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POLYMER CONCENTRATION, BRINE SALINITY AND TEMPERATURE EFFECTS ON XANTHAN GUM SOLUTIONS RHEOLOGY – A COMPLEMENTARY AND CHECK-UP STUDY

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Abstract. The polymer flooding is an Enhanced Oil Recovery technique used to improve the mobility ratio between the injection fluid and the oil in the reservoir. The solutions of the biopolymer xanthan gum exhibit a pseudoplastic behavior that can increase sweep efficiency. Some of the main factors influencing the flow behavior of injecting polymer solutions through the oil reservoir formation are the salinity of the solvent (the brine salinity used to make the polymer solution) and temperature. Therefore, this paper focuses on the viscosity behavior of the injection polymer solution as a function of polymer concentration, temperature, and salinity of the solvent. The impact of those variables in the xanthan gum solutions was evaluated according to the concentration regimes (dilute and semi-dilute) aiming to understand the performance of the polymeric solutions on reservoir conditions. The rheological measurements in xanthan gum solutions cover a range of polymer concentrations from 4000 ppm to 10 ppm, temperature varying between 25°C to 78°C, and two types of solvents (synthetic seawater and deionized water). The power law is the expression used to fit the data of viscosity as a function of shear rate in the pseudoplastic region for the whole range of temperatures, until 100 ppm. At lower concentrations, a Newtonian behavior is assumed. The analysis of the critical concentration (C^*) and both diluted and semi-diluted conditions allow understanding the solution behavior in some exploitation sceneries.

Keywords: enhanced oil recovery, polymer injection, biopolymers, Xanthan Gum, rheology, regime concentration.

1. INTRODUCTION

The implementation of enhanced oil recovery methods in oil reservoirs is crucial to reach the objectives of increasing hydrocarbon reserves and reduce production cost. These technologies are based on the injection of materials that are not initially naturally present in the reservoir (Russel, 2003). Enhanced Oil Recovery (EOR) techniques such as miscible gas injection and chemical injection (polymer, surfactant) are used to displace additional oil, incremental to that obtained by the secondary waterflood recovery process (Don W. Green, 1998). Among the EOR methods, the injection of a polymer solution in the reservoir is one of the most used techniques to control water production. For this reason, natural polysaccharides biopolymers are attractive for viscosity enhancement due to their low cost, high resistance to mechanical, thermal degradation and susceptibility to biodegradation which makes them environmentally friendly (AlQuraishi & Alsewailem, 2011).

The microorganism *Xanthomas Campestris* produces the biopolymer Xanthan Gum. The primary chain structure (Figure 1) consists of a cellulose-like chain of glucose monomers with β (1-4) glycosidic linkages (Sorbie & Kanneth, 1991). Some non-Newtonian analytical expressions are used to describe the polymeric solutions flooding through porous media, such as xanthan solutions, all of them focused on a pseudoplastic behavior (Don W. Green, 1998). The Xanthan Gum conformation in solutions has a strong dependence on the ionic content of the solvent. Due to electrostatic repulsions between the charges on the side chains, the Xanthan backbone is disordered and highly extended in deionized water. However, it retains some degree of flexibility. When salts are present in the solvent, the helical conformation is stabilized but keeps less degree of flexibility (Wyatt & Liberatore, 2009).

The temperature is one of the aspects that has a significant influence in viscosity of a polymeric solution. This effect has been studied by Abhijit, et al. (2010), Maity & Mahapatro (1988), Ghosh (1997) and Zhou, et al. (2000). All of them used the Arrhenius equation **Eq. (1)**, as proposed by Mayadunne, et al. (1996) to show the relationship between the temperature and the viscosity of the polymeric solution. In **Eq. (1)**, η is the apparent viscosity of the polymer solution, ΔE_η is the viscous activation energy, R is the gas constant and T is the temperature in absolute units. As a consequence, the viscous activation energy is related to the dependence of the viscosity on the temperature of the

polymer solution (Abhijit, et al., 2010). In general terms, the temperature decreases the viscosity of the solution following an exponential behavior.

$$\eta = A \exp\left(\frac{\Delta E_{\eta}}{RT}\right) \dots\dots\dots \text{Equation (1)}$$

Another relevant property is the molecular weight of the polymer. It is possible to determine an approximate value to that using the rheology data. According to Sorbie (1991), the viscosity of a polymer solution is related to the size and extension of the polymer molecule in that particular solution. This way, Billmeyer (1971) and Rodriguez (1983) define other quantities related to the viscosity and necessary to find the molecular weight of the polymer (see Table 1).

Table 1. Quantities related to the viscosity.

