ABSTRACT: Polymer solutions flowing in the porous media during enhanced oil recovery (EOR) processes are subjected to both shear and extensional rheological deformation. However, the previous rheological studies conducted on a surfactant–polymer (SP) system or polymer systems were only shear-based. In this paper, the extensional rheological performance of hydrolyzed polyacrylamide (HPAM) in the presence of an anionic surfactant at various concentrations (0, 0.01, 0.05, 0.1, 0.2, and 0.3%) is studied with deionized water and 1% NaCl. Further, the extensional rheological behavior of HPAM in the presence of NaCl and CaCl₂ is studied at varying ionic strengths (1−10%). A capillary break-up extensional rheometer is used for performing extensional rheological characterization. Results revealed that the extensional resistance of HPAM is enhanced in the presence of a surfactant. Particularly, around the critical micelle concentration value of the surfactant (0.1%), HPAM showed higher extensional resistance. Higher extensional resistance for the SP system is observed with deionized water when compared to 1% NaCl. HPAM showed improved performance at 1% NaCl salinity when compared to the higher concentration of NaCl salinity. However, the presence of even 1% of calcium ions is detrimental to the extensional properties of HPAM.

1. INTRODUCTION

The viscosity contrast between injected water and crude oil results in reduced oil recovery (∼40%) from the reservoirs.¹ Water flooding leaves most of the crude oil (∼60%) behind as residual oil, which can be produced by controlling the mobility ratio between water and oil and/or by increasing the capillary number. One of the effective ways to control the mobility ratio is the use of water-soluble high-molecular-weight polymers, which not only increase water viscosity but also push additional oil by blocking high-permeable water-flooded pores as polymer chains can entangle to form cross-linked 3D networks. Thus, polymer fluids are viscous in nature and exhibit better rheological properties than water; therefore, polymer fluids are often used to improve the rate of oil recovery from reservoirs. However, for the reservoirs with high salinity, salinity of reservoir brine significantly affects the rheological properties of polymer fluids. Therefore, high-concentration polymer fluid is required to recover oil from saline reservoirs. In addition, polymer adsorption increases with increasing formation salinity, which will restrict the polymer fluid injectivity in the reservoir, conductive to high water cut and poor sweep efficiency.¹⁶ Despite the recognition of the fact that the polymer solutions flowing in the porous media are subjected to both shear and extensional rheological deformation,¹⁷−¹³ the role of extensional rheology during polymer flooding was often overlooked. Erincik et al.¹⁴ conducted oil recovery experiments in a 24,000 ppm NaCl using viscoelastic polymer solutions. Higher oil recovery shown by higher saline polymer solutions could not be explained by oscillatory rheology. Recently, Azad and Trivedi¹¹,¹⁵ summarized the consequences of overlooking a viscoelastic polymer’s extensional rheology at higher salinity. Thus, for the development of such reservoirs, the effect of varying salinity on extensional rheology of polymer methods needs to be analyzed, on which we focus here.

Hydrolyzed polyacrylamide (HPAM) is the most commonly used water-soluble polymer in oil recovery applications⁴,⁸−¹⁰ and it exhibits viscoelastic properties which are dependent upon shear rate, temperature, concentration, molecular weight of the polymer, and ionic strength.¹⁶ The increased viscosity of these polymers is derived from high molecular weight and negative charges along the polymer chain,¹⁷ which produces improved flow properties to increase sweep efficiency of water flood. Therefore, the viscosifying capacity of PAM-based

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polymers is highly appreciated to recover the bypassed oil in water-flooded reservoirs. There are some other studies stating that polymer solutions possessing viscoelastic characteristics can recover the residual oil in the water-flooded reservoir.

HPAM is also used in combination with surface active agents, for example, surfactants in surfactant—polymer (SP) flooding, where the surfactant can help to provide additional advantages of (1) IFT reduction, (2) wettability alteration, and (3) higher residual oil recovery. In addition, the interaction between the surfactant and the polymer is analogous to mixed micelle formation, which increases the technical applications of SP systems. Therefore, various surfactants such as alkylmethylnaphthalene sulfonate surfactants, alkyl-branched sulfate and ethoxy sulfate surfactants, branched alcohol propoxylated sulfates, alkyl polyglycoside surfactants, viscoelastic surfactants, and gemini surfactant have been extensively utilized in the oil and gas industry. Sodium dodecyl benzenesulfonate (SDBS) is an anionic surfactant and it is used to promote increase in rheology of HPAM; anionic surfactant micelles orient themselves along the polymer chain and form SP entanglements of improved rheological properties. It has been reported that surfactant interaction with the polymer becomes strong and rheologically advantageous in case of a hydrophobically modified polymer. Therefore, it is vital to study the extensional rheological properties of the SP systems.

