

**EFFECT OF Cr^{3+} ON THE RHEOLOGY OF XANTHAN FORMULATIONS
IN POROUS MEDIA: BEFORE AND AFTER GELATION**

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ABSTRACT

Experiments were performed to probe the rheology of chromium(III)-xanthan gels and gels in porous media. For a large portion of the time before gelation, the presence of Cr^{3+} did not significantly affect the rheology of xanthan solutions in Berea sandstone. During brine injection after gelation, gels that contained 4000-ppm xanthan and 154-ppm Cr^{3+} provided substantial residual resistance factors (30 to 714). Residual resistance factors decreased significantly with increased fluid velocity and could be described by a power-law relation for superficial velocities ranging from 0.0076 to 4.79 m/d. Laboratory measurements of gel properties in $0.065\text{-}\mu\text{m}^2$ Berea and in $0.477\text{-}\mu\text{m}^2$ Berea were used during example calculations to show that the apparent "shear-thinning" nature of residual resistance factors will not eliminate the need for zone isolation during gel placement in unfractured injection wells.

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For many years, researchers have been aware of the non-Newtonian rheology of polymer solutions in porous media.¹⁻⁴ This has prompted some to speculate that non-Newtonian rheology could be exploited to maximize gelant penetration into high-permeability zones while minimizing gelant penetration into less-permeable, oil-productive zones.⁵ In an earlier paper,⁶ the potential of this idea was examined using eight different models of non-Newtonian rheology (including shear-thickening models,

shear-thinning models, and various combinations). The analysis indicated that the rheology of existing polymers will not eliminate the need for zone isolation during gel placement in unfractured injection wells. However, the validity of these calculations rests on several assumptions that need to be examined experimentally. First, the calculations assume that prior to gelation, the rheology of polymer solutions with crosslinkers is the same as that of polymer solutions without crosslinkers. Second, after gelation, the residual resistance factors are assumed to be independent of velocity and permeability. Third, the calculations assume that the wells are shut-in at the time gelation occurs. The calculations also assume that gels are not mobile. This paper reports the results of experiments that were designed to test some of the above assumptions for chromium(III)-xanthan gels in porous media.

RHEOLOGY BEFORE GELATION

Our first experiments were designed to test whether the rheology of xanthan solutions in porous media before gelation depends on the presence of chromium. These experiments were performed using aqueous solutions that contained 3000-ppm xanthan, 3% NaCl, and 0.3% CaCl₂ at 29°C. When chromium(III) was included, a concentration of 90 ppm was used. Pfizer provided both the xanthan (Flocon 4800[®]) and the chromium (X-link 2000[®]) that were used in this work. In these core experiments, Berea sandstone cores were used that had diameters of 1.27 cm and lengths of 15.24 cm. All cores had one internal pressure tap that was located 2.54 cm from the inlet sand face. The cores were not fired.

Viscosity vs. shear-rate data are shown in Fig. 1a for five 3000-ppm xanthan solutions:

- (a) without chromium, before injection;
- (b) without chromium, effluent from 0.289- μm^2 core after 10 pore volumes (PV);
- (c) with 90-ppm Cr³⁺ (freshly mixed), before injection;
- (d) with 90-ppm Cr³⁺, effluent from 0.289- μm^2 core (after injecting 10 PV, 0.7 hours after gelant preparation); and
- (e) with 90-ppm Cr³⁺, effluent from 0.289- μm^2 core (after injecting 53 PV, 4.5 hours after gelant preparation).

