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Enhanced Viscosity of Poly(acrylamide) Solution in the Presence of Chromium Citrate Triggered by Release of CO₂

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Partially hydrolyzed polyacrylamide (HPAM) has been widely used for water shut-off and profile control to enhance oil recovery. Herein, we reported a novel technique by which the crosslinking between HPAM and Cr³⁺ in aqueous solutions at 60 °C can be delayed effectively. Citric acid was selected as an organic complexing agent of Cr³⁺ so that the crosslinking between HPAM and Cr³⁺ can be prevented completely. Due to the decomposition of the bicarbonate (HCO₃⁻) embedded in solution, CO₂ released from solution and the pH value of solution increased gradually. The degree of ionization of HPAM and its ability to complex with Cr³⁺ increased accordingly. When the complexation of Cr³⁺ with HPAM is stronger than that with citric acid, the viscosity of the HPAM solution increased significantly. Under the closed condition, together with the existence of potassium dihydrogen phosphate (KH₂PO₄), the release of CO₂ was very slow and under a controlled condition so that the ionization of HPAM was prevented initially. Furthermore, the hydrogen bonding interactions between HPAM and melamine embedded in solution previously also postponed the ionization of HPAM. As a result, the crosslinking between HPAM and Cr³⁺ can be delayed for almost one month, completely meeting the requirements for deep water shut-off and profile control to enhance oil recovery.

Key words: Polyacrylamide, Delayed cross-linking, pH responsibility, Complexation

I. INTRODUCTION

Intelligent fluids and their response to external stimuli have drawn significant attention in recent years. For the study of intelligent fluids, different kinds of adjustable stimuli are widely used, including: light [1–3], heat [4–6], pH [7–11], redox [12, 13], CO₂ [14–16], and so on. Compared to other stimulation methods, the regulation of pH is simple and more effective. For example, Feng *et al.* [7] prepared a ready-to-use intelligent fluid fabricated by a pH-switchable wormlike micelle system, of which the viscosity could be adjusted by tuning the pH through the addition of few acids or bases. Under acidic conditions, *N*-erucamidopropyl-*N*, *N*-dimethylamine (UC22AMPM) and maleic acid complexed with each other by electrostatic interaction to form worm-like micelles, as a result, the viscosity of the solution maintain a high level. When the pH exceeded 6.51, due to the deprotonation of UC22AMPM, wormlike micelles dissociated and the viscosity decreased

rapidly. In addition, by combining the hydrophobic polyacrylamide with potassium oleate, and based on the assembly behavior of potassium oleate controlled by pH, Hao *et al.* [11] obtained an intelligent fluid that showed fascinating pH-responsive abilities. Furthermore, if the hydrophobic polyacrylamide was complexed with the cyclodextrin, an intelligent fluid [6] responsive to temperature can be obtained.

Zhang *et al.* [16] reported an intelligent fluid that can respond to CO₂, which was fabricated by mixing a tertiary amine-containing triblock polymer with nanoclay. In fact, this smart fluid, by its nature, showed pH responsive ability. Liu *et al.* [9] also reported a pH-responsive wormlike nanostructured fluid. However, the method to change the pH of the systems was confined due to the addition of acids or bases, which greatly limited their applications in a closed system such as in reservoir. Water shut-off and profile control is an important and effective method to enhance oil recovery. Due to its low-cost, water solubility, and excellent ability to crosslink with a variety of metal ions [17–22], partially hydrolyzed polyacrylamide (HPAM) has been widely used for water shut-off and profile control. To facilitate the injection and transportation of aqueous HPAM

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solution in the presence of metal ions, the crosslinking between HPAM and metal ions has to be delayed. The traditional technique is to add organic complexing agent such as lactic acid to trap the metal ions in advance to delay the release of the metal ions. For this purpose, the usual method is adding organic complexing agents such as lactic acid and citric acid to the system, through the introduction of the complexation of lactic acid or citric acid with metal ions, the time required to form crosslinking structure between polyacrylamide and metal ions can be prolonged and controlled [23–26]. Willhite *et al.* [23] embed Cr^{3+} in the nanoparticles formed by self-assembly of polyethyleneimine and dextran sulfate, and the chromium efficiently sequestered in nanosuspensions could be slowly released to crosslink the HPAM based on the change of temperature, ionic strength and so on, resulting in a significant delay in crosslinking. So far, the time required to delay the crosslinking of polyacrylamide and metal ions is usually limited within one week. Moreover, with the increase of temperature, the shrinkage in the time of delaying crosslinking made it more difficult to meet the technical requirements for enhanced oil recovery.

