosity same as the gravel-pack screen part. The fourth part was the stratum of the simulation device. To study the influence of the stratum, 0.2 m,

**Figure 1.** The molecule structural formula of HAWP.

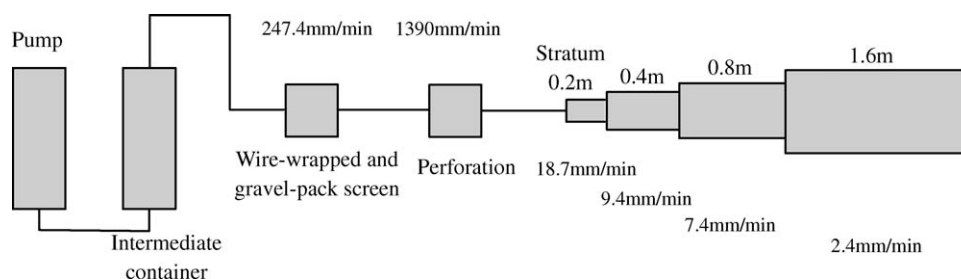


Figure 2 . The engineering schematic graph of the wellbore simulation device with the shearing rates.

0.4 m, 0.8 m, and 1.6 m apart from the perforation were chosen as the sample points. The shearing experiments by this simulation device were carried on at 65°C.

Apparent Viscosity Measurements

Viscosity measurements were performed by Brookfield DV-III Programmable Rheometer at 65°C and 7.34 per s, and the temperature was controlled by circulating-water bathing. Experiments were carried out with special care to avoid water evaporation. The viscosity value was the average data of the 3 min, 5 min, and 8 min.

Intrinsic Viscosity Measurements

In order to reflect the change of molecular weight by shearing effect of wellbore to the two polymers, the change of intrinsic viscosity was measured by Ubbelohde viscometer. Because HAWP was a special polymer with three-dimensional structure, the constants of K and α could not be found from literatures. Intrinsic viscosity was only chosen to reflect the change of molecular weight. The Ubbelohde viscometer was cleaned by water many times until there was no residual polymer on the inner surface of glass. All the polymer solutions were diluted to five times of mother liquid by distilled water. The polymer solution was injected into Ubbelohde viscometer, and the flowing time through the ball was recorded for parallel three times until flowing time difference was less than 0.2 s. The average value of three times was chosen as the final data.

DSL Measurements

DSL measurements were performed on BI-200SM (Brookhaven Instruments, NY, US). If the sample has no obvious impurity, the filtering step was omitted. If not, the quantitative filter papers are used to remove impurities. Then, the samples are injected into quartz observation bottle. The water bath circulation are opened and heated to 65°C. The laser is opened and stable for a half hours, and the laser attenuation mirror is

adjusted. Each sample was measured three times parallel. The data were calculated by CONTIN algorithm. The final data were the average of the three measured value.

AFM Measurements

AFM measurements were performed on a Nanoscope IIIa microscope (Digital Instruments, Santa Barbara, CA) in air at the ambient temperature with standard silicon tips (RTESP Type) in the tapping mode. AFM images were measured at the resonance frequency of the tips with 125 μm -long cantilevers (280–310 kHz). All the images were collected with the maximum available number of pixels (512) in each direction. The scanning speed was at a line frequency of 1.1 Hz. Stock solution of HAWP sheared by the model was taken out quickly and then stirred at low velocity for 30 s to obtain a homogeneous polymer concentration. For the AFM measurements, 0.1 mL of the stock solution was dropped onto freshly cleaved mica, and the solution was blown off simultaneously with a stream of high purity nitrogen.

Viscoelasticity Measurements

Viscoelasticity measurements were carried out by a Physica MCR301 rheometer (Anton Paar Instruments, Austria) with the cone plate testing and strain model measurements at 65°C \pm 0.5°C. Oscillatory measurements were performed by measuring the absolute value of the complex modulus $|G^*|$, the storage modulus G' , the loss modulus G'' , at a constant frequency of 0.01–10 Hz and a constant strain of 0.03. These magnitudes of frequency and strain were chosen so as to provide a stress of reasonable magnitude for purpose of sensitivity.

