Experimental investigation of effect of salts on rheological properties of non-Newtonian fluids

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ABSTRACT

The interaction between different polymers and brines is very common in some stages of the drilling process. The effects of salts on polymer solutions were studied at two different temperatures, $21°C$ and $42°C$. Poly-Anionic Cellulose (PAC) solutions with different concentrations were applied to analyze the rheological hysteresis and also identifying the base fluid for further experiments with brines. It was found that with increase of polymer concentration the liquid became more shear thinning and more stable over time and also that the rheological hysteresis decreased. A dominating role of the effect of metal ions on viscosity of polymer solutions was confirmed. Brines were found to have a strong influence on the rheology of PAC solutions, and transition to Newtonian behavior was observed in some cases for low salt concentrations at ambient temperature, and for all tested solutions at $42°C$.

INTRODUCTION

Water based drilling mud has become more and more common for industrial applications. Such muds get increasingly complex, containing a substantial amount of different additives such as viscosifiers, shale inhibitors and weighting agents. Shale inhibitors are introduced in drilling operation to avoid swelling of shale formations. Mud engineers nowadays have a possibility to precisely regulate viscosity, density, surface tension and other properties of mud by a large number of different components and their combinations. This paper aims to investigate the effects of salts on polymer solutions at two different temperatures, $21°C$ and $42°C$. The interaction of polymer solutions and different brines can happen in different stages of the drilling process. One example of this is drilling mud components mixing with formation water influxes. The effects of brines depend on its ionic composition, (mostly on cation valence, e.g. if it is mono-, di- or trivalent cations), salt concentration, temperature and also on the type of the polymer they interact with[1, 2].

For this purpose Poly-Anionic Cellulose (PAC) solutions with concentrations of 0.1-0.4 $wt\%$ were applied to analyze the rheological hysteresis. The PAC 0.4 $wt\%$ were identified as a base fluid to be mixed with $CsCOOH$, $KCOOH$, $NaCl$ brines of typical industrial concentrations for running further experiments and studying the effects of these salts on a polymer solution.

Poly-Anionic Cellulose

PAC is a semi-natural anionic Sodium Carboxy-Methyl Cellulose (CMC), with a high degree of substitution, high uniformity and high quality. Due to these qualities it is usually referred to as a premium quality additive[3, 4]. PAC is one of the most commonly used polymers[5] in drilling and completion fluids with a wide range of total solid content, salinity and pH. Small concentrations of PAC provide efficient reduction of filtration rates and good rheology stabilization[3].

The PAC molecular formula is $[C_6H_7O_2(OH)_2OCH_2COONa]_n$ and the structural formula is shown in Figure 1.

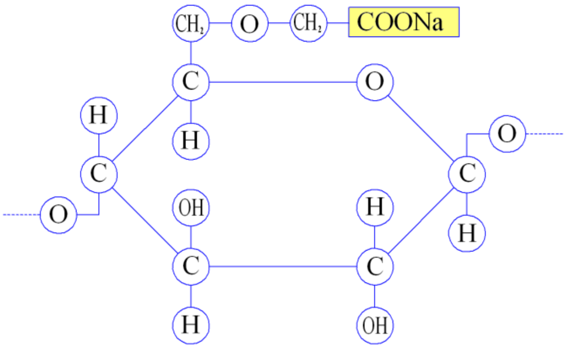

Figure 1. Structural formula for PAC[6].

PAC is a non-toxic, odorless, white or yellowish powder that is soluble in both hot and cold water. Water-PAC mixtures are viscous, transparent and tend to have neutral pH (6.0-8.5)[7].

Brines

Formate brines are widely used in the industry, mostly during reservoir drilling and completion stage, and also as weight material. The effect of Formate brines on polymers is considered to be an interesting study topic. A number of scientific works have been dedicated to investigation of the effect of Formate brines on Xanthan gum (Xg) solutions. In particular, significant increase of melting temperature and thermal stability of Xg has been reported [8, 9]. However, PAC-Formate / brine interaction has been poorly investigated. From literature and suppliers' catalogues[10], it was concluded that Potassium and Cesium Formates and their combinations were mostly used in industry in concentrations higher than 50 $wt\%$.

