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Rheological Studies of a Cross-Linked Polymer Gel System

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Abstract

The petroleum accumulations are found associated with water and it is rarely obtained without accompanying water production. Water production is a major technical, environmental, and economic challenge associated with oil and gas extraction. Rheological findings are of fundamental importance for the development, manufacture and processing of innumerable products. Polymeric gels exhibit a wide range of rheological properties and they are stable both mechanically and thermally in most cases thus are suitable for a variety of applications including controlled drug recovery, soft contact lenses and gelatin foods. The flow curves of different polymer gelants at different concentrations have been presented at different temperatures. In this study, the increment of gelant solution viscosity during gelling reaction was considered as an indication of development of gelation kinetic. Thus, a thorough knowledge of the viscosity changes with gelation is important for obtaining better results in the field. The data obtained from rheological study helps to roughly analyze the gelation time which can be determined from the viscosity buildup of the gelant. Thus, this is an indirect method to determine the gelation time which is an important characteristic of the polymer gel system. investigation on the bulk gelation studies and the effect of different parameters such as temperature, salinity, pH, concentration of polymer and cross-linkers on the gelation time and the thermal stability of the modified gel system in bulk. Gelation time is one of the most important characteristics of a polymer system which helps to determine the depth up to which the gelant can flow before gelation because if gelation occurs before reaching the target zones than the purpose of using the gel will remain unfulfilled.

Key word Polymer gelation, rheological properties, water production control, gelation time, thermal stability, cross-linking agents, enhanced oil recovery

INTRODUCTION

Polymer gels are very good examples of viscoelastic fluid which is a type of time independent non-Newtonian fluid. Viscoelastic fluids, as the name suggests, exhibit properties that are intermediate to both viscous liquids and elastic solids. Usually, the elastic nature dominates over short time periods while the viscous nature dominates over longer time scales.

The Herschel-Bulkley equation (Eqn. 1), obtained from a generalized model of Non-Newtonian fluid, is commonly written as:

$$\tau = \tau_0 + K\gamma^n \quad (1)$$

where τ = shear stress, γ = shear rate, τ_0 = yield stress, and K and n are regarded as model factors.

Polymeric gels are obtained by cross-linking high and/or low molecular weight polymers with a suitable cross-linker. Recent work showed that a combination of both levels can enhance the mechanical and thermal properties of the gel. These materials exhibit a wide range of rheological

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properties, making them suitable for a variety of applications including controlled drug recovery, soft contact lenses and gelatin foods. Gels are utilized in the oil industry to minimize water production [1–6].

Rheology tests are being used to quantitatively study the viscoelastic properties of PAMHQ/HMTA polymer gels. Gel strength is an indication of the maximum drawdown that polymer gels can sustain. Initially, the viscosity of the gelant solution was very low and gel strength was almost the same as that of polymer solution strength; therefore, no three-dimensional gel polymer network structure was detected. However, with the passage of time and through ageing at a specific temperature, the rate of gelling reaction increased. Consequently, when the cross-linking reaction took place in the gelant solution, it finally led to the formation of a three-dimensional gel polymer network structure. The objective of this study is to investigate the effect of salinity, gel composition, temperature and gel age on the viscoelastic properties of this system [7–12].

INSTRUMENTATION

The apparent viscosity of the different polymer gel samples as a function of shear rate was measured with the help of Brookfield Viscometer (Model DV-1+) shown in Figure 1.

The polymer gelants are viscoelastic fluids and with gelation their viscosity increases hence with higher range of viscometer we can measure their viscosity for longer time interval. The range of shear rate for this viscometer for is from 0.3–100 rpm. Figure 2 depicts the change in viscosity with shear rate for the viscometer mentioned above.

Figure 1. Brookfield viscometer.

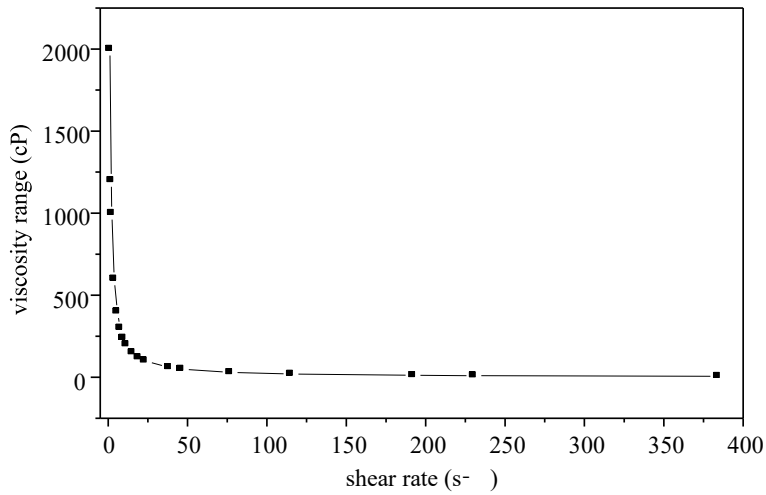

Figure 2. Viscosity range graph of Brookfield viscometer.