Quantity	Symbol	units
Polymer concentration	C	cm ³ /g
Polymer solution viscosity	η	cP
Solvent viscosity	η _s	cP
Relative viscosity	η _r = $\frac{\eta}{\eta_s}$	Dimensionless
Specific viscosity	η _{sp} = η _r - 1	Dimensionless
Reduced viscosity	η _R = $\frac{\eta_{sp}}{C}$	cm ³ /g
Inherent viscosity	η _I = $\frac{\ln \eta_r}{C}$	cm ³ /g
Intrinsic viscosity	[η] = $\lim_{C \rightarrow 0} \eta_I$	cm ³ /g

The relation between the intrinsic viscosity [η] and the molecular weight (Mw) is given by **Eq. (2)**, which corresponds to Mark-Houwink expression. In **Eq. (2)**, the intrinsic viscosity corresponds to the extrapolation of the inherent viscosity when the polymer concentration tends to zero. Finally, the constants K' and a are representative for each polymer in a specific solvent at the temperature of the rheological test.

$$[\eta] = K' Mw^a \dots\dots\dots \text{Equation (2)}$$

Analytical models can represent the flow behavior of xanthan gum solutions for apparent viscosity (η) as a function of shear rate (γ̇) or shear stress. Some studies apply models such as Eyring (Kincaid, et al., 1941), Ellis (Bird, et al., 1960 and Reiner, 1960), Carreau (Carreau, 1972 and Chauveteau, 1981) and Cross Model (Cross, 1965). The Power Law model, proposed by Bird et al. in 1960, is used to fit the pseudoplastic region and is given by **Eq. (3)**.

$$\eta = k \dot{\gamma}^{n-1} \dots\dots\dots \text{Equation (3)}$$

Where k is the consistency index (mPa s), and n is the behavior index, which typically ranges from 0.4 to 1.0 (Sorbie & Kanneth, 1991) for pseudoplastic fluids.

This paper aims to study the effects on the rheological behavior of the xanthan gum solutions caused by the change on the type of solvent from deionized water to synthetic seawater (30,099 ppm) at temperature levels ranging from 25 °C to 78°C, and polymer concentrations ranging from 4,000 ppm to 10 ppm. The performed study complements and serves as a check-up of a previous work developed by Veiga & Moreno (2019). The performed characterization is based on the concept of overlap concentration (C*). Some authors define this concept in different ways. According to Graessley (1980), C* refers to the polymer content at which the average spacing between the polymer molecules is twice the radius of gyration at zero concentration. In this way, the thickening mechanism changes at levels higher than (C*), where the curvature of the flow curves around the individual polymer coil is responsible for the increase in viscosity (Vadodaria & English, 2016). In contrast to that, at concentrations lower than C*, the network composed of overlapping coils resists the flow (Vadodaria & English, 2016).

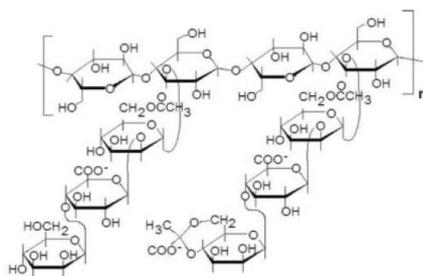


Figure 1. Structure of Xanthan Gum. (Jansson, et al., 1975)

2. EXPERIMENTAL

2.1 Materials

The polymeric solutions were prepared using Xanthan Gum from *Xanthomonas Campestris* supplied by SIGMA® Life Science without further purification. Deionized water with a conductivity of 0.05 $\mu\text{S}/\text{cm}$ and the salts shown in Table 2 were used to make the synthetic seawater (SSW) with total dissolved solids (TDS) of 30,099 ppm. SIGMA-ALDRICH® supplied all salts.

Table 2. Synthetic seawater composition.

Salt	Concentration (mg/l)
KCl	749.3
CaCl ₂ .2H ₂ O	726.3
MgCl ₂ .6H ₂ O	1271.3
SrCl ₂ .6H ₂ O	5.2
BaCl ₂ .2H ₂ O	2.0
FeCl ₂ .4H ₂ O	2.0
NaBr	82.4
Na ₂ SO ₄	57.7
NaCl	28252.5

2.2 Solutions preparation

In the case of enhanced oil recovery applications, the American Petroleum Institute recommends some practices to the laboratory preparation of polymer solutions from polysaccharide products (see norm API-RP 63 American Petroleum Institute, 1990). The recommended blender is a Waring® Commercial Blender™ Model 7011 S, or equivalent, fitted with a 40-oz glass or steel container. According to the norm, the concentration of the stock solution is 5,000 ppm. The mixing process consists of sifting dry Xanthan Gum in the make up water, while stirring at high speed with rheostat at approximately 40% (44-45 volts) during two minutes; after increase the velocity to 60% of the power during three minutes, and finally during two minutes mixing at 80 % of the stirrer power. This methodology was used in the work development by Veiga & Moreno (2019) to hydrated the powder of Xanthan Gum. However, according to the API, this procedure may or may not be applied to all dry based polysaccharide products, and the manufactures could recommend mixing processes.