The formation salinity plays an important role in polymer selection as it affects the viscosity and chain entanglements. Thus, this may reduce HPAM injectivity in saline reservoirs where salt ions tend to produce greater attraction to its chain entanglements, rheological properties, and injectivity. The ionic strength of formation water mostly dominated by monovalent ions such as Na⁺ and divalent ions such as Ca²⁻ and Mg²⁺ are highly undesirable for the successful performance of aqueous suspensions. It has been reported that monovalent salts can significantly affect the flow behavior of aqueous solutions used for flooding projects. Na⁺ ions tend to screen the negative charges on polyelectrolytes, which eventually reduces the stretching of polymer chains. Moreover, the effect of divalent ions on the viscosity of polymer solutions is much higher than that of monovalent ions; divalent ions can associate along the polymer back bone to form intrachain pairs and a manning condensation, in which a polymer behaves like a tight coil, is obtained. Despite the vital importance for salinity effect on polymer systems, previous studies (experimental or theoretical) focused on either steady shear or oscillatory shear properties and limited information is available for extensional flow behavior. Azad and Trivedi conducted experiments (filament diameter vs time) to develop extensional rheological data for various HPAM solutions of different concentrations (200–1500 ppm) and salinities (1000–25,000 ppm) and later developed a novel viscoelastic model to predict HPAM flow behavior in a porous reservoir using the extensional rheological measurements. Experimental evidence has shown that elastic turbulence at the pore scale could result in anomalous injectivity, shear thickening, and oil recovery behavior that cannot be explained by shear rheology. Although the recently developed core-scale and pore-scale viscoelastic models developed based on the shear rheological parameters underestimated the apparent viscosity in porous media and residual oil recovery during viscoelastic polymer injection, models developed based on the extensional rheological parameters predicted them fairly well. Therefore, there is a growing consensus that the extensional rheological aspects of polymers should not be overlooked.

Previous experiments performed on surfactant–HPAM polymer systems concluded that with the increase in surfactant concentration, the shear viscosity of the SP systems shows a monotonic decrease. The extensional rheological aspect of SP systems has not been reported in the literature. This study aims to understand the fundamental interactive behavior of most common SP systems (SDBS–HPAM) in the extensional rheological field. Cationic polyacrylamide (CPAM) with better thermal stability has also been investigated for mobility control applications in recent times. Research conducted by Al-Shajalee et al. revealed that cationic polymer at low salinity conditions could be a potential option to selectively reduce the water relative permeability without affecting gas relative permeability during the water-shut off treatment in gas sandstone reservoirs.

The focus of this work is to investigate (a) the effect of surfactant on the shear and extensional rheological behavior of an HPAM polymer solution and (b) the extensional rheological behavior of HPAM in the absence/presence of monovalent and divalent salts (NaCl/CaCl₂ 1–10 wt %). In the presence of divalent salts, SDBS and HPAM precipitated. Therefore, the SP interaction is done only in the monovalent conditions of 1% NaCl concentration. Several shear rheological experiments of HPAM-based systems were done at higher temperatures. Although the extensional rheological aspects of HPAM and other polymers were investigated at room temperature, no

Table 1. Details of Chemicals Used in the Study

| Chemical                      | Linear formula                  | CAS number | Supplier       | Purity, % |
|-------------------------------|--------------------------------|------------|----------------|-----------|
| Water                         | H₂O                             | 7732-18-5  | Self           | 100       |
| Sodium chloride               | NaCl                            | 7647-14-5  | Fisher chemical| 99        |
| Calcium chloride              | CaCl₂                           | 10043-52-4 | Fisher chemical| 99        |
| Hydrolyzed polyacrylamide     | (CH₂=CH)ₙ(CH₃)₄COOH              | 9003-06-9  | SNF floerger   | 99        |
| Sodium Dodecyl benzene sulphonlic acid | CH₃(CH₂)₁₂CH₃H₂SO₄Na | 25155-30-0 | Acros organics | 98.5      |
literature is available about the extensional rheology of the SP system. Therefore, the focus of this work is to investigate the extensional rheological aspect of a commonly used SP system, that is, SDBS–HPAM, at room temperature to gain a fundamental understanding in the shear-free field. Please note that only an available high-temperature extensional rheological study is reported by Sur et al.\(^6^1\) and can be studied for the SP system in future work.