Figure 3. Rheometer (Physica MC1) for rheology measurement.

This graph is important because it gives an idea about the variation of viscosity of fluids measured by it, so that we can judge the reliability of the readings taken for different samples of the polymer gels [13–17].

EXPERIMENTAL STUDIES

Rheology of Polymer Gelant

Freshly prepared cross-linkers solutions of HQ and HMTA in brine were added to the pre-determined quantity of polymer solution in fixed ratios as per requirement of the study to form polymer gelants at room temperature. The different gelants were prepared for temperatures of 85°C, 100°C, 110°C and 120°C. Measurement of viscosity of different polymer gelant samples was also determined at room temperature under different shear rates. There was no change in the apparent viscosity due to these measurements, which indicated that the shear rates encountered with this viscometer were not high enough to cause any mechanical damage to the polymer chains. The rheology of the polymer gel is measured using Rheometer (Physica MC1) as shown in Figure 3.

RESULTS AND DISCUSSION

Gelation Kinetics

When gelation starts the gel network starts forming and the viscosity of the gelling solution builds up. Therefore, it is possible to follow gelation reaction with viscosity measurements. Steady shear viscometry is reported in the literature for measuring reaction parameters using Avrami-type equation (eqn. 2), which is defined as:

$$\boldsymbol{x = 1 - exp(-kt^n)} \tag{2}$$

Where,

X = Fractional conversion
K = Reaction rate constant, 1/hour^n
t = Time, hours
n= Reaction order

The fractional conversion 'X' is defined as follows:

$$x = \frac{n(t)-n(0)}{n\,(max)-n(0)} \tag{3}$$

where,

$\eta(t)$ = Viscosity at time t, mPa.s

η_0 = Initial viscosity at time t=0, mPa.s

η max = Maximum viscosity measured by viscometer, mPa.s

The Avrami equation has been applied to this polymer gel system for the first time and the result obtained has been depicted in Figure 4. The data obtained is not in the range of first to second order as expected for many chemical reactions; the reason behind this is the dependence of classical reaction orders on the direct measurement of concentrations of the reactants and/or products. This is not the case in viscosity measurements where the concentration dependence of viscosity is not simple.

Effect of Polymer Concentration on Viscosity of Polymer Gel

Due to cross-linking a lattice like structure is formed which consists of two domains: dilute domain (free draining space) and dense domain (non-draining space). The dense domains are centered on the cross-linked regions where the polymers are bound together and extend outward by the entanglement of the free polymer onto the cross-linked regions. They are much less permeable than the dilute domains.

Even at the lowest polymer concentration, all the cross-linker reacts with polymer to form gel hence an increase in polymer concentration will mainly increase the number of free polymer coils which increases the viscosity of the gelant.

In order to check the effect of polymer concentration, five solutions were prepared in brine of 1 wt% salinity with PAM concentrations of 1, 1.5, 2, 2.5 and 3 wt% with the same concentration of HQ (0.2 wt%) & HMTA (0.3 wt%). Figure 5 depicts the change in viscosity with increase in shear rate of polymer gelants having different polymer concentrations.

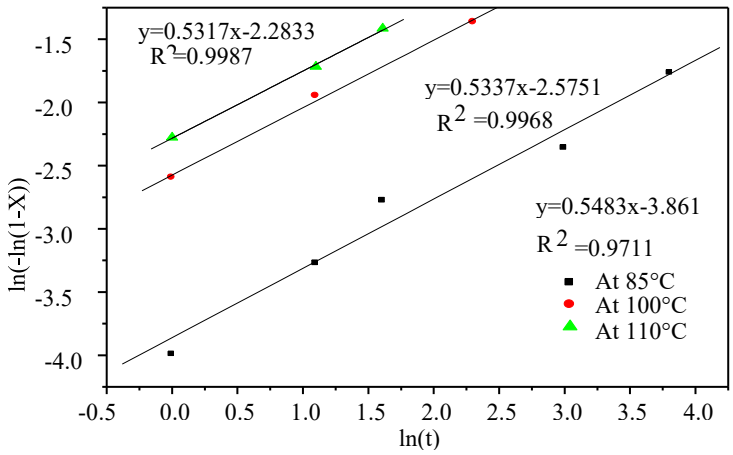

Figure 4. Avrami plot at shear rate of 1.5 rpm.