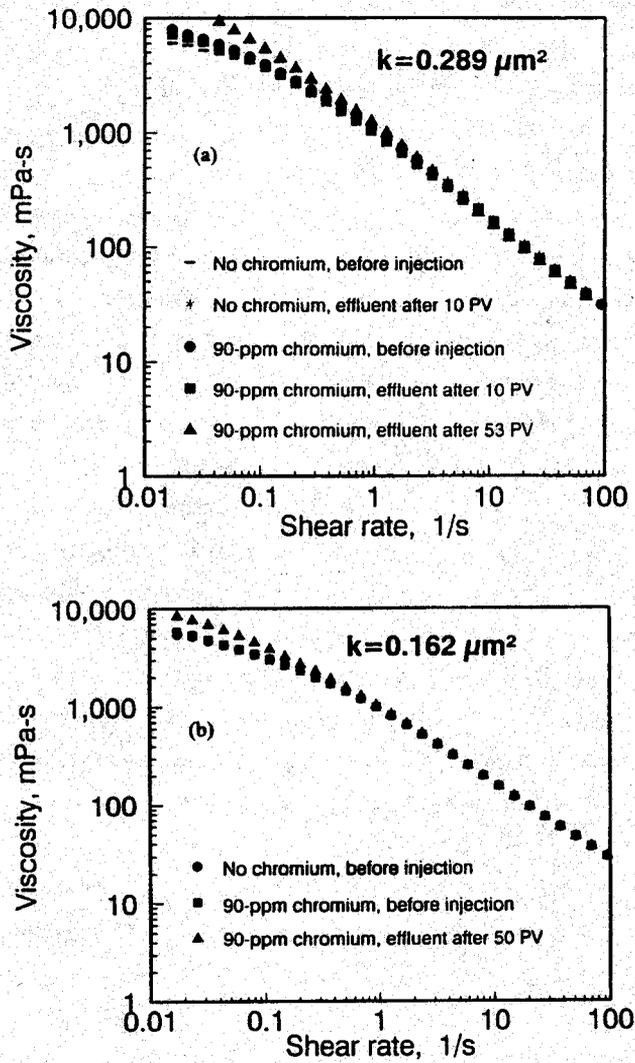


FIG. 1. Effects of chromium and flow through Berea sandstone on the viscosity of 3000-ppm xanthan.

Except possibly at very low shear rates, there is no significant difference in rheology among the five different solutions—at least not in the first several hours of the gelation process. The gelation time (as measured by monitoring viscosity at 1.75 s^{-1}) was 10.5 hours for gelant that was not injected into a core. The gelation time was about 8 hours for effluent that was collected after injection at a superficial velocity of 68.6 m/d. A firm gel formed in both cases. Figure 1b shows results from a similar experiment that involved flow through $0.162\text{-}\mu\text{m}^2$ Berea sandstone.

The effect of Cr^{3+} on xanthan rheology in Berea sandstone (during the first 7 hours of the gelation process) is shown in Fig. 2. Two experiments were performed with no oil present (in Berea sandstone with permeabilities of $0.289 \mu\text{m}^2$ and $0.162 \mu\text{m}^2$, respectively). One experiment was performed with a residual oil (*n*-dodecane) saturation of 0.31. For the core experiments, the rheology of xanthan solutions without Cr^{3+} was determined first. Then, brine was injected (at least 75 pore volumes) to displace the xanthan and to determine residual resistance factors as a function of flow rate. Next, xanthan solutions with 90-ppm Cr^{3+} were injected to determine resistance factors as a function of flow rate. This step was performed as early in the gelation process as was practical. Then, the core was shut in for several days to allow gelation to proceed. Gel and gelant were removed from the flow lines, and gel was scraped from the sand faces. Finally, brine was injected to determine residual resistance factors as a function of flow rate. Resistance factors and residual resistance factors were monitored in both core segments. The values that are reported here apply to the second core segment, so effects from plugging of the inlet sand face are excluded. A detailed listing of all results is included in Ref. 7.

Figure 2 shows that, during the first 7 hours of the gelation process, the presence of 90-ppm Cr^{3+} does not significantly affect the rheology of a 3000-ppm xanthan solution in Berea sandstone.

RHEOLOGY DURING GELATION

Researchers at the University of Kansas^{8,9} have examined the flow of Cr^{3+} -xanthan gels in sandpacks. They believe that filtration of polymer aggregates plays an important role in permeability reduction during gel treatments.^{8,10} A filtration

