

# Viscosity and Retention of Sulfonated Polyacrylamide Polymers at High Temperature

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**ABSTRACT:** The viscosity and retention of several copolymers of acrylamide (AM) with sodium salt of 2-acrylamido-2-methylpropane sulfonic acid (PAMS), and also hydrolyzed polyacrylamide (HPAM) have been studied under aerobic condition with and without the sacrificial agent, isobutyl alcohol (IBA) added at a temperature of 80°C. Parallel experiments have been performed in synthetic seawater (SSW) and 5 wt % NaCl. The viscosity at high temperature has been studied as a function of aging time, shear rate, sulfonation degree, molecular weight, and concentration of IBA. The retention in porous medium for sulfonated polyacrylamide polymers was measured in core floods using outcrop Berea sandstone. For the studied polymer sacrificial agent may protect polymer structure at high temperature. Higher sacrificial agent concentration gives better thermal stability in both 5 wt % NaCl and SSW solvents. Sulfonation degree also has a direct effect on thermal stability, i.e., higher sulfonation

degree lead to better thermal stability in terms of viscosity. By increasing temperature, less relative reduction in polymer solution viscosity was observed for the polymer with lower molecular weight. The presence of divalent ions at high temperature leads to strong reduction of HPAM polymer solution viscosity, but the viscosity is better maintained for PAMS copolymer solution at high temperature. The precipitation of HPAM first occurred after 3 months at 80°C and for PAMS copolymer with lowest sulfonation degree precipitation started after 7 months. For the studied polymers the retention was found to be relatively independent of temperature and compared to HPAM a much lower retention is observed for the sulfonated copolymers. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 3623–3629, 2011

**Key words:** sulfonated polyacrylamide polymers; HPAM; thermal stability; retention; sacrificial agents

## INTRODUCTION

The main purpose of using polymer in enhanced oil recovery is to increase the viscosity of water.<sup>1,2</sup> In this respect maintaining the viscosity of aqueous polymer solution in harsh environment conditions like high temperature and/or high salinity is a challenge.

Another parameter of importance for application of polymers for water flood mobility control is to minimize the loss of polymer due to adsorption to the rock, and more general retention during flow in porous medium. Results of static adsorption and retention at high temperature of HPAM and PAMS copolymers have shown that at high temperatures the adsorption and retention for both polymers increases moderately.<sup>3</sup>

The polymer degradation processes is classified by several elements such as chemical degradation, mechanical degradation, and biological degradation, which is the significant degradation process with

biopolymer e.g., Xanthan.<sup>4,5</sup> Chemical degradation is mainly due to the effect of oxygen and oxidation reactions is accelerated at high temperature. The effect of high temperature may increase polymer hydrolysis degree and also precipitation process with divalent ions present. These mechanisms, especially degree of hydrolysis, have been studied by other authors for different types of polyacrylamide-based polymers like PAM, HPAM, copolymers of polyacrylamide with some cationic and anionic comonomers, e.g., sulfonated polyacrylamide polymers (PAMS), and hydrophobically associating polymers.<sup>2–4,6–15</sup> Degree of hydrolysis can also affect rheological parameters.<sup>7</sup> Study of degree of hydrolysis effect on relative viscosity of polyacrylamide polymer solution, showed that in fresh water and NaCl solutions, polyacrylamide polymers with high degree of hydrolysis (range up to 35 mol %) display the most viscous solutions.<sup>16</sup> The reason for this is that the electrostatic repulsion of the  $-\text{COO}^-$  group makes the molecular chains of HPAM extend and thereby increase the solution viscosity.<sup>17</sup> Increased degree of hydrolysis may also lead to polymer precipitation in the presence of divalent ions.<sup>7</sup> At higher divalent ion concentrations electrostatic attraction between divalent cations and carboxylate groups increases and precipitation also may occur.<sup>11</sup>

