6ª Jornada de Recuperación Mejorada de Petróleo – EOR

Title: Thermal Stability Evaluation of EOR Polymers : Best Practices & Guidelines

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Polymer flooding is the most successful EOR method consisting in increasing the viscosity of the aqueous phase injected in a reservoir to improve its efficiency to sweep the oil to producer wells. EOR polymers are high molecular weight partially hydrolyzed polyacrylamides. The degree of hydrolysis and the polymer chemistry must be selected carefully according to the brine composition to avoid any incompatibilities and prevent from precipitation with divalent cations. The residence time of the polymer solution in the reservoir can range from several weeks to several months or even years, especially offshore. During this lapse of time, and upon the action of the temperature of the reservoir, acrylamides moieties can hydrolyze into acrylates increasing the degree of hydrolysis of the polymer and the risk of precipitation with the divalent cations.

In the literature, the only official protocol is the one given by the American Petroleum Institute. This technique recommends the use of several cycles of vacuum and nitrogen flushes with frozen and unfrozen of the polymer solution to decrease the concentration of dissolved oxygen below 100 ppb. The ampoules are then sealed under vacuum using a blue torch. The quality of the sealing and of the deoxygenation is strongly operator dependent and oxygen ingress can be observed through micro cracks during the melting and cooling of the glass seal.

In the field, polymer solutions are generally prepared under Nitrogen blanketing and remaining oxygen in solution is rapidly striped out by the reservoir acting as a strong reducing agent. Therefore, O_2 concentration is lower than 5 ppb. To reproduce such level of dissolved oxygen, the use of a glove box has been progressively generalized to the main laboratories. Results remain however quite variables depending on the protocol that has been followed.

To align procedures so that to generate more homogeneous results, this paper presents the most appropriate practices developed upon the last decade. It is based on deep analysis of failures and successes in conducting thermal stability tests. The aim is to bring experimental inputs to draw new guidelines for the thermal stability evaluation of EOR polymers. The following items have been considered:

- Preparation of a polymer solution for thermal stability;
- Selection of the most appropriate material to be used for aging, for instance type of containers (glass versus stainless steel), types of plugs.

Introduction

During the last decade polymer flooding has been implemented on different oilfields and has demonstrated to be the most efficient technology to improve oil recovery at the lowest expense possible (1) (2) (3) (4) (5) (6) (7) (8) (9) (10). Projects now move from pilot trials to field expansions and some time even to full fields. However, one of the main concerns remain the long-term stability of the polymer solutions during its stay in the reservoir. This concern becomes more and more frequent since the conditions of the field candidate for polymer injection are becoming more and more difficult; increase of the salinity with the presence of divalent cations and/or increase of the reservoir temperature up to 120°C. Some of these new fields are also located offshore where the spacing can be large and the residence time of the polymer in reservoir can varied from 1 to 8 years.

In the literature contrasting results can be found and can leave wrong beliefs in the mind of the reader. Authors usually refer to the API protocol but practically a very few followed it carefully (11) (12). Indeed, according to the API procedure (13), the polymer solution must be prepared a make-up water previously deoxygenated by bubbling of oxygen-free research grade Nitrogen then place the polymer in glass ampoules, connect the ampoules to a vacuum ramp, cool down the ampoules (do not freeze), evacuate to < 0,1 mm Hg, let equilibrate the ampoules during 2 to 16 hours, return the manifold to atmospheric pressure using Nitrogen and repeat the steps of cooling down, vacuum and Nitrogen flush several times to reach O_2 concentrations below 1 ppm, usually about 100 ppb. If some authors apply carefully this method, other clamming using such protocol rather either perform a single or no more than two or three cycles of vacuum and nitrogen flush (14), some others (15) (16) remove the step of cooling down the solution to further decrease the concentration of dissolved oxygen leading to a final oxygen content of about a ten to a hundred of ppb, way above the oxygen level encountered in the reservoir, usually below 5 ppb. Some authors also reported the risk of cracks formation when sealing the ampoule with the blue torch. It was in particular evidenced by Levitt *et al.* (17) where resazurin were used to identify any O_2 ingress during the aging. Indeed, during the sealing and the cooling of the glass afterwards, stress is applied on the glass and cracks can be formed. This operation is strongly operator dependent and even the best ones make mistakes from time to time. In some cases, authors are also bubbling the polymer solution with either ultra-pure Nitrogen or Argon (16) but the impact of the bubbling on the viscosity is not addressed and mechanical may or may not occur. In other papers, thermal stability is evaluated while no information is given about the protocol of deoxygenation (18).

