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CHARACTERIZATION AND SELECTION OF POLYMERS FOR FUTURE RESEARCH ON ENHANCED OIL RECOVERY

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Abstract. Polymer flooding has been applied for petroleum recovery and the main results of this method are the effective increasing in oil production and the reduction of water circulation The objective of this work is to present a methodology for pre-selecting a polymer to be used in future research on enhanced oil recovery (EOR) by injecting polymer solution. A reservoir was selected and characterized. Seven samples of commercial partially hydrolyzed polyacrylamide (PHPA) were also selected and characterized. Polymer solutions were prepared and characterized in terms of filterability, viscosity, stability (under reservoir conditions) and mechanical degradation. Polymer-reservoir interaction was also investigated. The results showed that it is very useful to establish a methodology to pre-select the more suitable polymer for fluid injection operations in oil field. Besides, for the conditions used in this study, the best polymer presents hydrolysis degree of 30%, molar mass of $5 \cdot 10^6$ g /mol and intrinsic viscosity of 10 ml/g.

Key words: petroleum recovery, polymeric solutions, polymeric characterization, rock characterization, rheological behavior, filterability tests, static adsorption test.

1. Introduction

Polymer flooding comes forward as a sustainable method for petroleum recovery [1]. Its principle is based on the injection of a bank of polymeric solution in the injection well, which travels through the reservoir toward the production wells [1]. These results are consequences of the physico-chemical interactions of the polymer solution, the reservoir-rock system and the fluids [2-5]. In such operations, the polymer solution should travel the rock formation pushing the oil toward the production wells. In order to achieve this technical function, viscosity of the displacing phase (polymer solution) must be close to the viscosity of the displaced phase, improving thus the sweeping efficiency and, consequently, the amount of oil recovery. In spite of the principle being relatively simple, the operation success basically depends on two critical points: i) the selection of the polymer appropriating to the reservoir and ii) the design of the polymeric solution to be injected. The choice of the polymer is usually made starting from the characteristics of the polymer molecule and of the reservoir system and it is confirmed by specific laboratory tests for this purpose [6-10]. The present paper is focused on the elements required for a proper polymer selection.

2. Experimental

2.1. Materials

Seven commercial samples of polyacrylamide with different characteristics were used. The samples are named: Flopaan 2030 S, Flopaan 2130 S, Flopaan 2230 S, Flopaan 2435 S, Flopaan 2630 S, Flopaan 3230 S and Flopaan 0430 S. All of them were gently supplied by Floerger, France [11], as a powder with 99 % of purity. Other solvents and chemical products were also used. Glacial acetic acid P.A., sodium hypochlorite (5 % of purity), acetone P.A., toluene P.A. and methanol P.A. were supplied by Merck. Brazilian Petroleum Company – PETROBRAS has gently supplied the rock samples of a Brazilian petroleum reservoir.

2.2. Methods

The methodology includes the selection/ characterization of the polymer material and polymeric solutions and the selection of the target reservoir. The characteristics of the reservoir were compared with well-established literature criteria for a good reservoir for polymer flooding and the characteristics of the target field.

2.2.1. Polymer characteristics

Hydrolysis degree

Since the methodology to determine hydrolysis degree (%) by titration introduces a very high uncertainty, in this paper it was calculated based on the elementary analysis of nitrogen by using the Elementary Analyzer C-H-N-2400, Perkin Elmer. The hydrolysis degree was calculated by the ratio between the percentage of nitrogen measured by the elementary analysis and the theoretical value calculated from the original formula for hydrolysis degree equal to zero.

Water content

The water content (%) was determined by liofilization. A mass of 5–10 g of the polymer was accurately weighed. The sample was being frozen for 5–10 minutes using a dry ice bath and acetone. Then, it was heated to 233.15 K and pressured to 10^{-1} mbar. The weight of so treated sample was determined. The entire process was repeated until two consecutive weight measures differed no more than 5 %. The amount of water was calculated based on the weights determined before and after the treating.

Thermal stability

The polymers thermal stabilities under storage conditions were determined at a heating rate of 278.15 K/min, from 303 K to 423.15 K, under nitrogen, using a Thermogravimetric Analyzer Perkin Elmer – TGA 7.