Herein, we reported a novel technique by which the crosslinking between HPAM and Cr^{3+} in aqueous solutions at 60 °C can be delayed for almost one month. In our experiments, citric acid was selected as an organic complexing agent of Cr^{3+} so that the crosslinking between HPAM and Cr^{3+} can be prevented completely. Due to the decomposition of the bicarbonate (HCO_3^-) embedded deliberately in solution, CO_2 released from solution and the pH value of solution increased gradually. The degree of ionization of HPAM and its ability to complex with Cr^{3+} , therefore, increased accordingly. When the complexation of Cr^{3+} with HPAM is stronger than that with citric acid, the viscosity of the HPAM solution increased significantly. However, under the closed condition, together with the existence of potassium dihydrogen phosphate (KH_2PO_4), the release of CO_2 was very slow so that the ionization of HPAM was prevented effectively. Furthermore, the hydrogen bonding interactions between HPAM and melamine embedded in solution also postponed the ionization of HPAM. As a result, the crosslinking between HPAM and Cr^{3+} can be delayed for almost one month. The similar results have never been reported previously.

II. EXPERIMENTS

A. Materials

Partially hydrolyzed polyacrylamide (the degree of hydrolysis 21 mol%), citric acid chromium, potassium dihydrogen phosphate, sodium bicarbonate and melamine were purchased from Sinopharm Chemical Reagent Co. Ltd. All materials were analytical reagents and used as received.

B. Preparation of HPAM solutions

NaCl (0.5 g) was dissolved in 100 mL deionized water, followed by addition of HPAM (0.05 g) slowly. After stirring 2 h, a transparent and uniform solution was obtained. Then, thiourea (0.05 g), chromium citrated (0.2 g), melamine (0.05 g), sodium bicarbonate (0.3 g), and potassium dihydrogen phosphate (0.2 g) were added to the above solution. After stirring for 30 min, the solution was transferred into the glass bottle, and finally the bottle was sealed.

C. Rheological measurements

Rheological measurements were conducted on a TA AR-G2 rheometer using a cone-plate of 40 mm diameter with a cone angle of 1°. The shear rate was set to be 0.451 s^{-1} , 6 data points per minute, 5 min in total, and was taken the average.

D. pH variation measurements

A mixed solution of 0.3 wt% sodium bicarbonate and 0.2 wt% potassium dihydrogen phosphate was prepared. The pH was measured every one hour with an electronic pH tester at different temperatures.

III. RESULTS AND DISCUSSION

A. Effects of lactic acid and citric acid on the gelation properties of HPAM-Cr(III) system

As a common thickener, HPAM-Cr(III) crosslinking system has been widely used in oil extraction engineering. When Cr^{3+} dissolved in water, Cr^{3+} will go through hydration, hydrolysis, hydroxylation, further hydrolysis and further hydroxylation process, finally form multi-core hydroxyl bridge chromium ions, as shown in FIG. 1(a). If HPAM exists, the chromium-containing ligand will complex with the carboxyl group in HPAM as shown in FIG. 1(b), then different polyacrylamide molecules will be crosslinked to achieve the purpose of thickening [18, 27, 28]. In the process of water shutoff and profile control, in order to inject the crosslinked HPAM into the target layer, it is necessary to control the crosslinking degree between HPAM and Cr^{3+} to reduce the initial viscosity, after it was injected into the target layer, the crosslinking between HPAM and Cr^{3+} will come about and increase gradually to raise the final viscosity, for this purpose, the most common practice is to add lactic acid or citric acid into the solution, then the Cr^{3+} acting as a cross linker will complex with citric acid or lactic acid at first, as an alternative method, lactic acid chromium or lemon chromium can be prepared as a crosslinking agent from the start.