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For sulfonated copolymers of PAM, in addition to the hydrolysis of amide group, the AMPS group also may hydrolyze at temperature higher than 100°C.<sup>6,8,13</sup> Hydrolysis of PAMS group occurs above 120°C at pH 8, and at lower pH the rate of hydrolysis will increase, e.g., at pH 6 the rate of hydrolysis is five times faster than pH 8. This is due to the net negative charge on the polymer, which is greater at pH 8.<sup>8</sup> Results for introducing PAMS group into the polyacrylamide polymer molecular structure improve stability of polyacrylamide at least up to 120°C. But at even higher temperature, more than 120°C, PAMS can not protect acrylamide against thermal hydrolysis.<sup>6,8,10</sup>

Different authors have studied the viscosity of polyacrylamide polymers in anaerobic conditions, with and without oxygen scavenger and sacrificial agent and some other additives.<sup>8-14</sup> In the presence of divalent ions, a maximum temperature for use of HPAM is reported to be about 75°C.<sup>10</sup> There is a need for modified synthetic polymers that can maintain viscosity at high temperature in presence of divalent ions. In this respect, one possibility is to replace acrylate monomer of HPAM with other monomers either nonionic monomers, e.g., *N*-Vinyl Pyrrolidone (NVP) and diacetone acrylamide (DAAM), or anionic comonomers, e.g., sodium 2-acrylamide-2-methylpropane sulfonate (AMPS).<sup>6,7,10-15</sup>

Our previous publication<sup>18</sup> about viscosity study of sulfonated polyacrylamide include a range of sulfonation degree, from 5 to 32 mol %. The results showed that PAMS with high sulfonation degree are more salt tolerant and divalent ions have less effect on viscosity for PAMS copolymer compared to HPAM. Further the solubility of PAMS copolymers in brine with divalent ion present, was found to improve for polymer with higher sulfonation degree.<sup>18</sup>

Alcohols can prevent oxidization of the polymer, because the alcohol can be easily oxidized and thereby as sacrificial agent protect the polymer against oxidization.<sup>15</sup> The purpose of the sacrificial agent is mostly for transport and handling of the polymer, while the oil reservoir is regarded mainly as a closed and anaerobic environment. Several authors have used different kinds of alcohols, but IBA showed better protection results of the viscosity of polyacrylamide-based polymers.<sup>15,19,20</sup> Oxygen solubility in water equals  $\sim 2 \times 10^{-4}$  mol L<sup>-1</sup><sup>21</sup> and several percents of IBA is able to eliminate all of this oxygen and to protect the polymer from oxidation.

One of the objectives in this work is to quantify the effect of IBA on the polymer solution stability, in terms of viscosity, at 80°C. Furthermore, the effect of sulfonation degree and molecular weight on the viscosity of polymer solution at the aforementioned conditions, have been studied.

## EXPERIMENTAL

### Materials

#### Polymers

Six sulfonated polyacrylamide polymers with different sulfonation degree and molecular weight, and HPAM 3330s with 25–30 (mole %) hydrolysis and 8 MDa molecular weight, were used in this research. The characteristics of the studied polymers are given in Table I. All the polymers used were supplied by SNF Floerger.

#### Bulk solutions

The solvents used were 5 wt % NaCl in water, and to study the effect of divalent ions simplified seawater with contribution of the main ions (SSW), was made and used. The composition of SSW is given in Table II. The temperature here is 80°C.

#### Adsorbent

Retention was studied by injecting polymer solution into Berea sandstone cores mounted in core holder kept in an oven at 80°C. The length of these cores was around 10-cm long and 3.7 cm in diameter. The permeability was around 500–700 mD, and the porosity  $\sim 20\%$ .