Molar mass

The molar masses of the polymer samples were determined by intrinsic viscosity, following API Standard [6]. The viscometric average molar mass was calculated using the Mark-Houwink-Sakurada equation. The values of K and a used for all calculations were $6.75 \cdot 10^{-5}$ and 0.830, respectively, corresponding to the constants for polyacrylamide 30 % hydrolysis degree and water 3 % salinity [6], which is close to the salinity of the target reservoir.

2.2.2. Polymer solutions

Preparation of polymer aqueous dispersions

All the solutions were prepared following the American Petroleum Institute – API Standard (Recommended practices for evaluation of polymers used in enhanced oil recovery operations, 1990), starting from a stored solution with 5000 ppm, taking into account the recommendations regarding agitation, speed, total time of solubility, *etc*.

Filterability tests

The quality or homogeneity of all used solutions was verified through the filterability test (API Standard [6]). In this procedure the samples are submitted to filtration in a 10 µm steel filter with a controlled flow rate and monitored pressure. The increase of pressure indicates the presence of heterogeneities, known as "fish eyes", in the solution. When an increasing in pressure was observed, the solution was discarded, and a new solution was prepared accordingly to the standard. This procedure is mainly important before the polymer-reservoir interaction tests.

Determination of pH

Using a pH Analyzer Thermo Orion 290 the pH values of all polymer solutions were obtained.

Rheological behavior

Curves of viscosity against concentration were obtained for prepared solutions using all polymers. Since it is indicated by API Standard [6] and is used for field applications, thus a Brookfield LVT/ULA viscometer was used. The analyses were carried out at 298.15 and 323.15 K. The shear rate was 7.3 s^{-1} since it is close to the shear rate applied to the fluid in the reservoir [3, 4].

Stability under reservoir conditions

A stability test of polymer solutions was performed under the target reservoir conditions, that is, measuring viscosity of polymer dissolved in injection water, at reservoir temperature (323.15 K) and shear rate of 7.3 s^{-1}, using a Haake RheoStress RS 600 rheometer, sensor DG 41.

Mechanical degradation

Mechanical degradation (shearing) of the polymer solutions was determined according to the API Standard [6]. The polymer solutions flowed (high flow rate) under pressure through a capillary tube at different velocities. The shear rate was then calculated as a function of capillary diameter and flow rate – shear rate = $4 \cdot$ flow rate/ ($\pi \cdot$ (capillary radius)^3). By the comparison of the viscosity before and after the flowing, it was possible to get a curve of viscosity decreasing percentage against a shear rate. The viscosity values were obtained using a Brookfield LVT/ULA viscometer.

Determination of polymer solution concentration

The PHPA concentration in the effluents of the flow tests was accomplished using the turbidimeter method according to API Standard [6]. Due to this method, the polymer reacts with sodium hypochlorite resulting in an insoluble precipitate. This system is analyzed by using the Spectrophotometer HP 8452A.

2.2.3. Polymer/reservoir interaction

The interaction between polymer and reservoir was established by static adsorption, which was performed with the original reservoir rock to preserve the lithological characteristics, even though it is being in a disaggregated form. The disaggregation of the rock was made using a special mill that separates the grains, maintaining their integrity. After disaggregation a granulometric separation was made to remove the very small material, named silt, which is a consequence of the disaggregation of natural cement that joints the grains and may harm the analysis. Then, the disaggregated rock was dried and samples were put in contact with the polymer solution. After the equilibrium time, the final concentration of the solution was measured

and compared with the initial one. The physico-chemical adsorption was calculated (in µg of polymer for each gram of rock) as the ratio between the difference of initial and final concentrations and the total mass of the rock. This procedure was repeated for different concentrations of polymeric solutions, making possible to draw an adsorption curve as a function of concentration. The obtained data were adjusted by the Langmuir isotherm, which is the well-known and recognized mathematical model to represent the phenomenon and its reversibility.

3. Results and Discussion

3.1. Evaluation of the Target Reservoir

The evaluation of the reservoir was done using basic criteria defined in literature [1-4]. These criteria vary a little depending on the source from where they are extracted. Table 1 presents: the parameters being evaluated (first column), the average value for these criteria that may be found in the mentioned literature (second column), the values of these relative parameters for the target reservoir located in the northeast part of Brazil (third column) [12], and the evaluation of each item identifying if it is good for the test or not (fourth column). As expected, the selected reservoir does not present the ideal profile. However, it is inside the accepted range for petroleum recovery with polymer injection.