In the water, lactic acid chromium or lemon chromium will gradually ionize to produce trivalent

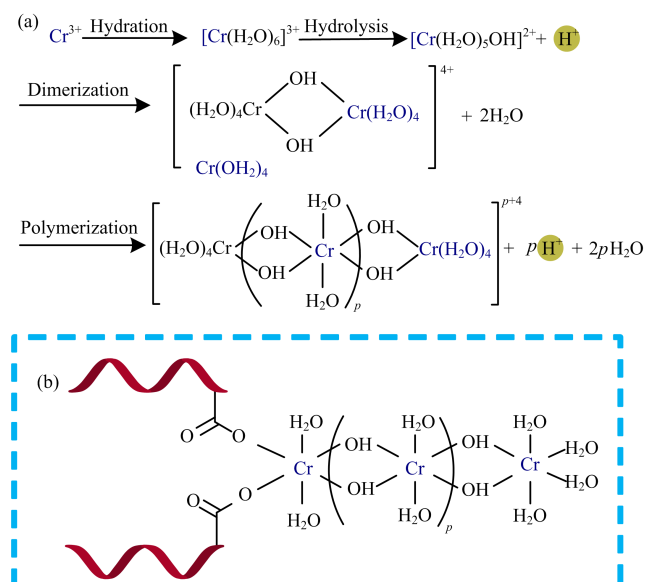
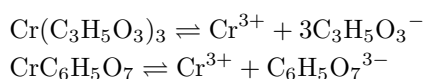


FIG. 1 (a) The formation process of multi-core hydroxyl bridge chromium ions. (b) The gelling mechanism of HPAM and multi-core hydroxyl bridge chromium ions.

chromium ions, as follows:



The Cr^{3+} then hydrolyzes stepwise as shown in FIG. 1 and crosslinks with the carboxyl groups in the HPAM. The ionization process of lactic acid chromium or lemon chromium is slow, as a result, adding lactic acid or citric acid can effectively delay the gelation time of Cr^{3+} and HPAM. Adding organic complexes can delay the crosslinking of HPAM and Cr^{3+} , and the ability to delay the crosslinking depends on their complexation constant and temperature. The complexation constants are 1.2, 3.3, 3.7, 4.7, 5.4, and 7.69 for salicylic acid, lactic acid, acetic acid, propionic acid, malic acid, and citric acid, respectively. We can see that the complexation constant of lactic acid is relatively small and the complexation constant of citric acid is the largest. The stronger complexation between organic complexes and Cr^{3+} , the longer the time required to delay the crosslinking of HPAM and Cr^{3+} . On the other hand, the higher environment temperature is, the shorter delay time will be realized. According to the current literature [23, 26], the addition of citric acid can prolong the crosslinking time of HPAM- Cr^{3+} system to 1–2 days at 90 °C. Considering the situation in Daqing Oilfield, the temperature of most reservoirs is about 60 °C. According to the actual engineering requirements, the time needed to crosslink HPAM and Cr^{3+} should be prolonged to over one month. To solve this problem, we here used lactic acid and citric acid to control the crosslinking time of HPAM- Cr^{3+} system. It was demonstrated that the system would be cross-linked rapidly by using lactic

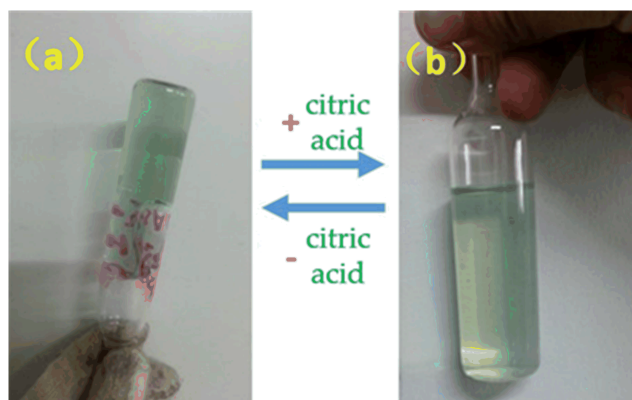


FIG. 2 The comparison pictures of HPAM- Cr^{3+} system (a) without citric acid and (b) with citric acid.

acid as organic complex in the oven at 60 °C, while the system did not produce cross-linking for one month by using citric acid. And there would not be any cross-linking with the further prolongation of time (FIG. 2), by adding both lactic acid and citric acid into the system and further adjusting their ratios, it showed that there existed neither immediate cross-linking, nor delaying cross-linking indicating that no matter what ligand was selected, the time needed for the crosslinking of HPAM and Cr^{3+} could not be prolonged to over one month at 60 °C in the closed conditions. In order to solve this technical problem, we should find another method.