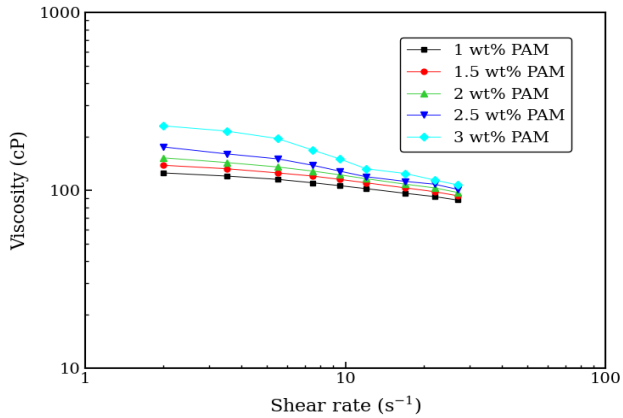

Figure 5. Change in viscosity with shear rate for gelants with different polymer concentration at room temperature (25°C)

Figure 6 illustrates the relation of viscosity with an increase in PAM concentration at different shear rates at room temperature. After gelation at 85°C the gels formed showed greater mechanical strength with PAM concentration of 30,000 ppm and this strength decreased gradually with reduction in PAM concentration. The gels at higher concentration were very stiff in nature and formed in lesser time interval. Syndask's code was used to assign the nature of each gel. This suggests that the intensity of cross-linking is low at low levels of polymer concentration.

Effect of Temperature on Viscosity of Polymer Gel

The polymer gelant comprised of 1 wt% PAM, 0.3 wt% HQ and 0.4 wt% HMTA in 1 wt % brine was kept for gelation at three different temperatures of 85, 100 and 110°C. An observation for the changes in the viscosity of the gelant in each case is shown in Table 1 (a)–(c).

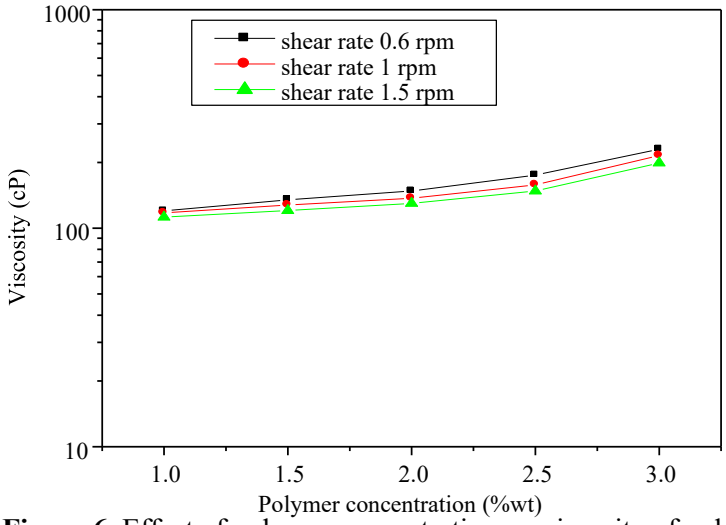

Figure 6. Effect of polymer concentration on viscosity of polymer gelant at 25°C.

Table 1. Change in viscosity of polymer gelant (1 wt% PAM, 0.3 wt% HQ, 0.4 wt% HMTA)

(a). At 85°C						
Shear rate (s^{-1})	**Viscosity at room temperature (cP)**	**Viscosity (cP) of polymer gelant at 85°C**				
		After 1 hr	***After 3 hrs***	***After 5 hrs***	***After 20 hrs***	***After 45 hrs***
3.84	77.4	92.2	96.6	100.5	105.6	133.2
5.76	71.6	77.6	84.8	101.2	101.2	123.2
7.68	70.8	74.4	82.5	97.8	97.8	116.7
9.6	67.9	73.4	80.4	95.8	95.8	112.1
11.52	66.8	69.8	75.8	79.8	87.8	108.4
15.36	62.7	66.2	72.2	75.4	85.7	103.6
19.2	59.6	63.2	68.5	71.2	82.4	98.6

(b). At 100°C				
Shear rate (s^{-1})	**Viscosity at room temperature (cP)**	**Viscosity (cP) of polymer gelant at 100°C**		
		After 1 hr	***After 3 hrs***	***After 10 hrs***
3.84	77.4	99.6	129.2	154.8
5.76	71.6	95.2	115.2	145.6
7.68	70.8	89.1	103.2	128.2
9.6	67.9	85.4	97	116.2
11.52	66.8	83.4	93.6	104.4
15.36	62.7	77.9	86.6	95.3
19.2	59.6	73.4	79.3	90.6

(c). At 110°C				
Shear rate (s^{-1})	**Viscosity at room temperature (cP)**	**Viscosity (cP) of polymer gelant at 110°C**		
		After 1 hr	***After 3 hrs***	***After 5 hrs***
3.84	77.4	115.8	154.2	159.6
5.76	71.6	103.6	125.6	142.4
7.68	70.8	96.6	113.4	136.2
9.6	67.9	92.4	108.5	128.9
11.52	66.8	89.8	101.2	125
15.36	62.7	83.7	94.6	113.4
19.2	59.6	76.9	88.5	104.9