On the other hand, several authors (19) (20) (21) proposed the use of a glove box or anaerobic chamber to ensure reaching lower final dissolved O_2 concentrations and prevent the sample under aging from any oxygen contamination during the measurement of its viscosity. This method is progressively replacing the current API recommendation, but the protocol is still not sufficiently described, and methodology can differ significantly from one lab or one company to the other. For example, different types of container are used for the aging such as glass ampoules, glass bottles, stainless steel containers, containers made of different alloys, and containers having different coatings. Some laboratories or companies prepared the tests in the glove box but exposed the sample to oxygen just before measuring its viscosity. This can be dramatic especially if using alloy-made containers releasing iron in solution during the aging.

In this paper, we propose to share our knowledge acquired along 8-years+ of glove box practice to help drawing the basis of a more uniform method for the evaluation of long-term stability of EOR polymers. The use of the glove box allows to achieve reliable ultra-low O_2 concentrations and to reduce the number of ampoules prepared by sample. Indeed, using the vacuum ramp method, an ampoule must be prepared for each measurement performed after aging. The number of ampoules must be anticipated depending on the number of measurements over time and some spare must be planned in case of leakage (cracks formation) or suspicion of poor deoxygenation. When using a glove box, the same ampoule or container can be used for the all study, reducing significantly the time of preparation for the aging test. This paper focuses on how to prepare the polymer solution, the impact of the type of ampoules, using ampoules

made of stainless steel, different alloys, or coated with different “inert” materials. Glass ampoules have also been tested, using the different glassware ampoules/bottles available on the market.

Experimental part

Fluids and chemicals

Different EOR polymers from SNF were used in the different tests presented at continuation. In this paper we focus on polymers under powder form reported in Table 1.

Table 1 : *Summary of the different EOR polymers used in this work.*

Polymer	Type	Form
FP 3630 S	Copolymer AMD/AA	Powder
AN 105 VHM	Copolymer AMD/ATBS	Powder
AN 125 VHM	Copolymer AMD/ATBS	Powder

Glove box description

To achieve total anaerobic conditions, the glove box is the best option. In the present paper a Jacomex GP concept T-4 was used. It was equipped with copper catalyst to scavenge any trace of oxygen. The method has been described in previous works (22) (23) (24) (25). Oxygen measurements were performed using an Oxy IQ transmitter for the O_2 present in the atmosphere of the glove box while O_2 concentration in solution was measured using a Presens Fibox4 Trace equipped with a PST6 probe. The sensitivity of the probe is 0.1 ppb in liquid phase.

Figure 1 : *Glove box GP concept T-4 from Jacomex.*

The use of a glove box is critical, since it allows reaching very low O_2 concentration in the atmosphere. Indeed, the O_2 in solution equilibrates with the O_2 in the atmosphere. Figure ***2*** shows the evolution of the dissolved O_2 concentration as a function of the O_2 concentration in the atmosphere. The level of O_2 in the glove box being usually below 100 ppb allowing reaching dissolved O_2 concentration in solutions prepared in the glove box lower than 20 ppb, usually between 5 and 15 ppb.

