# High-temperature-resistant polymer gel system with metal–organic mixed cross-linking agents

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**ABSTRACT:** A high-temperature (200°C)-resistant polymer gel system was developed from partially hydrolyzed polyacrylamide (HPAM), chromium lactate (CrL), and water-soluble phenol/formaldehyde resin (WPF) mixed cross-linkers. Rheological measurements indicated that the gelation process of the gel system could be divided into four successive steps: induction, first cross-linking with metal cross-linker, secondary cross-linking with organic cross-linker, and stabilization. Effects of various parameters that affect the gelation time and gel strength including polymer concentration, cross-linker concentration, salinity, pH, and the gelation temperature were evaluated. Gelant formulated with 0.5 wt % HPAM, 0.1 wt % CrL, and 0.9 wt % WPF and treated at 80°C for 48 h showed sufficient gelation time, high rigidity, and good thermal stability. Morphology observation by scanning electron microscopy (SEM) and atomic force microscopy (AFM) revealed that the gel had compact network microstructure. A cross-linking mechanism for the gel system was proposed based on the gelation process and experimental results. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 42261.

## INTRODUCTION

Owing to the ongoing depletion of light oil resources and the increasing global energy demand, major oil companies around the world have been paying much more attention to heavy oil for the last two decades.[1–4] Several effective thermal techniques have been developed in oil industry to enhance heavy oil recoveries, such as cyclic steam stimulation (CSS), steam flooding, and steam-assisted gravity drainage (SAGD).[5] However, after a certain period of steam injection, steam breakthrough becomes a common problem which results in a narrow steam sweep area, poor heat efficiency, and a reduction in ultimate oil recovery.[6,7] Thus, injection of plugging materials is required to block breakthrough channelings.[8,9] The use of steam foam, phenolic resin, and superfine cement has been reported.[10,11] However, the effect of steam foam is limited due to the poor foam stability. Although the phenolic resin can be cross-linked at the formation temperature and turn to be a rigid plug, it is too expensive for large-scale applications. The disadvantage of the superfine cement is the permanent plug that may lead to damage to the formation. Hydroxypropyl methylcellulose-based thermo-reversible gel has been reported for in-depth conformance control in steam-stimulated well,[12] but the gel strength and thermal stability are also limited.

At present, partially hydrolyzed polyacrylamides (HPAM) gels are widely used for water shutoff in production wells and for profile modification in injection wells.[13–15] There are two types of cross-linkers used in HPAM gel technology, namely, metallic cross-linkers and organic cross-linkers. The different types of cross-linkers lead to discrepancies in rheological properties, salt resistance, and temperature resistance. Gels prepared with metallic cross-linkers such as $Cr^{3+}$, $Al^{3+}$, or $Zr^{4+}$ salts have lower stability at high temperatures and their short gelation time preventing proper placement in deep, high temperature reservoirs. While gels with organic cross-linkers such as polyethyleneimine, phenol/formaldehyde, and resol normally need higher gelation temperature and the gel strength is weak. Polyethyleneimine cross-linked copolymer of acrylamide and tert-butylacrylate gel was reported to be stable up to 130°C,[16] but this gel system requires high polymer concentration, and the gel strength is weak. An HPAM gel system using the combination of resorcinol and phenol/formaldehyde as primary and secondary cross-linker was reported and showed thermostability at 90°C after 30 days.[17]

The thermostability and the gel strength are critical issues for the polymer gels to be used as steam channeling plugging agents.

Either metal-cross-linked or organic-cross-linked polymer gel has been seldom applied in blocking the steam breakthrough because the temperature of steam is much higher than the temperature of water. Most of the polymer gel becomes totally soluble when the temperature is above 150°C. Polyacrylamide-based gel system that can withstand more than 150°C temperature has never been truly realized in oil and gas industry. Therefore, it has been necessary to develop a thermally stable polymer gel system that has low viscosity at lower temperatures in order to be pumped into the well, but able to be cross-linked at the formation temperature and remain rigid gel after prolonged exposure to steam injection temperature. The high-temperature-resistant property cannot be realized by just simply elevating the concentration of polymer and cross-linkers, because the viscosity of the gelant will be too high and the overdose of the cross-linking agent will lead to gel syneresis, besides the high cost.

In order to overcome the aforementioned limitations, we focused our attention on the gel system composed of polyacrylamides, metal, and phenolic mixed cross-linking agents. The aim of this article is for better gel strength and high-temperature stability via the collaborative cross-linking of metallic and organic cross-linkers. Hence, investigations of the gelation process, the effects of various parameters on the gelation properties, the thermal stability, and the microstructure were addressed. Finally, a field test result was briefly presented.

