

Rheological Evaluation of HPAM fluids for EOR Applications

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Abstract-- The interest in techniques for enhanced oil recovery has grown due to the need to improve oil productivity reducing water injection and production. Although waterflooding is the most widely used method to recover oil, this process becomes less effective when the mobility ratio is unfavorable and the displacement efficiency is low. Furthermore, great volumes of injected and produced water lead to a premature economic limitation of the production wells and the field itself.

In this context, the polymer flooding appears as a good alternative method to reduce water production. The addition of polymer increases fluids viscosity and, therefore, a more favorable mobility ratio is achieved. This improvement on mobility ratio leads to a better swept efficiency, so, the water breakthrough is postponed and also the oil production is anticipated. On the other hand, there are concerns about the effectiveness of the polymer when formation water with high-salinity and hardness are considered.

The main objective of this work is to analyze the procedures for tailoring a polymer fluid from a given synthetic formation water composition aiming to reach a target viscosity to further displacement experiments. Moreover, the polymer solutions properties, such as, overlapping concentration, molecular weight, salt influence and ionic strength are evaluated under rheological analysis.

As a result, the target viscosity for a specific reservoir condition and polymer were determined. Additionally, it was possible to identify two concentration regions, a dilute and a semi-dilute, and the overlapping concentration (C^*) as a function of the shear rate. It was found that all salts added to the synthetic formation water composition negatively affect the apparent viscosity of the polymer fluids. Also, this effect is more pronounced when salts with divalent ions content are considered.

Index Term-- Polymer solution, rheology, polymer flooding, enhanced oil recovery (EOR)

INTRODUCTION

Recently, techniques to enhance oil recovery (EOR) have received great attention from the oil industry. This activity has been stimulated by oil price, uncertainties of supply, depletion of known reserves and low efficiency associated to traditional recovery methods (RAMIREZ, 1987). Therefore, oil demand will be satisfied not only by new explorations but also by the improvement of the recovery of reservoirs already known.

There are several techniques to increase oil recovery (thermal, chemical, miscible, microbial), and the key to assess the potential use of each technique is based on a clear understanding of the physical phenomena and the mechanisms involved. It is important to highlight that any technique depends on specific conditions. Therefore, its application must be evaluated case by case.

The polymer flooding is a chemical method to enhance oil recovery. Water-soluble polymers are added to the displacing fluid to be injected in order to increase its viscosity. Although residual oil saturation remains essentially the same, the addition of polymer lowers the total water volume needed to achieve this residual saturation due to a better mobility control which improves the sweep efficiency (RAMIREZ, 1987, SORBIE, 1991).

Several polymers have been considered for EOR application. However, the polymers often studied and applied in chemical EOR methods are Xanthan gum (XG) and hydrolyzed polyacrylamide (HPAM) (NEEDHAM & DOE, 1987; LAKE, 1989; SORBIE, 1991, DU & GUAN, 2004).

Xanthan gum is known by its excellent viscosifying properties, even for high-salinity brines. It also presents high resistance to shear degradation, making it easier to handle and inject (NEEDHAM & DOE, 1987). Despite the benefits, the Xanthan gum is less available and also more susceptible to biodegradation, and safety concerns about biocide addition, hamper its use (OSTERLOH & LAW, 1998).

HPAM is the polymer most often used for EOR applications especially because of its availability and relatively low cost comparing to polysaccharides. It is less susceptible to biodegradation, has good viscosifying properties, and it also reduces water permeability unlike the Xanthan gum (LAKE, 1989; OSTERLOH & LAW, 1998; MOREL, *et al.*, 2007; ABIDIN, PUSPASSARI & NUGROHO, 2012). Therefore, the HPAM was chosen as the focus on this research.

POLYACRYLAMIDE

Polyacrylamide is a synthetic water-soluble polymer. It usually consists of copolymers of various ratios of acrylic acid and acrylamide (CAENN & CHILLINGAR, 1996). The most widely used form of the HPAM for recovery methods is the partially hydrolyzed (LAKE, 1989; SORBIE 1991; SHENG, 2011). The HPAM is a polyelectrolyte with negative charges on the carboxylate groups (FIGURE 1), which implies strong interaction between the polymer chains and any cation present in the solvent, especially for higher hydrolysis degrees (NASR-EL-DIN, HAWKINS & GREEN, 1991).