### Method and measurements

#### Solution preparation

The 5 wt % NaCl solution or SSW was stirred by propeller stirrer at 720 rpm and dry polymer powder was slowly introduced into the side of the vortex to avoid formation of fisheyes which can be formed if the powder is not wetted evenly. The solution was then stirred slowly for almost 90 min to ensure complete dissolution. A standard polymer solution (5000 ppm) was made in the two different solvents with the same ionic strength, 5 wt % NaCl and SSW. IBA was added with different concentrations ranging from 0.1 to 5 wt %. The prepared sample with 5000 ppm polymer concentration was then put inside 8 mL sample vials with black caps with PTFE/faced liner inside and sealed well before putting them inside oven at 80°C.

#### Viscosity measurement

The viscosity measurement was performed by using a rotary rheometer, Anton Paar Physica MCR 300, with parallel plate system for these with low viscosity (e.g., viscosity less than 10 mPa s<sup>-1</sup>).

#### pH measurement

pH was measured at ambient temperature by using Orion 960 autochmeistry pH meter.

**TABLE I**  
Polymers Characteristics

Polymer <sup>a</sup>	Molecular weight (Million Dalton)	Sulfonation degree (Mole %)
AN105	6	5
AN113	8	13
AN125VLM	2	25
AN125	8	25
AN125VHM	12	25
AN132	8	32
HPAM	8	–

<sup>a</sup> All studied polymers were supplied by SNF FLOERGER.

#### Retention measurement at 80°C

For retention test at high temperature the cores were mounted in a core holder and saturated with the appropriate solvent (SSW) by using a vacuum pump. The core holder and the two cylinders filled up with polymer solution and solvent were placed in the oven at 80°C for 1 day. After being aged for 1-day injection of two pore volumes of the polymer solution started, and it was post flushed by three pore volumes of the solvent. Produced effluent was collected in another cylinder which was outside at ambient temperature. During the injection because of the expansion of the core holder at high temperature, which would increase the sleeve pressure, the core holder was connected to a back pressure regulator to keep the sleeve pressure constant. At the end of injection the polymer concentration was measured and then adsorbed amounts were calculated by subtracting the total amount produced from the total amount polymer was injected.

#### Concentration measurements

To measure concentration the starch triiodide method as described by Scoggins and Miller was used.<sup>22</sup> Starch forms a color complex with iodine and the absorbance amount at peak wavelength, 568 nm, which can be detected by using a UV-vis spectrometer. This method gives a linear response in a range of 0–10 ppm.

**TABLE II**  
Synthetic Seawater Composition Containing the Main Ions with the Same Salinity of Seawater—Grams of Salts in 1 kg Solvent

Salt	Amount in g kg <sup>-1</sup> solvent
NaCl	25.0
MgCl <sub>2</sub> ·6H <sub>2</sub> O	11.1
CaCl <sub>2</sub> ·2H <sub>2</sub> O	1.7
KCl	0.7

#### Hydrolysis degree determination

There are different methods to determine degree of hydrolysis, e.g., potentiometric, conductometric titration, colloidal, and spectroscopy methods, e.g., C<sup>13</sup> nuclear magnetic resonance.<sup>23,24</sup> Here in this research, to measure degree of hydrolysis for sulfonated polyacrylamide polymers and HPAM potentiometric titration was used. Polymers concentration must be known to use this method, and a starch triiodide method was used, as explained before. Also for potentiometric titration an 809 series Titrand from Tiamo titration and More company have been used. To remove impurities polymer solutions were dialyzed by dialysis tube which is a semi permeable membrane tube.