## EXPERIMENTAL

### Materials
HPAM with a molecular weight of $5 \times 10^6$ Dalton and 9–10% degree of hydrolysis (HBHT-13), chromium lactate with a Cr content of 5% and delaying agent of 0.35% (CrL), and water-soluble phenol/formaldehyde resin with 30 wt % of active matter content (WPF) were from Shengli Oilfield Huabin Chemical Industry Co. Ltd. and were used as received. The dosage mentioned in this work was weight percent of the received chemical in gelant solution.

### Preparation of Gelant Solution
A gelant solution was prepared by mixing the polymer solution of required concentration and a certain amount of cross-linker at room temperature. The polymer concentration was varied from 0.2 to 0.7 wt %. The concentration of the CrL cross-linker was varied from 0.05% to 0.2 wt % and the WPF cross-linker was varied from 0.2 to 0.9 wt %. In this study, the cross-linking reaction was initiated by heating the gelant solution at different temperatures in an oven.

### Characterization Methods
**Determination of Gelation Time.** Gelation time was determined by the tube inversion method.[18] The gelant solution was sealed in a bottle and put into an oven at the desired temperature. The samples were monitored periodically by inverting the bottle, and a gel code was assigned to each sample based on observation. Gel strength during development of gelation kinetic was expressed as an alphabetic code of A–I (Sydansk code).[13,19] Code A means that there is no detectable gel formation and the gel appears to have the same viscosity as the original polymer solution. Whereas code I indicates that there is no gel surface

deformation by gravity upon inversion. Gelation time was defined as the time required attaining a Sydansk code C. The gel strength code was also used to evaluate the gel strength and its stability. The effects of different parameters on the gelation time of the polymer gel were determined.

**Determination of Gel Strength.** The gel strength was measured by using the breakthrough vacuum method.[20] The procedure was as follows: the gel was placed into a cuvette, then the tube connected to a pump was quickly put into the gel. The maximum pressure during the process was recorded as the gel strength.

**Viscosity Measurement.** The viscosity of samples was measured using a Brookfield Viscometer (model DV-1C). The viscosity reading was taken at regular intervals of time using spindle number 42.

**Microstructure Characterization.** The microstructures of the samples were investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM). SEM was recorded on a Hitachi S-4800 Cold Field Scanning Electron Microscope. Samples were preprocessed to freeze-drying to maintain their natural state. Determinations were conducted at accelerating voltage of 10 kV and working distance from 5 to 10 mm. AFM was performed on an Agilent 5400, using antimony (n) doped silicon cantilevers (SCM-PIT, Veeco) in tapping mode. The scanning rate was 1.0 line/s.

## RESULTS AND DISCUSSION

The thermostability of polymer gel is influenced by many factors. Among them, the structural characteristics of the polymer and the cross-linker are the most important issues. For the partially hydrolyzed polyacrylamides, sample with higher Mw has larger hydrodynamic volume, exhibiting a larger viscosity in solution than that with low Mw. In addition, the degree of hydrolysis is also a key factor to the viscosity of the polymer solution. Based on a vast number of preliminary screening tests, a HPAM sample with Mw of $5 \times 10^6$ Da and 8–10% hydrolysis degree was selected as the polymer in this work. CrL was employed as the first cross-linker, and WPF as the secondary cross-linker.

### Gelation Performance
**Effect of Polymer Concentration.** The polymer concentration is critical to the structure and properties of the gel networks. Figure 1 shows the curve of gelation time and gel strength versus various polymer concentrations (0.2–0.8 wt %). All samples composed of the same amount of cross-linkers (0.1 wt % CrL and 0.9 wt % WPF) and were subjected to the same gelation temperature of 80°C. It can be found that the lower the polymer concentration, the weaker the gel. As polymer concentration increased, the resulting gels became stronger. But polymer concentration higher than 0.8 wt % is not recommended because the initial viscosity of the gelant would be too high to be pumped into the formation.

As shown in Figure 1, the gelation time decreased with increasing the polymer concentration. This is due to the increased cross-linking sites and reaction chance. High polymer concentrations result in greater cross-linking density in the gel networks and more cross-linking sites (carboxylate group). The
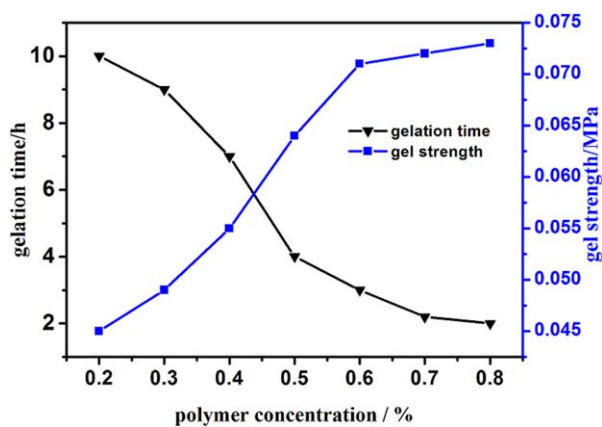
**Figure 1.** Effect of polymer concentration on gelation performance. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