Aiming to have full hydration of the stock solution of Xanthan Gum, a study was carried out to test some modifications to the API-RP 63 according to previous works. Different methodologies were studied to evaluate the rheological properties of the solutions due to alterations in the following variables: Temperature (25 °C and 80 °C), blender type (magnetic and impeller) and solvent (deionized water). Obtained the best results by combining the methodologies proposed by Dakhil et al. (2019) and Reinoso et al. (2018). Firstly, the xanthan gum powder was added slowly (180 minutes) to deionized water at 25°C, stirred in a magnetic mixer at high frequency, and after the mixing process, the solution was left still during 24 hours at room temperature to reach the full hydration of the polymer. Then, the stock solution was stored at 8°C, and the dilutions were done along 1 to 14 days after the stock solution preparation.

Figure 2.a shows the validation of the protocol that presented the best rheological behavior compared to the results obtained by Whitcomb & Macosko, 1978, Couvelier & Launay, 1986 and Papagiannopoulos, et al., 2016 at 2000 ppm for xanthan gum solutions. Additionally, time degradation was studied at one, seven, and fourteen days after the preparation. The solution did not show significative degradation during this period (Figure 2.b.).

Another essential difference from the API-RP 63 relates to the storage the stock solution of Xanthan Gum at 8 °C to delay the biological degradation. Table 3 shows a parallel between the API norm and the methodology used in this work to obtain a stock solution of Xanthan Gum.

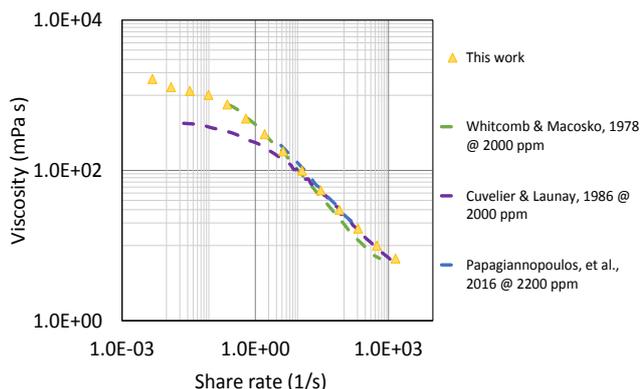


Figure 2.a. Methodology validation.

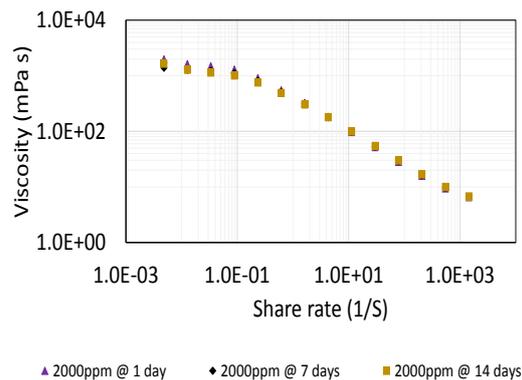


Figure 2.b. Time degradation.

The brine stock solution was prepared considering TDS of 90297 ppm or three times the original synthetic seawater (SSW) concentration. Firstly, were added the dry base salts and the deionized water in a glass baker at room temperature. Then, we use an ultrasonic agitation for some minutes to improve the dilution process. The solution was storage at the same conditions of the polymer stock (8 °C).

Dilutions were done using two types of solvent, deionized water, and brine stock solution. The samples were prepared at concentrations of Xanthan Gum of 4000, 2500, 1000, 500, 250, 100, 50, 25, and 10 ppm. In the first case, dilutions were salt free, and in the second one, we used SSW as a solvent, i. e., the concentration of salts was maintained constant at 30,099 ppm.

Table 3. Stock preparation methodologies.

	API-RP 63	This work
Stock concentration	5000 ppm	4000 ppm
Solvent	Brine	Deionized water
Stirrer	Waring® Commercial Blendor™	Magnetic
Sprinkle time	7 minutes	180 minutes
Mixing time	2 – 3 hours	24 hours
Mixing temperature	25 °C	25 °C
Storage temperature	25 °C	8 °C

2.3 Rheology

The rheological characterization was carried out using a rheometer HAAKE MARS III equipped with a coaxial cylinder geometry Z41 Ti-B08031 (41.35 mm inner diameter, 43.25 mm outer diameter and 55 mm length). The temperature was controlled by a modular temperature controller MARS (TM-LI-C). Temperature stabilization was reached after approximately one hour of water circulation through the equipment. In each case, the stabilized temperature was registered and corresponded to 25 °C ± 0.1 °C, 60 °C ± 0.1 °C, 69 °C ± 0.1 °C and 78 °C ± 0.1 °C, respectively. The apparent viscosity curve against shear rate for each case was collected using a CR (control rate) mode in a share rate range from 0.0001 s⁻¹ to 10000 s⁻¹, with 30 seconds of acquisition for each of the 20 points for the whole curve. The pressure of the cell corresponded to the atmospheric conditions.