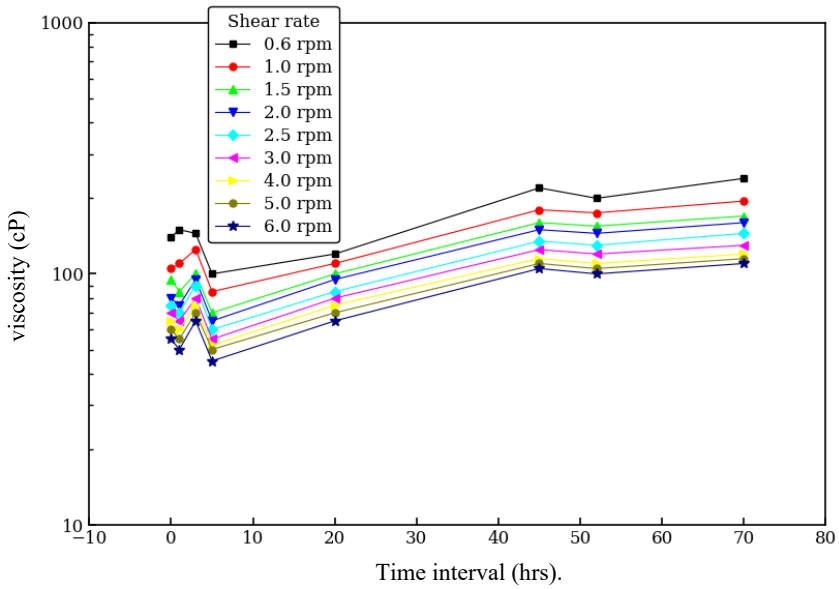

Figure 7. Change in viscosity with time at different shear rates at 85°C (PAM 1 wt%, HQ 0.3 wt%, HMTA 0.4 wt%)

Figure 7 shown illustrates the viscosity evolution as a function of time for gelling solutions containing 1 wt% PAM and 0.35 wt% HQ with 0.35 wt% HMTA prepared in brine with readings taken at different shear rates. The apparent viscosity increases with the increase in temperature at any particular shear rate. The gels formed at higher temperature are more viscous hence has greater ability to shut-off water in reservoirs and enhance oil production.

Before gel formation, the viscosity of the gelling solution is relatively low but after gel formation it is hard to obtain accurate readings. From the Tables 1(a–c) it can be noted that the viscosity of the gel formed at 85°C can't be measured after 50 hours since it surpasses the measurement range of the viscometer. Similarly, readings were not obtained after 10 hours and 5 hours at gelation temperatures 100°C and 110°C respectively. Thus, the mechanical strength of cross-linked polyacrylamide gel increases with the increase in temperature. The elastic modulus increased gradually upon the addition of the crosslinkers until it reached a constant level indicating that the gel formation reaction is over [22–24].

When the measurements were taken it was observed in each case that initially the reading remains constant for some time known as the induction time and then it starts increasing indicating the onset of gelation. Figures 8 and 9 represent a typical tonguing and stiff gel respectively formed after gelation of the polymer gelant.

Figure 8. Tonguing gel.

Figure 9. Stiff gel.

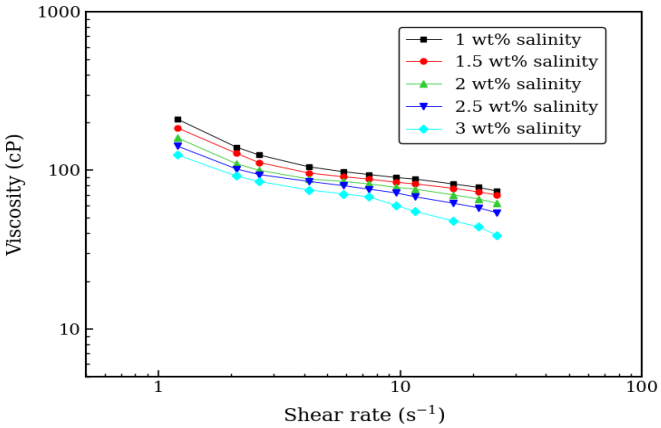

Figure 10. Change in viscosity of polymer gelant (PAM 1 wt%, HQ 0.35 wt%, HMTA0.35 wt%) at 25°C for varying salinities.