In commercial products, the hydrolysis degree ranges from 15 to 35%. In EOR applications, the general hydrolysis degree is close to 25% (MELO, *et al.*, 2002). Zeynali, Rabii & Baharvand (2004) and Choi (2008) concluded that the higher the hydrolysis level, the higher is the viscosity until values around 30 to 40%, after which the viscosity decreases. According to Sheng (2011), this reduction on viscosity for hydrolysis degree up to 40% is the result of the severe

compression and distortion of the flexible chains of the polymer.

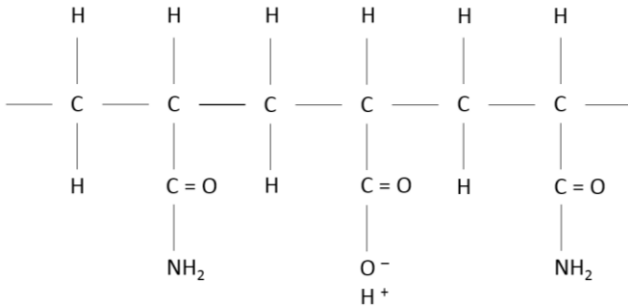


Fig. 1. Molecular structure of a partially hydrolyzed polyacrylamide (LITTMAN, 1988).

Several relations are used to designate the viscosity of a polymer solution (η), such as, polymer solution viscosity at very low shear (η_0) and limiting value at high shear (η_∞), solvent viscosity (η_s), relative viscosity (η_r), specific viscosity (η_{sp}), reduced viscosity (η_R), inherent viscosity (η_I) and intrinsic viscosity [η].

The intrinsic viscosity is the limit of the inherent viscosity when polymer concentration tends to zero. According to Sorbie (1991), the intrinsic viscosity can be used to estimate the molecular weight of the polymer by Mark-Houwink equation (Eq. 1):

$$M_w = ([\eta]/K')^{\frac{1}{a}} \quad (\text{Eq. 1})$$

where K' and a are constants and are dependent upon the polymer, the solvent and the temperature. Their values are typically in the range $3-700 \times 10^{-5}$ and 0.5 to 1.0, respectively.

Salt influence

When ionic compounds like salts are dissolved into water, they dissociate into ions. A measure of the ion concentration in the solution is called ionic strength. The ionic strength of the solution is obtained according to Eq.2:

$$I = \frac{1}{2} \sum m_i z_i^2 \quad (\text{Eq. 2})$$

where m is the molar concentration of ion i , and z is the charge number of that ion.

As a polyelectrolyte, the flexible chains of the HPAM may interact with the ions in solution. In this way, the salinity of the formation water is a great obstacle to polymer flooding application, since these interactions between the salts and the charged polymeric molecules are able to reduce the viscosity of the polymer solutions (SORBIE, 1991; MAIA, BORSALI & BALABAN, 2009; ALQURAIISHI & ALSEWAILM

2012; ABIDIN, PUSPASSARI & NUGROHO, 2012).

Many researchers (MUNGAN, SMITH & THOMPSON, 1966; MUNGAN, 1972; CHANG, 1978; NASR-EL-DIN, HAWKINS & GREEN, 1991; MELO, *et al.*, 2002; MANDAL & OJHA, 2008) investigated the effect of salts on the viscosity of partially hydrolyzed polyacrylamides. They state that the increased salt concentration reduces significantly the viscosity of the polymer solution, especially for low shear rates.

When HPAM is dissolved into water, the groups COO^- of the molecular chain repel each other causing its structure to remain extended. This feature gives the molecular chain a higher hydrodynamic volume, increasing the viscosity of the polymer solution. However, when salts are added to the solution, the negative charges of these side chains are neutralized by the cations of the salt, resulting in a molecular shrinkage and, consequently, decreasing fluids viscosity. (SHENG, 2011).