3. RESULTS AND DISCUSSION

3.1 Deionized water solvent

The flow curves data were taken covering a range of polymer concentration from 4000 ppm (stock solution) to 10 ppm at 25°C and 60°C. Xanthan Gum solutions exhibited a Newtonian plateau with a high viscosity value at low share rates. After that, a pseudoplastic behavior took place. This behavior is typical of the polymeric solutions, where the viscosity is a function of the shear rate. Below 50 ppm, the xanthan gum solutions exhibited only a Newtonian plateau (Figure 3). At 25°C, significant pseudoplastic behavior could be observed, because xanthan gum diluted in deionized water adopts a partial self-associated conformation due to polymer entanglements and hydrogen bonds (Rochefort & Middleman, 1987 and Ross-Murphy, 1995). On the other hand, xanthan gum becomes less viscous and less

pseudoplastic when the temperature increases from 25°C (figure 3.a) to 60°C (figure 3.b). That is a consequence of the beginning of the transition from a partially ordered random broken helix conformation to disordered random coil conformation (Pelletier et al., 2001 and Reinoso, et al., 2018).

In this work, the Power Law model, Eq. (3), was used to fit the rheological behavior of the solution in the pseudoplastic region. According to the expected results, the value of n increases when the polymer concentration of the solution is lower. Besides that, k increases when the Xanthan Gum concentration is higher. Figure 4 shows the values of each constant according to the power law in the whole range of polymer concentration at 25 °C and 60 °C.

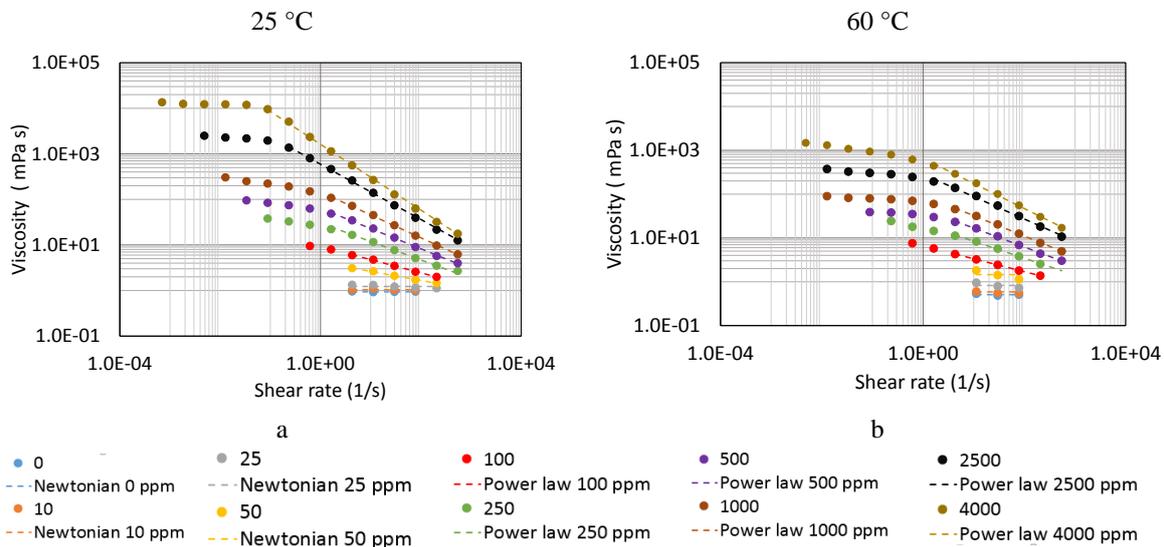


Figure 3. Xanthan Gum solutions viscosity at 25°C and 60°C in deionized water.

In the studies developed by Ouaer & Gareche (2018), the method used to calculate the overlap concentration (C^*) consists of plotting the apparent viscosity versus polymer concentration at specific shear rates in logarithmic scale, (Figure 5). As can be seen, two concentration regions can be identified in each curve, which refers to dilute and semi-dilute regimes. In this way, the interception of the two slopes of apparent viscosity as a function of polymer concentration corresponds to C^* . Table 3 shows the results for both temperatures. One can note that the C^* at 25°C is close to 28 ppm at different values of shear rate. This value is smaller than the overlap concentration at 60°C, which is 31 ppm. According to the empirical observation done by Wyatt & Liberatore (2009), the value of C^* usually occurs at about twice the viscosity of the solvent, in our case for a temperature of 25°C the viscosity of the solvent is 0.94 mPa s and the apparent viscosity at C^* is 1.80 mPa s. At 60 °C, the viscosity of the deionized water was 0.51 mPa s, and the viscosity at C^* was 0.95 mPa s. This way, our values of C^* are in accordance with the literature.