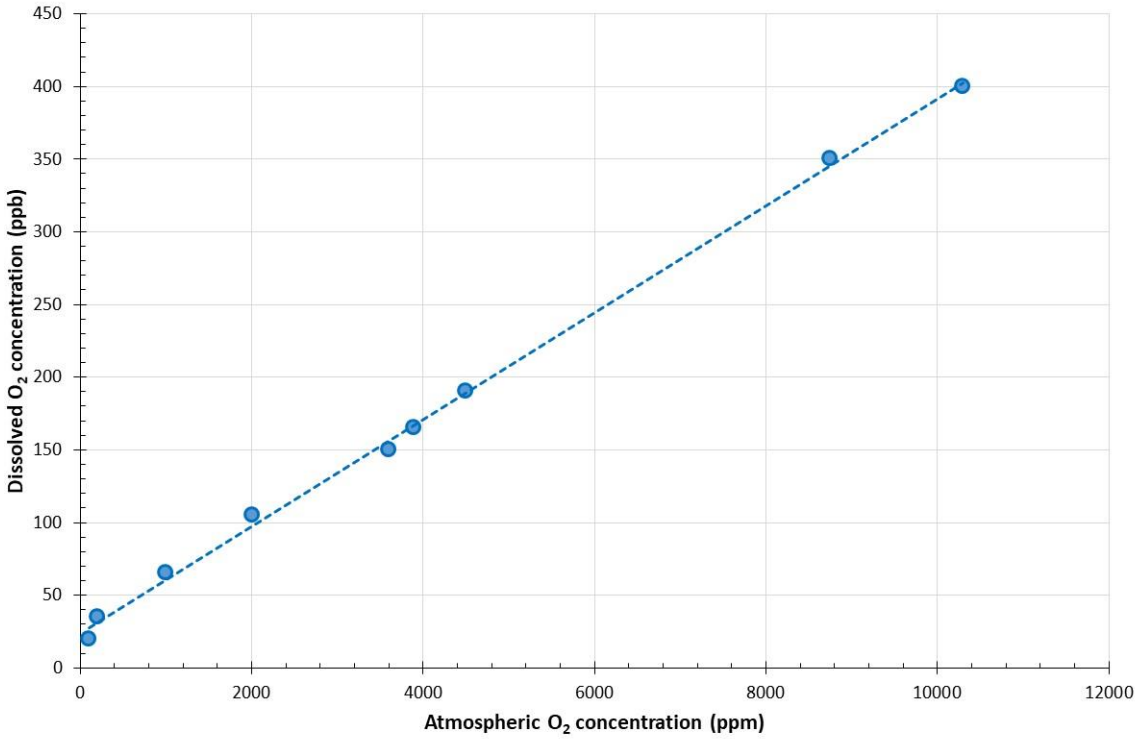

Figure 2 : *Evolution of the dissolved O_2 concentration as a function of the O_2 concentration in the atmosphere.*

Viscosity measurements

Viscosity measurements were performed using a Brookfield LVT™ viscometer equipped with an UL adapter™ at 6 rpm (7.34 s^{-1}) connected to water bath set at 25°C.

Figure 3 : *Brookfield LVT™ viscometer (left), UL adapter (right).*

Results and Discussions

Solutions preparation

There are mainly two options for the preparation of the polymer solutions, either the brine is first deoxygenated, and the polymer is dissolved in the brine directly in the glove box, or the polymer solution is first prepared outside the glove box and then deoxygenated in the glove box. In both cases, the material, the products and the fluids must be introduced in the glove box via the glove box airlock according to the instructions from the manufacturer.

Brine deoxygenation then polymer dissolution:

The deoxygenation of the brine is performed by bubbling oxygen-free research grade Nitrogen. In Figure 4 is reported the evolution of dissolved O_2 concentration in 1 liter of brine placed in a glove box during the bubbling period. Accordingly, it is recommended to bubble the solution at least 20 min to reach dissolved O_2 concentrations below 20 ppb.