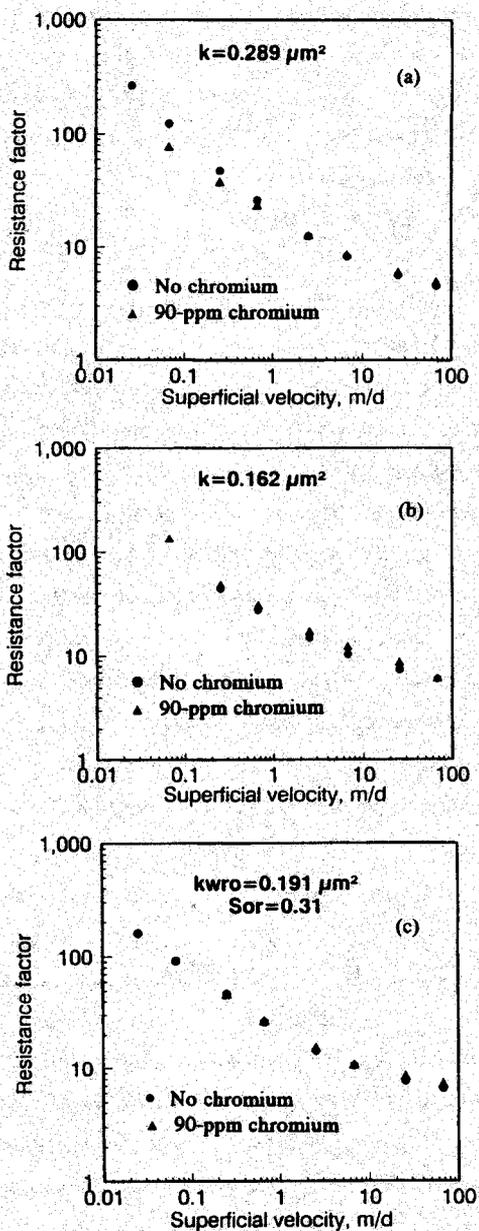


FIG. 2. Effect of chromium on the rheology of 3000-ppm xanthan in Berea sandstone. (\bullet No chromium; \blacktriangle 90-ppm chromium)

mechanism is most likely to be important if flow is occurring at the time that polymer aggregates grow to the size of pore throats. However, early in the gelation process (when polymer aggregates are small or are present in very low concentrations), filtration may not be important. Our resistance-factor data show that, early in the gelation process, the rheology of Cr^{3+} -xanthan gelants is not significantly different from that of a xanthan solution without Cr^{3+} . Data from the University of Kansas do not contradict this

How long after the gelant is prepared does the filtration mechanism become important? Our data indicate that the rheology in porous media remains unchanged for at least half of the time required for the onset of gelation to be noticed in a beaker or viscometer. Data from the literature^{8,9} suggest that even longer time periods may be required for the filtration mechanism to be important for Cr^{3+} -xanthan gelants.

After gelant injection, wells are commonly shut in. If the well is shut in relatively early in the gelation process, a filtration mechanism may not be important. If flow is still occurring at the time when significant polymer aggregates develop, of course, filtration may be important. Is any advantage to be gained by the flow of polymer or gel aggregates? More specifically, will polymer aggregates plug high-permeability zones to a greater extent than low-permeability zones? Hejri *et al.*⁸ noted that resistance factors for Cr^{3+} -xanthan polymer aggregates increased with decreasing permeability. More work is needed to fully assess the impact of filtration on the effectiveness of gel treatments.

EFFECT OF FLOW THROUGH POROUS MEDIA ON GELATION TIME

We noted that flow through Berea at high rates (68.6 m/d) decreased the gelation time for a gelant that contained 3000-ppm xanthan and 90-ppm Cr^{3+} . In contrast, Hejri *et al.*⁸ and Garver *et al.*¹¹ found that flow through sandpacks and Berea sandstone increased the gelation time. Hejri *et al.* used gelants with 1500-ppm xanthan and 50-ppm Cr^{3+} , while Garver *et al.* used gelants with 500-ppm to 1000-ppm xanthan and with 50-ppm to 150-ppm Cr^{3+} . Interestingly, Jousset *et al.*⁹ found cases where flow through sandpacks did not change the gelation time significantly for a gelant that contained 1500-ppm xanthan and 50-ppm Cr^{3+} . Clearly, more work is needed to resolve how flow through porous media affects gelation.

RHEOLOGY DURING BRINE INJECTION AFTER GELATION

Our most disturbing finding was that **residual resistance factors were very low** during brine injection after gelation. In general, **residual resistance factors for the Cr^{3+} -xanthan gels** were not much greater than those **for xanthan solutions without Cr^{3+}** (having values that were **slightly greater than unity**).