RESULTS AND DISCUSSION

The viscosity of the polymer solution was one of the most important factors to polymer flooding. It can regulate the mobility ratio of the oil and water (polymer solution), which could

Table II. The Apparent Viscosity of HAWP and HPAM Passing Through Wellbore Simulation Device

	Original polymer solution	Wire-wrapped and gravel-pack screen	Perforation	Stratum 0.2 m	Stratum 0.4 m	Stratum 0.8 m	Stratum 1.6 m
Shearing rate (per s)	0	798.2	1874.8	214.7	79.3	12.7	11.6
Viscosity of HAWP (mPa·s)	212.4	160.6	58.1	43.4	93.9	103.8	118.0
Viscosity of HPAM (mPa·s)	200.4	87.9	2.5	2.6	2.4	2.5	2.6
Retention percent of HAWP (%)	100	75.6	27.4	20.4	44.2	48.9	55.6
Retention percent of HPAM (%)	100	43.9	1.2	1.3	1.1	1.2	1.3

Table III. The Intrinsic Viscosity of HAWP and HPAM Passing Through Wellbore Simulation Device

	Original polymer solution	Wire-wrapped and gravel-pack screen	Perforation	Stratum 0.2 m	Stratum 0.4 m	Stratum 0.8 m	Stratum 1.6 m
Intrinsic viscosity of HAWP (mL/g)	909.43	864.6	478.98	485.2	457.3	441.4	438.67
Intrinsic viscosity of HPAM (mL/g)	1466.89	245.9	25.9	19.8	16.9	17.4	17.6

enlarge the sweeping volume. The porous media and high flowing rate would bring high shearing effect to polymer solution. The apparent viscosity was first measured to reflect this shearing thinning.

Table II shows the viscosity changes of HAWP and HPAM at the seven sample points. It is found that the viscosities of two polymers have different changing trends. The viscosity of the original polymer solutions were both about 200 mPa·s. From the wire-wrapped and gravel-pack screen, the viscosity begins to decrease. HPAMs viscosity is down to minimum at perforation and it no longer reduced in the next part. While the viscosity of HAWP touches the bottom at the stratum 0.2 m. After this point, it recovered to 118 mPa·s at stratum 1.6 m. To explain this data further, the retention percentage of remaining viscosity was also shown in Table II. At stratum 1.6 m, it was found that the viscosity retention percent of HAWP was 55.6%, whereas HPAM was only 1.3%. We can infer that HAWP could also be used for the next flooding but HPAM was not.

This recovery of viscosity and the high retention percentage of HAWP aroused our interest to study polymer change process in porous media. First, the intrinsic viscosity was measured to reflect the change of molecular weight shown in Table III. The relationship of molecular weight and viscosity would be explained.

It is found that the intrinsic viscosities of the two polymer solutions decreased when flowing through wellbore with different degree. This results show that the molecular chain of two polymers are both severely destroyed by the wellbore.

The intrinsic viscosity of HPAM is 1466 mL/g in original solution, 25.7 mL/g at perforation, 19.7 mL/g at stratum 1.6 m. There is no recovery or reassembling of the molecular chain in HPAM. This matches the results of the viscosity data shown in Table II.

From Table III, the intrinsic viscosity of HAWP is reduced from 909.7 mL/g (original solution) to 478.9 mL/g (perforation), and then to 438.6 mL/g at stratum 1.6 m. The depressed degree of intrinsic viscosity is higher than that in HPAM, which shows the molecular chain is not so destroyed severely as HPAM. The

cause of the difference was explained that: the essence of shearing was drag and stretching by porous media to coil of molecular chain. Under the action of shearing, the coil with disordered freely state was first stretched to linear chains. If the shearing continued, then the molecular chain would be broken and degraded. For the linear polymer chain (HPAM), the molecular chain was longer, the more contact points between porous media and per polymer chain. The polymer with higher molecular weight was easier to be dragged and stretched even to be sheared. For HAWP, its molecular weight was lower than HPAM. The molecular chain length was shorter than it, so the shearing effect to HAWP was not severely as HPAM. This is explained why the intrinsic viscosity decreased degree of HAWP was less than HPAM.

It found that the viscosity of HAWP decreased from perforation and stratum 0.2 m, then increased from stratum 0.4 m to 1.6 m, whereas the intrinsic viscosity was decreasing in the whole flowing process. The degraded molecular weight of HAWP was not newly reassembly. Then what did arouse the viscosity to recovery? The question was studied in the next part by DSL and AFM.

The DSL experiments were first carried out to explain further the shearing effect of near-well bore simulation to the polymer aggregate size.