## RESULTS AND DISCUSSION

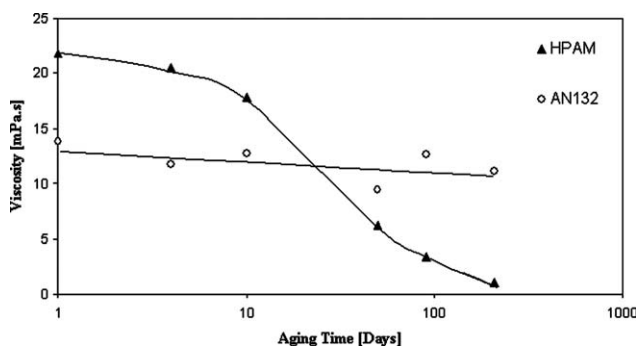
#### Aging time dependence of polymer viscosity at 80°C

Under anaerobic condition without divalent ions in the brine, and without sacrificial agents, HPAM as well as PAMS polymer with 25 mol % sulfonation degree are reported to be stable, in terms of viscosity, at 120°C for up to 2 years.<sup>12</sup> In the presence of divalent ions the maximum temperature at which HPAM can be effective is about 75°C.<sup>10</sup>

In this work, all test and measurements were performed under aerobic condition. These studied have been performed both, with and without added IBA. The viscosity results of sulfonated polyacrylamide polymers and HPAM being aged at 80°C for 90 days without IBA present are shown in Table III. As can

**TABLE III**  
The Viscosity of Sulfonated Polyacrylamide Polymers and HPAM Under Aerobic Condition at 80°C, 5000 ppm Polymer Concentration, Without IBA, at 100 s<sup>-1</sup> Shear Rate

Polymers Solvent	Initial viscosity (ambient temp.) (mPa s <sup>-1</sup> )		1 day		7 days		90 days	
	5 wt % NaCl	SSW	5 wt % NaCl	SSW	5 wt % NaCl	SSW	5 wt % NaCl	SSW
HPAM	33.5	24.6	14.2	8.0	4.4	1.2	0.8	0.5
AN105	32.6	29.7	16.0	10.3	3.9	2.3	0.9	1.1
AN113	28.6	27.9	10.7	8.5	4.4	1.9	0.8	0.7
AN125	27.1	27.0	6.1	5.6	4.7	1.7	2.0	1.2
AN132	27.4	24.6	7.19	5.1	5.7	2.8	1.2	0.9

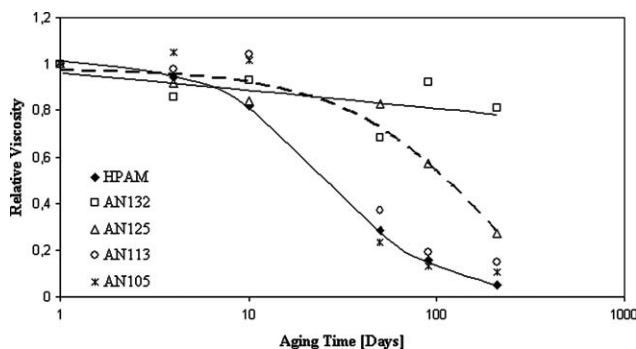


**Figure 1** Viscosity as a function of aging time for HPAM and AN132, at 80°C, 5 wt % NaCl included 3 wt % IBA, and at 100 s<sup>-1</sup> shear rate.

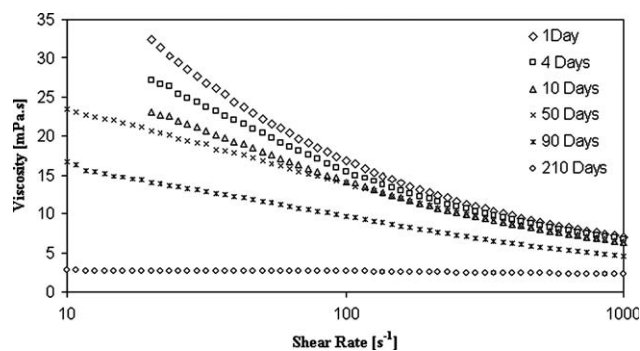
be seen, all the polymers more and less loose their viscosity after 90 days without IBA present. Further experiments that will be presented are with IBA present.

Aging time dependence of viscosity for the sulfonated polyacrylamide polymers and HPAM is shown in Figure 1. As seen in Figure 1, the viscosity of HPAM solution decreases abruptly as function of aging time, but for the sulfonated polyacrylamide polymer with highest sulfonation degree, AN132, the viscosity is more and less independent of time within studied time scale.