Why were residual **resistance factors so low**? We note that the pH of freshly prepared Cr^{3+} -xanthan gelant was 3.8. We also note that if pH was maintained at a value of 7 (using buffers), gelation did not occur. **Reservoir rocks usually have a tremendous capacity to buffer aqueous solutions near neutral pH.**¹²⁻¹⁶ Therefore, contact with reservoir rock could render Cr^{3+} -xanthan gelants **ineffective** by changing pH and inhibiting gelation.

Do our low residual resistance factors contradict **previous literature reports**? **Several researchers^{8,9,17}** noted substantial residual resistance factors for Cr^{3+} -xanthan gels (as high as 9200). These researchers often used lower polymer and Cr^{3+} concentrations (e.g., 1500-ppm xanthan, 50-ppm Cr^{3+}) than those used in our study. **Thus, our low residual resistance factors appear surprising upon first consideration.** However, the previous researchers **performed their** studies using sandpacks (with Ottawa sand). **These sandpacks do not contain clays or carbonate minerals** (e.g., dolomite, calcite, siderite) that are responsible for the buffering capacity of reservoir rocks. Therefore, it is not surprising that gelation in these sandpacks is not inhibited to the extent observed in consolidated sandstones.

In order to achieve higher residual resistance factors, additional core experiments **were performed** using a gelant that contained 4000-ppm xanthan **and 154-ppm Cr^{3+}** . The **brine for these** experiments contained 0.5% KCl, and the temperature was 41°C. The viscosity of this brine **was 0.65 mPa·s** at 41°C. After saturation with brine and determination of permeabilities and porosities, tracer studies **were performed** to determine dispersivity values (α_f) for the cores. Gelant was then placed in the cores using a superficial velocity of 4.79 m/d. The cores were then shut in for several days to allow **gelation to occur**. After gelation, brine was injected to determine residual resistance factors. Finally, tracer studies were again performed to determine the final dispersivity values (α_f) and the relative changes in pore volume (V_{pi}/V_{pi}) that were caused by the gel.

One core experiment (in $0.076\text{-}\mu\text{m}^2$ Berea) was performed using a Cr^{3+} -xanthan gelant without adjustment of pH. **This gelant had an initial pH value of 4. The gelation time for this gelant (at 41°C) was 6 hours, and a rigid gel was formed.** However, we noted that this gelant had very little buffering capacity (i.e., its pH could be changed very easily). During injection of **gelant at pH=4, we** also noted that the pH of the effluent **remained** near neutral after several pore volumes. We were concerned that a pH value of 4 was not representative of the pH experienced by the gelant in the core. Therefore, we performed two core experiments (in $0.065\text{-}\mu\text{m}^2$ Berea and in $0.477\text{-}\mu\text{m}^2$ Berea) in which the pH was 7 for the injected gelant.

Residual resistance factors after gel placement are shown in Fig. 3 for three different Berea cores. In all three cores, residual resistance factors decreased significantly with increased fluid velocity. Equations that describe the relation between residual resistance factor (F_{rr}) and superficial velocity (u , in m/d) are included in Fig. 3. Power-law exponents for the relations vary from -0.26 to -0.55 . Of course, brine is a Newtonian fluid, so the apparent "shear-thinning" behavior must be attributed to the gel in the core rather than to the brine. The apparent shear-thinning behavior generally is not due to gel mobilization upon exposure to successively higher flow rates. Stable pressure drops were quickly achieved at a given injection rate. Also, residual resistance factors at a given injection rate in a given core were fairly reproducible.

We noted that Cr^{3+} -xanthan gelants that were buffered at pH=7 did not appear to gel during beaker tests. Based on this observation, we expected to see low residual resistance factors for the gelants that were injected at pH=7. Thus, we were somewhat surprised that residual resistance factors shown in Figs. 3b and 3c (associated with gelant injected at pH=7) were generally as high as those shown in Fig. 3a (associated with gelant injected at pH=4).