Initially with an increase in the cross-linker concentration, the polymer chains begin to connect more frequently. This increases the cross-link density, turning a liquid solution into a semi-solid, three-dimensional network that traps the solvent (usually water). Once the optimal point is reached, the network becomes over-cross-linked. The "bridges" become so numerous and short that the polymer network starts to contract or "shrink." As the network tightens, it literally squeezes out the trapped solvent. This process of the gel contracting and expelling liquid is called syneresis. At very high concentrations, cross-linkers can prevent a gel from forming altogether [25].

Effect of Salinity on Viscosity of Polymer Gel

Gelants were prepared in brines containing various salts concentrations. In this study, NaCl was used to examine the effect of sodium ions on the gelation time of this system. To study the effect of salinity, samples were prepared with a constant polymer content of 1 wt% PAM, 0.35 wt% HQ, and 0.35 wt% HMTA in brine with salinity varying from 10,000 ppm to 30,000 ppm. The viscosity of these solutions was measured at 25°C across different shear rates, as illustrated in Figure 10. It was observed that at any given shear rate, the apparent viscosity diminished as the NaCl concentration increased." The gels formed in brine have higher elastic modulus than those formed in distilled water due to rapid cross-linking reaction. Greater cross-linking occurs in presence of cations present in brine which further increases the gel strength. However, an examination of the samples showed that the gel strength decreased with increase in brine salinity. The gels formed after gelation at different temperatures were not viscous rather they were watery in nature [26–28].

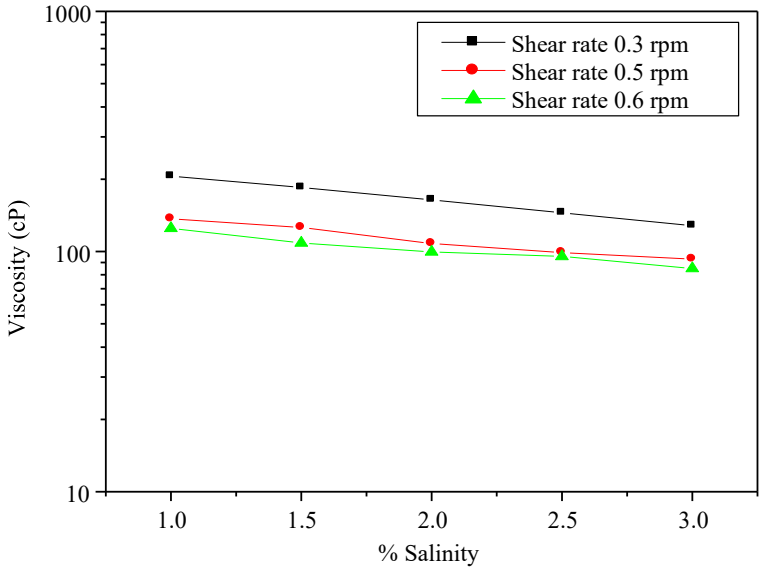

Figure 11. Decrease in viscosity with increase in salinity at room temperature (PAM 1 wt%, HQ 0.35 wt%, HMTA 0.35 wt%).

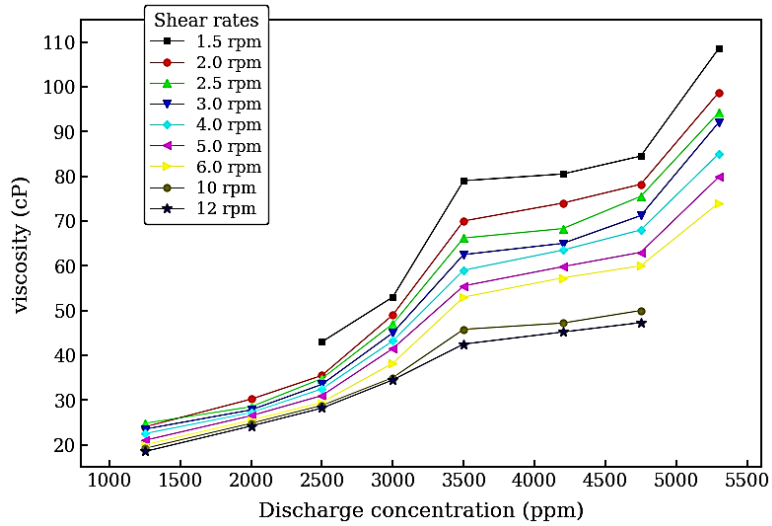

Figure 12. Change in viscosity with variation in cross-linker concentration.

The presence of excess monovalent cations in the gel system screens the negative charges on the polymer backbone, making it difficult for the positive cross-linker ions to access and react with those sites. Consequently, the cross-linking density decreases, leading to a significant reduction in the gel's elastic modulus. The results obtained for the variation in viscosity of polymer gelant comprised of 1 wt% PAM, 0.35 wt% HQ and HMTA each at room temperature are shown graphically in Figure 11. The elasticity of the gelants decreased with increase in salinity.

