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**EFFECTS OF DEGREE OF HYDROLYSIS AND SHEAR ON
GELATION REACTION KINETICS AND GEL STRENGTH**

Topical Report

**By
Hong W. Gao**

February 1991

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**IIT Research Institute
National Institute for Petroleum and Energy Research
Bartlesville, Oklahoma**

**Bartlesville Project Office
U. S. DEPARTMENT OF ENERGY
Bartlesville, Oklahoma**

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EFFECTS OF DEGREE OF HYDROLYSIS AND SHEAR ON GELATION REACTION KINETICS AND GEL STRENGTH

By Hong W. Gao

ABSTRACT

Gelation tests were conducted to investigate the effect of the degree of hydrolysis on gelation reaction kinetics and gel strength using four low-molecular-weight polyacrylamides (MW = 400,000 daltons), which were 10% (HPAM1-10), 20% (HPAM1-20), 30% (HPAM1-30), and 40% (HPAM1-40) hydrolyzed, and Cr(III) (pH = 4.8) and Al(III) (pH = 7.0) crosslinkers.

Results showed that for polymer/Cr(III) gel systems, samples prepared with a low-molecular-weight polyacrylamide polymer, which was 20% hydrolyzed, gelled at a faster rate and retained higher gel strength than those prepared with a low-molecular-weight polyacrylamide polymer, which was 10% hydrolyzed. Under the screening conditions, no viscosity enhancement was observed in samples prepared with polymers having a degree of hydrolysis equal to or greater than 30%.

For polymer/Al(III) gel systems, samples prepared with a low-molecular-weight polyacrylamide polymer, which was 20% hydrolyzed, gelled at the fastest rate and retained the strongest gel strength among the polymer/Al(III) gel systems prepared with four low-molecular-weight polyacrylamide polymers, which were 10, 20, 30, and 40% hydrolyzed, respectively.

Gelation tests of gel systems in glass bead packs showed that high shear favored the gelation of a gel system that had a fast rate of gelation, but had an adverse effect on the gelation of three gel systems that had a slow rate of gelation. Weak gels were found to be injectable through porous media. Weak gels were degradable under high shear condition and regained viscosity under low shear conditions.

INTRODUCTION

Mechanical degradation of high-molecular-weight polyacrylamides is a practical problem in field applications.¹⁻¹¹ A mobility-control system based on crosslinking a low-molecular-weight polyacrylamide may be much less susceptible to mechanical degradation. Because of the characteristics of crosslinked polymers,¹² low-crosslinking-density gelled polymers under high shear conditions (near the wellbore) should provide a better injectivity than do the conventional polyacrylamides. The ability of the crosslinked gels to reheal at low shear conditions will permit the gel to develop the necessary viscosity to maintain a favorable mobility ratio in reservoir formations where the shear is low.

Previous work¹³⁻¹⁴ has shown that potential gel systems for mobility control can be formulated using a low-molecular-weight polyacrylamide polymer (molecular weight = 400,000 daltons), which was

10% hydrolyzed, and Cr(III) (pH = 4.8), Cr (VI)/thiourea (pH = 6.8) and Al(III) (pH = 7.0) as crosslinkers. Study on two partially hydrolyzed low-molecular-weight polyacrylamides has revealed that gelation can be affected by the degree of hydrolysis.¹³ The degree of hydrolysis has also been shown to affect the gelation rate of a conventional polyacrylamide with a metal crosslinking agent.¹⁵ In this work, the study of the effect of the degree of hydrolysis on gelation reaction kinetics and gel strength was undertaken to identify the optimum degree of hydrolysis for making weak gels for mobility control.

To investigate the flow behavior of crosslinked low-molecular-weight gels in porous media, some of the gel systems that were formulated before¹³⁻¹⁴ and some new formulations were tested in porous media to determine the effect of shear on gelation reaction and gel strength, and their injectivity, shear degradation, and reheelability.