**Figure 2.** Effect of CrL cross-linker concentration on gelation process. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

observation is in good accordance with those well documented in the literature.[21] It is worth mentioning that at a polymer concentration of 0.5 wt %, the gel exhibits strength of 0.065 MPa. The value is close to a previously reported gel with polymer concentrations of 0.8 wt %.[20] Besides, syneresis was not observed in all gel samples with various polymer concentrations.
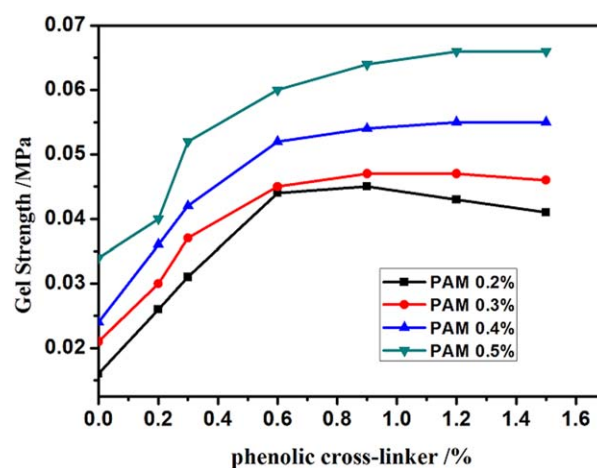
**Effect of Cross-Linker Concentration.** Figure 2 shows the gelation process of the gelant solutions formulated with 0.5 wt % polymer, 0.9 wt % WPF, and various amount of CrL (0.05–0.2 wt %). The viscosity of the sample was monitored at a constant temperature (80°C). Gel samples cross-linked with CrL or WPF solely were also measured for comparison. As it can be seen, the curve A shows that with WPF as cross-linker, the system results only a weak flowing gel after 13 h thermal treatment. While curve B indicates that with CrL only, the cross-linking reaction takes place quickly and completes with 6 h. The gel viscosity cannot be further increased with prolonged time. Curves C, D, E, and F are from the gels with the same concentration of WPF cross-linker, but with increased CrL concentrations. For the gelant solution with very low CrL concentration (curve C), the viscosity of the system gradually increased in the period of 5 −15 h, finally giving a cross-linked gel with moderate gel strength. Comparison of the curve B with curve D shows that the WPF cross-linker does not have much effect on the CrL controlled cross-linking reaction during the early process. On the contrary, the CrL cross-linker does have strong influence on the later stage of cross-linking, which is supposed to be a WPF-controlled process. Without sufficient CrL-controlled cross-linking (curve A and C), the gelant solution cannot be well cross-linked to a rigid gel, even with same WPF concentration. When the CrL concentration is increased to 0.15 wt % (Line E) and further to 0.2 wt % (Line F), the cross-linking reaction becomes faster and the gel turns to be stronger. For Lines D, E, and F, similar cross-linking profiles can be observed. The final gel viscosity of the gel system with 0.20 wt % of CrL is only a little bit higher than the gel with 0.15 wt % CrL. Considering the cost and to avoid the gel syneresis, 0.1 wt % was chosen as an optimal CrL concentration.
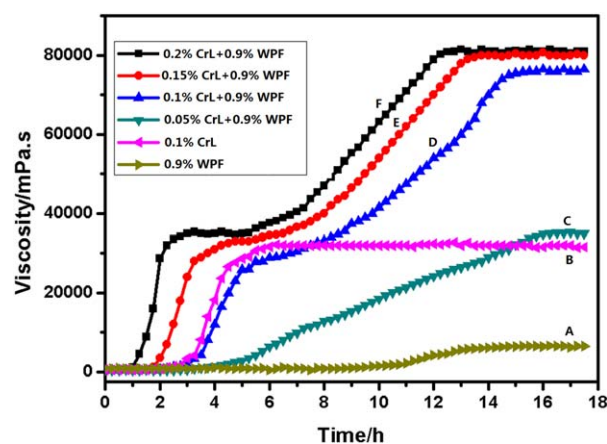
Figure 3 shows the effects of WPF concentration on the gel strength. The gelant solutions contain 0–1.5 wt % WPF while the concentration of CrL was kept constant 0.1 wt %. All samples were measured at 80°C. As shown in Figure 3, the gel strength is greatly improved when WPF is added into the gel system. Gelation strength increases with the increase of concentration of WPF, except the gelant formulated with 0.2 wt % of polymer concentration. When the polymer concentration is low, larger WPF concentration will lead to the decrease in the strength of gel possibly due to syneresis caused by excess cross-linking. In consideration of the gel performance and the gel cost, the optimum formula for the gel system is obtained: 0.5 wt % HPAM, 0.1% CrL, and 0.9 wt % WPF, which is used for the following studies.