Effect of Concentration of Cross-Linker on Viscosity of Polymer Gel

The variation of apparent viscosity of different polymer gelants having the same concentration of both the cross-linkers with a constant polymer concentration of 1 wt % at room temperature is shown in the flow curve of Figure 12.

The concentration of HQ and HMTA was in the ratio of 1:1 and varied from 2,000 ppm – 5,000 ppm (i.e. 0.2 wt% - 0.5 wt%) to study the variation in gel strength at room temperature. Thus, greater concentration of the cross- linker results in the increase of gel strength of the polymer gel.

Figures 13 and 14 shown below depicts the increasing trend of viscosity with an increase in concentration of HQ / HMTA during 5 hrs of gelation. This shows that the mechanical strength of the gel formed increases with an increase in concentration of cross-linkers. The lower the cross-linkers concentration, the worse the degrees of cross-linking. The solubility of the polymer gel increases with low cross-linking density; therefore, the degree of water swelling of the gel gets reduced. Hence the gels of weak strength are formed. The shear stress increases with an increase in concentration of cross-linkers since greater cross-linking takes place on the polymer chain. However, high concentrations of the cross-linker can result in gel syneresis, which was noted at temperatures above 120°C which contained cross-linkers concentration above 0.6 wt% each of HQ and HMTA and pH greater than 9.0 [29].

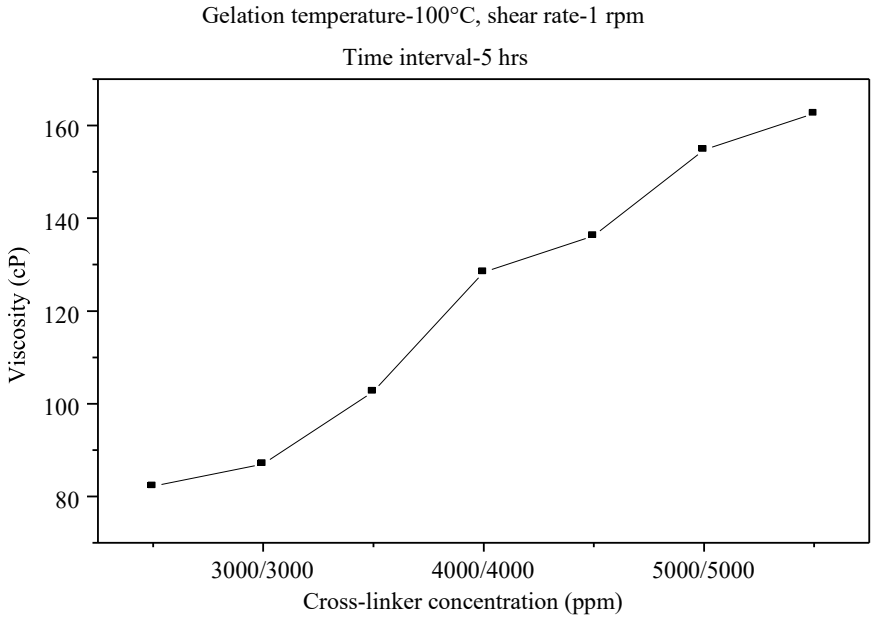

Figure 13. Change in viscosity after 5 hrs of gelation at 100°C for same concentration of HQ/HMTA.

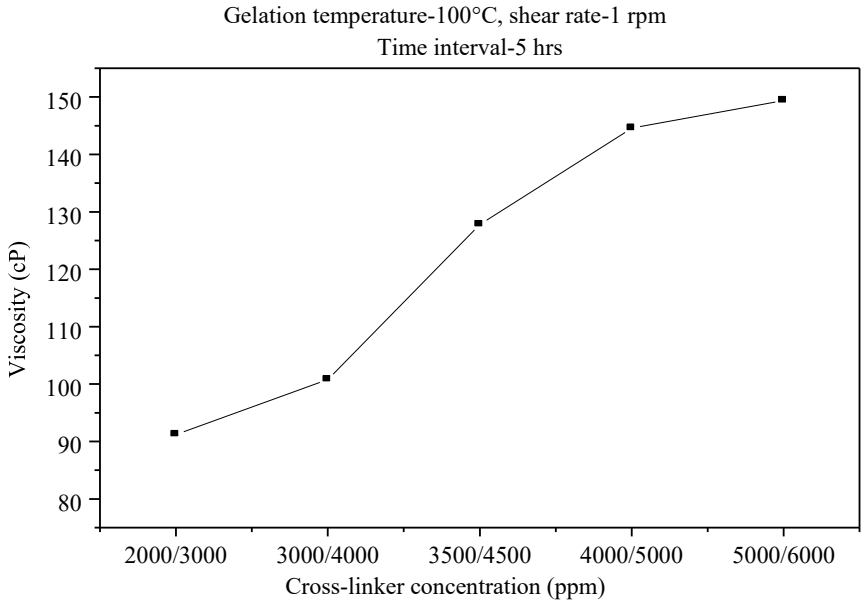

Figure 14. Change in viscosity after 5 hrs of gelation at 100°C for different concentration of HQ/HMTA

Syneresis is an undesirable phenomenon as has already been mentioned in previous sections, especially when treating naturally fractured reservoirs.