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HYDROLYSIS EXPERIMENTS

To investigate the effect of the degree of hydrolysis on gelation kinetics and gel strength, three batches of low-molecular-weight polyacrylamides having different degrees of hydrolysis were prepared. Before preparing these polymers, hydrolysis experiments were conducted in alkaline media at room temperature (22.4° C) using a low-molecular-weight polyacrylamide (HPAM1-10, MW=400,000 daltons), which was 10% hydrolyzed, to investigate the effects of NaOH concentration and aging time on the degree of hydrolysis. Seven different concentrations of NaOH equivalent to 21 to 80% of the amounts stoichiometrically required for complete conversion of HPAM1-10 to sodium polyacrylate were used. Actual amounts of polymer and NaOH used in each experiment are listed in table 1. The increased degree of hydrolysis of each sample was determined at three different aging times using a titration method.¹⁶⁻¹⁷ Results are shown in table 1 and figure 1. After 120 hours of aging, final degrees of hydrolysis ranging from 15.4 to 40.8% were obtained.

Based on the results of hydrolysis experiments, a batch of low-molecular-weight polyacrylamide, which was 20.4% hydrolyzed, was prepared by aging a sample that contained 3,380 gm of 50,000-ppm HPAM1-10 solution (169 gm of HPAM1-10) and 650 meq of NaOH for 38 hours at room temperature (22.4° C). A second batch of low-molecular-weight polyacrylamide, which was 29.5% hydrolyzed, was prepared by aging a mixture that contained 3,180 gm of 50,000-ppm HPAM1-10 solution (159 gm) and 1,060 meq of NaOH for 50 hours at room temperature. A third batch of low-molecular-weight

polyacrylamide, which was 40% hydrolyzed, was prepared by aging a mixture that contained 2,920 gm of 50,000-ppm HPAM1-10 solution (146 gm) and 1,440 meq of NaOH for 166 hours at room temperature. After the desired degree of hydrolysis was obtained, further hydrolysis of each sample was stopped by adding concentrated acetic acid to neutralize unreacted sodium hydroxide. The hydrolyzed polymers were then purified by precipitation twice from aqueous solution by use of menthol and dried under vacuum at 50° C. These three batches of polymers were used in the following gelation tests.

TABLE 1 - Hydrolysis experiments at 22.4° C

Sample No.	Weight of 40,000-ppm HPAM1-10, gm	meq of -COO- and -CONH ₂	meq of NaOH added	Final degree of hydrolysis after 120 hr of aging
1	100.00	55.93	1.47	15.4
2	92.99	52.01	15.71	22.8
3	88.99	49.77	19.93	29.6
4	85.01	47.55	23.70	33.6
5	81.01	45.31	27.53	37.4
6	77.99	43.62	31.59	39.3
7	75.01	41.96	33.70	40.8

GELATION TESTS – EFFECT OF DEGREE OF HYDROLYSIS ON GELATION REACTION KINETICS AND GEL STRENGTH

To investigate the effect of the degree of hydrolysis on gelation kinetics and gel strength, screening tests were conducted on samples prepared with four low-molecular-weight polyacrylamide polymers (MW=400,000 daltons), which were 10% (HPAM1-10), 20% (HPAM1-20), 30% (HPAM1-30), and 40% (HPAM1-40) hydrolyzed, and a Cr(III) crosslinker in 53 meq/L NaCl at pH = 4.8 and an aluminum citrate crosslinker in 53 meq/L NaCl at pH=7.0 at room temperature, 22.4° C. Polymer concentrations used in screening tests ranged from 13,000 to 17,000 ppm for polymer/Cr(III) systems and from 6,000 to 17,000 ppm for polymer/Al(III) systems. Cr (III) concentrations used ranged from 50 to 175 ppm and Al(III) concentrations used ranged from 25 to 2,800 ppm. All samples were agitated and aged under a nitrogen atmosphere in bottles. Viscosity measurements were used to monitor the progress of gelation.