Figure 4 shows results of tracer studies that were performed before and after gel placement in the three cores, respectively. (The tracer bank included potassium iodide that was monitored spectrophotometrically at 230 nm.) In all three cores, the gel increased dispersivity values by factors ranging from 5.5 to 17.8. The 50% concentration level for tracer effluent provides an indication of the fraction of original pore volume (V_{pf}/V_{pi}) that remains after gelation. This fraction ranged from 0.55 to 1. In $0.076\text{-}\mu\text{m}^2$ Berea, the gel formed from gelant at pH=4 apparently occupies 45% of the original pore volume (see Fig. 4a). In contrast, in $0.477\text{-}\mu\text{m}^2$ Berea, the gel formed

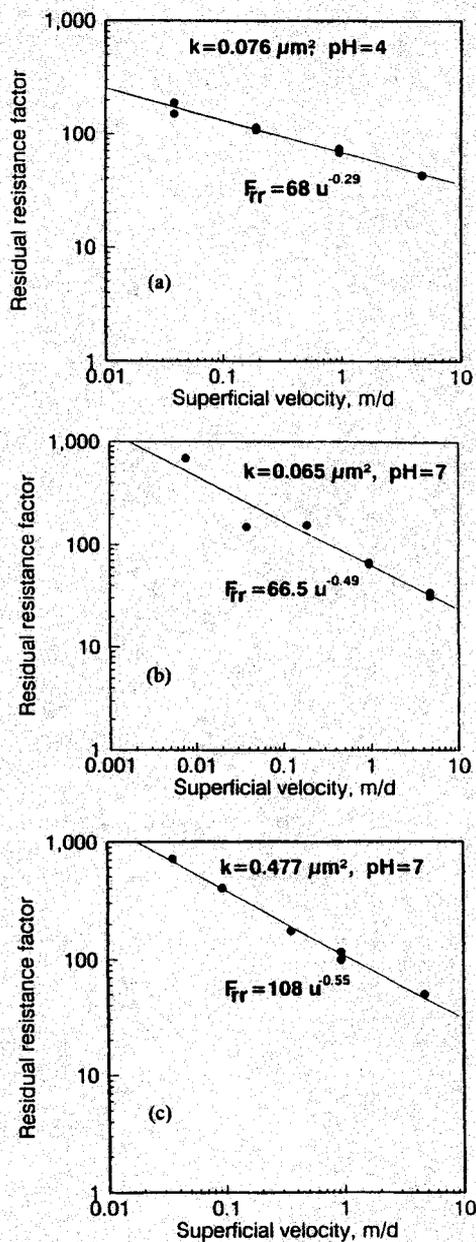


FIG. 3. Residual resistance factor (F_{rr}) vs. superficial velocity (u). (4000-ppm xanthan, 154-ppm chromium)

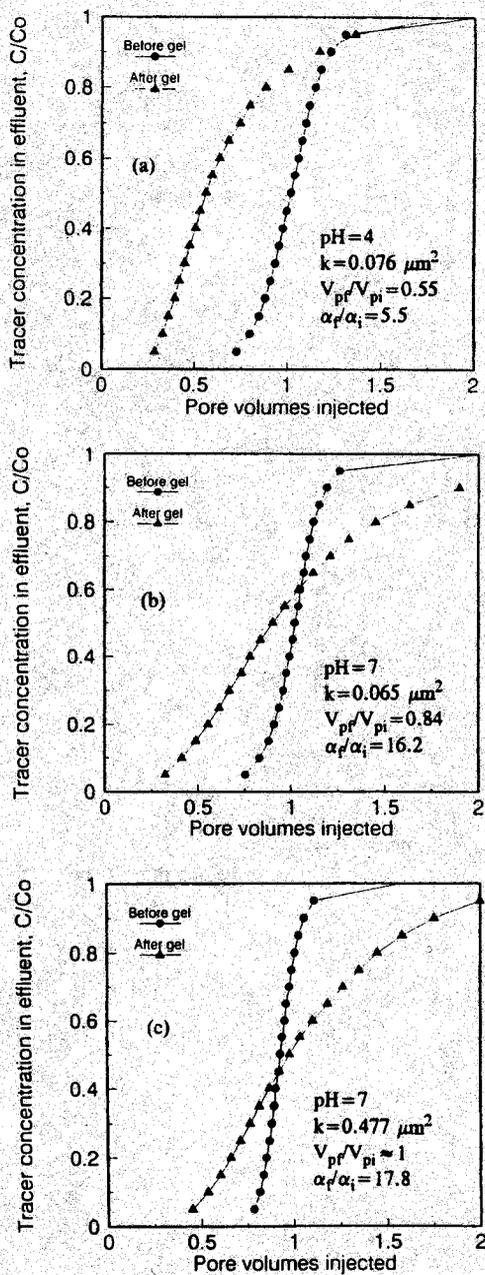


FIG. 4. Tracer effluent curves during brine injection before (●) vs. after (▲) gel placement in Berea sandstone. (4000-ppm xanthan, 154-ppm chromium)

from gelant at pH = 7 apparently occupies an insignificant fraction of the original pore volume (see Fig. 4c). How can a gel reduce permeability substantially and yet occupy a very small fraction of the pore volume? Perhaps, small gel **particles lodge in pore throats**—thereby, dramatically reducing brine permeability without **occupying much** volume. This behavior requires further study.