Sodium Chloride ($NaCl$) is also a very common drilling mud additive[11], mostly used as an active shale inhibitor, a gas hydrate formation minimizer, and a bridging agent for lost circulation in saturated salt systems, but rarely as a weight material[12, 13]. The typical concentration of $NaCl$ is about 3-8 $wt\%$.

Therefore experimental concentrations were defined to be 20 $wt\%$ and 60 $wt\%$ for Potassium-Formate ($KCOOH$), 7.6 $wt\%$ and 60 $wt\%$ for Cesium-Formate ($CsCOOH$) and 5 $wt\%$ for Sodium chloride ($NaCl$).

THEORY

This section provides general information about tested mixtures, including functions, typical concentrations and why they were chosen.

Water

The understanding of polymer-brine water mixtures could hardly be achieved without understanding properties of water as a dispersing medium. Water in general, and especially liquid water is a unique substance that can behave as an atomic, ionic, and molecular material. These types of behaviors may be defined by domination of covalent, electrostatic, and dispersion forces, respectively.

Two different co-existing structures could be distinguished in liquid water: polymeric and monomeric. The polymeric portion is represented by molecules connected with hydrogen bonding with spatial structuring, while monomeric is more or less separate water molecules, filled in between a loose 3D "grid" of the polymeric structures [14]. The proportions of the monomeric and polymeric parts are temperature dependent, with a strong increase of polymeric structure close to the freezing point, and predominantly monomeric at higher temperatures.

Shear thinning

Shear thinning behavior of PAC solutions may be explained by the behavior of the polymer macromolecules, as shown in Figure 2.

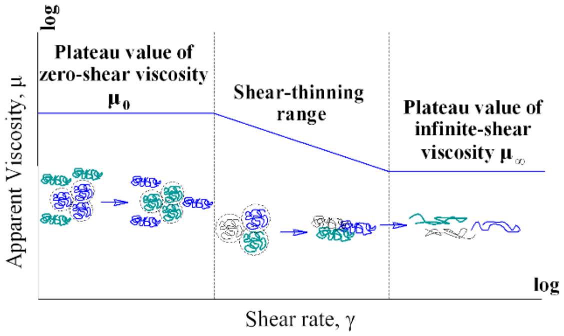

Figure 2. Shear thinning polymer macromolecules behavior[15].

At the state of rest, when no shear is applied, some molecules have a complex entangled spherical form, while others are partially disentangled. When a low shear is applied, the molecular collisions increase the viscosity, while for entangled molecules collisions contribute to molecular disentanglement. They become oriented in the shear direction and thus decrease the viscosity. The overall viscosity, however, remains the same. This situation is often referred in literature as a plateau value of zero shear viscosity (μ_0). With further increase of shear rate, disentanglement and orientation in the shear direction happens for more and more molecules, and resistance to shear and viscosity decreases. After reaching a certain shear rate value, the population of macromolecules achieve a state of maximum disentanglement. An additional increase of shear rate will not cause the viscosity to decrease. This is called plateau value of infinite shear viscosity [15]. From a chemical point of view, such behavior of macromolecules is caused by hydrogen bonding, acting between parts of a molecule, different molecules, and finally between water and polymer. Sarafutdinow et al. [14] applied a set of viscosity measurements on different polymer additives and concluded that for PAC, hydrogen bonding is equally distributed in volume and the observed pseudo-plastic behavior could be explained by fast recovering of broken hydrogen bonds.

Hydration

In general, the effects of salts on water structure are mostly defined by effects of cations. There are, however, some exceptions. Some ions like K^+, Cs^+, Cl^- interact with water molecules in the polymeric part of the water "grid" with the effect of distorting the structure. This effect is called negative hydration. Ions like Na^+, SO_4^{2-}, OH^-, Ca^{2+}, on the other hand, interact with the monomer part of the water e.g. with free water molecules, by creating hydrate envelopes around ions, decreasing the activity of the free water molecules, and hence protecting the water grid from destruction. This effect is called positive hydration. However, by increase of salt concentration in water, the water grid will be fully destroyed [14].