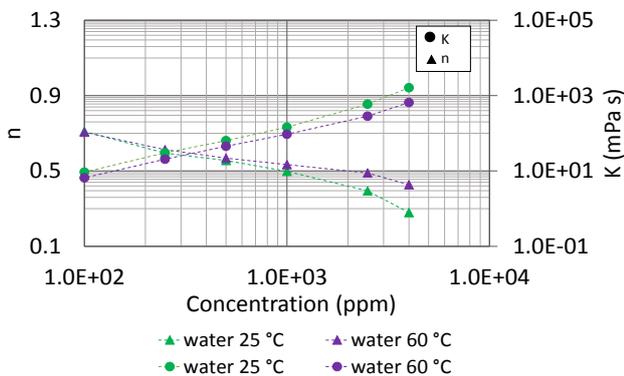


Figure 4. Values of n and k in deionized water

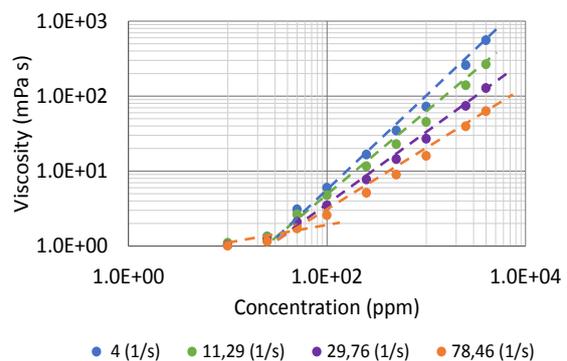


Figure 5. Overlap concentration of Xanthan Gum at 25 °C in deionized water.

Table 4. Overlap concentration at different shear rate values for Xanthan solutions in deionized water and two temperature levels.

Temperature (°C)	25				60		
Shear rate (1/s)	4.28	11.3	29.8	78.5	11.3	29.8	78.5
Critical concentration (ppm)	22.9	25.2	29.1	34.0	28.8	29.7	34.6
C* average	28				31		

3.2 Synthetic seawater solvent

In the second part of the study, the Xanthan Gum dilutions were prepared using synthetic seawater with TDS of 30,099 ppm. The polymer concentration of the samples remained in the range of 4000 ppm to 10 ppm. Each measurement was done at four temperatures (25°C, 60°C, 69°C and 78°C) to have a broader study of the temperature effect on the rheological behavior of the polymeric solutions. According to Wyatt & Liberatore (2009), the salt content in the solvent produces dramatic changes in the polymeric structure of the solution and its rheology. See the flow curves in Figure 6.