EXAMPLE CALCULATIONS FOR AN UNFRACTURED INJECTION WELL

The above information can be used to estimate the effects of a gel treatment in an unfractured injection well. For example, consider a reservoir with two noncommunicating layers. Layer 1 has a permeability (k_1) of $0.477 \mu\text{m}^2$ and a porosity (ϕ_1) of **0.219**. Layer 2 has a permeability (k_2) of $0.065 \mu\text{m}^2$ and a porosity (ϕ_2) of **0.185**. The wellbore radius is 0.15 m. The pressure drop (downhole) between this **unfractured injection well** and a nearby production well (in a 16-ha five-spot pattern) is maintained constant at 6895 kPa. During brine injection before the **gel treatment**, the superficial velocities (u_1 , u_2) at the wellbore radius can be shown to be **207 m/d in Layer 1** and 28.5 m/d in Layer 2.⁷

A gelant with 4000-ppm xanthan and 154-ppm Cr^{3+} at pH = 7 is injected (without isolating zones) until the gelant reaches a radius of 15.24 meters from the injector in Layer 1. We assume that chemical retention and inaccessible pore volume are the same in $0.477\text{-}\mu\text{m}^2$ rock as in $0.065\text{-}\mu\text{m}^2$ rock. Diffusion and dispersion were neglected. (For discussions of the impact of chemical retention, inaccessible pore volume, diffusion, and dispersion, see Refs. 6, 18, 19, and 20.)

We can estimate the minimum depth of penetration of gelant in Layer 2. The minimum penetration into Layer 2 will occur if the resistance factor (F_r) of the gelant is equal to one.^{6,18} If $F_r = 1$ and if the gelant penetrates 15.24 meters into Layer 1, then the minimum radius of penetration into Layer 2 will be 6.15 meters.⁷ In reality, a **viscous xanthan gelant** will penetrate a greater distance into Layer 2.⁶ However, we will use the optimistic value of 6.15 m in our subsequent calculations.

After injecting the gelant, the well is shut in to allow gelation. When brine injection is resumed, Fig. 3 can be used to determine the residual resistance factor (F_{rr}) at a given superficial velocity. In particular, $F_{rr} = 108u^{-0.55}$ in Layer 1 and $F_{rr} = 66.5u^{-0.49}$ in Layer

2. For the case of constant injection pressure, the fluid velocities at the wellbore radius after the gel treatment are $u_1 = 1.64$ m/d and $u_2 = 0.323$ m/d.⁷ We note that these velocities are within the range where the F_{rr} relations are valid in Fig. 3. If fluid velocities are too high, unrealistic F_{rr} values can be predicted. In particular, F_{rr} values cannot be less than one. When making calculations, checks should be made to insure that F_{rr} values are realistic.

Finally, the gel treatment is found to reduce brine injectivity in Layer 1 to 0.79% of the original injectivity. This value is determined by dividing u_1 obtained after the gel treatment (1.64 m/d) by u_1 obtained before the gel treatment (207 m/d). In the same way, the gel treatment is estimated to reduce brine injectivity in Layer 2 to 1.13% of the original injectivity (0.323/28.5). This latter estimate of injectivity reduction is actually optimistic. Recall that we assumed that the gelant would penetrate the minimum distance of 6.15 m into Layer 2. Since a viscous xanthan gelant will probably penetrate a greater distance into Layer 2, the final brine injectivity in Layer 2 will be somewhat less than 1.13% of the original injectivity.