Crosslinking With Chromium Chloride

Results showed that under the concentrations of polymer and Cr(III) used, precipitate was formed in all samples prepared with HPAM1-40. Precipitate was also formed in samples prepared with HPAM1-30 at a polymer concentration of 13,000 ppm and Cr(III) concentrations greater than 100 ppm, and at a polymer concentration of 15,000 ppm and Cr(III) concentrations greater than 150 ppm. For the samples prepared with HPAM1-20, precipitate formed only in two samples that contained 13,000 ppm of polymer and Cr(III) concentrations greater than 150 ppm. These results indicated that at the same polymer concentration, the maximum concentration of Cr(III) that could be used to crosslink the polymers without forming the precipitate in the solution decreased with the increase in the degree of hydrolysis.

Viscosity measurements on samples that did not form precipitate showed that no viscosity enhancement was observed in samples prepared with HPAM1-30. Viscosity enhancement was observed in samples prepared with HPAM1-10 and HPAM1-20, which were 10 and 20% hydrolyzed, respectively. Comparison of the measured viscosity as a function of time showed that at the same polymer and Cr(III) concentrations, the gel systems prepared with HPAM1-20 gelled at a faster rate and gave higher gel strength than those prepared with HPAM1-10. Typical results are shown in figures 2 and 3 for two gel systems (13,000-ppm polymer/100-ppm Cr(III) and 15,000-ppm polymer/100-ppm Cr(III)). As shown in these two figures, gelation was complete within 3 days in samples prepared with HPAM1-20, which was 20% hydrolyzed, under continuous agitation. Gelation was also complete within 3 days in other samples prepared with HPAM1-20 under continuous agitation. Results also showed that gel strength increased with polymer concentration (figs. 2 and 3) and Cr(III) concentration (fig. 4).

Crosslinking With Aluminum Citrate

Typical results of the effect of the degree of hydrolysis on gelation kinetics and gel strength for polymer/Al(III) systems are shown in figure 5 for four gel systems. Each of which contained 12,000 ppm of polymer and 350 ppm of Al(III). Among the four gel systems, the one prepared with HPAM1-20 gelled at the fastest rate and retained the highest gel strength. Similar results were observed for all other gel systems prepared at polymer concentrations from 12,000 to 17,000 ppm using HPAM1-10, HPAM1-20, HPAM1-30, and HPAM1-40, and at Al(III) concentrations from 350 to 1,400 ppm. Among the samples prepared at a polymer concentration of 6,000 ppm and Al(III) concentrations from 50 to 2,800 ppm, only samples prepared with HPAM1-20 formed gels after 53 days of continuous agitation under a nitrogen atmosphere. These results indicated that the optimal degree of hydrolysis for making weak gels was about 20%. Of these systems, those that contained 15,000 to 17,000 ppm of HPAM1-10 and 350 to 700 ppm of Al(III), 12,000 to 15,000 ppm of HPAM1-30 and 350 ppm of Al(III), 12,000 ppm to 17,000 ppm of HPAM1-40 and 350 ppm of Al(III), and 12,000 ppm of HPAM1-40 and 700 ppm of Al(III) formed weak gels (viscosity between 10 and 100 cP at 5.96 sec^{-1}) after 102 days of continuous agitation under a nitrogen atmosphere. Gel systems prepared with 12,000 ppm of HPAM1-10 and 350 to 1,400 ppm of Al(III) formed very weak gels (viscosity lower than 10 cP at 5.96 sec^{-1}). These gel systems had the potential for use as mobility-control agents.

Figures 6 and 7 show the effects of polymer and crosslinker concentrations on gelation rate and gel strength for HPAM1-20/Al(III) gel systems. Increasing the polymer (fig. 6) or crosslinker concentration (fig. 7) increased the gelation rate and gel strength. After 53 days of continuous agitation under a nitrogen atmosphere, those gel systems that contained 350 ppm of Al(III) and 12,000 to 17,000 ppm of HPAM1-20 (fig. 6), and 8,000 ppm of HPAM1-20 and 1,400 ppm of Al(III) (fig. 7) formed visible gels (viscosity higher than 100 cP at 5.96 sec^{-1}). These systems were too viscous to be used as mobility-control agents. Compared with Cr(III) crosslinker, the Al(III) crosslinker gelled the polymer at a much slower rate.