B. Effects of pH on gelation properties of HPAM/lactic acid chromium system

It can be seen from FIG. 1(b) that the complexation between HPAM and Cr^{3+} is essentially a coordination cross-link of carboxylate ions rather than carboxylic acid with Cr^{3+} , similarly, the complexation between lactic acid or citric acid and Cr^{3+} is also a coordination cross-link of carboxylate ions with Cr^{3+} . Therefore these interactions coexist and compete with each other in the aqueous phase. Because the charge repulsion between carboxylate ions in HPAM's polymer chain is evidently larger than that of small molecules, it is difficult for carboxylic acid's ionization degree of HPAM to match that of small molecules, which limits the complexation of HPAM and Cr^{3+} . Then the added organic complexes such as citric acid can complex with chromium ions strongly, resulting in limited ionized chromium ions, so that the HPAM- Cr^{3+} system does not produce any cross-linking at 60 °C in the presence of citric acid and the crosslinking time can be controlled for more than one month. By raising the pH of the system, the carboxyl groups in HPAM will be increasingly ionized to form carboxylate ions, and the complexation between the polyacrylamide and chromium ions will be significantly enhanced, and the resulting complexes

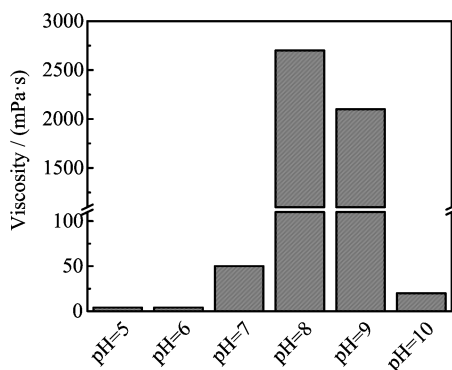


FIG. 3 The change of the viscosity of HPAM-Cr³⁺ system with the pH.

tend to be more competitive. Therefore, HPAM-Cr³⁺ system has pH responsive ability. In order to verify our judgment, we determined the change of the viscosity of HPAM-Cr³⁺ system with pH after 24 h in an enclosed environment at 60 °C. The results are shown in FIG. 3, it can be seen from the figure that when the pH was low (pH=5 or 6), the viscosity of the system was almost the same as that of water, indicating that the cross-linking reaction didn't happen in the system. When the pH was increased to 7, the viscosity of the system rose slightly to about 50 mPa·s, indicating that only a slight cross-linking reaction occurred at this time. Further increasing the pH of the system to 8, the viscosity of the system increased significantly after 24 h, reaching about 2700 mPa·s. With the further increase of pH, the viscosity of the system decreased from 2200 mPa·s at pH=9 to about 20 mPa·s at pH=10. The above results showed that pH had a significant effect on the gelation properties of HPAM/lactic acid chromium system.

Due to the much stronger complexation interaction between citric acid and Cr³⁺ than that between HPAM and Cr³⁺, a higher pH condition should be achieved which is in favor of the crosslinking between HPAM and Cr³⁺. The experimental results of FIG. 3 have shown that the HPAM-Cr³⁺ system did not produce any cross-links at 60 °C in the presence of citric acid, even if the time prolonged to over one month. If bicarbonate ions were engaged in the HPAM-Cr³⁺ system, the pH of the system could be changed resulting from the gradually released CO₂ by bicarbonate ions under heating conditions, which possibly triggered the cross-linking of HPAM with Cr³⁺. If pH buffer such as potassium dihydrogen phosphate was added into the system, and in a completely closed system, the time required to delay the crosslinking of HPAM-Cr³⁺ system in the presence of citric acid could possibly be prolonged to over one month.

C. Effects of release of CO₂ on the pH

In order to use the release of CO₂ to change the pH, we dissolved sodium bicarbonate (NaHCO₃) in the

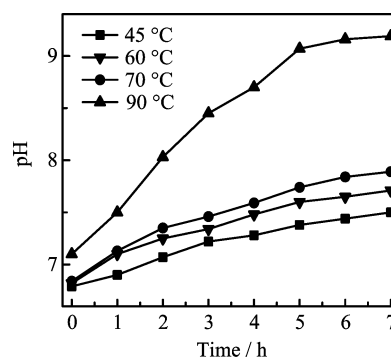


FIG. 4 The change of the pH with time for the solution consisting of 0.1% sodium dihydrogen phosphate and 0.3% sodium bicarbonate at different temperature.