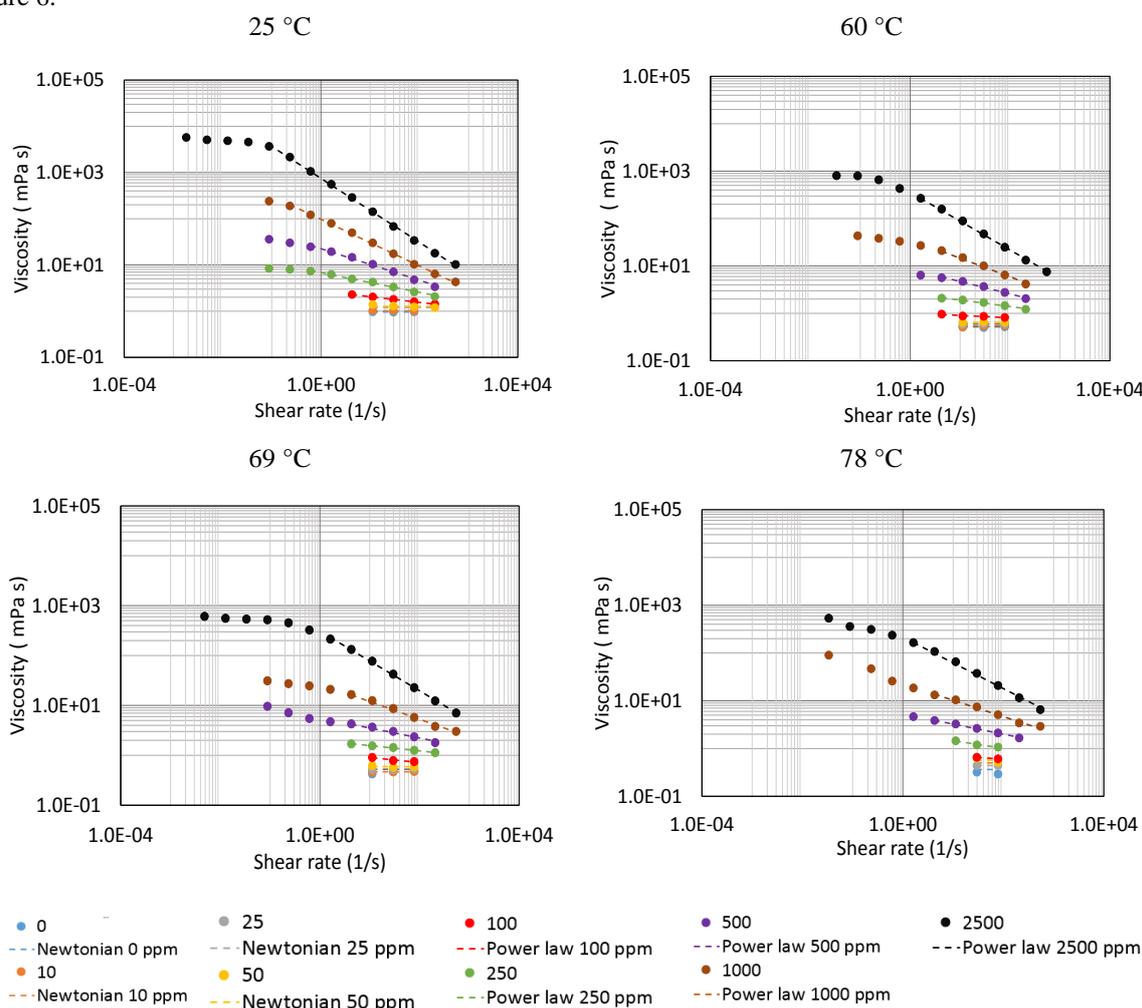


Figure 6. Xanthan Gum solutions viscosity at 25°C, 60°C, 69°C and 78°C in SSW.

One can note a decline in thickness due to the presence of salt by comparing the results obtained for solutions prepared using SSW and deionized water. Wyatt & Liberatore (2009) suggest that the shielding due to the salt ions allows the polymer chain to assume a more compact structure in solution. Because the domains of the polymer chains decrease in size, the number of interactions with neighboring chains also decreases. In other words, for an increase in the viscosity is necessary more polymer chains in the solution to have the same quantity of interactions between the molecules. However, at 2500 ppm of polymer, the solution viscosity in SSW is higher than that in deionized water at both temperatures, 25°C and 60°C. An explanation for that could rely on the possibility of the salts ions shield the polymer chains, increasing the ionic charges among polymer molecules, and, as a consequence, protecting them from the adverse effect caused by temperature increase.

The power law (Bird et al., 1960) was also used to fit the pseudoplastic region obtained at each temperature. That is the same methodology already applied to polymer solutions prepared with deionized water. Figure 6 shows the trend of the consistence index K and behavior exponent n as the function of polymer concentration at each temperature in the SSW solvent. The effect of the salt concentration in the solution is more clearly when analyzing the tendency of the curves in Figures 4 and 8. At 25 °C, the values of n in deionized water are low in comparison with the solutions in SSW. That means the solution loses pseudoplastic properties in the presence of salts. Also, the effect of temperature reduces the solution viscosity in the same proportion when it is free of salt or is prepared with SSW.

Another way of study the effect of the synthetic seawater solvent in the solution is the impacts on the overlap concentration (C^*). The values of each critical concentration were determined using the same methodology of the water solvent (Ouaer & Gareche, 2018) at 25 °C, 60 °C, 69 °C and 78 °C in different shear rates. Therefore, at 25°C, the overlap concentration is close to 77 ppm (Figure 9). Table 5 shows the values of the overlap concentration C^* for each temperature of the study. At 25 °C, the ions in the solution generated an increase of more than three times the value of the critical concentration when the solution was prepared with SSW against that one using only deionized water.

Similarly, at 60 °C, the influence of SSW solvent increased the value of C^* more than two times. In general, when the solvent has ions, a higher concentration of Xanthan Gum is necessary to reach the semi-dilute regime. Besides, the temperature also affects the critical concentration in both scenarios (deionized water and SSW), demanding more polymer into the system for a higher level of temperature to overcome the dilute regimen.

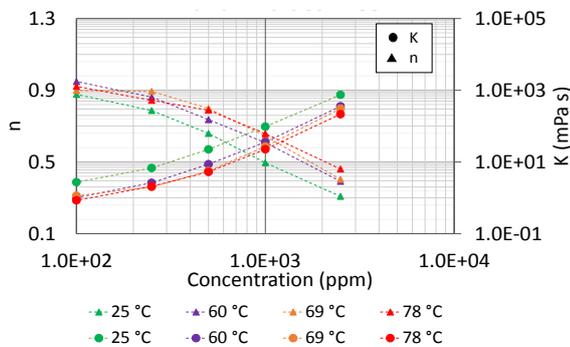


Figure 7. Values of n and k in synthetic seawater.

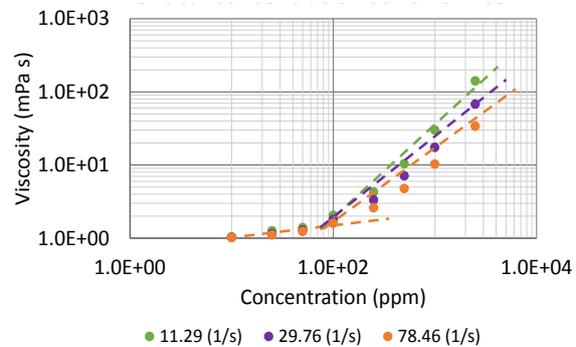


Figure 8. Overlap concentration of Xanthan Gum at 25 °C in SSW.