EXPERIMENTAL

This section will describe the mixing procedure, experimental setup, and the test matrix tested in this study.

Mixture preparation

Preparation of experimental mixtures was conducted in the following way: at first, aqueous solutions of polymer and salt were prepared separately, and then precise controlled volumes of these solutions were mixed. In this way a best mixing result was ensured, while also keeping track of the polymer concentration. This procedure was implemented based on published literature with intention to let both polymer and salts face all possible processes (e.g. hydration, water molecules orientation, hydrogen bonding) separately and completely before mixing. Otherwise, for example in case of adding the polymer powder into brine, there was a possibility of incomplete hydration of PAC macromolecules, because of all or most of the free-water molecules engaged by salt ions.

PAC mixture preparation was conducted according to the Kelco CMC manual[6]. Figure 3 illustrates the mixing technique.

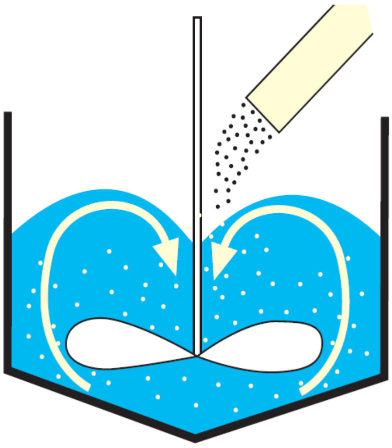

Figure 3. PAC dissolution technique [6].

A pre-calculated amount of PAC powder was carefully and evenly added to deionized water in the mixing facility in order to permit all particles to become individually wetted. Also it was crucial to keep the rotation speed of the agitator high during mixing in order to keep the viscosity low. The rotation speed was 3000 rpm during mixing and then decreased to 2000 rpm for 30 $minutes$. Afterwards, the solutions were placed in a dark place at ambient temperature 21°C for 48 $hours$. This settling also allows for eventual small entrained air bubbles to separate out.

Brine solutions were obtained from Potassium-Formate (75 $wt\%$, 1.578 $s.g.$), Cesium-Formate (82 $wt\%$, 2.248 $s.g.$), and mixing of dried powder of Sodium Chloride salt in deionized water. The decision to use only monovalent salts was based on literature investigation[2, 16, 17]. Also, a number of trial attempts to prepare PAC and $CaCl_2$ mixture resulted in salt precipitation even for $CaCl_2$ concentration less than 5 $wt\%$.

After the sequence of calculation and dilution steps, five testing mixtures were prepared. Table 1 shows the test fluids and the test matrix in this study. So called base fluids were also prepared in order to achieve reference values (Table I.1 in Appendix I). It should be noticed that 60 $wt\%$ $KCOOH$ brine mixed with 60 $wt\%$ $CsCOOH$ brine, and also 5 $wt\%$ $NaCl$ brine mixed with 7.6 $wt\%$ $CsCOOH$ brine have approximately similar molar concentrations of metal ions.

The rheology model used for curve fitting was a power-law model ($\mu = \kappa\dot{\gamma}^{n-1}$) where $\dot{\gamma}$ is the shear rate. The consistency factor (κ) and the power-law index (n) are calculated by using the curve fitting model in Matlab software and represented for each solution in Table 1. The density of the fluids was measured by density measuring module (DMA 4500) – Anton Paar.

Experimental methodology and set up.

Rheology measurements were conducted by means of Anton Paar MCR 302 apparatus (see Fig. 4). A Concentric Cylinder modification (CC27) was used because of higher measuring accuracy and minimal liquid evaporation. That was extremely important due to the considerable time required for running some of the experiments more than 7 hours. It was decided to set the shear rate ramp up as a logarithmic sequence and use a fixed number of measurement points in each shear rate decade interval. The measuring point duration was also changing logarithmically from 600 sec at the lowest shear rate to 5 sec at the highest shear rate.

All experiments were conducted at ambient pressure. There was an attempt to conduct tests at 55 bar by using a pressure cell modification, but based on obtained results it was considered less successful.