**Gelation Process.** To further study the gelation process, the gel formulated with a combination of 0.5 wt % polymer, 0.1 wt % CrL, and 0.9 wt % WPF at 80°C was taken as an example. Figure 4 shows the rheological curve (i.e., line D in Figure 1) and the color changes of the gelant.



**Figure 3.** Effect of WPF cross-linker concentration on gelation performance. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
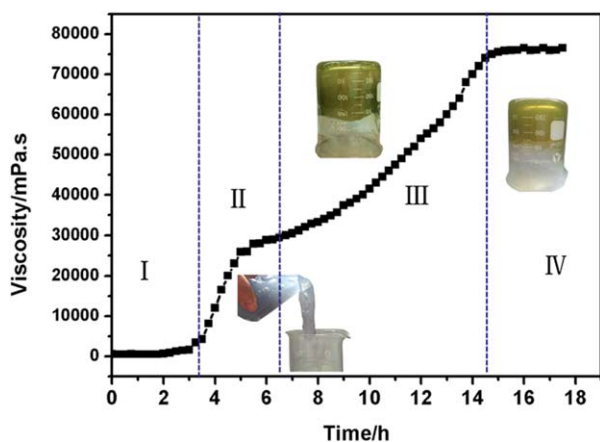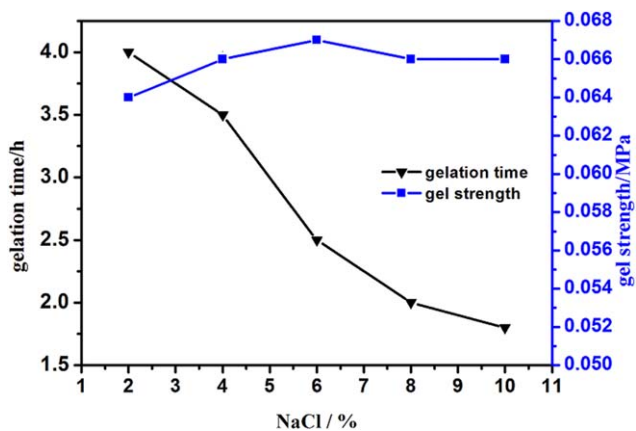
**Figure 4.** The four steps of gelation process. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



**Figure 5.** The effect of sodium chloride on gelation time and gel strength. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

As shown in Figure 4, the gelation process of the systems can be divided into four successive steps with the viscosity ranging between 45 and 80,000 mPa·s. Step I (0–3 h), there was no obvious viscosity increasing in this period. The viscosity was mainly resulting from the macromolecules in the solution. Step II (3–6 h), the viscosity increased rapidly. This was a rapid cross-linking period. The increased viscosity was mainly caused by CrL-initiated intermolecular cross-linking reactions, yielding a gel of code B. Step III (6–15 h), WPF cross-linker took the control of the cross-linking reaction, and gel strength rose from flow state to low deformation condition (code H). It was interesting to note that the color of the gel changed from blue to green in this period. Step IV (15–18 h), the gelling process continued to a stabilization period. The viscosity of the gel reached maximum and the gel became rigid (code I). Therefore, it is reasonable that the secondary cross-linker (WPF) takes effect based on the gelation status of the first cross-linker (CrL). This previously unrevealed phenomenon can be explained based on the gelation mechanism with mixed cross-linkers, which is described in the latter section.

**Effect of Salinity.** Salinity is an important factor for gelation performance.[13,20] The effect of salinity of polymer gelant on gelation rate depends on many factors, including gel system, polymer concentration, brine concentration, gelling temperature, etc. Several investigators found that gelation occurred rapidly in presence of salt compared to distilled water,[22,23] while gel samples prepared with constant polymer and cross-linker concentration and varying brine salinity showed that the gelation time increases with the increase in concentration of brine.[24] There were also some studies showed that increasing total dissolved salts (TDS) had positive effect on the gelation time delay of gel system.[15,25] The gelation time increases and the initial gelation strength becomes weak with increasing the TDS of gelling solution.