The influence of the salts on fluid viscosity is also dependent upon the hydrolysis level of the polymer and the cation of the salt itself (SORBIE, 1991; CHANG, 1978, WARD & MARTIN, 1981). Ward & Martin (1981) also showed that the viscosity reduction resulted from the addition of divalent cations is stronger for polymer solutions more concentrated.

Although the monovalent cations, especially sodium (Na^+), are prevalent in the formation water, there is a negligible amount of divalent cations (mainly calcium, Ca^{2+} , and magnesium, Mg^{2+}), especially in formation water from offshore reservoirs. The presence of these divalent ions has a more significant effect on polymer solution properties than monovalent species when they are added in equivalent mass percentage (CHANG, 1978; SORBIE, 1991; SHENG, 2011). This is the result of the stronger binding forces for the divalent ions.

Overlapping concentration

Experimental and theoretical studies concerning the critical overlapping concentration (C^*) have been reported in the literature. In these studies, two concentration regions, a dilute and a semi-dilute, are addressed and the parameters associated with the dimensions of a polymer chain, the viscosity and the molecular weight, were evaluated (PAPANAGOPAULOS, PIERRI, & DONOS, 1998).

The overlapping concentration indicates the region of transition between the dilute and the semi-diluted regimes. In the dilute regime, the macromolecules are separated from each other and behave independently. In the semi-dilute regime, the macromolecules are entangled and thus, frictions are imposed on each other, increasing the viscosity of the fluid. The transition between the regimes is characterized by a change in the shape of the viscosity-concentration curve as shown in Figure 2 (AL HASHMI, *et al.*, 2013).

According to Sorbie (1991), the higher the polymer concentration on the solution, the higher is the interaction

among them and the solvent. As the polymer concentration decreases, the fluid loses its pseudoplastic characteristic, resembling to a Newtonian fluid, once the viscosity of dilute solutions presents negligible changes with shear rate increase.

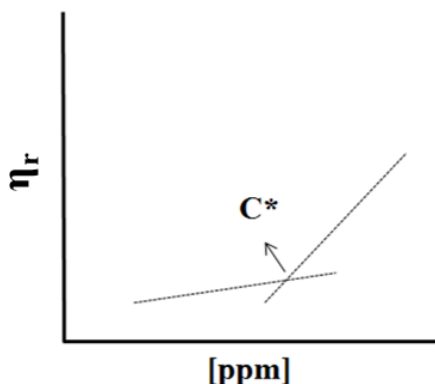


Fig. 2. Critical overlapping concentration, C^* .

Methodology

The method to prepare the polymeric fluids was based on the API-RP-63 except by the fact that, instead of the brine, distilled water was used to dilute the polymer. According to this API, a stock solution containing 5000 ppm of polymer (HPAM) should be first prepared and, from this concentrated solution, dilutions should be made until the desired polymer concentration is reached. It's also required that the solution exhibits a homogeneous aspect, i.e. no insoluble particle (fish eyes) should be present.

To prepare the stock solution, the dried polymer was uniformly sprinkled into distilled water with vigorous stirring within 30 seconds. Then the stirrer speed was reduced in order to avoid the mechanical degradation of the polymer. The homogenization was kept for three hours. After that, the stock solution was stored overnight in a Becker. The diluted solutions were prepared by adding distilled water to the stock solution until it reaches the desired polymer concentration. After water addition, the solution was stirred for 10 minutes using a magnetic stirrer, also in the low speed.

The polymer used in this research was the Flopaam AN110 SH (HPAM) which is an ATBS-based polymer less sensitive to temperature and salinity. The molecular weight of the polymer was provided by the supplier as roughly 8-10 million g/mol with 10% anionicity. The hydrolysis degree was not reported. In order to perform displacement tests, the polymer fluid was tailored for a given synthetic formation water composition and the target fluid viscosity was set as ~ 10 cP for $7,848 \text{ s}^{-1}$ of shear rate.