A comparison of the relative viscosity, i.e., the viscosity at different aging time divided by the viscosity after 1 day at 80°C, of the sulfonated polymers and HPAM is shown in Figure 2. As results show, the relative viscosity for the polymers with lower sulfonation degree, AN105 and AN113, behave in the same manner as HPAM polymer solution, but for the polymers with higher sulfonation degree, AN125 and especially AN132, the viscosity is more stable. This shows that introducing the large amount of PAMS group into the polyacrylamide polymer molecular structure may increase polymer stability limit, in terms of viscosity at least up to 80°C.<sup>6,8,10,13</sup>



**Figure 2** Relative viscosity ( $\frac{\eta}{\eta_{1\text{day}}}$ ) as a function of aging time for different sulfonated copolymers and HPAM, at 80°C, 5 wt % NaCl included 3 wt % IBA, and at 100 s<sup>-1</sup> shear rate.



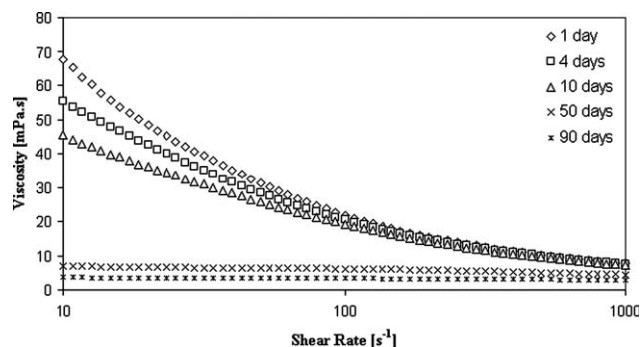
**Figure 3** The viscosity as a function of shear rate for AN125 being aged up to 210 days at 80°C, and at 5 wt % NaCl included 3 wt % IBA.

Shear viscosity was measured at 80°C on polymers from low to high shear rate and *vice versa*. For all solutions studied, no shear rate hysteresis was observed even at high temperature.

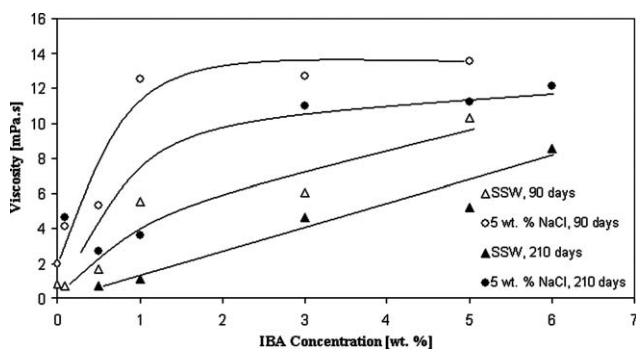
Aging time may change the fluid viscosity behavior, as results show in Figure 3 for AN125, which qualitatively illustrate the results observed for all other polymer. Similarly Figure 4 shows results for HPAM. As seen in Figures 3 and 4 the viscosity behavior changes from shear thinning, i.e., viscosity decreases when shear rate increases, to the Newtonian behavior, i.e., the viscosity is shear-independent. For HPAM it only takes 10 days to induce this transition in viscosity behavior, while for AN125 it takes about 7 months before we observe viscosity behavior changes toward Newtonian behavior.

#### Effect of IBA concentration on the viscosity

Without IBA present all the studied polymers loose viscosity after 90 days at 80°C, as already shown by results given in Table III. In Figure 5, the effect of IBA concentration on the polymer stability, in terms of viscosity, for the polymer with the highest sulfonation degree, AN132 at 80°C is shown. In all cases, viscosity is found to increase upon addition of IBA.