Table 2. Gelant with 0.25 wt% HQ and HMTA each in 1 wt% PAM.

S.N.	Shear rate (s^{-1})	Viscosity (cP)
1	5.76	42.4
2	7.68	35.4
3	9.6	35
4	11.52	34.2
5	15.36	32.4
6	19.2	31.8
7	23.04	29.9
8	38.4	28.8
9	46.08	28.4
10	76.8	24.8

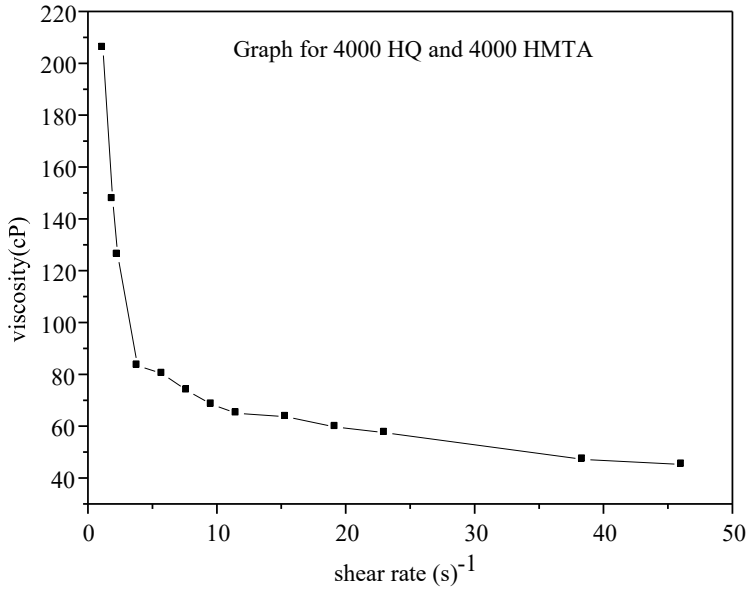

Figure 15. Viscosity of polymer gelant (1 wt% PAM, 0.4 wt% HQ and 0.4 wt% HMTA) with shear rate at room temperature.

Effect of Shear Rate on Viscosity of Polymer Gel

The viscosity of polymer gels at different shear rates reveals the fact that viscosity decreases with the increase in shear rate of the viscometer.

The change in viscosity at room temperature for different polymer gelants having same concentrations of cross-linkers is shown in Table 2.and Figure 15 and 16. Decrease in viscosity may be attributed to the uncoiling and aligning of the polymer chains on exposure to shear flow.

Figure 17 is a flow curve of viscosity with change in shear rate for polymer gelant comprised of 0.2 wt% HQ and 0.3 wt% HMTA at 100°C. Polymer gels at low shear rates are more viscous than those formed at high shear rates. The nature of the graphs shown in Figures 16 and 17 resembles the graph shown in Figure 17 showing the change in viscosity with shear rate of the viscometer.

At high speeds, the long polymer chains no longer stay in tangled balls. Instead, they stretch out and align themselves straight, pointing in the same direction that the liquid is moving. Because the chains are now neatly lined up and offering less resistance, the liquid becomes "thinner" and flows more easily. This drop in thickness is what scientists call shear-thinning behavior.

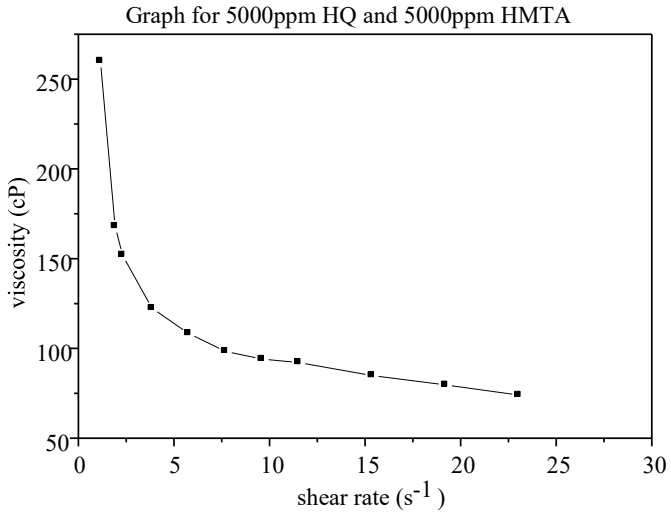

Figure 16. Change of viscosity with shear rate of polymer gelant (1wt% PAM, 0.5 wt% HQ and 0.5 wt% HMTA) at room temperature.