2. EXPERIMENTAL WORK

2.1. Materials. HPAM (flopaam 3630), with \(M_c\) of 18–20 MDa in powder form, was received from SNF Floerger France and it was used as received without any modification. SDBS was purchased from Acros Organics. A magnetic stirrer (IKA-C-MAG-HS7) was used to dissolve SDBS in HPAM solution. To study the effect of salts, NaCl and CaCl\(_2\) were purchased from Fisher Chemicals. Deionized (DI) water, obtained from Millipore Elix-10 purification apparatus (electrical conductivity of water = 0.0054 mS cm\(^{-1}\)), was used to prepare aqueous HAPM and SP solutions. The details about the chemicals used in the study are given in Table 1. The compositional details of different systems are given in Table 2.

Table 2. Compositional Details of Different Solutions Prepared in This Study

| solution | HPAM concentration (ppm) | SDBS (wt %) | NaCl (wt %) | CaCl\(_2\) (wt %) |
|----------|--------------------------|-------------|-------------|-----------------|
| HPAM     | 1000                     | 0           | 0           | 0               |
|          |                          | 1           | 1           | 0               |
|          |                          | 5           | 5           | 0               |
|          |                          | 10          | 0           | 0               |
| SP       | 1000                     | 0.01        | 0           | 0               |
|          |                          | 0.05        | 0           | 0               |
|          |                          | 0.1 (cmc)   | 0           | 0               |
|          |                          | 0.2         | 0           | 0               |
|          |                          | 0.3         | 0           | 0               |
| SP       | 1000                     | 0.01        | 1           | 0               |
|          |                          | 0.05        | 1           | 0               |
|          |                          | 0.1 (cmc)   | 1           | 0               |
|          |                          | 0.2         | 1           | 0               |
|          |                          | 0.3         | 1           | 0               |

2.2. Synthesis of SP Solutions and SFT Measurements. First, HPAM solution of 1000 ppm was prepared by dispersing 0.1 wt % of HPAM in desired salinity by stirring at 250 rpm for a few minutes. HPAM concentration is fixed at 1000 ppm throughout the work. Next, SDBS with a concentration of 0.01, 0.05, 0.1, 0.2, and 0.3 wt % is added in HPAM solution at a stirring speed of 150 rpm for 48 h to ensure complete dissolution of SDBS in HPAM solution. During the entire process, samples were sealed using a paraffin tap. NaCl and CaCl\(_2\) saline solutions were prepared at the concentration of 1, 5, and 10 wt %. 1000 ppm HPAM solutions were added to the prepared saline solutions (Table 2).

For SP solution, surface tension (SFT) of SDBS–HPAM systems is determined by SFT measurements using the Du Nouy ring. The measured SFT values for the SP system in the absence and presence of NaCl is reported in Figure 1.

It is clear that adding salts reduces the SFT value of SP systems. However, critical micelle concentration (cmc) appears to be ~0.1 wt % for both the systems. Generally, adding salts reduces the SFT of SP systems.\(^6^2\)\(^,\)\(^6^3\) At a higher salinity, reduction in the electrostatic repulsion between the ionic surfactant’s head groups occurs, which in turn promotes the micelles formation and binds the SP complexes. Therefore, the system becomes more surface-active in the presence of salts.

2.2.1. Extensional Rheology Experiments. A capillary break-up extensional rheometer (CaBER, Thermo scientific Canada) was used for extensional rheological characterization of different aqueous solutions. Each sample was placed between the bottom and top plates of the rheometer. The filament will be formed when the top plate is separated from the bottom plate. For the separation of plates, a strike time of 50 ms is given (8–10). After stretching, the fluid was squeezed by the capillary forces. Filament diameter will continue to show decrease until the filament breaks, which is monitored by an inbuilt laser micrometer. The imposed step strain stretches the droplet between two plates, which results in filament drainage that is governed by the balance between driving capillary force and the resistive viscous and elastic forces. Following the viscous dominated fluid drainage in the early phase, the intermediate time scale of viscoelastic fluids, which have been reported to be governed by the balance between elasticity and SFT, is represented by the exponential decline in the filament diameter. The upper convected Maxwell model accounting for elasticity is used to estimate the extensional relaxation time for two samples by regression. The solution to the upper convected Maxwell model is given in eq 1. The linear data representing the elastic region is extracted from the filament diameter versus a time semilogarithmic plot for the samples. The extracted data are then fitted with the solution of the upper convected Maxwell model (eq 1) using regression and the extensional relaxation time is determined using the match. SFT value of water (73 mN/m) is used as a reference for the HPAM system. For the SP system, measured SFT values shown in Figure 1 are used. The operational parameters for these experiments are reported in Table 3.