HPAM/citric acid chromium system. To better control the change rate of the pH, we added potassium dihydrogen phosphate (KH₂PO₄)/sodium bicarbonate (NaHCO₃) as a pH buffer into this system. This pH buffer could not only maintain the system at a low pH for a long time, but also produce CO₂ through the reaction of KH₂PO₄ with NaHCO₃, after CO₂ escaped from the solution, the pH of the system increased gradually.

To verify the assumptions mentioned above, the change of the pH with time for the mixed solution of KH₂PO₄ and NaHCO₃ at different temperature was measured, of which the concentration of KH₂PO₄ and NaHCO₃ was 0.1% and 0.3%, respectively. For the mixed solution at 45 and 70 °C, respectively, the initial pH was about 6.8, which was comparable to the pH of the solution prepared *in situ*, and the results are shown in FIG. 4. It can be seen from the figure that the pH of the mixed solution gradually increased to 7.5 at 45 °C and 7.9 at 70 °C in 7 h, respectively. These results verified the correctness of our idea that the pH buffer solution consisting of KH₂PO₄ and NaHCO₃ could initially maintain the system at a low pH, as time went by, the CO₂ generated by the reaction of KH₂PO₄ and NaHCO₃ escaped from the solution, then the pH of the system increased gradually. As the temperature was further increased to 90 °C, the pH of the system changed faster.

It is important to note that in order to facilitate the measurement in the above-mentioned verification test, the airtightness of the system was not good, and this factor accelerated the escape speed of the generated CO₂, as a result, the pH of the mixed solution of KH₂PO₄ and NaHCO₃ increased fast. While the system was airtight, due to the slow escape speed of CO₂, the pH buffer consisting of KH₂PO₄ and NaHCO₃ maintained the system at a low pH for a longer time, and thus the gelling process of HPAM-Cr³⁺ could be delayed and controlled effectively. FIG. 5 shows the change of viscosity with time for the HPAM / citric acid chromium system with 0.1% KH₂PO₄/0.3% NaHCO₃ at 60 °C. It can be seen from the results that the vis-

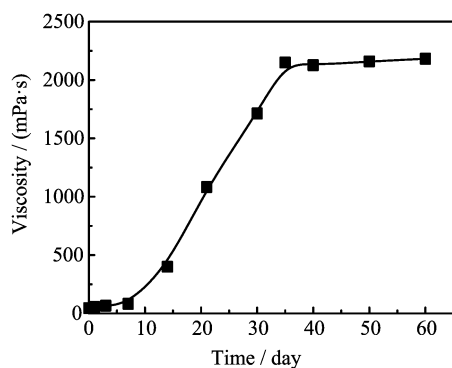


FIG. 5 The change of the viscosity of the HPAM/citric acid chromium system with time.

cosity of the system at the initial state was less than 40 mPa·s, and it remained almost constant or increased only a little over time. However, when the time was prolonged to 30 days, the viscosity of the system was obviously larger than that at the beginning, and the viscosity reached a maximum of 2299 mPa·s after 40 days. With the further prolongation of time, although the viscosity of the system decreased slightly, it was still above 2000 mPa·s. Along with the measurements of the viscosity, we also measured the pH of the sealed sample, the results indicated that the pH of the sample maintained at 7 to 8 for the first 20 days while it increased to 8 to 9 after 1 month, which was consistent with our expectation. It can be seen that there still existed small amount of CO₂ escaping from the solution to the cavity of the tube. As a consequence, the pH of the solution increased slowly, and the complexation between HPAM and Cr³⁺ was gradually activated, then the viscosity of the system accordingly increased. The escape rate of CO₂ could be decelerated in the presence of KH₂PO₄ in a closed system at 60 °C. In turn, the complexation between HPAM and Cr³⁺ was delayed effectively. This novel method we report here can prolong the cross-linking time to over one month in a closed system at 60 °C, and amazingly the final viscosity of the system can be maintained above 2000 mPa·s, which was much more excellent than previously reported ones and, more importantly, meet the engineering requirements.