Table 1. Test Matrix

Fluid name	**Fluid composition**	**Density ($s.g.$)**	**pH**	**Apparent viscosity**		**Temp [°C]**
				K	**n**	
Base Fluid	PAC 0.4 $wt\%$	1.0017	6.37	0.175	0.79	21
Fluid A	PAC 0.4 $wt\%$ and $KCOOH$ 20 $wt\%$	1.1217	7.6	0.028	0.98	21
Fluid B	PAC 0.4 $wt\%$ and $KCOOH$ 60 $wt\%$	1.4303	9.0	0.038	0.98	21
Fluid C	PAC 0.4 $wt\%$ and $CsCOOH$ 60 $wt\%$	1.7214	11.2	0.012	1	21
Fluid D	PAC 0.4 $wt\%$ and $NaCl$ 5 $wt\%$	1.0376	5.0	0.035	0.97	21
Fluid E	PAC 0.4 $wt\%$ and $CsCOOH$ 7.6 $wt\%$	1.0602	9.9	0.018	0.99	21
Base Fluid	PAC 0.4 $wt\%$	-	-	0.053	0.95	42
Fluid B	PAC 0.4 $wt\%$ and $KCOOH$ 60 $wt\%$	-	-	0.015	1	42
Fluid C	PAC 0.4 $wt\%$ and $CsCOOH$ 60 $wt\%$	-	-	0.007	1	42
Fluid D	PAC 0.4 $wt\%$ and $NaCl$ 5 $wt\%$	-	-	0.013	1	42
Fluid E	PAC 0.4 $wt\%$ and $CsCOOH$ 7.6 $wt\%$	-	-	0.006	1	42

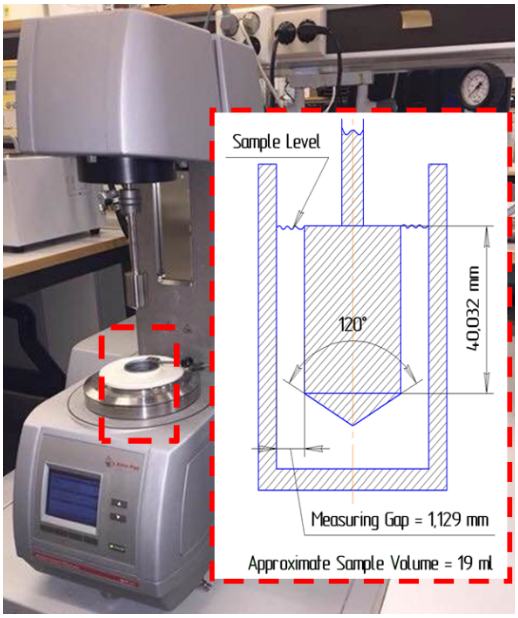

Figure 4. Rheometer set up with measuring modification dimensions [18].

ANALYSIS

The results of rheology tests are presented in this section to characterize the rheological hysteresis of PAC solutions and analyze the effects of ions on the polymer chain.

Hysteresis analysis.

Hysteresis analysis was performed on PAC solutions with different polymer concentrations. Another target was to define the base fluid for further experiments with brines. Figure 5 represents the results for PAC 0.1-0.4 $wt\%$. It indicates that hysteresis is decreasing with increasing shear rate.

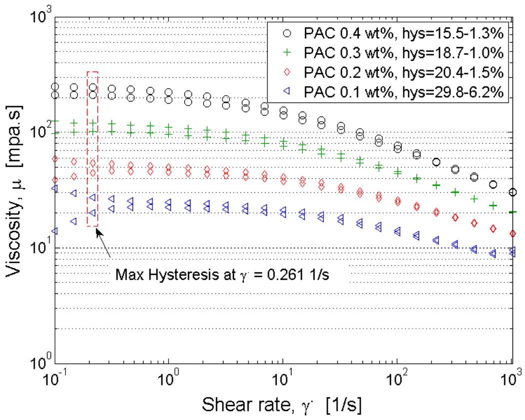

Figure 5. Hysteresis of PAC mixtures with different polymer concentration.