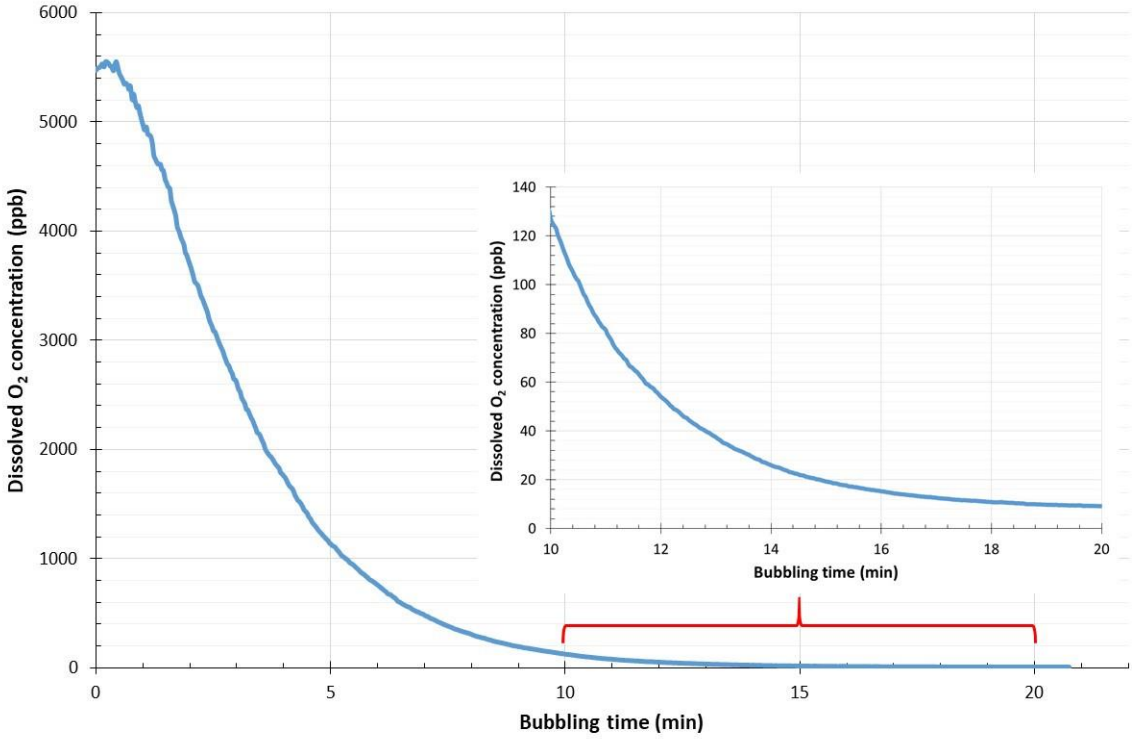

Figure 4 : *Evolution of the dissolved O_2 concentration as a function of the bubbling time using oxygen-free research grade Nitrogen in a glove box.*

The polymer solution is then prepared in the glove box following the recommendation given in the API RP 63 (13). First a mother solution is prepared at a concentration of 5 000 to 15 000 ppm depending on the brine composition (the higher the brine salinity, the higher the concentration). The brine is placed in beaker and initially stirred at 500 rpm and the powder is gently sprinkled into the wall of the vortex. After initial hydration of the polymer solution (observed via a reduction in the vortex after 30 minutes), the stirring rate is reduced to 200 rpm and maintained for 2 hours to ensure a complete dissolution. The mother solution is then diluted to the desired concentration using the same deoxygenated brine. The dilute solution is maintained under gentle stirring (100-200 rpm) for 2 hours to ensure homogeneity.

Direct preparation of the polymer solution and post-deoxygenation:

The alternative consists in preparing the polymer solution outside the glove box, following the same protocol than the one given in the previous paragraph. The polymer solution is then introduced in the glove box and deoxygenated. The deoxygenation can be performed either maintaining the polymer solution under agitation or bubbling Nitrogen as previously mentioned for the brine. The impact of these two methods is discussed at continuation. Two polymers (FP 3630 S and AN 125 VHM) prepared under the same conditions (2 000 ppm in a brine composed of 30 000 ppm of NaCl and 3 000 ppm of $CaCl_2,2H_2O$) where degassed applying either, a gentle magnetic mixing, a strong magnetic mixing, a gentle bubbling or a strong bubbling as shown in Figure 5.