From Table IV, it was found that the HAWP mean particle size sheared by wire-wrapped and gravel-pack screen point reduced 6.5% of original solution. At perforation, it reduced to 66.3%. At stratum 0.2 m, the particle size recovered to 103.0%. After this point, the particle size continued to fall. It was also found that HPAM mean particle size reduced to 65.1% of original solution. After this point, the particle size did not recovery. When compared the shearing effect of wellbore to the two polymers, the perforation was the most strong action to polymer. The recovery only existed in HAWP for its special molecular structure. This result was consistent with the viscosity and intrinsic viscosity.

Figure 3 was the particle size distribution of HAWP (A) and HPAM (B) to further show the particle size change shearing by

Table IV. The Mean Particle Size of HAWP and HPAM Passing Through Wellbore Simulation Device

	Original polymer solution	Wire-wrapped and gravel-pack screen	Perforation	Stratum 0.2 m	Stratum 0.4 m	Stratum 0.8 m	Stratum 1.6 m
Mean particle size of HAWP (nm)	1600.2	1496.1	504.7	1648.7	1574.4	1431.9	1237.5
Mean particle size of HPAM (nm)	277.9	221.1	180.9	187.9	182.3	184.7	177.8

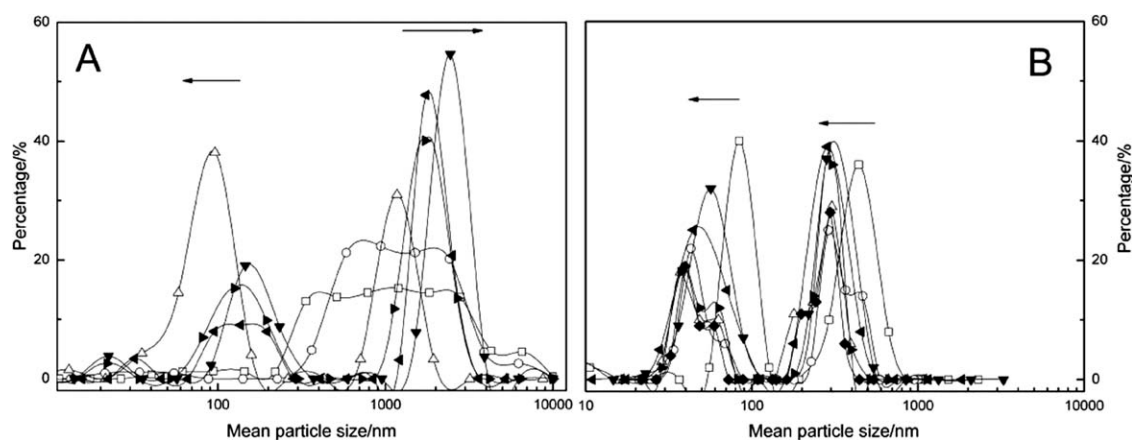


Figure 3. Particle size distribution of HAWP (A) and HPAM (B) by DSL: original polymer solution (\square), wire-wrapped and gravel-pack screen (\circ), perforation (\triangle), stratum 0.2 m (\blacktriangledown), 0.4 m (\blacktriangleleft), 0.8 m (\blacktriangleright), and 1.6 m (\square).

wellbore. As for HAWP, the particle size distribution was only one main peak at 335.93–2801.20 nm (original polymer solution) and 363.66–6228.52 nm (wire-wrapped and gravel-pack screen). At perforation, a twin peak first appeared at 35.23–158.43 nm (main peak) and 712.54–1941.43 nm (the second peak). At the next stratum points, the twin peaks always kept. Along the polymer solution moving forward, the two peaks changed as shown the arrow direction in Figure 3(A). The main peak shifted right, and the second peak shifted left. As for HPAM, it was different that there always was a twin peak from the original polymer solution to stratum 1.6 m. Along the solution moving forward, the main and second peaks both shifted right.

The difference of mean particle size and particle size distribution between the two polymer solutions attributed to their different aggregate state in aqueous solution. Because of the existence of hydrophobic groups between molecular chain and intramolecular, the HAWP molecular chain tended to form network structure. In the porous flowing process, there coexist two effect actions: unbinding of association caused by the shearing and stretching; the association by hydrophobic groups. The two roles are competitive at the porous flowing process. When the shearing is stronger than the association by hydrophobic groups, the particle size became smaller, just as the perforation. When the association is stronger than shearing, the network structure will be recovered. The particle size became bigger slowly. Although the molecular chain of HPAM has association role by hydrophobic groups, it tended to form random coil. During the porous flowing process, the random coil was destroyed to smaller coil.