Rheological apparatus and procedure

The rheological tests were performed in a HAAKE MARS III rheometer, which is a high precision instrument usually used in rheological characterization of Newtonian and non-

Newtonian fluids (Figure 3). In this study, the rheometer was used to evaluate the apparent viscosity versus shear rate of the polymeric fluids. The temperature was set as $23 \text{ }^\circ\text{C}$ and the sensor system used was the double gap (DG41) because this sensor is preferred for low-viscous fluids and/or small sample volumes.



Fig. 3. HAAKE MARS III rheometer.

RESULTS AND DISCUSSIONS

Many tests were performed aiming to evaluate the influence of the polymer concentration and the salt content on the rheological parameters (τ_0 , K , and n) of the fluids. The flow curves and the results are presented as follows.

The stock solution was formulated twice so that the methodology to prepare the polymer fluids could be validated. The flow curves of both fluids, formulated as stock solution, are shown in Figure 4 and 5.

It can be seen that the rheological curves for both fluids are similar and also, their rheological behavior can be described by the Herschel-Bulckley (HB) model (pseudoplastic with yield stress, $\tau = \tau_0 + K\dot{\gamma}^n$) or the Ostwald de Waele (OW) model (pseudoplastic without yield stress, $\tau = K\dot{\gamma}^n$) (MACHADO, 2002). The rheological parameters of each fluid, according to each model, are summarized in Table I.

It can be seen that the model that best fits the behavior of the polymeric fluids is the Herschel-Bulckley. Silveira & Moreno (2013) obtained HPAM fluids with rheological curves described by Ostwald de Waele model, i.e., without yield stress.

The presence of the yield stress may be result of the absence of salts in the stock solution composition. As known, the salt content negatively affects the viscosity of the polymer fluids, and this includes the yield stress, i.e., a lower shear stress is required for the fluid to flow. As the technique to prepare the stock polymer solution presented repeatability, we proceeded to further analyzes.

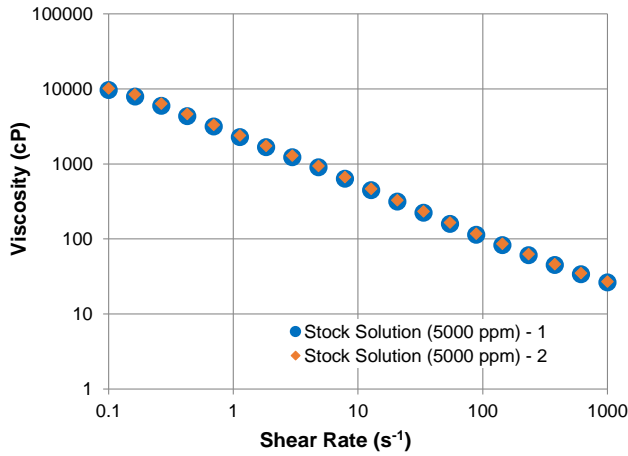


Fig. 4. Viscosity-shear rate of both stock solutions (@23 °C).

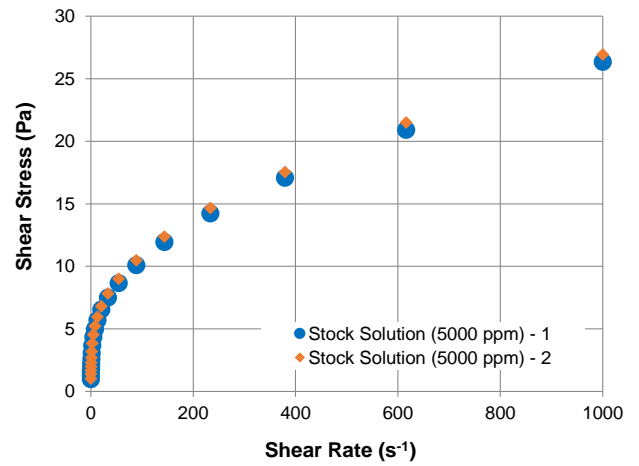


Fig. 5. Shear stress-shear rate of both stock solutions (@23 °C).