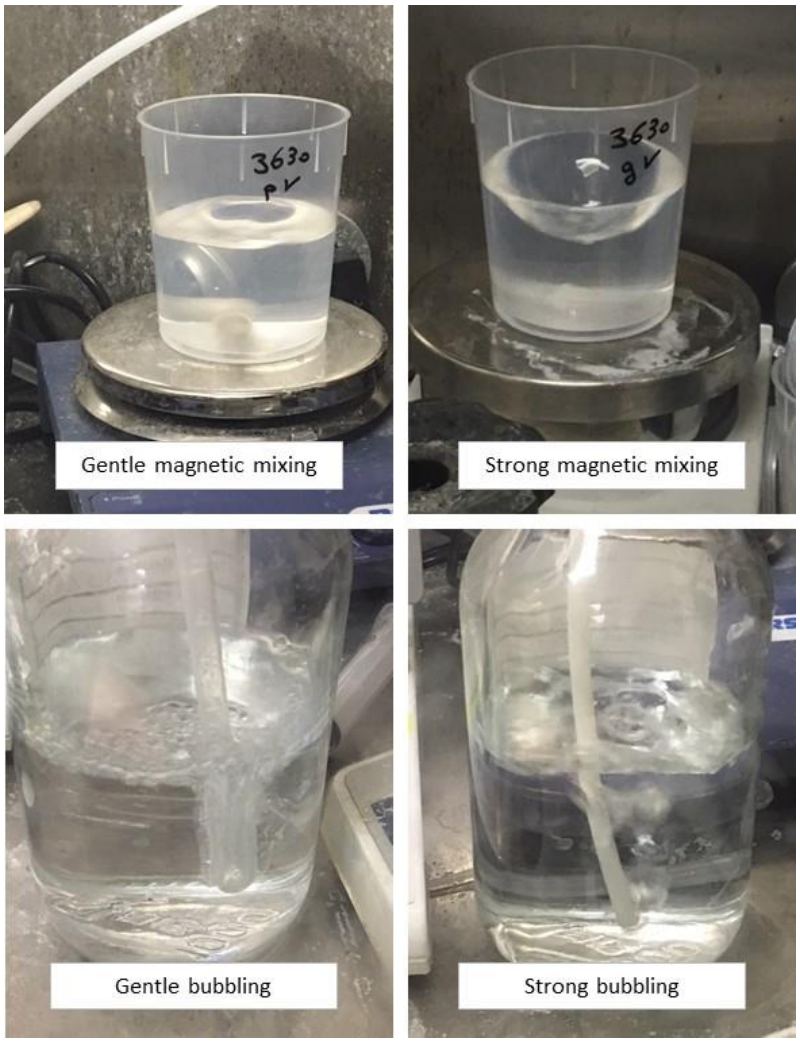

Figure 5 : *Different procedures to remove oxygen from the polymer solution in the anaerobic glove box.*

Viscosity was measured before and after 2 hours of mixing/bubbling using a Brookfield LVT viscometer equipped with an UL spindle at 6 rpm (7.34 s^{-1}). Results are reported in Figure 6.

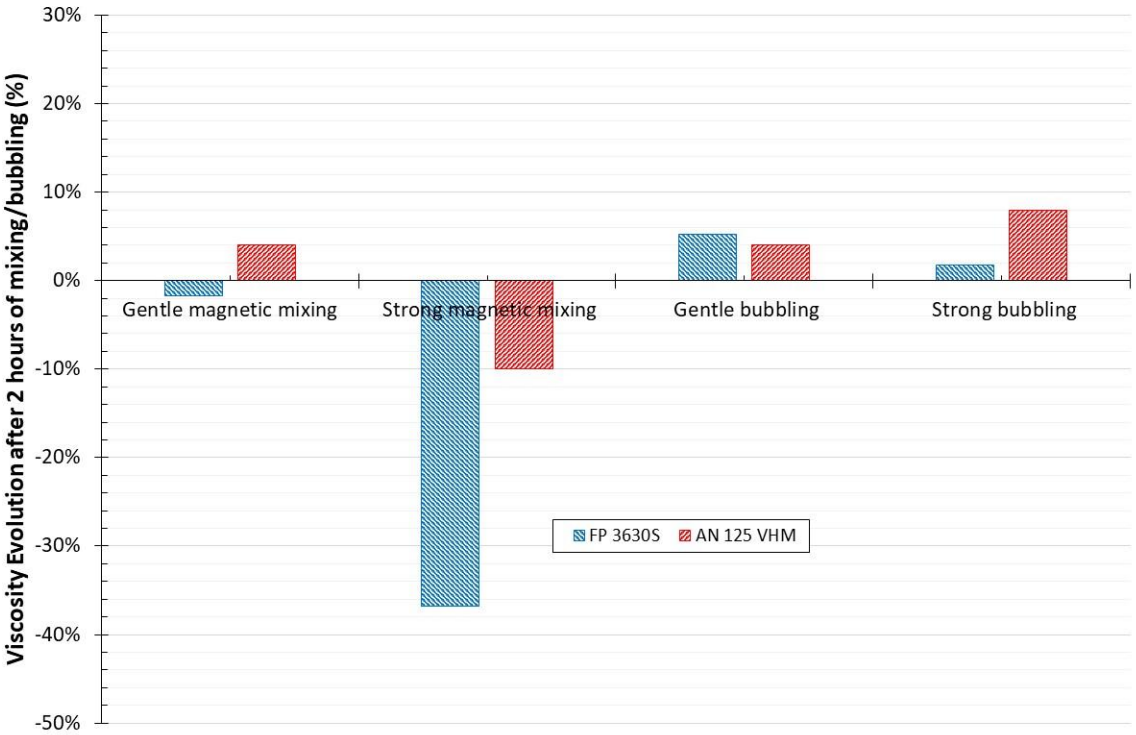

Figure 6 : Impact of the type of deoxygenation procedure on viscosity after 2 hours of mixing/bubbling.