In this work, the effect of sodium chloride on the gelation time and gel strength was investigated (Figure 5). The gelants were formulated with a combination of 0.5 wt % polymer, 0.1 wt % CrL, and 0.9 wt % WPF cross-linker at 80°C. The gelation time decreases with increasing sodium chloride concentration, but

the gel strength fluctuates in a small range of 0.064–0.067 MPa. So, it is hard to say that the increase of NaCl concentration has positive or negative effect on the gel strength. However, the gelation time decreases rapidly with increasing the salinity. This is because that the inorganic salts compress the electric double layers of polymers. Accordingly, the repulsion forces of charged groups are decreased, making the reactions occur more easily.[26]

**Effect of pH.** The effects of pH on the gelation time and gel strength were investigated. Figure 6 shows the gelation time and gel strength of the gelling solution containing 0.5 wt % HPAM, 0.1% CrL, and 0.9 wt % WPF with different pH values at 80°C. The initial pH value of the gelant is about 7.5. The pH range over which the experiments were carried out was adjusted from 4 to 10 by adding aqueous NaOH or HCl solution. It is found that the gelation time first decreases with pH, and then increases after pH = 8. While the gel strength, initially, reaching a maximum value of 0.068 MPa at pH = 8, later it starts to decrease. Therefore, the optimum pH value of this gel system is 7–8. Above pH = 8, the gel strength decreases rapidly, which may be an indication that the status of metallic and organic cross-linkers is influenced by the stronger basic environment.
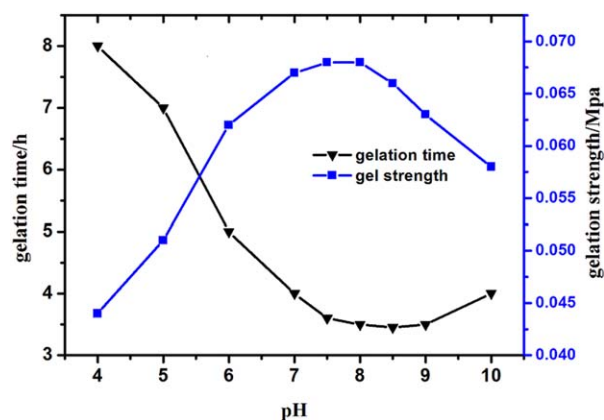


**Figure 6.** The effect of pH on gelation time and gel strength. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
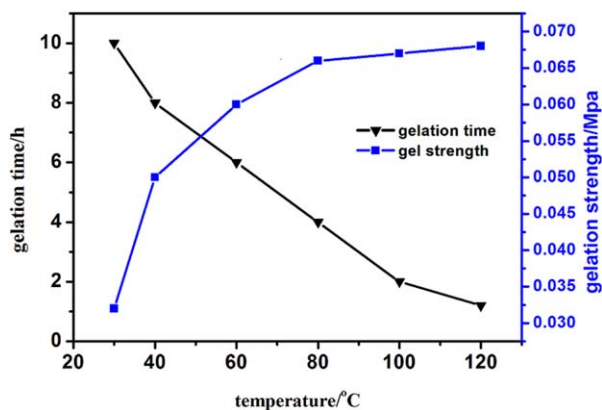
**Figure 7.** The effect of temperature on gelation time and gel strength. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

**Effect of Gelation Temperature.** The effect of temperature on the gelation time and gel strength of the gelant solution containing 0.5 wt % HPAM, 0.1 wt % CrL, and 0.9 wt % WPF was determined. The solutions were kept for gelation at different temperatures ranging from 30 to 120°C for 48 h. The gel strength increases and gelation time decreases with temperature as shown in Figure 7. This may be due to the increase in the thermal motion of the molecules in the solution, promotes the intertwining reaction between polymer molecules and cross-linker molecules, and increases their opportunity to agglomer-
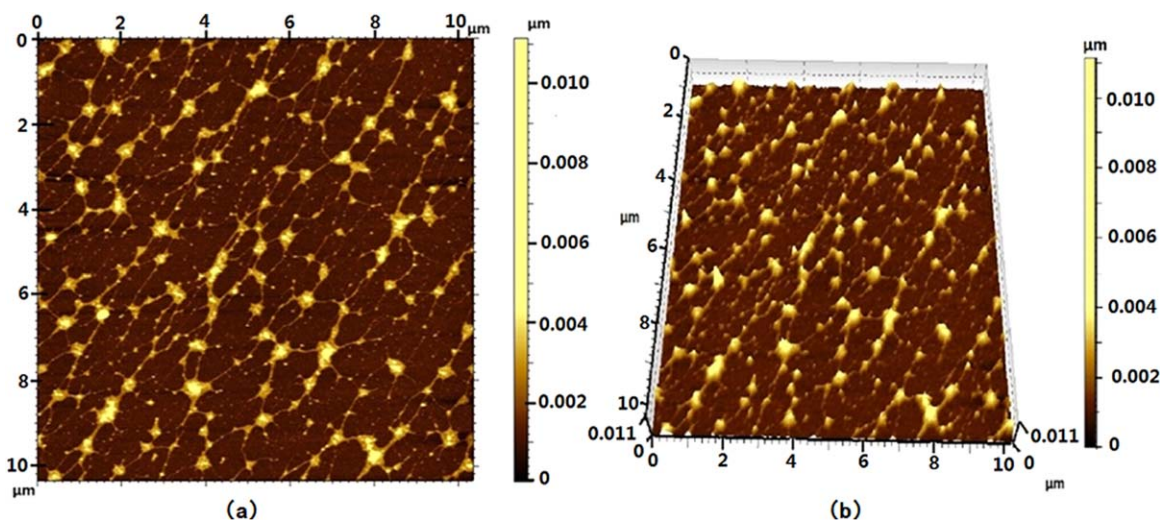