In polymer flooding, temperature and salinity are key parameters, because if they are above certain values the behavior of the polymeric solution can suffer alteration due to thermal and saline conditions, and the success of the operation is not guaranteed. The oil properties (viscosity and degree API) are also important for the selection of the suitable polymer to be injected, since the selection depends on the viscosity correction that is intended to reach. Other important parameters are the remaining oil saturation, that has to be above a minimum value, and the water/oil mobility (or viscosity) ratio, which will be adjusted by adding polymer in the aqueous phase, allowing the increase in swept efficiency and, as a consequence, in oil recovery [1-5]. The polymer performance is directly related to the water/oil mobility ratio of the reservoir before its application: high original mobility ratio meaning higher polymer flooding efficiency.

The characteristics of the reservoir, such as rock type, permeability, absence of gas cap, aquifer and natural fractures, should meet recommendations given in literature [2-5]. The high clay content of the target reservoir (Table 1) must be considered with great attention since the physico-chemical adsorption of the polymer in the rock can be significantly increased, generating a pressure increase and, in extreme cases, leading to a blockage of the porous path [2-5, 8-10]. This problem can be eliminated, or minimized, by adapting the polymer solution to the rock conditions by means of laboratory tests. The high heterogeneity is a concern in all recovery methods, particularly in the case of polymer flooding, since an improvement of the swept efficiency is its goal [2-4]. In spite of this, the target reservoir reasonably satisfies the required properties.

3.2. Polymer Characterization and Selection

Due to a relatively low cost and high availability, PHPA was selected for the present work. Moreover, PHPA

Table 1

Evaluation of the target reservoir

Parameter	Literature [3, 4, 9]	Target reservoir [12]	Evaluation *
Temperature, K	≤ 353.15	323.15	Good
Total salinity, ppm	≤ 50.000	30.000	Good
Oil viscosity, cP	≤ 80	50	Good
API degree (oil)	≥ 25	22	Reasonable
Move oil saturation, %	≥ 10	32.2	Good
Permeability, mD	≥ 100	100	Good
Mobility ratio	≥ 1	21	Good
Rock	Sandstone	Sandstone	Good
Heterogeneities	Low	High	Bad
Clay content, %	Low	High	Bad
Gas cap	Non-existent	Non-existent	Good
Natural aquifer	Non-existent	Non-existent	Good

* This indicates how good the selected rock is for the flow tests

Table 2

Characterization of PHPA samples

Flopaam sample	Hydrolysis degree, %	Molar mass, g/mol	Water content, %	Thermal stability*, K	pH (1 %, 298.15 K)	Intrinsic viscosity**, ml/g
2030 S		$5.0 \cdot 10^5$				-
2130 S	25	$3.0 \cdot 10^6$				-
2230 S		$5.0 \cdot 10^6$				-
3230 S	30	$5.0 \cdot 10^6$	<1	433.15	7.5	10
0430 S	10	$1.0 \cdot 10^7$				-
2435 S	25	$1.2 \cdot 10^7$				-
2630 S		$1.6 \cdot 10^7$				-

* Temperature of the beginning of the degradation obtained in TGA
** The values of the constants *K* and *a* were 6.75E-05 and 0.830, respectively [6], in the intrinsic viscosity equation $[\eta] = K \cdot (MM)^a$ [6]
(-) Not measured

is used in more than 95 % of world polymer applications for EOR [2-4]. For all samples, the powder form was chosen since it is easier to handle with. An anionic load was selected to reduce the physico-chemical adsorption in the reservoir rock, as suggested by literature [2-5, 7-10]. The polymer samples were characterized and based on those characteristics and only one sample was pointed for using in future research on EOR, particularly on the development and validation of a mathematical model for polymer flooding. Table 2 presents the PHPA characterization results.

The polymer samples presented the stability compatible to petroleum reservoir applications. The hydrolysis degree and mass molar results show that the samples present distinct characteristics, which will be later compared with the rheological behavior of their solutions.

The polymer solution quality or homogeneity was verified through the filterability test (API Standard [6]). Fig. 1 presents the pressure-time plot for 1000 ppm solutions of the largest molar mass sample (Flopaam 2630) and the lowest molar mass sample (Flopaam 2030 S). Distilled water is also presented as a reference. The other polymeric solutions presented homogeneous solutions without insoluble residues with curves between these two, which means that all solutions present very low probability to blockage the porous media.