Results show that no significant degradation is obtained when bubbling the solution with Nitrogen whatever the intensity of bubbling. However, applying a constant mixing speed using a magnetic stirrer can lead to significant viscosity loss (up to 37%) probably due to shear degradation. Bubbling also allowed to reach a low level of O_2 in a shorter time compare to deoxygenation by stirring.

Importance of the ampoule/bottle material

After preparation, viscosity is measured, and the polymers solutions are poured in ampoules or bottles for aging. The containers are then transferred to an oven set a reservoir temperature. Obviously, the best solution is to have the oven inside the glove box, but this is not practical when hundreds of samples have to be evaluated at different aging temperatures. In the present paper, aging containers are transferred to ovens out of the glove box. Consequently, the containers must remain perfectly sealed during the aging to avoid any ingress of oxygen and avoid any evaporation. When applying the vacuum ramp protocol, specifically designed borosilicate glass ampoules are used. Here, the glove box protocol gives more freedom regarding the choice of the container and the laboratories/companies running thermal stability used different types of ampoule or bottle. At continuation we have selected the most commonly ones and compare their performance with a glass ampoule specifically designed by a local glass blower as well as with a glass ampoule flame sealed in the glove box (technic recently adapted in-house for the glove box O_2-free environment). The different ampoules and bottles tested in this section are reported in Table 2 below.

Table 2 : *List of the ampoules and bottles commonly used for thermal stability tests.*

#	Material	Type	Manufacturer	Treatment
Amp_1	316 L Stainless Steel	Ampoule	Swagelok	Passivation
Amp_2	Glass	Ampoule	SNF/Local glass blower	-
Amp_3	Glass	Ampoule	SNF/Local glass blower	Sealed in the GB

Amp_4	Glass	Ampoule	Chemglass	-
Bot_1	Glass + PTFE-coated silicon seal in the tap	Bottle	Duran	-
Bot_2	Glass + Aluminium crimp seal	Bottle	Perkin Elmer	-
Bot_3	Glass + Aluminium crimp seal	Bottle	Manufacturer 2	-
Bot_4	Glass + Aluminium crimp seal	Bottle	Manufacturer 3	-

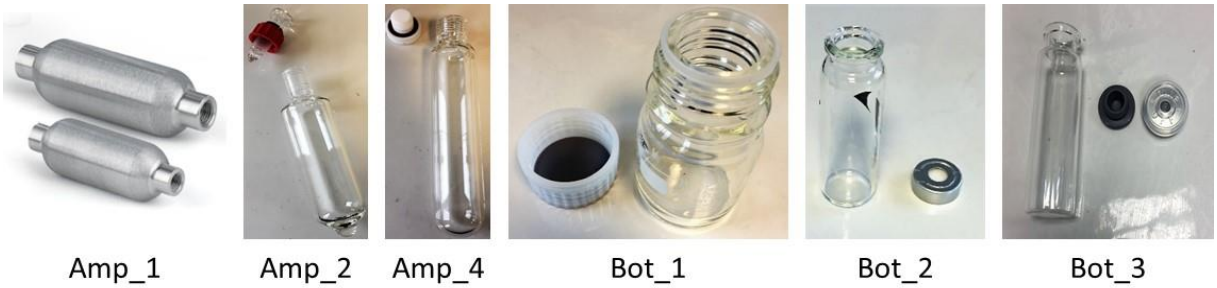

Figure 7 : *Illustration of the ampoules mentioned in this section.*

In order to assess the impact of each container on the thermal stability of a polymer solution, a benchmark solution was prepared using AN 105 VHM at 2 000 ppm in a brine containing 30 000 ppm of NaCl. Aging was performed at 78°C for 90 days. Results reported in Figure 8 show an increase of viscosity over 30 days aging whatever the container. This behavior is due to the hydrolysis of the polymer in the absence of divalent cations. For the 316 L stainless-steel ampoule (Amp_1), the homemade glass ampoule (Amp_2) and the glass ampoule sealed in the glove box (Amp_3) viscosity reached a plateau afterwards. The maximum hydrolysis rate of the polymer was reached and the brine being free of divalent cations, the polymer remains stable. At the contrary for all the other ampoules and bottles, degradation starts to be observed between 30 and 60 days of aging. We have evidenced that those ampoules leaked during the cooling phase before entering the cells in the glove box allowing O_2 ingress. Another experiment was performed to assess this for Amp_2, Amp_4 and Bot_1. O_2 level was checked after 7 days of aging at 105°C (see Table 3).