**Figure 8.** The status of the gel after 30 days at 200°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary. com.]

ate, thus the gelation process is accelerated. Considering that the polymer formulated in the gelant solution has a relatively low degree of hydrolysis, an increase in gelation temperature may also result in an increase in the degree of hydrolysis of the
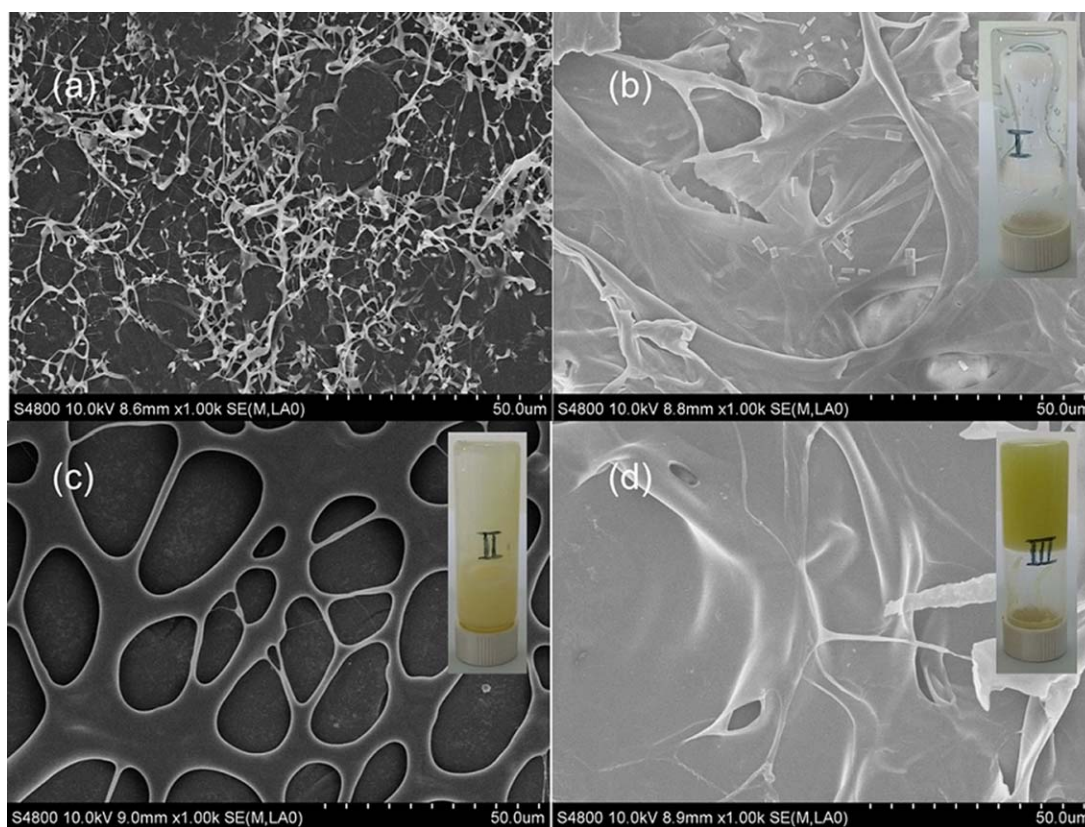


**Figure 9.** SEM images of different kind of gel systems: (a) gel systems before cross-linking; (b) CrL-cross-linked polymer gel; (c) WPF-cross-linked polymer gel; (d) CrL and WPF cross-linked polymer gel. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

**Figure 10.** Microstructure of the polymer gel with CrL and WPF mixed cross-linkers: (a) two-dimensional diagram and (b) three-dimensional diagram. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

polymer, which in turn will increase the number of cross-linking sites. As a result, the gels are formed in shorter time due to rapid cross-linking and the gel strength increases.