Table I
Rheological parameters of the stock solutions

Fluid	τ_0	K	n	R ²	Model
1	-	2.163	0.3548	0.9974	OW
	0.8946	1.582	0.3975	0.9984	HB
2	-	2.299	0.3492	0.9975	OW
	0.9117	1.696	0.3907	0.9984	HB

Dilution of stock solution

The dilute solutions were also prepared according to the API-RP-63 and, again, with no salt-content. The stock solution was diluted until the desired concentration of polymer was achieved. The viscosity versus shear-rate of the dilute solutions is shown in Figure 6.

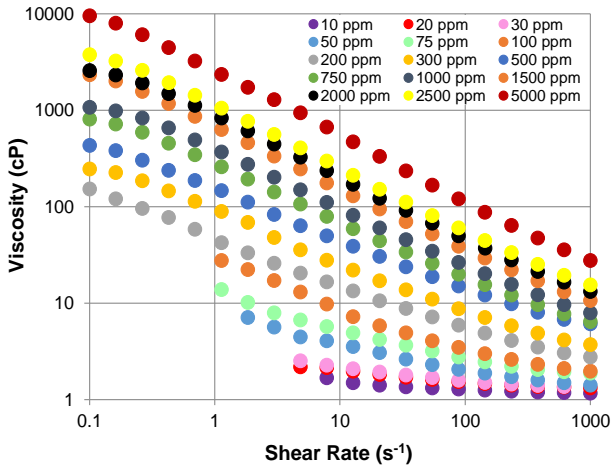


Fig. 6. Viscosity vs. shear rate of the dilute solutions @23 °C.

viscosity decreases with increasing the shear rate. This is a consequence of the uncoiling and aligning of polymer chains when exposed to shear flow (NASR-EL-DIN, HAWKINS & GREEN, 1991).

When the viscosity versus polymer concentration is plotted for a specific shear rate, it is possible to identify the two concentration regions, the dilute and the semi-dilute. In Figure 7 the dilute region is represented by the dashed lines and the semi-dilute region is represented by the continuous lines.

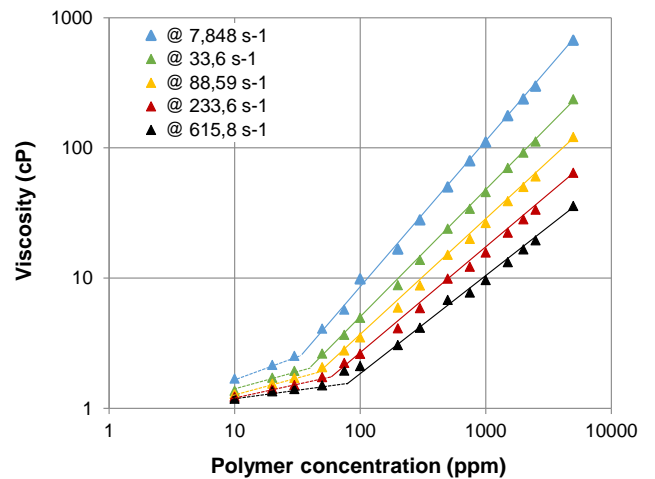


Fig. 7. Critical overlapping concentration (C*) for several shear rates @23°C.

As expected, the higher the polymer concentration, the higher is the viscosity of the solution, for all shear rates. It can also be seen that, for a fixed polymer concentration, the apparent

The overlapping concentration is the result of the intersection of both lines. They were plotted by potential trend lines ($y = ax^b$) where x is the polymer concentration and y is the apparent viscosity. The results obtained for different shear rates are seen in Table II.

It can be seen that the critical overlapping concentration (C^*) is a function of the shear rate. The higher the shear rate, the higher the overlapping concentration is, and, as expected, the correspondent apparent viscosity decreases with the increase in the shear rate.