Table 3 : *Oxygen ingress depending on the type of ampoule. Aging time = 7 days at 105°C.*

Ampoule	**Dissolved O_2 concentration**
Amp_2	27 ppb
Amp_4	458 ppb
Bot_1	1 200 ppb

Also, the seal of Duran bottle is made of Silicon and coated with PTFE but only on one side. It appears that by placing the seal at the reverse (Silicon exposed directly to the solution, BOT_1_Rev) the degradation was faster than when the seal was placed properly (PTFE side exposed to the solution). By using an already used Duran bottle (meaning a bottle already used for a thermal stability test of 1 year) degradation was found to be faster too. Some components or adjuvants used in the formulation of the seal may diffuse through the PTFE coating and contaminate the solution causing an accelerate degradation of the polymer solution. These observations have also been made using similar glass bottles from other manufacturers. Since we were suspicious about the potential contamination from adjuvants used in the materials of the lids of the conventional glass bottles or to their potential leakage during cooling, we have decided to use ampoules without those materials. In the case of the stainless-steel ampoule, the sealing with the plug is ensured by a thin film of Teflon around the screw thread while in

the case of the homemade glass ampoules the sealing is only ensured by a specific glass to glass contact between the ampoule and the plug.

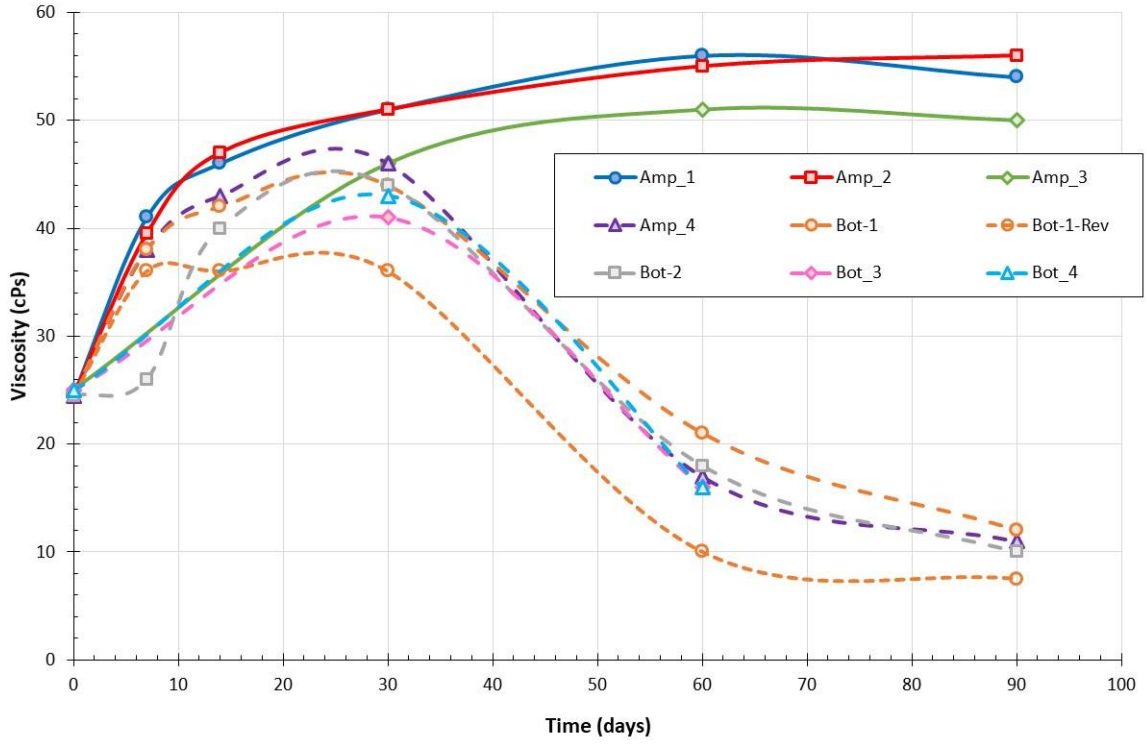

Figure 8 : *Impact of the type of container on the evolution of the viscosity a solution of AN 105 VHM prepared at 2000 ppm in 30 000 ppm NaCl and aged at 78°C.*