Table 5. Overlap concentration at different shear rate levels for xanthan solutions in synthetic seawater.

Temperature (°C)	25			60			69			78	
Shear rate (1/s)	11.3	29.8	78.5	11.3	29.8	78.5	11.3	29.8	78.5	29.8	78.5
Critical concentration C^*	78.4	76.0	76.9	84.0	83.0	80.7	74.5	78.0	83.2	108	82.8
Average C^* (ppm)	77			82			79			95	

Figure 10 shows the plot of the reduced and the inherent viscosity as the function of the polymer concentration in the dilute regime. According to **Eq. (2)** the Mark-Houwink equation, (Sorbie & Kanneth, 1991), the extrapolation to zero concentration gives $16589 \text{ cm}^3/\text{g}$. Considering the values of $K' = 1.7 \cdot 10^{-4}$ and $a = 1.14$ for a particular Xanthan sample in 5800 ppm of NaCl brine at 25 °C, we obtained a molecular weight of 10.2×10^6 . Those values for K' and a are typically in the range of $3 - 700 \times 10^{-5}$ and 0.5 to 1, respectively, as reported by Milas et al. (1985).

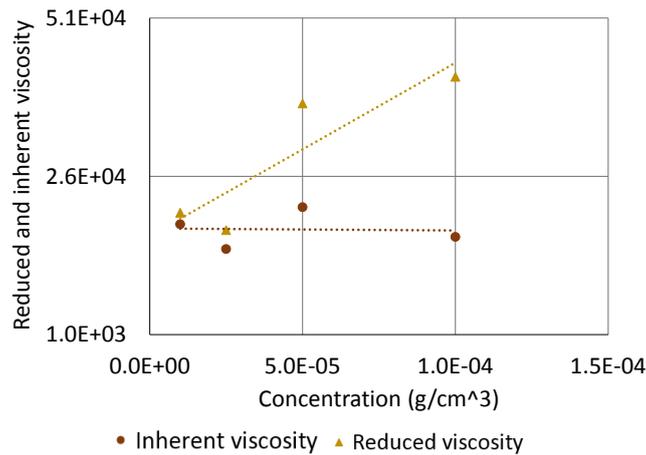


Figure 9. Determination of the intrinsic and inherent viscosity for Xanthan Gum.

Figure 11.a shows the semilogarithmic Arrhenius plot of the apparent viscosity, in Pa*s, versus the reciprocal of the absolute temperature (k). The plotted values correspond to the rheological results at an arbitrary shear rate of 29.76 s^{-1} . The dash lines correspond to the best fit. The viscous activation energies were calculated at different concentrations from zero to 2500 ppm using the slope of each straight line. Table 6 shows the values of the activation energy for each polymer concentration, as a result, the activation energy had not a significative change in those solutions.

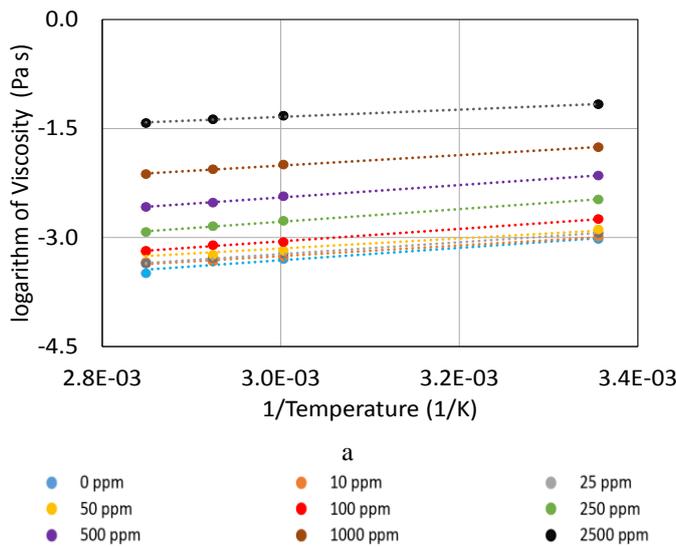


Table 6. Values of activation energy for viscous flow.

Sample concentration (ppm)	Slope	ΔE_{η} (KJ/mol)
10	728.3	13.95
25	813.7	15.58
50	690.5	13.22
100	855.5	16.38
250	867.4	16.61
500	850.6	16.29
1000	716.7	13.72
2500	495.9	9.497

Figure 10. Plot at a constant shear rate of 29.76 s^{-1} using Arrhenius equation, Eq. (1).