\[
D_{\text{mid}}(t) = D_o \left( \frac{G D_o}{4 \sigma} \right)^{1/3} e^{(-t/\lambda)}
\]

where \(D_{\text{mid}}(t)\) is the midpoint diameter at time \(t\), mm, \(D_o\) is initial diameter of the sample loaded, mm, \(G\) is elastic modulus, \(P_a\), \(\sigma\) is SFT of polymer samples, mN/m, \(t\) is the time, s, and \(\lambda\) is the relaxation time of polymer sample, s.

The axial force balance detailed in Anna and McKinley\(^5^8\) is given by eq 2.

![Figure 1. SFT as a function of surfactant concentration for the SDBS–HPAM system at deionized water and 1% NaCl conditions.](https://dx.doi.org/10.1021/acsomega.0c00481)
where \( \eta_s \) is the solvent viscosity, Pa·s, \( \tau_{zz} \) is the first normal stress, Pa, \( \tau_{rr} \) is the second normal stress, Pa, and \( \dot{\varepsilon} \) is the strain rate, s\(^{-1}\).

The term \( 3\eta_s\dot{\varepsilon} \) represents viscous Newtonian stress. A multiplicative factor of 3 to the first term of the right-hand side of eq 2 implies the induction of extensional stress to the term as the Trouton’s ratio for the Newtonian solvent is 3. Non-Newtonian elastic stresses are represented by a normal stress difference (\( \tau_{zz} - \tau_{rr} \)). Both these terms suggest the existence of only elongational flow during filament drainage in CaBER experiments. Capillary action is resisted only through the elongational stresses, and viscosity calculated out of them represents the apparent extensional viscosity. Hencky strain and strain rate were defined as per the standard analysis of Anna and McKinley of CaBER experiments by eqs 3 and 4.

\[
\varepsilon(t) = 2 \ln \left( \frac{D_0}{D_{mid}(t)} \right)
\]

\[
\dot{\varepsilon}(t) = -\frac{2}{D_{mid}(t)} \left( \frac{dD_{mid}(t)}{dt} \right)
\]

The comprehensive analysis of extensional flow in the neck indicates that the apparent extensional viscosity incorporating a nonzero tensile force is needed and the viscosity represented by eq 5 without correction factor is insufficient. Considering nonzero tensile stress in a force balance equation, the apparent extensional viscosity used by Kim et al. is represented by eq 6.

\[
\tilde{\eta}_{app}(\varepsilon) = \left( 2X - 1 \right) \sigma \frac{d\varepsilon}{dD_{mid}}
\]

where \( X \) is the axial correction factor required to account for axial variation. The value of \( X \) depends on the local shape of the filament and 0.71 is assigned for the axial correction factor \( X \) in the Table 3. Operational Conditions during Extensional Rheology

| parameters          | values          |
|---------------------|-----------------|
| temperature         | ambient         |
| initial gap distance | 3 mm            |
| final gap distance   | 8.2 mm          |
| aspect ratio        | 2.73            |

Figure 2. (a) UCM fit to filament diameter data for determination of extensional relaxation time of HPAM at deionized water conditions, (b) ratio of calculated initial diameter to measured diameter as a function of time, (c) extensional viscosity as a function of the strain rate plot showing the maximum extensional viscosity around the critical Deborah number, and (d) power law fit to the extensional viscosity versus Hencky strain values to determine the strain hardening index.
CaBER experiment. The extensional viscosity versus strain rate plot can be generated using eqs 4 and 6. Entov and Hinch derived that fluid relaxes at a two-third rate of strain using the finite extensible nonlinear elastic (FENE) theory. Filament drainage is constant at the critical Deborah number of 0.66, and it represents the maximum elastic limit where elongational viscosity tends to exhibit maxima. The maxima around the critical Deborah number will be used as maximum extensional viscosity. Strain can be calculated using eq 3. Extensional viscosity versus strain, around the critical Deborah number value, is fitted with the power law, resulting in the determination of the strain hardening index.