The use of stainless-steel ampoules can also be problematic since it may release iron in the solution through corrosion. It has already been demonstrated that in absence of oxygen the release of iron had no impact on the viscosity. However, if some O_2 contamination occurred or if the viscosity is measured outside the glove box the degradation will be significant. Brand new and already used stainless-steel ampoules from two manufacturers (Swagelok and Parker) have been filled with a deoxygenated brine containing 50 000 ppm of NaCl and aged at 105°C. At regular interval of time (4, 15 and 30 days) iron content was determined using a Chemets kit. Results are given in Table 4.

Table 4 : *Iron released in 50 000 ppm NaCl when aged in stainless-steel ampoules at 105°C.*

Manufacture	New/Used	4 days	15 days	30 days
Swagelok	New	1 ppm	2 ppm	3.5 ppm
	Already used	0,3 ppm	0,3 ppm	1 ppm
	Passivated	n.d.*	n.d.	1 ppm
Parker	New	7 ppm	> 10 ppm	> 10 ppm
	Already used	10 ppm	> 10 ppm	> 10 ppm
Homemade Glass Ampoule	New	n.d.	n.d.	n.d.

*n.d.: not determined – under limit detection.

Results show that Parker ampoules released much more iron than the Swagelok ones and are not recommended. Swagelok ampoules that have already been used to perform stability tests allow reducing even more the quantity of iron released in solution. Therefore, systematic passivation of the Swagelok ampoules is recommended to reach very low level of iron during aging. The passivation step is also performed before re-using an old ampoule. The homemade glass ampoule was used as reference and showed no iron release as expected.

It was as well interesting to see the impact of different coating inside the stainless steel ampoule on iron release. In Table 5 are reported a series of ampoules from Swagelok having different coatings. It appears that the coating only increases the cost of material without bringing any improvement compared to passivation regarding iron release in solution. Surprisingly the ampoule having a Sulfinert coating release even more iron than the ampoule with no coating and no passivation.

Table 5 : *Impact of the coating on Iron release at 105°C in 50 000 ppm NaCl.*

Material	Coating	4 days	15 days	30 days
Stainless-steel	-	1 ppm	2 ppm	3.5 ppm
Stainless-steel	Passivated	n.d.	n.d.	1 ppm
Stainless-steel	Nickel	1 ppm	1 ppm	1 ppm
Stainless-steel	Sulfinert	1 ppm	2 ppm	5 ppm
Stainless-steel	Teflon	0.4 ppm	1 ppm	2 ppm
Monel	-	1 ppm	1 ppm	1 ppm

Conclusions

In this paper, new recommendations are given on how to evaluate the long-term stability of EOR polymers in reservoir conditions (brine, temperature) using a glove-box. Main conclusions can be summarized as follow:

- Glove-box preparation is more reliable and more efficient than preparing sealed ampoules using a vacuum ramp. It allows reaching quickly low dissolved O_2 concentrations (5 to 15 ppb) and it avoids the formations of cracks frequently observed when using sealed glass ampoules. The same aging container can be used for the all study allowing saving time and storage space.
- The polymer solutions can be prepared either in the glove-box using a brine previously deoxygenated by bubbling oxygen-free research grade Nitrogen during 20 min, or outside the glove-box and bubbling the polymer solutions in the glove-box afterwards during 30 min.
- The important parameter is the quality of the container used for the aging. In particular, the container needs to remain perfectly sealed during the aging.
    - The majority of the glass ampoules or glass bottles available on the market leak during the cooling period when taking the sample out of the oven. They also have lids made of silicon or composite materials which may diffuse in solution and degrade the polymer.
    - A homemade glass ampoule was developed with a local glass blower. The sealing of this ampoule is ensured by specific glass to glass contact between the ampoule body and the plug avoiding any leak or contamination of the polymer solution.
    - 316 L stainless-steel ampoules can be used providing the viscosity of the polymer solution is monitored in the glove box. Swagelok ampoules were identified as the ampoules releasing less iron in solution, however a passivation treatment performed prior to any aging test is highly recommended.
    - The use of stainless-steel ampoules having a specific coating has not shown any improvement compared the passivated ampoules. The use of more expensive alloys does not seem to bring any improvement neither.

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