Fig. 2 presents the variation of the consistence index as a function of concentration for the aqueous solutions prepared with PHPA of different molar masses (*MM*) and hydrolysis degrees (*HD*). The analyses were performed at 298.15 K and shear rate of 7.3 s^{-1}. By analyzing only the PHPA curves presenting hydrolysis degree of 25 %, it

is observed, as expected [2-5], that the consistence index increases more strongly with the increase of the concentration for the samples of the higher molar mass. Comparing the solutions prepared with the polymer presenting the same molar mass ($5 \cdot 10^6$ g/mol) and different hydrolysis degrees (25 and 30 %), it is observed that higher viscosity values are reached at the same concentration for the sample with larger hydrolysis degree, as a consequence of greater electrostatic repulsion in the polymeric yarn that induces remarkable increase of the macromolecule hydrodynamic volume [8-10].

Since the viscosity is of paramount importance for operations involving injection of aqueous fluids in petroleum reservoirs and it varies as a function of different parameters (such as, molar mass, hydrolysis degree and concentration), a mathematical fitting was performed using the data of Fig. 2. In spite of the small number of experiments, which does not allow an accurate fit, a linear relationship among the three parameters in a simultaneous way gives an idea of the relative contribution of these parameters to the final viscosity value. This indication is given by the coefficient values of Eq. 1 that represents a linear fitting of the viscosity (μ) against molar mass (*MM*), hydrolysis degree (*GH*) and concentration (*C*). One can notice that the molar mass presents the highest contribution followed by the hydrolysis degree and, finally, by the concentration. This analysis confirms the importance of the specification / selection stage of the appropriated polymer for field processes.

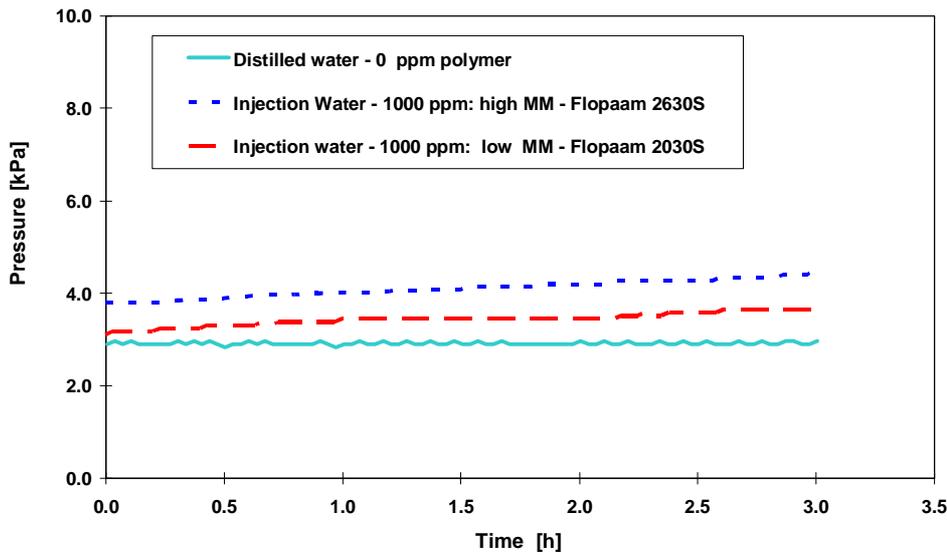$$m = 24.02MM + 12.57HD + 0.43C - 480.15 \qquad (1)$$

Fig. 1. The filterability data: pressure against time for the solutions of Flopaam 2630 S and Flopaam 2030 S, at 298.15 K
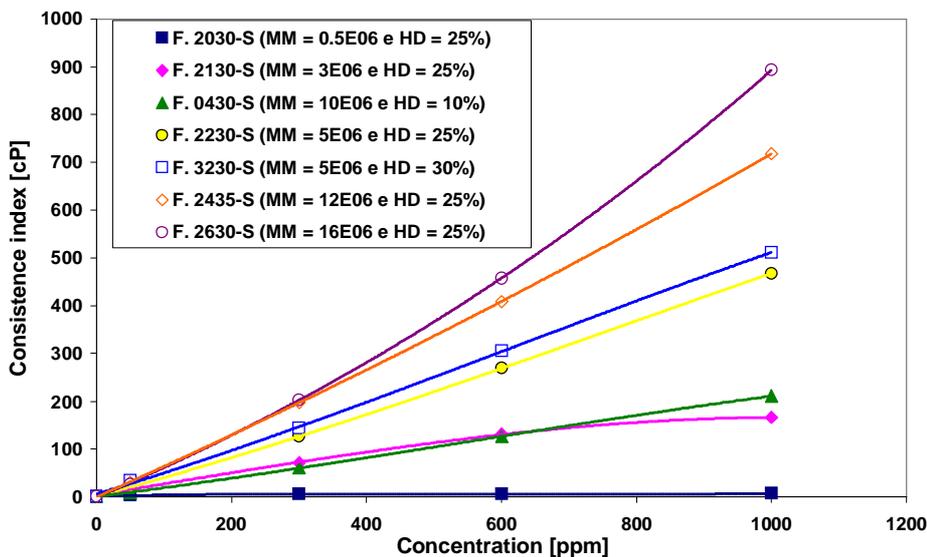