The final result of the gel treatment is that the injection profile has not been improved to any significant extent. Injectivity in both layers has been reduced by about 99%. This injectivity reduction may be desirable in the most-permeable layer, but it would be very harmful in the oil-productive layer (i.e., Layer 2). Thus, zone isolation would be needed during gel placement. The same conclusion is reached if residual resistance factors are assumed to be constant after gelation.¹⁸ Thus, the apparent non-Newtonian behavior of residual resistance factors for this Cr^{3+} -xanthan gelant will not eliminate the need for zone isolation in unfractured injection wells.

The above analysis can be applied in a similar manner to gel placement in vertically fractured injection wells. To some extent, the analysis is simplified in fractured wells because the flow geometry is linear.⁷ However, in fractured wells, two other factors should be considered. First, injection of viscous polymer solutions or gels may extend the length of the fracture. Second, a large part of the beneficial effects that are derived from the gel may come from reducing the flow capacity of the fracture rather than from selectively reducing permeabilities in the different strata that are cut by the fracture.

CONCLUSIONS

1. For a large fraction of the time before gelation, the presence of 90-ppm Cr^{3+} did not significantly affect the rheology of a 3000-ppm xanthan solution in Berea sandstone.
2. During brine injection after gel placement, residual resistance factors in Berea sandstone were quite low. Clay and carbonate minerals in Berea may have forced the gelation reaction to occur near neutral pH rather than at the injection pH (3.8). The buffering action of reservoir rocks should be considered when evaluating gel performance in the laboratory.
3. Using 4000-ppm xanthan and 154-ppm Cr^{3+} , a rigid gel was formed in a beaker at pH = 4, but gelation was not evident at pH = 7. Even so, the composition injected at pH = 7 provided substantial residual resistance factors (30 to 714) in Berea sandstone.
4. Tracer studies indicated that the gel (4000-ppm xanthan, 154-ppm Cr_3^+) occupied between 0% and 45% of the original pore volume, depending on the initial pH of the gelant and the core permeability. Tracer studies also revealed that the gel increased dispersivity values in Berea by factors ranging from 5.5 to 17.8.
5. For a gel with 4000-ppm xanthan and 154-ppm Cr^{3+} at pH = 7 in $0.477\text{-}\mu\text{m}^2$ Berea, residual resistance factors were quite high (50 to 714), even though tracer studies indicated that the pore volume occupied by the gel was near zero. Perhaps, small gel particles lodge in pore throats—thereby, dramatically reducing brine permeability without occupying much volume.
6. Residual resistance factors provided by gels (4000-ppm xanthan, 154-ppm Cr^{3+}) decreased significantly with increased fluid velocity and could be described by a power-law relation for superficial velocities ranging from 0.0076 to 4.79 m/d.
7. Laboratory measurements of gel properties in $0.065\text{-}\mu\text{m}^2$ Berea and in $0.477\text{-}\mu\text{m}^2$ Berea were used during example calculations to show that the apparent "shear-thinning" nature of residual resistance factors will not eliminate the need for zone isolation during gel placement in unfractured injection wells.

ACKNOWLEDGEMENTS

This work was financially supported by the U.S. Department of Energy, the New Mexico Research and Development Institute, Amoco, Conoco, Marathon Oil, Mobil Research & Development Corp., Oryx Energy, Oxy USA, Phillips Petroleum, Shell Development Co., Elf Aquitaine, and Texaco. This support is gratefully acknowledged. Richard Schrader performed most of the experimental work, and his contribution is greatly appreciated. We also thank Jeff Albers for his help during some of the experiments. The authors also appreciate the help of the staff of the New Mexico Petroleum Recovery Research Center in preparing and reviewing this paper.

NOMENCLATURE

- C = tracer concentration in effluent, kg/m^3
 C_o = injected tracer concentration, kg/m^3
 F_r = resistance factor (brine mobility divided by gelant mobility)
 F_{rr} = residual resistance factor (brine mobility before gel placement divided by brine mobility after gel placement)
 k_i = permeability of Layer i , μm^2
 k_{wro} = permeability to water at S_{or} , μm^2
 S_{or} = residual oil saturation
 u = fluid flux or superficial velocity, m/d
 u_i = superficial velocity at the wellbore radius in Layer i , m/d
 V_{pf} = apparent remaining pore volume, cm^3
 V_{pi} = initial pore volume of the core, cm^3
 α_f = dispersivity of the core after gel placement, m
 α_i = initial dispersivity of the core, m
 ϕ_i = porosity in Layer i

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