For polymer gel used in blocking steam breakthrough channeling, appropriate working time is required for the placement of the gelant solution into the problem zone before it gels. From Figure 7, it can be seen that the gelation time is about 4 h at 80°C. It is sufficient for the steam channeling blockage because the volume of the gel solution is relatively small in such operations.

**Thermal Stability**

The main consideration for a polymer gel system being used in steam channeling lies in its high-temperature stability. To the gel system cross-linked with either CrL or WPF only, overdose of the cross-linker would not increase its thermostability. Contrarily, severe syneresis can occur because of excess cross-linking. In addition, the problem associated with HPAM/WPF gel system is that the gel is too weak to be used in steam channeling control, even the gelant solution formulated with high concentration of polymer and cross-linker. To test the high-temperature stability of the gel system with CrL and WPF mixed cross-linkers, a gelant solution containing 0.5 wt % HPAM, 0.1 wt % CrL, and 0.9 wt %

WPF was first treated at 80°C for 48 h, then the cross-linked gel was further treated at 200°C for 30 days. After thermal treatment, the gel was removed from the vessel and cooled to room temperature, as shown in Figure 8, the gel kept rather good shape without any syneresis, indicating that the gel had good long-term thermal stability. This excellent high-temperature-resistant property of the HPAM/CrL/WPF gel system may be a result of (1) appropriate structure of the selected HPAM, (2) suitable water-soluble phenol/formaldehyde resin, and (3) the collaborative works of the metal and organic mixed cross-linkers.

**Microstructure of the Gel**

SEM and AFM were employed to investigate the microstructure of the gels. Figure 9 shows the micrographs of the polymer gel before and after cross-linking. The PAM solution (0.5 wt %) shows a filamentous structure [Figure 9(a)].The microstructure of the CrL-cross-linked gel [Figure 9(b)], which has a Sydansk code E. shows a segmented three-dimensional structure. The gel system cross-linked with WPF alone has a porous mesh-like network structure with pore sizes ranging from several to approximate 20 μm [Figure 9(c)]. The microstructures between CrL-cross-linked polymer gels and WPF-cross-linked polymer gels are significantly different. This difference may be attributed
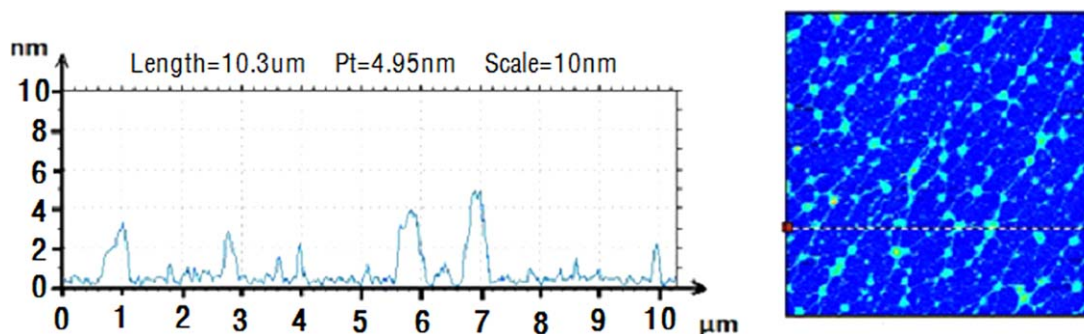


**Figure 11.** The thickness of microscopic cross-linking points of the gel sample. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

to their different cross-linking mechanism. CrL-cross-linked gel is much stronger than WPF-cross-linked gel. When the CrL and WPF were used together, a code I gel thus formed shows a compact and more uniform dense structure [Figure 9(d)]. AFM micrograph shows the network structure of the polymer gel with the mixed cross-linkers (Figure 10). The mesh width is around 2 μm. The cross-linking points with irregular heights ranging from 0.5 to 5.0 nm seem rather firm (Figure 11). This dense structure provides high mechanical strength that is beneficial to the gel strength and thermal stability.