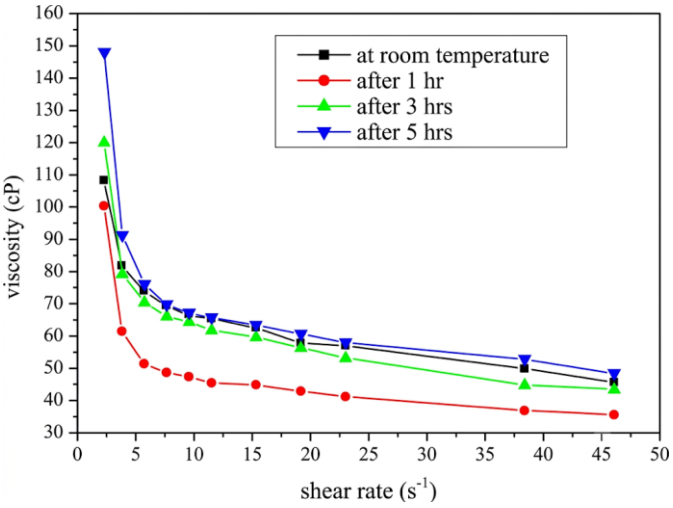

Figure 17. Gelant with 0.2 wt% HQ and 0.3 wt% HMTA in 1 wt% PAM at 100°C.

Effect of Degree of Hydrolysis on Viscosity of Polymer Gel

Degree of hydrolysis (D) is defined as the fraction of the carboxyl residue (n) replacing acrylamide units (m) over the total number of the polymer macromolecule.

$$D = \frac{n}{n+m} \tag{4}$$

Degree of hydrolysis is important and can affect the physical properties of the polymers, e.g. polymer rheological properties, solution viscosity etc. The rate of hydrolysis increases if the pH of the polymer gelant decreases and this is due to the net negative charge on the polymer. Therefore, direct effect of hydroxide ions on amide group is more difficult because of greater electrostatic repulsion [30].

The acrylamide-based polymers are known to hydrolyze under alkaline conditions producing carboxylate groups and ammonia. The hydrolyzed polymer has a large effective diameter which leads

to a higher resistance factor. The viscosity of solution increases with an increase in degree of hydrolysis of polymer solution. The carboxyl groups of polyacrylamide solution bear negative charges thus become an anionic polyelectrolyte. Due to the repulsion forces between ionized carboxyl groups, polymer chain extends and uncoils in the solution which causes an increase in hydrodynamic volume of the polymer chains. In other words, because of the repulsion forces between the negative charges (carboxyl groups), the original coiled molecules of PAM uncoil as hydrolysis proceeds and molecules become more extended thereby increasing viscosity of solutions. Thus, PAM with greater degree of hydrolysis upto a certain limit is more efficient and can yield better results in enhanced oil recovery [31].

CONCLUSION

The polymer gels of PAM/HQ/HMTA have high mechanical strength with a thick rubber like appearance. They can be applied to high temperature reservoirs and are thermally stable. The viscosity of the different gels prepared with different concentrations were measured at pH ranging from 8.0-9.0. Avrami equation was used to obtain the reaction order for the present gel system and the order of the reaction was within 0.5483 at 85°C and 0.5317 at 110°C for 1 wt% PAM, 0.3 wt% HQ and 0.4 wt% HMTA.

The viscosity of gels at higher polymer concentration at room temperature was high compared to that at low polymer concentration. The gels at higher gelation temperatures (varied from 85 – 120°C) were very stiff in nature and attained viscosity rise in lesser time interval. Gawish et al., (1999) studied this polymer gel system but details of the viscosity changes were not reported. The gels formed at higher temperature are more viscous hence has greater ability to shut-off water in reservoirs and enhance oil production.

It was observed that at a given shear rate, the apparent viscosity diminished as sodium chloride concentration increased. The elasticity of the gelants decreased with increase in salinity. Thus, at high salinities of brine the polymer and cross-linkers concentration should be increased to get highly viscous gel for efficient water shut-off in reservoirs. The viscosity of solution increases with an increase in degree of hydrolysis of polymer solution. Thus, PAM with higher degree of hydrolysis is more efficient to yield better results in enhanced oil recovery.

The final gel strength and stability of the present organically cross-linked PAM-based gel can be controlled by adjusting the concentration of cross-linkers, polymer concentration, salinity, gelation temperature, shear rate, and the degree of hydrolysis to obtain high-viscosity gels. Therefore, the gel system investigated in the present study demonstrates excellent potential for effective water shut-off in reservoirs, thereby improving sweep efficiency and enhancing oil recovery.

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