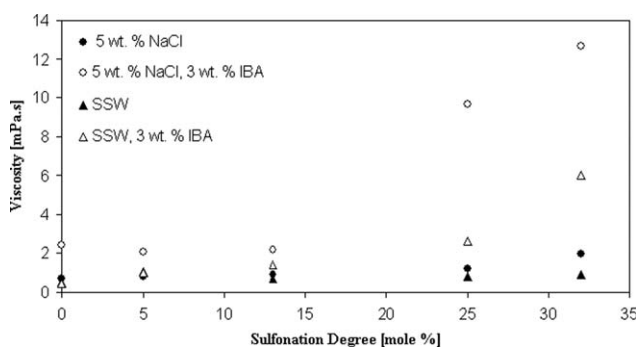
**Figure 4** The viscosity as a function of shear rate for HPAM exposed 90 days at 80°C and at 5 wt % NaCl included 3 wt % IBA.



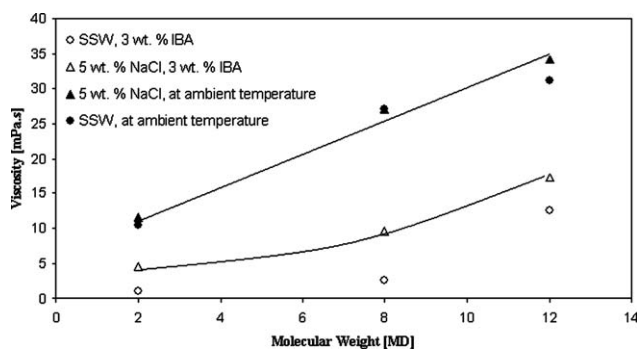
**Figure 5** The viscosity of AN132 as a function of IBA concentration for two different aging times at 80°C, SSW and 5 wt % NaCl, and at  $100 \text{ s}^{-1}$ .

In 5 wt % NaCl solution, the viscosity stabilizes and levels off at about 3 wt % IBA concentration. Study by others have only been made for a fixed concentration of IBA, for example, Willington, has studied the effect of adding 3 wt % IBA, and in our case this concentration seems to be effective for polymer stability, in terms of viscosity.<sup>15</sup> In the brine containing divalent ions (SSW) the viscosity is increasing with IBA concentration for the range in concentration tested. Thus, the highest IBA concentration is the best choice for achieving a maximum solution viscosity in solvent with divalent ions (SSW).

To investigate the impact of the sulfonation degree on the stability of the polymer solution viscosity a comparison was made between the studied polymers at a fixed IBA concentration, 3 wt %. The results are presented in Figure 6 where also the viscosity without IBA added is shown. As seen, high viscosity is achieved for the polymers with sulfonation degree more than 25 mol % in 5 wt % NaCl solvent. Although, the results in Figure 6 are for viscosity after 90 days, Figure 2 shows this also may apply after 1 year for the polymers with higher sulfonation degrees, AN125 and AN132.



**Figure 6** The viscosity as a function of sulfonation degree exposed 90 days at 80°C, for the polymers with the same molecular weight, e.g., 8 MD, with (3 wt %) and without IBA, at SSW and 5 wt % NaCl, and at  $100 \text{ s}^{-1}$ .



**Figure 7** Viscosity as a function of molecular weight for the polymers with the same sulfonation degree, 25 mol %, exposed 90 days at 80°C and ambient temperature,<sup>18</sup> at 5 wt % NaCl and SSW, and at  $100 \text{ s}^{-1}$  shear rate.

With divalent ions present, the difference in viscosity is not very big, but still we have an increase in viscosity particularly for the polymer with the highest sulfonation degree, AN132.

Figure 7 shows the results of the effect of molecular weight on the viscosity changes for polymers with the same sulfonation degree (25 mol %), in SSW and 5 wt % NaCl solvents. This is a comparison of fresh polymer solutions viscosity at ambient temperature,<sup>18</sup> with the viscosity of polymer solutions exposed for 3 months at 80°C. As shown in Figure 7, in NaCl solvent, viscosity loss is higher in absolute values for the polymer with higher molecular weight. Similar observation with a larger effect for high MW polymers is also seen for solvent with divalent ion presence (SSW).