## Proposed Mechanism for the Gelation with Mixed Cross-Linkers

The cross-linking mechanism involved in either metal or phenolic resin cross-linker has been well documented.[27,28] As reported previously, the carboxylate group ($-COO^-$) in the polymer chain could be chelated with the metal Cr (III) species, resulting in the formation of a cross-linked network structure.[29,30] When WPF was used as a cross-linker, either in a mixture of phenol and formaldehyde or a prereacted resin format, the amide groups of the polymer provide cross-linking sites reacting with multiple hydroxymethyl groups on the phenolic ring, forming the gel system.[31–33] Generally, the gelation time of the Cr/HPAM gel system is short, while the organic cross-linker provides a longer gelation time. Based on the fact that the WPF cross-linker took effect on the basis of CrL cross-linker, the mechanism for this mixed cross-linker system was proposed. As show in Figure 12, the CrL cross-linker reacts first with $-COO^-$ groups in the polymer chains to form a preliminary network structure. Owing to the low CrL concentration, the cross-linking degree is not very high and the gel strength is relatively weak. Then the WPF cross-linker reacts with the polymer through the condensation of $-CH_2OH$ and $-CONH_2$, and reinforced the network structure. Here the WPF cross-linker is represented by a simple trihydroxymethyl phenol molecule for simplicity. The gel strength increases as a result of the compact structure. It is worth mentioning that the first cross-linker provides the basis for the secondary crossling reactions. This means that the secondary cross-linking needs a stable and constrained environment to take place. Zhu et al. reported that cross-linking
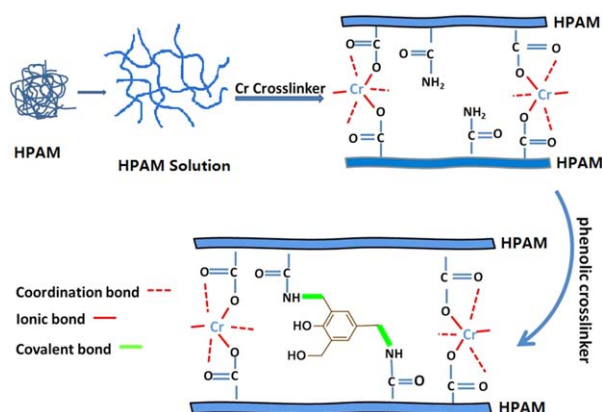


**Figure 12.** Proposed mechanism for the gelation with CrL and WPF cross-linkers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

**Table I.** Gel System for Blocking the Steam Breakthrough Channeling

| Property | HPAM + CrL + WPF |
|---|---|
| Polymer concentration | 0.6 wt % HBHT-13 |
| CrL concentration | 0.10–0.11 wt % CrL |
| WPF concentration | 0.9–1.0 wt % |
| Initial viscosity (170 s$^{-1}$) | 65 mPa·s |
| pH of the gelant solution | 7.2 |
| Well temperature | 68°C |
| Gelation time | 6 h |
| Final Sydansk gel code | I |
| Total volume | 350 m$^3$ |

could be disturbed with the flow of the gelant, which resulted in decrease of gelation rate and cross-linking degree, or even failure to form the gel.[34] Ni et al. suggested that a reaction distance of 0.6 nm was required for the condensation of $-CH_2OH$ and $-CONH_2$ groups.[35,36] All these observations support the hypothesis that the preformed network structure by the first cross-linker is essential to the effective cross-linking with the phenolic cross-linker. In such a case, the use of secondary cross-linker can significantly increase the gel strength and improve the high-temperature resistance.

### Field Testing

To evaluate the practical industrial application potential of the studied gel system, a field test was carried out in Binnan Heavy Oil Reservoir well 83X078. Prior to the test, cyclic steam stimulation (CSS) technique was utilized to assist the oil production for this well. After a long period of CSS operation, the well was found serious steam breakthrough on six directions toward its neighbor wells. Thus gel treatment has applied to block the steam channeling, which was performed in November, 2013. The gel system was formulated as shown in Table I. After gel treatment, 3000 m$^3$ of high temperature steam (270°C) was injected and not any breakthrough was detected, indicating all breakthrough channeling were successfully sealed with the gel and the gel could sustain high temperature. The oil production of the well has more than doubled, compared to its previous status. Primarily, this gel technology is feasible in blocking steam breakthrough channeling and has the potential to enhance heavy oil production.

## CONCLUSIONS

This work presented a high-temperature-resistant gel system. It was found that the gel formed with HPAM and CrL/WPF mixed cross-linkers had high gel strength and excellent high-temperature stability. Rheological measurements indicated that the gelation process could be divided into four successive steps in which the CrL cross-linker and the WPF cross-linker took effect cooperatively. A gel system formulated with 0.5 wt % polymer, 0.1 wt % CrL, and 0.9 wt % WPF cross-linker and treated at 80°C for 48 h showed sufficient gelation time (4 h), high rigidity (code I), and good thermal stability (200°C ageing for 30 days). SEM/AFM images of the gel showed a compact network structure. This system is cost-effective and applicable

for blocking steam breakthrough channeling in heavy oil production. Laboratory long-time curing and primary field test revealed that the gel system has high industrial application potential.

## ACKNOWLEDGMENTS

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