Second, the microstructures of the two polymers were measured by AFM shown in Figure 4 with three points as representative: original solution, perforation, and stratum 1.6 m. It is found that the microstructures of the two polymers have large difference from the images. In the original solution of HAWP before shearing, it is found that the polymer is a cross network spread all over. On the 1 cm² mica substrate, it was found an intact network without break. It deduced that the network was the inherent reason of the high viscosity of the original solution.

The network included the bigger and smaller microstructure, the bigger circle made the framework with the diameter was about 240 nm of the polymer and the smaller circle acted as the reinforcement with diameter was about 80 nm, which presented regular six-membered or seven-membered rings [Figure 4(A)]. The similar network microstructures were also observed by Feng et al.²⁶ From their experimental results, the diameters of the mesh range from 2 μm to more than 100 μm . The network structure is not observed in HPAM solution. The randomly coil is obtained from Figure 4(A'). To get similar viscosity of original polymer solution, the concentration of HPAM is 6000 mg/L and the concentration of HAWP only needs 1750 mg/L.

When the two polymer solutions flowed through perforation and the stratum 1.6 m, the microstructure happened to great changes. The strong network structure of the HAWP original polymer solution is sheared to the many domains with weak linking between them. As the solution flowing along the stratum to 1.6 m, the structure was composed of domain with weak linking, which was similar as the perforation, but the grain number of domains per unit increases. For the microstructure of HPAM, it changed from randomly coil [Figure 4(A')] to sparse globular without any linking between the particles at perforation [Figure 4(B')] and the stratum 1.6 m [Figure 4(C')].

It is speculated that the recovery of the network bring the recovery of viscosity in HAWP solution. The hydrophobic molecular is easy to congregate in the liquid solution driven by hydrophobic interaction. When compared with the long chain of HPAM, the hydrophobic group of the HAWP was the key to form the steady network structure. The hydrophobic effect and the entropy driving process enhanced the interaction in the molecular chain, which led to the conversion of single molecular chain to aggregate molecular, the big molecular aggregate to molecular chain bunch, and to random coil, finally to network in the solution. The chain entanglement and hydrophobic effect existed at arbitrary molecular aggregate or chain bunch, so the network was three dimensional in the solution.

The data shows that the viscosity and microstructure have certain association. The polymer with strong and whole network spread all over the solution has higher viscosity than polymer