Fig. 2. Viscosity against concentration for PHPA samples dissolved in distilled water, at 298.15 K and shear rate of 7.3 s^{-1}

where, μ – viscosity, cP; *MM* – molar mass, million g/mol; *HD* – hydrolysis degree, %; *C* – concentration, ppm

Starting from the presented data a pre-selection of the polymer to be used in flow tests under the target reservoir conditions was made. Since the product type (PHPA), the ionic load (anionic) and the physical form (powder) were already selected, it was necessary to choose the hydrolysis degree and the molar mass.

The *MM* range usually adopted in operations of fluid injection in petroleum reservoir lies in the range of $1\cdot10^6$ and $30\cdot10^6$ g/mol [4]. At first sight, the higher molar mass, the higher the final fluid viscosity using smaller amount of polymer. However, the selection of the most

adequate molar mass must be based on the permeability average value for the target reservoir rock. Porous media presenting very low permeability, which in a certain way is related to the size of the pores, can be partially or completely blocked (mechanical trapping) by polymer molecules that present very high molar masses (for instance, in the upper limit of the previously presented range – $30\cdot10^6$ g/mol). Since the permeability of the selected rock is in the recommended lower limit (see Table 1), the Floopam samples presenting molar masses equal or greater than $10\cdot10^6$ g/mol were discarded, in spite of the higher viscosity reached. Among the four remaining samples, the one that presents larger viscosity value is Flopaam 3230 S, with hydrolysis degree of 30 % [13].

Then the Flopaam 3230 S was submitted to more specific tests that are ascribed here. In case of any sample satisfies the requirements, it would be necessary to select another polymers set to be evaluated, following the procedure presented before.

Fig. 3 shows the stability of the polymer solution in distilled water as a function of temperature: 298.15 K (standard condition) and 323.15 K (temperature of the target reservoir). The solution viscosity reduces with the temperature increase. Although such reduction is not very high it can slightly influence the result of the field operation.

Another important evaluation is connected with the polymeric solution behavior in the presence of ions. The injection water presents a total salinity concentration of less than 1000 ppm, including sodium (larger amounts) and divalent ions, such as calcium and magnesium. Fig. 4 illustrates the behavior of the viscosity as the function of the concentration for Flopaam 3230 S solutions in the target reservoir injection water. For comparison the curve for standard conditions (distilled water and temperature of 298.15 K) is also presented. It can be observed that the system, under the reservoir conditions, presents viscosity values much lower than those obtained under standard conditions. Of course, the temperature increase affects such reduction, as observed in Fig. 3. However the largest contribution for the remarkable reduction is the presence of ions (sodium and divalent ions) in the injection water, which interact with the polymer negative loads, neutralizing the effect of macromolecular expansion [2-5, 7-10].

Another important factor is the shear degradation that takes place in two different stages: 1) when the polymer flows from the surface until the porous media (through pipes, valves, *etc.*); and 2) when the polymer is submitted to the flow in porous media itself. The first stage represents the viscosity loss due to the breaking of the polymer chains caused by the mechanical shearing during the flow, particularly in restrictions, such as pumps and valves. In this work, such degradation was evaluated in the laboratory, submitting the polymer solution to a pressurized flow with a high flow rate in a capillary tube at different pressures [6]. Fig. 5 presents the viscosity against shear rate, for Flopaam 3230 S at 1000 ppm in injection water at 323.15 K, previously submitted to different shear rate in the capillary tube at 298.15 K. It was observed that the viscosity loss could be considered negligible, even when the solution was submitted to shear rates in the capillary as high as 165000 s^{-1}.