EFFECT OF SHEAR ON GELATION REACTION AND GEL STRENGTH

Previous studies¹³⁻¹⁴ showed that complete gelation of some polymer/Cr(III) and polymer/Al(III) gel systems was not obtainable with continuous agitation in a bottle. To develop a mixing procedure that will produce constant rheological properties for sufficient amount of gel sample for shear degradation tests, two HPAM1-10/Cr(III) gel systems in 53 meq/L NaCl at pH=-4.8 and two HPAM1-20/aluminum citrate gel systems in 53 meq/L NaCl at pH=7.0 were tested at room temperature (22.4°C) in a flow system that contained a glass bead pack (7.5 cm in length and 0.4 cm in diameter). The glass bead pack was wet packed with sieved glass beads having a particle size distribution of from 170 to 200 mesh. A 390-mesh

screen was placed at each end of the glass bead pack to retain the bead particles. Before injecting a gel system into the glass bead pack, the pack was flushed with brine at pH = 4.8 for tests with polymer/Cr(III) systems and 7.0 for tests with polymer/Al(III) systems until the pH of the effluent brine was equal to that of the injected brine. NaOH and HCl were used to adjust pH. Brine permeability was then measured. Each gel system was filtered through a 325-mesh screen before it was injected into the glass bead pack. Viscosity measurements using a Contraves viscometer or a Brookfield viscometer were made to monitor the progress of gelation.

Polymer/Cr(III) Gel Systems

The glass bead pack used for tests with a gel system that contained 15,000 ppm of HPAM1-10 and 100 ppm of Cr(III) had a brine permeability of 3,420 md. After injecting about 30 mL of the gel system at a flow rate of 0.4 mL/min (apparent shear rate $1,160 \text{ sec}^{-1}$) into the glass bead pack, the glass bead pack was completely plugged under an injection pressure of about 200 psi. After replacing the 390-mesh screen on each end of the pack with a new screen, the glass bead pack was still plugged, indicating that strong gel was formed inside the porous medium. In a separate shear experiment with a Brookfield viscometer, the viscosity of this system increased from 5.85 to 22.8 cP after being continuously sheared for 3 days at a shear rate of 46 sec^{-1} . A gel system with a viscosity of 22.8 cP should be injectable under a pressure of 200 psi. These results indicated that mixing the gel system that contained 15,000 ppm of HPAM1-10 and 100 ppm of Cr(III) at high shear condition formed gel at a faster rate and gave higher gel strength than did mixing the gel system continuously either in a Brookfield viscometer at low shear condition (46 sec^{-1}), or in a bottle.

Another gel system that contained 15,000 ppm of HPAM1-10 and 25 ppm of Cr(III) was tested in a glass bead pack having a brine permeability of 3,710 md. Before injecting this system into the glass bead pack, gelation reaction was monitored with a Brookfield viscometer. After 6 days of continuous shearing at a shear rate of 46 sec^{-1} , the viscosity increased from 5.65 to 6.2 cP, an increase of only 9.7%, indicating that the system had a slow rate of gelation. After continuously injecting this system through the glass bead pack at an apparent shear rate of greater than $1,000 \text{ sec}^{-1}$ for more than 23 times (5,000 PV), the system did not show any viscosity enhancement, indicating that high shear had an adverse effect on the gelation of a gel system that had a slow rate of gelation.

Polymer/Al(III) Gel Systems

A gelation test for a gel system that contained 12,000 ppm of HPAM1-20 and 200 ppm of Al(III) in 53 meq/L NaCl at pH = 7.0 was conducted in a glass bead pack having a brine permeability of 4,200 md. Before injection through the porous medium, the gel system had been agitated for 3 days in a bottle and was filtered through a 325-mesh screen. After one, two, and more than 15 times (3 days) of passing through the porous medium, no viscosity enhancement was observed, indicating that no gelation occurred in the porous medium at high shear conditions. However, for the same gel system agitated for 3 days in a bottle, the viscosity measured at a shear rate of 5.96 sec^{-1} increased from 16.2 to 18.5 cP, an increase of 14%. A separate gelation test using the same gel system in a Brookfield viscometer also showed that the viscosity increased by 20% after 3 days of continuous shearing at a shear rate of 46 sec^{-1} . Compared with the gel system that contained 15,000 ppm of HPAM1-10 and 100 ppm of Cr(III), this gel system had a slower rate of gelation. These results indicated that high shear also had an adverse effect on the gelation of a polymer/Al(III) gel system that had a slow rate of gelation.