Table II
Potential trend lines parameters ($\eta = a.c^b$)

Shear Rate	Trend line 1 (Dashed line)		Trend line 2 (Continuous line)		C^* (ppm)	Apparent Viscosity η (cP)
	a	b	a	b		
@ 7.848 s ⁻¹	0.728	0.365	0.054	1.104	33.99	2.639
@ 33.60 s ⁻¹	0.649	0.324	0.049	0.993	48.10	2.271
@ 88.59 s ⁻¹	0.720	0.254	0.051	0.909	57.84	2.018
@ 233.6 s ⁻¹	0.795	0.190	0.057	0.819	67.01	1.770
@ 615.8 s ⁻¹	0.829	0.159	0.071	0.720	80.01	1.660

Estimating the molecular weight of the polymer

In order to estimate the molecular weight of the polymer used, the laboratory data were plotted in Figure 8 as inherent viscosity versus polymer concentration. The graph data are taken from the dilute concentration region at shear rate of 7,848 s⁻¹.

Extrapolating the polymer concentration to zero, the resulting intrinsic viscosity is $[\eta] = 61,242 \text{ cm}^3/\text{g}$. According to Eq. 1.1 and adjusting the K' and a coefficients for higher values presented in the literature (700×10^{-5} and 1,0, respectively) the minimum molecular weight estimated for the HPAM used was 8.75 million g/mol, which is included in the molecular weight range provided by the supplier.

Salt influence on polymer viscosity

In order to evaluate the effect of the type of salt in the apparent viscosity of the polymer fluid, each salt of a synthetic formation water composition was individually evaluated. It has been reported polymer concentrations, at field applications, ranging from 500 to 2500 ppm (ALUHWAL, 2008; XIAODONG, & JIAN, 2013). To perform this study, a

polymer concentration of 1250 ppm was chosen. The type of salt and its correspondent concentration for the formation water composition are given on Table III

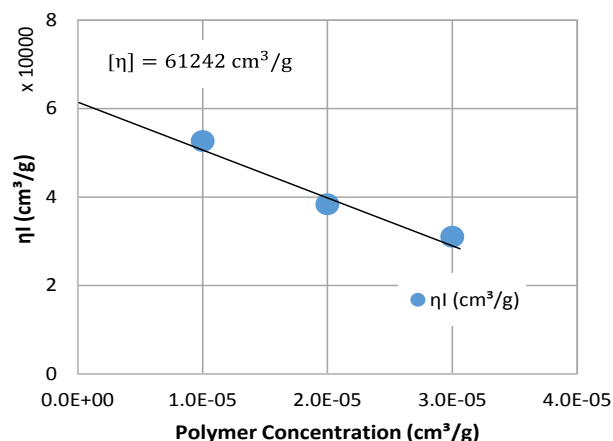


Fig. 8. Determination of the intrinsic viscosity for an HPAM sample.

Table III
Concentration of each salt for a given synthetic formation water composition.

Solutions (HPAM + Salt)	Salt	Concentration	Ionic Strength of Individual Solution	Ionic Strength (Proportion to NaCl)
		mg/L	mol/L	mol/L
1	NaHCO ₃	234	0.003	0.18%
2	KCl	4974	0.067	4.20%
3	MgCl ₂ . 6 H ₂ O	6264	0.092	5.82%
4	CaCl ₂ . 2 H ₂ O	10208	0.208	13.11%
5	SrCl ₂ . 6 H ₂ O	1737	0.020	1.23%
6	BaCl ₂ . 2 H ₂ O	196	0.002	0.15%
7	NaCl	92829	1.588	100%
	Total	116442	1.98	

The ionic strength of each salt and its proportion related to the NaCl are also included for further comparison among them. The results are seen in Figure 9.

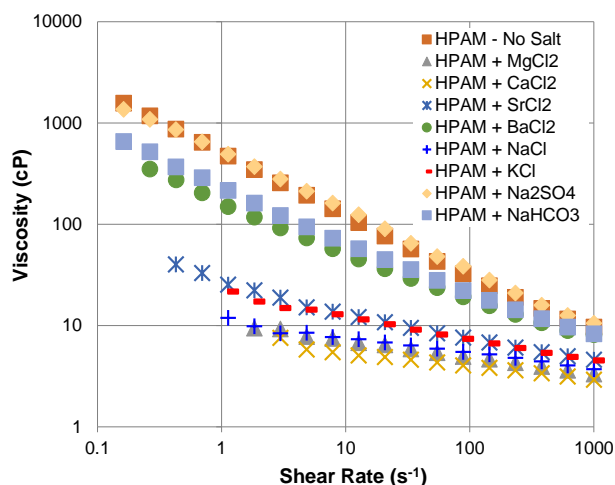


Fig. 9. Influence of each salt addition on the original viscosity of the fluid (1250 ppm HPAM).