Finally, the polymer-reservoir rock interaction was evaluated. Literature [2-5, 7-10] is unanimous when considering the importance of the polymer injection process adsorption for petroleum recovery. This is the main parameter controlling the process and it is necessary, therefore, it would be known and quantifiable previously in the laboratory in order to support the design and evaluation steps. The adsorption may be separated into the mechanical retention (irreversible) and physico-chemical adsorption (reversible).

The irreversible adsorption is associated with polymer molecule sizes related to the pore throats. This can be easily determined by the mass balance in a conventional porous flow test. On the other hand, the physico-chemical or reversible adsorption, which is related to chemical and physico-chemical characteristics of the polymer and the rock, is not so easy to be experimentally measured by flow tests [3-4]. One of the options discussed in literature is to perform that determination in a static way [4, 6]. In these works the Flopaam 3230 S solution was evaluated by static test with two kinds of rock (Berea
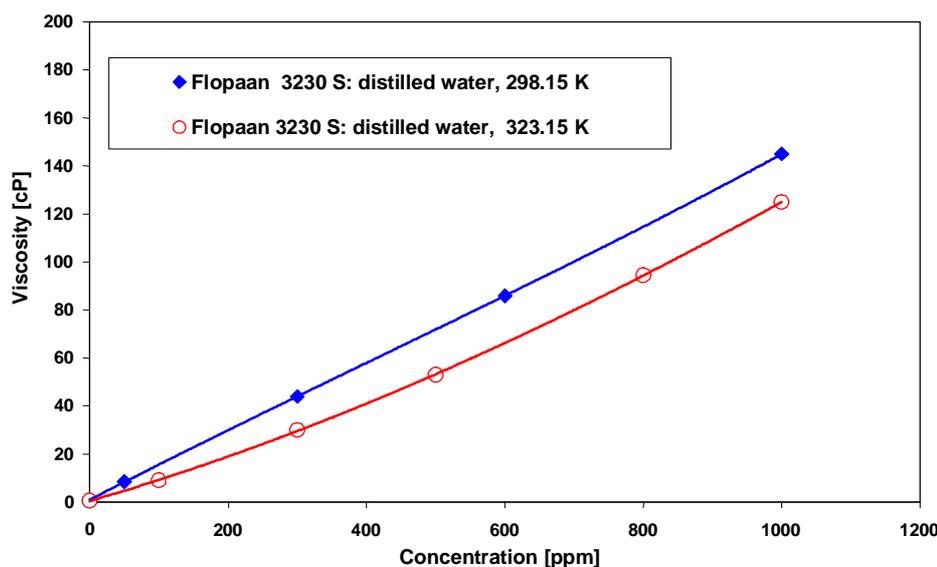