A gelation test for another gel system that contained 10,000 ppm of HPAM1-20 and 200 ppm of Al(III) in 53 meq/L NaCl at pH = 7.0 was conducted in a flow system that contained about 1,200 cm of 0.125-in OD tubing at apparent shear rates between 50 and 195 sec^{-1} . Results are shown in figure 8. The average apparent shear rate used during the 1st to 6th day and 15th to 20th day was about 55 sec^{-1} . As shown in figure 8, the rate of gelation reaction for the gel system under shearing condition in the flow system was about the same as that in a bottle under continuous agitation. These results indicated that for a gel system that had a slow gelation rate, the rate of gelation reaction in a bottle under continuous agitation would be close to that in a flow system under low shear condition. These results indicated that low shear favored the gelation of the gel system that had a slow rate of gelation.

To test whether the gel system was degradable and injectable through a porous medium, the above gel system was injected through a glass bead pack having a brine permeability of 3,600 md at apparent shear rates higher than $1,000 \text{ sec}^{-1}$ (highest shear rate used was about $2,200 \text{ sec}^{-1}$) after 20 days of continuous circulation in the flow system. After more than six passes, the viscosity of the gel system measured at a shear rate of 5.96 sec^{-1} decreased from 24.4 to 19.4 cP, as shown in figure 8, indicating that some of the crosslinking bonds were broken under high shear condition. After the high shear condition was removed, gelation reaction resumed, and the viscosity increased from 19.4 to 23.42 cP (fig. 8) after another 4 days of shearing in the flow system at low shear condition. The higher gelation rate as indicated by the larger slope of the viscosity-aging time curve after the high shear was removed than that before the crosslinking bonds were broken indicated that some of the broken bonds were rehealed. This test also demonstrated that the weak gel system (more than 1,460 PV) was injectable through a porous medium.

CONCLUSIONS

Based on the results of this study:

1. For a low-molecular-weight polyacrylamide polymer (molecular weight = 400,000 daltons), the optimum degree of hydrolysis for making weak gels for mobility control is about 20% when using Cr(III) at pH = 4.8 or Al(III) at pH = 7.0 in 53 meq/L NaCl as the crosslinker.
2. High shear favors the gelation of gel systems that have a fast rate of gelation and low shear favors the gelation of gel systems that have a slow rate of gelation.
3. Crosslinking bonds are broken under high shear condition and reformed under low shear condition.

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FIGURE 1. Effects of concentration of NaOH and aging time on the hydrolysis of HPAM1-10 at 22.4°C

FIGURE 2. Effect of the degree of hydrolysis on gelation rate and gel strength of polymer/Cr(III) gel systems at 22.4° C.

FIGURE 3. Effect of the degree of hydrolysis on gelation rate and gel strength of polymer/Cr(III) gel systems at 22.4°C.

FIGURE 4. Effect of Cr(III) concentration on gelation rate and gel strength at 22.4°C

FIGURE 5. Effect of the degree of hydrolysis on gelation rate and gel strength of polymer/Al(III) gel systems at 22.4°C.

FIGURE 6. Effect of polymer concentration on gelation rate and gel strength of HPAM1-20/350-ppm Al(III) gel systems at 22.4°C.

FIGURE 7. Effect of crosslinker concentration on: gelation rate and gel strength of 8,000-ppm HPAM1-20/AI(III) gel systems at 22.4°C.

FIGURE 8. Effects of shearing and agitation on the gelation rate of a 10,000-ppm HPAM1-20/200-ppm Al(III) gel system at 22.4° C.

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