A representative hysteresis value ("hys") was calculated for every shear rate step using the following equation:

$$Hysteresis(\%) = \frac{|\mu_{max} - \mu_{min}|}{\mu_{avg}} \cdot 100 \qquad (1)$$

The hysteresis calculations were conducted for datasets containing 8 sequential measurement series.

For low shear rates the possibility of error is high because of experimental noise and also lower accuracy of measurement related to very small angles of torque and angular displacement. For this reasons the comparison between hysteresis values for PAC mixtures with different polymer concentrations began first from the third shear rate step at 0.216 $1/s$.

PAC 0.4 $wt\%$ had the lowest hysteresis effect of 15.5% at low shear rate, while PAC 0.1 $wt\%$ had the highest. The higher the polymer concentration, the higher stability the system achieves.

From Table 1, PAC 0.4 $wt\%$ has the lowest power-index ($n = 0.79$) which means the shear thinning was more pronounced than that for other PAC solutions with lower polymer concentration. It is also shown in Figure 5 that decrease in viscosity is much higher for higher PAC concentrations at the same shear rates.

However, using PAC 0.4 $wt\%$ with higher degree of shear thinning and higher overall stability, it is possible to measure the effect of salts on PAC solution more precisely and consistently. Based on this, the PAC 0.4 $wt\%$ was considered as a base fluid.

Effects of brines on PAC solution $T = 21°C$

Experiments conducted on mixtures at 21°C are shown in Figure 6. Different brines with different concentrations were tested at ambient pressure and temperature $T = 21°C$. Instead of mass concentration, molar concentration is used for further analyses, since this is a better representative value (Appendix I, Table I-1).

A decrease of apparent viscosity was observed for all tested fluids compared to base fluid. This could be explained by a decrease of spherically structured macromolecules. As illustrated in Figure 6, Fluid C shows a nearly Newtonian behavior. That is probably related to the high salt concentration in the solution. High salt concentration implies huge amounts of active ions, which not only destroy water structure, but also break water or polymer bonding.

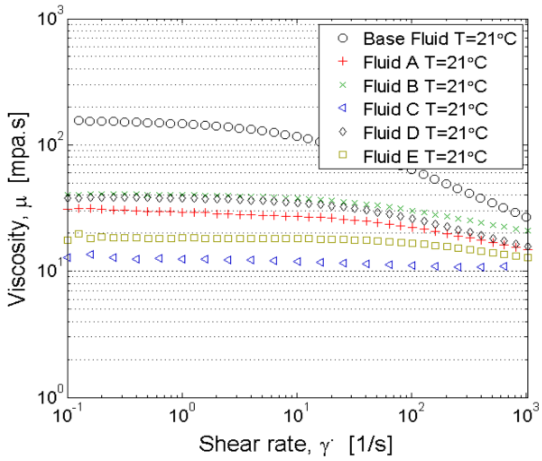

Figure 6. Rheology of PAC with salts at 21°C.

However, Fluid B with slightly higher molar concentration of metal ions (4.74 $mol/liter$) compared to Fluid C (4.33 $mol/liter$) did not affect on PAC properties that much. Taking into consideration that the anionic fractions are the same, a conclusion can be that Cesium ions impact more on rheological properties of PAC than Potassium ions do.

Another situation is observed with fluids A and B. Despite the fact, that the molar concentration of Potassium Formate is 3.8 times higher in fluid B, the viscosity curves dare only slightly higher for fluid B, while the slopes are practically identical ($n = 0.98$). Both fluids have significant increase in range of shear rate for the plateau value of μ_0 and decrease in slope of the shear thinning region (from $n = 0.79$ to $n = 0.98$). It is suggested that the increasing range of shear rate for μ_0 is related to a higher fraction of partially disentangled polymer macromolecules. This partial disentanglement is caused by salt ions which affect the polymer macromolecules, breaking their spherical structure. To finalize, the results for fluids A and B led to conclusion that there is a sort of critical Potassium Formate concentration, less than 2.67 $mol/liter$. Further increase of concentration did not cause any significant change in shear thinning region.