Fig. 3. Viscosity against concentration for Flopaam 3230 S in distilled water, at 298.15 K and 323.15 K (shear rate is 7.3 s^{-1})
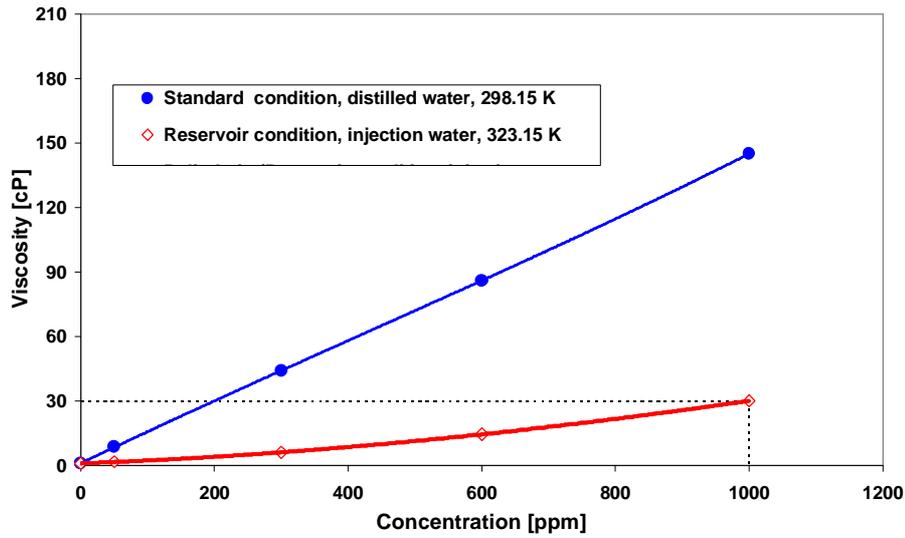
Fig. 4. Viscosity against concentration of Flopaan 3230 S under reservoir and standard conditions
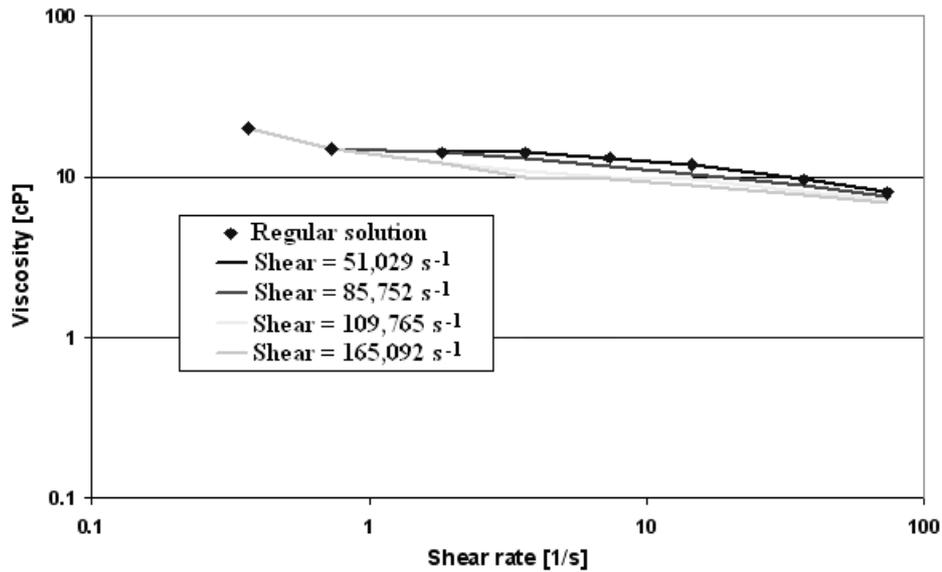(shear rate = 7.3 s^{-1})

Fig. 5. Mechanical degradation of Flopaam 3230 S after flowing in a capillary at 298.15 K with several shear rates.
The curves were obtained at 323.15 K

outcrop and the target reservoir rock), and as a function of the concentration. The samples of unconsolidated and previously dried rocks were put in contact with the polymeric solution and, after the equilibrium time being reached, the final concentration of the solution was measured and compared with the initial one [6].

Fig. 6 illustrates the experimental results and the respective adjusted curves using the Langmuir model. It can be noticed that the Langmuir equation fits well the experimental points showing the suitability of that model to the phenomenon. The isotherm for the Berea outcrop was run since several authors use this rock as a reference to represent a reservoir rock homogeneous and reasonably clean in terms of the clay content [3, 4]. For comparison, the expected range of adsorption for reservoirs in general is also highlighted in Fig. 6 for 1000 ppm concentration [9]. The curve of physico-chemical adsorption for the target reservoir is a little bit above the curve for the Berea outcrop and lies in the upper part of the literature range. It may confirm that the adsorption values that will be obtained in future adsorption tests under the flow will be high.

The authors believe that there is a correlation between the high clay content of the target reservoir (Table 1) and the high adsorption value found in the static test.
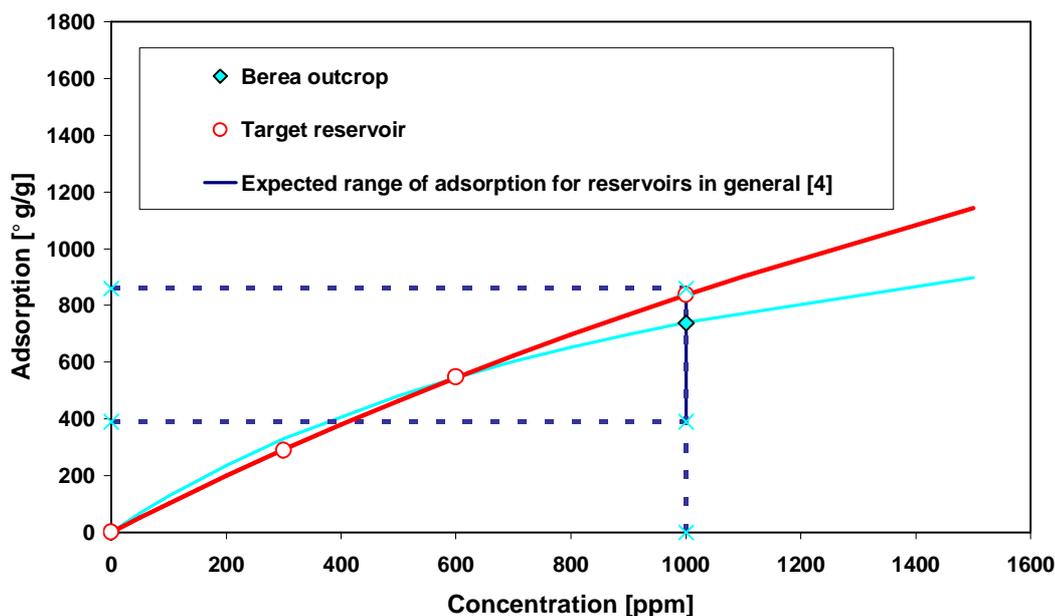
Fig. 6. Static adsorption of Flopaan 3230 S in Berea outcrop and target reservoir rock regarding their molar mass, hydrolysis degree and rheological behavior