D. Control of melamine on the proton hydrogen of partially hydrolyzed polyacrylamide (HPAM)

Although the cross-linking time of HPAM/citric acid chromium system could be prolonged to more than one month after adding NaHCO₃ and KH₂PO₄ in a closed system at 60 °C, the initial viscosity of the system was still relatively high, while the ideal water shutoff profile control agent should have both low initial viscosity and high final viscosity. For this purpose, a certain amount of melamine was added into the system. According to the literatures [29, 30], melamine can combine with the

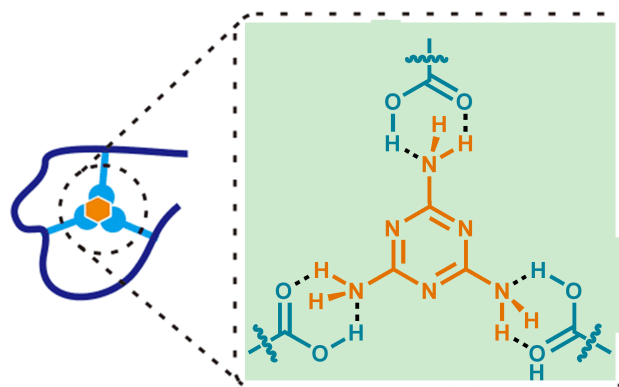


FIG. 6 Photographs and schematic illustration of the carboxylic acid groups in HPAM “bonded” by melamine through hydrogen bonds.

carboxyl groups in the polymer (-COOH) by hydrogen bonding interactions. If there was a certain amount of melamine in the system, the melamine will interact with HPAM through hydrogen bonds, as shown in FIG. 6, resulting in more curled polymer chains, and then the initial viscosity of the system will decrease. In addition, because the hydrogen proton in the polymer carboxyl group will be “confined” through the formation of hydrogen bond between melamine and polyacrylamide, it will be more difficult for the polyacrylamide to ionize. Therefore the complexation between polyacrylamide and chromium ions will be inhibited, and finally the cross-linking of the system can be delayed effectively.

In order to verify this idea, a certain amount of melamine was added to the HPAM/citric acid chromium system, which contained a pH buffer solution consisting of 0.1% KH₂PO₄ and 0.3% NaHCO₃, as shown in FIG. 7. The change of the viscosity with time for the mixture with and without melamine in a closed system at 60 °C was measured. And it can be seen that after adding melamine, the initial viscosity of the system decreased to 10 mPa·s, while the final viscosity remained almost the same, which can fully meet the requirements of engineering. Comparing the data points with and without melamine in FIG. 7, a significantly right shift can be observed after adding melamine, indicating that melamine could delay the complexation between polyacrylamide and chromium ions. Due to the positive correlation between the increase of the pH and the crosslinking of the carboxylate groups in the polymer chain with chromium ions, the carboxylic acid groups “bonded” by melamine through hydrogen bonds were gradually released and converted into carboxylate ions. And the final number of carboxylate ions that could participate in the cross-linking reaction, which was not affected by melamine. So the final gel strength of the system remained unchanged.

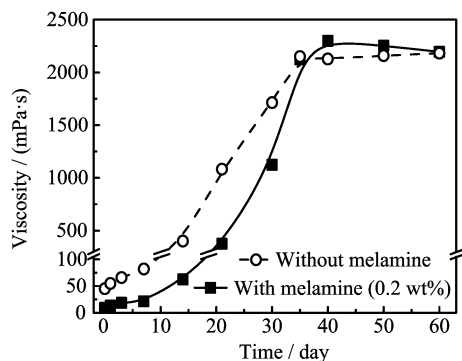


FIG. 7 The effects of the addition of melamine on the gelation properties of HPAM/citric acid chromium system.

IV. CONCLUSION

We have studied the viscosity of HPAM solution in the presence of Cr^{3+} at 60 °C. We find with astonishment that by dissolving citric acid into solution the crosslinking between polyacrylamide (PAM) and Cr^{3+} can be prevented completely. Due to the decomposition of the bicarbonate (HCO_3^-) embedded deliberately in solution, CO_2 released from solution and the pH value of solution increased gradually. The degree of ionization of HPAM and its ability to complex with Cr^{3+} , therefore, increased accordingly. When the complexation of Cr^{3+} with HPAM is stronger than that with citric acid, the viscosity of the HPAM solution increased significantly. Under the closed condition, together with the existence of potassium dihydrogen phosphate (KH_2PO_4), the release of CO_2 was very slow and under a controlled condition so that the ionization of HPAM was prevented initially. Furthermore, the hydrogen bonding interactions between HPAM and melamine embedded in solution previously also postponed the ionization of HPAM. As a result, the crosslinking between HPAM and Cr^{3+} can be delayed for almost one month, completely meeting the requirements for deep water shut-off and profile control to enhance oil recovery.

V. ACKNOWLEDGEMENTS

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