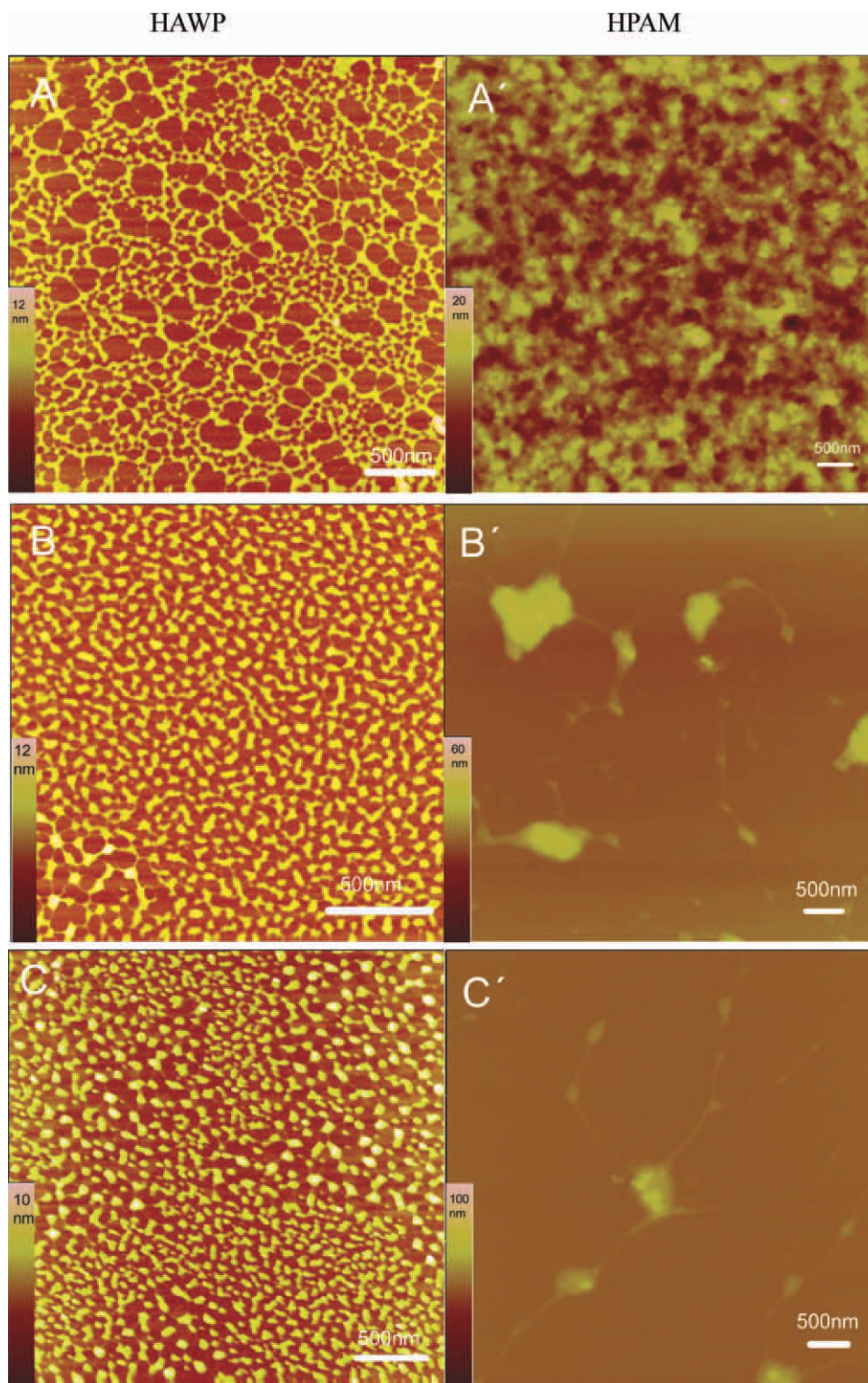


Figure 4 . Typical tapping-mode AFM images of HAWP (A) and HPAM (A') original solution, sheared by the perforation of HAWP (B) and HPAM (B') solution, and sheared by the stratum 1.6 m of HAWP (C) and HPAM (C') solution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

with sparse network with weak linking. It also be concluded that the viscosity and microstructure of the polymer solution would recover at a certain [Figure 4(A–C)] degree, but could

not restored to original structure. The wellbore area only represents the violent shearing to the polymer for flooding, which could not reflect the whole shearing and recovery process. So