It can be seen that the addition of any salt negatively affects the apparent viscosity of the fluid with no salt content. From the flow curves presented in Figure 9 it is also possible to see the divalent ion effect. Even the ionic strength of Solution 3 (HPAM + MgCl₂·6 H₂O) being around 17 times lower than the ionic strength of Solution 7 (HPAM + NaCl), both solutions presented similar effect of reducing the original viscosity of the fluid with no salt content.

Similar comparison can be made between Solution 2 (HPAM + KCl) and Solution 5 (HPAM + SrCl₂·6 H₂O). In both comparative cases, the ionic strength of the solutions with divalent ions content were lower, however, the viscosity reduction was similar. It confirms that the effect of viscosity reducing is more pronounced for divalent ions than for monovalent ones.

After the study of each salt individually, a polymer solution with 1250 ppm of polymer concentration (HPAM) containing all salts was prepared. The flow curve of the whole fluid composition is presented on Figure 10.

It can be seen that when all salts of the formation water composition were added to the polymer solution, the target fluid viscosity, initially set as approximately 10 cP for shear rate of 7,848 s⁻¹, was not reached (7.39 cP). Thus, there was a need to increase the polymer concentration into fluid composition and therefore, the next polymer concentration was chosen as 1500 ppm.

In Figure 10 is also presented the flow curve of this new whole fluid composition. It can be seen that the target viscosity was achieved with this new polymer concentration (10.09 cP), therefore, from now on this will

be the polymer concentration under study for further applications in core flooding experiments for EOR tests.

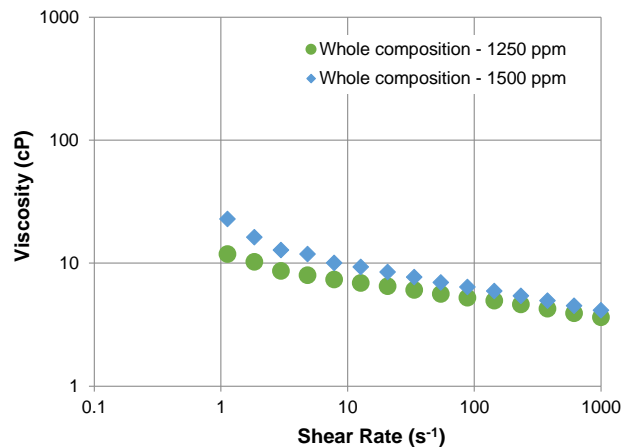


Fig. 10. Viscosity of the whole fluid composition

CONCLUSIONS

It could be seen that the method of preparing polymeric fluids based on API-RP-63 presented repeatability. The model that best fits the rheological behavior of the concentrated solution was the Herschel-Bulckley, which may have been the result of the absence of salts on the stock solution preparation.

Two concentrations regions, a dilute and a semi-dilute, were addressed for the polymer under study. It was also observed that the transition area, which is the intersection of both regions and represents the overlapping concentration (C*), is shear-rate dependent.

Finally, the effect of adding different salts of a given synthetic formation water was evaluated and it was concluded that all salts under analysis reduced the original apparent viscosity of the polymer solution with no salt content. Additionally, concerning the ionic strength of the solution with each salt individually added, it was possible to see that this effect was more pronounced for the salts containing divalent ions.

NOMENCLATURE

τ	Shear stress (Pa)
τ_0	Yield Point Stress
K	Flow consistency index
n	Flow behavior exponent
$\dot{\gamma}$	Shear rate (s ⁻¹)
K'	constant
a	constant
Mw	Molecular weight (g/mol)
η	Apparent viscosity (cP)
$[\eta]$	Intrinsic viscosity (cm ³ /g)
m	Molar concentration (mol/L)
R ²	coefficient of determination of statistical models
z	Charge number of the ion

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