The great efforts of several authors investigating the static adsorption test [3-6, 9] follow the belief that it is possible to apply, for consolidated rock, the adsorption value found for unconsolidated rock, through normalization by the superficial area. This procedure would be very useful since it is difficult to obtain the Langmuir isotherm from the flow tests, due to the reversible character of the physico-chemical adsorption and/or the presence of inaccessible porous volume [3-6, 14, 15]. However, in practice, such methodology does not work since there is an enormous error associated with the determination of the superficial area for the typical range of reservoir. That point strongly motivates further research on alternative methods to accomplish such adsorption evaluation.

4. Conclusions

This paper developed and presented a methodology for pre-selecting a polymer to be used in future research on enhanced oil recovery (EOR). For this purpose, the reservoir and the molecule characteristics were confronted and it was possible to select the suitable polymer specification for petroleum recovery in the target reservoir.

Tests to evaluate the rock-fluid interaction are indispensable for the final selection of a polymer for EOR. However, besides being long and difficult, they are expensive since they use samples of the specific reservoir rock. Then, it is very important to perform a pre-selection of the polymer based on some criteria to optimize that final flow tests stage. The methodology described in this paper consists of the following steps:

1. Selection of the polymer type with an ionic (negative) load based on the lessons learned alongside with previous field work.

2. Selection of the physical form (powder, due to the handling easiness).

3. Choice of a single supplier in order to guarantee the reproduction of the results.

4. Based on the fact that polymer retention/adsorption in the rock is a function of the relative size of polymer molecules compared with pore throats, polymer samples were selected in the range of molar mass that minimizes such retention/adsorption effects. Following the characteristics of the target reservoir, only polymer samples in the inferior/medium range of molar mass were selected.

5. For those polymer samples it was selected the one with the best rheological performance, which is related to the hydrolysis degree. Flopaam 3230 S was selected based on this criterion.

6. A more detailed characterization of Flopaam 3230 S was performed to support the tests modeling the retention/adsorption process in the transport of particles through porous media, which is of great scientific and industrial importance for several applications in the Petroleum, Chemistry and Environmental Engineering.

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ХАРАКТЕРИСТИКА І ВИБІР ПОЛІМЕРУ ДЛЯ МАЙБУТНЬОГО ВИКОРИСТАННЯ ПРИ ВИДОБУТКУ НАФТИ

Анотація. Для підвищення ефективності виробництва нафти та зниження циркуляції води при видобутку нафти використовують нагнітання у свердловину полімерів. В роботі представлено методику попереднього вибору полімеру, який надалі буде використаний в дослідженнях, пов'язаних з видобутком нафти з вприскуванням полімерного розчину. Вибрано і охарактеризовано пластовий резервуар, а також сім зразків промислового, частково гідролізованого поліакриламіду. Приготовлений полімерний розчин охарактеризований на фільтрованість, в'язкість, стабільність та механічну стійкість. Досліджена взаємодія між полімером і резервуаром. Показано, що найкращі результати отримані при використанні полімеру, в якого ступінь гідролізу становить 30 %, молекулярна маса $5{\times}10^6$ г/моль і внутрішня в'язкість 10 мл/г.

Ключові слова: видобуток нафти, полімерні розчини, характеристика полімеру, характеристика пласту, реологічна поведінка, тест на фільтрованість, тест на статичну адсорбцію.