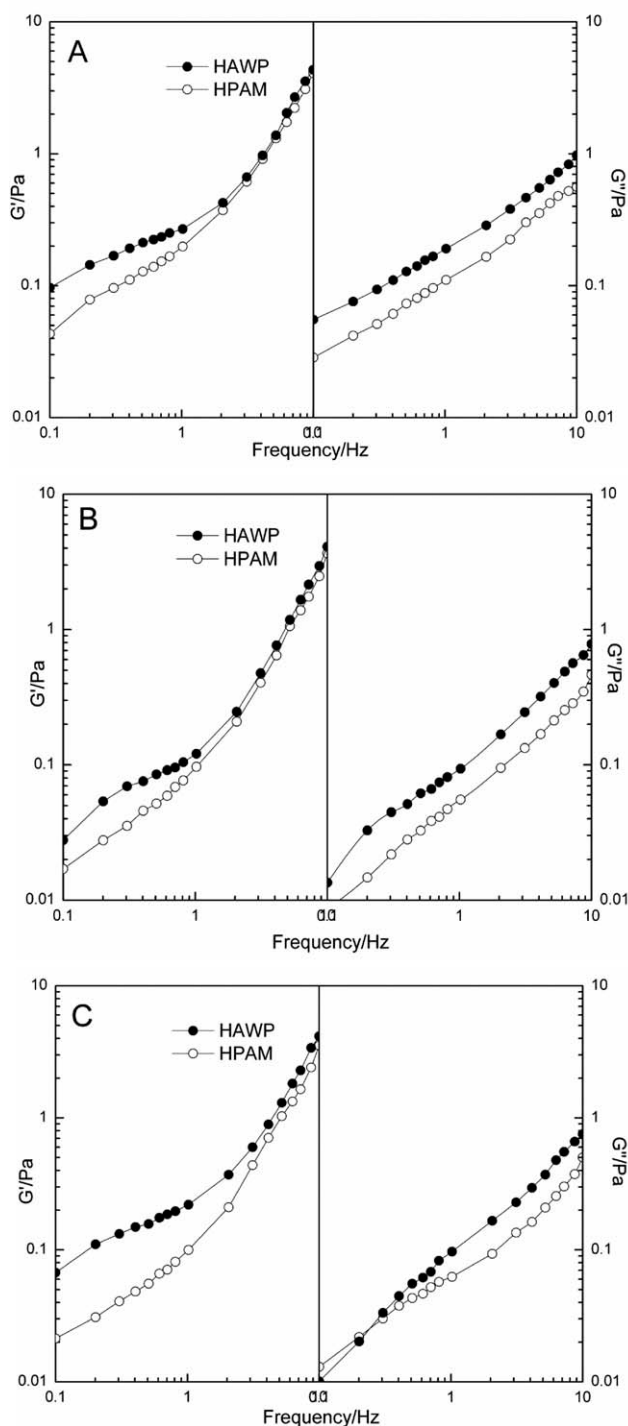


Figure 5 . Viscoelasticity of HAWP and HPAM original solution (A), sheared by the perforation (B), and sheared by the stratum 1.6 m (C).

the images by AFM only show several points during the long process. It is believed that the microstructure of HAWP network was repeatedly disjoined and reformed newly.

Third, as an important factor to flooding oil, the viscoelasticity was also studied to check the reformed process.

In Figure 5, data for the storage moduli (G') and loss moduli (G'') of HAWP and HPAM solution at three sample points as a

function of frequency are presented. From the data, we can find both G' and G'' decreased after shearing by perforation of the two polymers, until stratum 1.6 m both G' and G'' of HAWP recovered to a certain extent. Both G' and G'' could not recover any extent. The reformed polymer (HAWP) could be used to flood oil.

To improve the sweeping volume and obtain higher oil recovery, the viscosity is the most important factor to polymer flooding. There are two methods to improve the viscosity of the water soluble polymer: one is increasing the molecular weight, and the other is to form the three-dimensional network structure. HPAM is the former and HAWP was the latter. For linear chain type polymer, the direct result of the increasing molecular weight was the increasing particle size. When the random coil of HPAM was sheared by porous media, at the first time, the coil was disjoined to smaller one (wire-wrapped and gravel-pack screen). Further shearing acting, the smaller coil was stretched. When the shearing effect was further (perforation), the molecular chain was destroyed. This destroyed process was consistent with the experimental data: the viscosity, the intrinsic viscosity, and mean particle size also decreased sharply at perforation; at the same time, AFM images showed that the big coil disjoined to small ones even to smaller fragment. Summarized by the results, it can conclude that after shearing by wellbore, HPAM had no ability to flood oil from the reservoir. Although the polymer with three-dimensional network (HAWP) suffering the same strong shearing effect (perforation), but could reform network at slower shearing rate (AFM images showing) by self-assembly of hydrophobic groups. Although it could not recovery to the original solution, but the retention percent of viscosity was about 55%. It can inferred that the sheared polymer solution also be used to flood in oilfield.

CONCLUSIONS

Detailed study on the influence of the shearing effect by wellbore simulation model device on the observed rheological and morphology properties of HAWP and HPAM have been performed. As for HAWP, at the perforation, the viscosity began to decrease sharply, until to the stratum 0.2 m it reduced to minimum, after this point it recover slowly to a certain value but could not reach the original. The intrinsic viscosity data reflected that the molecular weight of the two polymers both decreased after sheared by near-well area. The recovery of viscosity was not based on the recovery of molecular weight. The DSL and AFM results showed the sheared polymer recovered to newly three-dimensional microstructure. The shearing process was explained that the compact network structure formed by the interchain or intrachain association and the molecular chain entanglement was disrupted to loosely structure at perforation, then it reformed to a new network structure gradually, which not the same as the original solution. The viscoelasticity had the same change with viscosity and morphology. When compared with HPAM, its viscosity, viscoelasticity, and intrinsic viscosity had no recovery flowing through the wellbore area. By the way, there was no reformation of structure by AFM. Because HPAM was linear chain polymer with high molecular weight and bigger particle size, it was easier to be stretched and

degraded by porous media. Meanwhile, the destroyed molecular chain could not reformed new structure due to the lack of effective linkage. HPAM was not suitable to flood in the stratum. Instead of it, even though severely sheared by the wellbore area, HAWP could keep its high viscosity to flood oil to enhance oil recovery.

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