Finally, the results of this work are compared with those obtained by Veiga & Moreno (2019). They prepared the Xanthan Gum solutions according to the methodology established in API RP 63 of the American Petroleum Institute (1990) and used the same polymer and a brine solvent of 20,000 ppm of NaCl at 23°C and 73°C . Figure 12 shows the results of both studies at similar temperature levels. The full points refer to the data of this work, and the empty points refer to the data of Veiga & Moreno (2019). The trends are the same, although there were some differences in the preparation methodology, temperature levels, and salinity of the solvents.

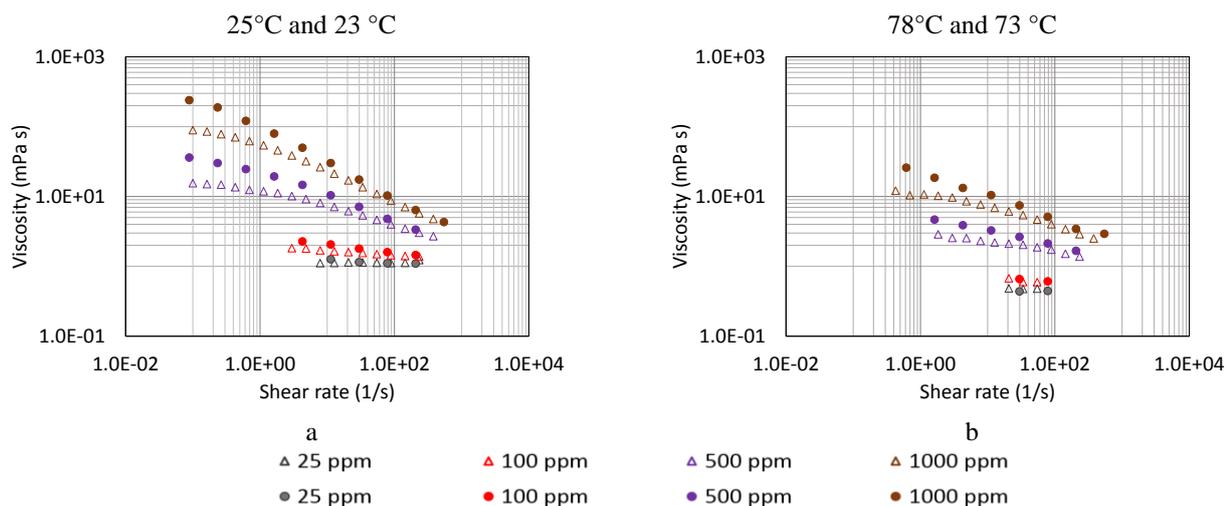


Figure 11. Xanthan Gum solutions checkup viscosity: full points refer to this work, and empty ones refer to Veiga & Moreno (2019).

3.3 Conclusions

The viscosity of the Xanthan Gum solutions was measured using two types of solvent (salt-free and synthetic seawater). The polymer concentrations varied from 4,000 ppm to 10 ppm. The curves of apparent viscosity as a function of the shear rate were adjusted using the power-law model to fit the pseudoplastic region. In the graphs of apparent viscosity versus polymer concentration, two concentration regimes (dilute and semidiluted) were identified and evaluated in each case. The best hydration of the power base Xanthan Gum was accomplished by including some modifications in the methodology established in the American Petroleum Institute (API). In this case, the solvent was deionized water, and the mixing time was 24 hours in a magnetic stirrer at room conditions. The stock solution was storage at 8°C to decrease the degradation, and the dilutions were prepared in 14 days. The temperature affected the behavior of the apparent viscosity reducing the value of the Newtonian plateau in each concentration, and as a consequence, the behavior index characterizing the pseudoplastic region also increased. On the other side, the content of salt in the solvent helped on the thickening role of the polymer solution at 2500 ppm. The presence of salts in lower polymer content decreased the viscosity of the solution; however, at high polymer concentration, the salt ions shield the polymer molecules and protected them from the adverse effect of the temperature increase. The study of the overlap concentration in SSW solvent showed higher values than those measured for the deionized water. This way, the semidiluted regimen was reached at low concentration when the solvent was the deionized water. The influence of temperature in the overlap concentration was studied, and under the conditions of this work, high temperature required high polymer concentrations to reach the semi-diluted regime and to obtain a target viscosity. Two more properties were calculated from the solution in SSW. Using the Mark-Houwink equation (Eq. (2)) and the constants for NaCl solvent, the Xanthan Gum molecular weight was determined as 10.2×10^6 . Finally, the viscous activation energy was calculated using the Arrhenius equation (Eq. (1)) and the results for each concentration did not show great variation. For EOR processes is vital to identify the transition between the dilute and semi-dilute regions, because, under the second regime, the increase in viscosity is faster as the function of polymer concentration. Besides that, the pseudoplastic behavior matters because at high injection rate, the viscosity is lower, helping the injection close to the wellbore, and decreasing the mobility ratio when the flow velocity decreases as the fluid advances through the formation.

4. ACKNOWLEDGMENTS

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