#### Divalent ions effect on polymer viscosity at 80°C

Recently, results have been reported that HPAM polymer solutions being aged for 2 years at 120°C in the solvent with no divalent ions, and under anaerobic condition can keep their viscosity. Similar results are observed for sulfonated polyacrylamide polymers with 25 mol % sulfonation degrees.<sup>12</sup> HPAM polymers are well known to be unstable at higher temperature with divalent ion presence.<sup>11,12,25</sup> For temperature above 70°C acrylamide group in HPAM hydrolyses, and changes to acrylate group. At high degree of hydrolysis HPAM are found to precipitate with divalent ions present ( $> 2000 \text{ mg L}^{-1}$ ) at 75°C.<sup>11</sup> For PAMS copolymer with 25 mol % sulfonation degree no precipitation has been observed at the same condition.<sup>6,7,10-15</sup>

In this work, at 3 wt % IBA concentration, with divalent ions present, precipitation has been observed for HPAM after 3 months and similarly for the polymer with the lowest sulfonation degree (AN105) after 7 months at 80°C. No precipitation was observed for the other sulfonated copolymers even after a year exposed to 80°C in SSW. In 5 wt %

**TABLE IV**  
Degree of Hydrolysis (%) at Ambient Temperature and for the Polymers Been Aged for 7 months at 80°C at SSW, with Measured Polymer Concentration of 1500 ppm

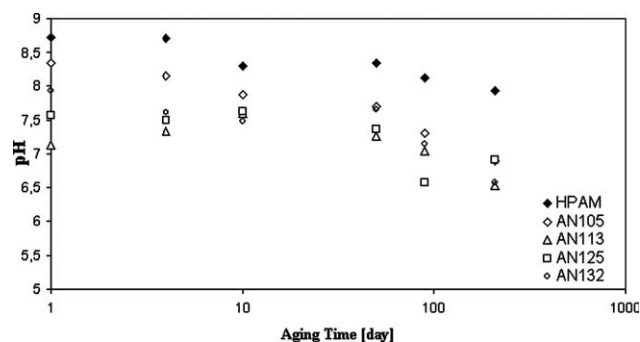
Polymers	Ambient temp.	At 80°C after 210 days
HPAM	33	65
AN105	25	55
AN113	24	46
AN125	27	45
AN132	22	42

NaCl solvent, no precipitation was observed for all the sulfonated copolymers, but HPAM was found to precipitate after 7 months.

As shown in Figures 5–7, the decrease in polymer solution viscosity at 80°C for HPAM and the PAMS copolymers at 5 wt % NaCl and at SSW is different. If the sulfonation degree increases (Fig. 6), in both 5 wt % NaCl and SSW solvents, then the effect of temperature on the viscosity loss of the polymers decrease. However, the difference is not very strong for the polymers in SSW solvent. As shown in Figure 7, the absolute value of the viscosity loss is higher for the polymer with higher molecular weight for both SSW and 5 wt % NaCl with a larger effect at SSW solution.

#### Hydrolysis of the sulfonated polyacrylamide polymers and HPAM

Table IV shows the degree of hydrolysis of the sulfonated polyacrylamide polymers and HPAM, been exposed 7 months at 80°C. As mentioned above, no precipitation was observed for the polymers with a higher sulfonation degree than 5 mol %, being exposed up to 1 year at 80°C. This means that precipitation is not a major cause for viscosity loss of PAMS copolymers. Therefore the major reason for viscosity loss of PAMS copolymers seems to be degree of hydrolysis. Based on the results, the hydrolysis degree of these polymers, is directly dependent on the sulfonation degree, and this is in line with measurements by other authors for sulfonated polyacrylamide polymers.<sup>6,25</sup> The results confirm that if sulfonation degree increases then the rate of hydrolysis decreases and levels off.<sup>6</sup> This can be explained due to the electrostatic effects, and strong repulsion between the charges on the poly-