Comparison between Fluids C and E shows some similarities with the same comparison for Fluids A and B. Although the concentration of metal ions is 13 times higher for Fluid C, the difference between their effects on PAC rheology is considered to be insignificant. The viscosity is slightly higher for fluids with smaller metal ion concentrations. The n value for Fluid E is 0.99 and indicates almost Newtonian properties.

The anionic part ($COOH^{-}$) in fluids A, B, C and E was the same. So, as expected, salts behavior was mostly defined by cations in their composition. Also as expected, measurements indicated a decrease of pH value (0.2-0.9) for tested fluids (A, B, C, E) compared to brines with the same concentrations.

The results for fluid D is considered to be the most interesting. Fluid D, containing only 0.88 $mol/liter$ of $NaCl$, has changed the polymer mixture properties to the same degree as other fluids with significantly higher salt concentrations. There are, however, some distinctive differences. First of all, the μ_0 did not increase that much compared to fluid A and B, and also the slope is slightly higher ($n = 0.97$) compared to the one for fluids A and B ($n = 0.98$). Another difference was a higher decrease in pH value (1.4) compared to brine with the same concentration.

For $NaCl$ brine, the pH value was 6.4, while for fluid D it was 5.0. Such a strong effects with such a small concentration of $NaCl$ (0.88 $mol/liter$) is most probably related to the fact that both Na^{+} and $Cl^{-}$are chemically very active ions. In case of fluids A, B, C and E the main role was played by cations, but in case of Fluid D both cations and anions affect the rheological properties.

To summarize, the dominant role of metal ions on rheological properties of PAC solutions was confirmed. Cesium ions have the highest effect from all tested fluids, while Potassium ions have the lowest effect.

Effects of brines on PAC solution $T = 42°\mathrm{C}$

Experiments conducted on mixtures at 42°C and ambient pressures are shown in Figure 7. For all fluids tested at this temperature, except for Base Fluid, a near Newtonian behavior was observed ($n = 1$). One can notice an increase in apparent viscosity value at the lowest shear rates for fluids B and D. The improved solution at low shear rates is due to the use of Concentric Cylinders (CC27). One of very few disadvantage of Concentric Cylinder is the possibility of transition to turbulent flow at high shear rates.

For the Base Fluid, results at 42°C shows as expected a decrease of viscosity. Other notable observations are slightly increased shear rate range of μ_0 and decreased slope (from $n = 0.79$ to $n = 0.95$) of the shear thinning region.

With increase of temperature, a decrease of viscosity values for fluid B was observed, together with transition to near Newtonian behavior. Increase in temperature leads to increase of chemical activity of ions in solution, so these ions at this temperature destroy water structure and force PAC macromolecules to full disentanglement.

For Fluid C, which had near Newtonian behavior at ambient temperature, showed a decrease of overall apparent viscosity. Fluid D obtained a near Newtonian behavior at $T = 42°\mathrm{C}$ and with the overall apparent viscosity decreasing in the same way as Fluid B. Another observation is that Fluids C and E have almost similar viscosity curves, despite a factor of 13 difference in metal ions concentrations.

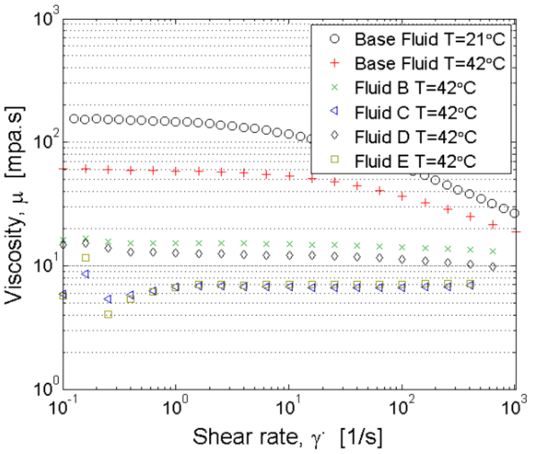

Figure 7. Rheology of PAC with salts at 42°C.

DISCUSSION AND CONCLUSIONS

This study was carried out with the intention of achieving better understanding of PAC solutions stability and possible effects of different brine solutions. The results led to the following conclusions:

From mainly instrumentation reasons, increase of polymer concentration from 0.1 to $0.4\,wt\%$ lead to more stable and consistent viscosity measurements. There might also be a physical stabilizing effect due to increase of effective viscosity. This could prevent flow fluctuations in the polymer solution even at low shear rates. Finally, also observed, fluids with higher polymer concentration showed higher degree of shear thinning (n changed from 0.98 to 0.79 with increasing of polymer concentration).

Every tested brine concentration made their individual significant impact on PAC viscosity. Potassium Formate brines, even at high concentrations, caused the least changes on PAC rheological properties. Highest effects were achieved with Cesium Formate brines, both on high and low brine concentration. Sodium Chloride appeared to be in between these extremes.

However, with increase of temperature all viscosified brines behaved as near Newtonian fluids. A strong relation between chemical activity of ions and their effects on polymer solution was observed together with a dominating role of metal ions.

To finalize, with Formate brines a clear relation appeared between reactivity of metals and effects of these metal ions on PAC rheology. A similar dependence was not found with Sodium-containing brines. They did not fit into “the metal reactivity – effects on PAC rheology”. There are two possible reasons for that: one is the presence of Sodium in PAC molecule and another is the presence of Chlorine ions in Sodium-containing brine. Chlorine ions have high chemical activity compared to Formate ions and expected to affect the PAC rheological properties in significantly higher degree.

Based on the results, it can be concluded that the interaction of PAC and brines in water based mud may impact negatively on rheological properties, in particular on the effective viscosity. This is the case especially for drilling in reservoirs where temperatures usually are significantly higher than 42°C.

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APPENDIX I

Rheology of brines and experimental test matrix.

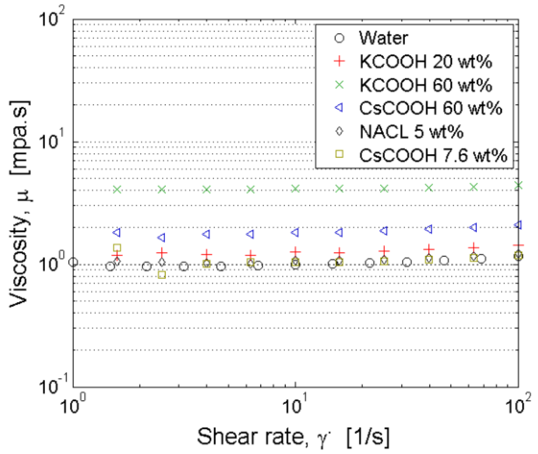

Figure I-1: Rheology of brines at T=21℃

Table I.1: PAC solutions and brines rheology properties.

Fluid name	**Fluid composition**	**Density ($s.g.$)**	**pH**	**C_m Salt**	**C_m Metal ions**	**Apparent viscosity**		**Temp [$°C$]**
						K	**n**	
Base Fluid	PAC 0.4 $wt\%$	1.0017	6.37	-	-	0.175	0.79	21
-	PAC 0.3 $wt\%$	1.0014	6.44	-	-	0.091	0.89	21
-	PAC 0.2 $wt\%$	1.0011	6.66	-	-	0.043	0.95	21
-	PAC 0.1 $wt\%$	1.0007	6.84	-	-	0.022	0.98	21
Brine	$KCOOH$ 20 $wt\%$	1.1195	8.5	2.67	1.23	0.001	1	21
Brine	$KCOOH$ 60 $wt\%$	1.4038	9.5	10.22	*4.74*	0.004	1	21
Brine	$CsCOOH$ 60 $wt\%$	1,6817	11.4	5.80	*4.33*	0.002	1	21
Brine	$NaCl$ 5 $wt\%$	1.0358	6.4	0.88	**0.34**	0.001	1	21
Brine	$CsCOOH$ 7.6 $wt\%$	1.057	10.2	0.45	**0.33**	0.001	1	21

*C_m: molar concentration [$mol/liter$].