**Figure 8** pH variations as a function of aging time for sulfonated polyacrylamide polymers and HPAM at SSW with 3 wt % IBA, exposed at 80°C.

mer chain and hydroxide anion.<sup>6,11,25</sup> Chemical additives and pH, also have important effects on the chemical degradation and degree of hydrolysis.<sup>7,20,26</sup> In our study, the pH of the solutions being aged at 80°C with IBA was monitored and the results are shown in Figure 8. As shown in Figure 8, pH was in the range of 6–8. In this range, pH has no significant effect on the hydrolysis degree.

#### The effect of temperature on the retention of the polymers

In the dynamic adsorption experiments at ambient temperature, a slug of 2 PV polymer solution flows through a cylindrical Berea sandstone core, followed by a 3 PV post flush of SSW or 5 wt % NaCl solutions. The concentration is measured from samples collected at the outlet of the core. Polymers may be retained in the core by other means than adsorption, for example trapping in small pore throats. Additionally, there is a constant shearing of the polymers due to flow, which may affect the adsorption process. The results of retention at ambient temperature show that with the increasing sulfonation degree, less polymer molecules are retained in the core. The level of retention is very low for AN125VLM, AN125, AN125VHM, and AN132, with HPAM having the highest retention.<sup>27</sup>

For retention experiments at 80°C after injecting polymers into the Berea sandstone core, polymer concentration and viscosity remained practically unchanged in the effluent as before injection. As the results show, in Table V, increasing temperature gives a small increase in the retention of these sulfonated

**TABLE V**  
The Retention of Polymer in Berea Sandstone at Ambient Temperature (20°C) and 80°C

Polymers	Retention ( $\mu\text{g g}^{-1}$ )						HPAM
	AN105	AN113	AN125VLM	AN125	AN125VHM	AN132	
SSW at 20°C	111.5	58.7	39.0	–	3.3	16.5	124.2
SSW at 80°C	111.7	60.0	46.8	–	10.5	20.0	139.0

polyacrylamide polymers and of HPAM. The retention either at ambient<sup>27</sup> or high temperature, of the sulfonated polyacrylamide is found to be less than the retention of HPAM. The results of this research confirm reported results from other studies of retention with the polymer with 25 mol % sulfonation degree at two different temperatures, e.g., 90 and 100°C,<sup>3,27</sup> which shows that the amount of retention at these two temperatures are the same. Generally temperature does not seem to affect the retention of these sulfonated polyacrylamide polymers.

### CONCLUSIONS

The main conclusions of this article on the behavior on viscosity and retention of sulfonated polyacrylamide polymers (sulfonation degree from 5 to 32 mol %) and HPAM at 80°C and aerobic condition are as below:

The presence of the sacrificial agent, IBA, can increase the thermal stability, in terms of viscosity, of sulfonated polyacrylamide polymers. For the studied polymers the viscosity is better maintained as a function of time for the polymer with higher sulfonation degree. Good results were obtained with 3 and 5 wt % IBA concentration in both 5 wt % NaCl, and SSW solvents.

For the polymer solution with IBA, at high temperature the presence of divalent ions lead to strong reduction of HPAM solution viscosity, but the viscosity is better maintained for PAMS copolymer.

Precipitation has been observed for HPAM which precipitated after almost 3 months, and also for PAMS copolymer with the lowest sulfonation degree (5 mol %) which precipitated after 7 months of aging at 80°C.

High temperature increases retention for both the sulfonated polyacrylamide polymers and HPAM, but only to minor extent. With regard to EOR applications, the effect of temperature on retention of polymer in porous medium thus seems not to be a critical issue for the polymers tested.

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