3. RESULTS AND DISCUSSION

First, the extensional rheological results of HPAM (1000 ppm) are presented followed by the discussion on extensional rheology of SP fluid, consisting of HPAM and varying SDBS concentrations at both deionized and 1% NaCl conditions. The extensional relaxation time is attained from the filament diameter versus time plot by fitting the upper convected Maxwell (UCM) model. Extensional viscosity results, determined from filament diameter data, are also presented. Finally, the effect of monovalent (NaCl) and divalent (CaCl2) salts (1, 5, and 10 wt %) on extensional rheology of the base case polymer is discussed.

Table 4. Experimental Extensional Data of HPAM Solution for Different Conditions

| system                  | HPAM concentration (ppm) | SDBS (wt %) | salt (wt %) | filament break-up time (s) | extensional relaxation time (s) | maximum extensional viscosity (Pa-s) | strain hardening index |
|-------------------------|--------------------------|-------------|-------------|-----------------------------|----------------------------------|--------------------------------------|------------------------|
| SP solution             | 1000                     | 0.01        | 0           | 3.2                         | 0.39                             | 91                                   | 2.4                    |
|                         |                          | 0.05        | 0           | 4.2                         | 0.53                             | 140                                  | 2.45                   |
|                         |                          | 0.1         | 0           | 4.9                         | 0.64                             | 220                                  | 3.18                   |
|                         |                          | 0.2         | 0           | 3.4                         | 0.41                             | 166                                  | 3.14                   |
|                         |                          | 0.3         | 0           | 2.8                         | 0.30                             | 80                                   | 2.23                   |
| SP solution             | 1000                     | 0.01        | 1% NaCl     | 1.25                        | 0.133                            | 35                                   | 3.23                   |
|                         |                          | 0.05        | 1% NaCl     | 1.32                        | 0.14                             | 50                                   | 3.22                   |
|                         |                          | 0.1         | 1% NaCl     | 1.74                        | 0.153                            | 73                                   | 3.07                   |
|                         |                          | 0.2         | 1% NaCl     | 0.79                        | 0.0835                           | 35.51                                | 3.07                   |
|                         |                          | 0.3         | 1% NaCl     | 0.754                       | 0.07                             | 21.83                                | 2.9                    |
| HPAM solution + NaCl    | 1000                     | 0           | NaCl 0      | 2.45                        | 0.299                            | 75                                   | 2.19                   |
|                         |                          | 1           | NaCl 0      | 1.86                        | 0.176                            | 149                                  | 3.96                   |
|                         |                          | 5           | NaCl 0      | 1                           | 0.099                            | 101                                  | 3.68                   |
|                         |                          | 10          | NaCl 0      | 0.65                        | 0.075                            | 56                                   | 3.63                   |
| HPAM solution + CaCl2   | 1000                     | 0           | CaCl2 1     | 0.51                        | 0.047                            | 48                                   | 3.50                   |
|                         |                          | 5           | CaCl2 1     | 0.22                        | 0.02                             | 24                                   | 3.38                   |
|                         |                          | 10          | CaCl2 1     | 0.08                        | 0.013                            | 13                                   | 3.13                   |

3.1. Effect of SDBS on Extensional Property of HPAM at Deionized and Monovalent Ion Conditions. 3.1.1. Effect of SDBS on Break-up Time and Extensional Relaxation Time of HPAM. First, HPAM solution is analyzed for extension at ambient condition and it was observed that the filament diameter of HPAM progressively decreases to a minimum size of 0.06 mm before it finally breaks at 2.47 s (see Figure 2a and Table 4).

Generally, the break-up time of less than 0.1 s is considered to have zero elasticity, which could not result in any residual oil saturation (Sor) reduction even at 1 to 2 ft/day flux rate in the reservoir. Purely viscous polymers such as glycerin and xanthan gum that possess this characteristic were reported to result in a Sor of 0.43–0.45. A low-molecular-weight polymer such as HPAM 3130 reported to possess the break-up time of less than ~0.5 s could result in a Sor of 0.42, only a marginal reduction because of its relatively lesser extensional resistance. A break-up time of ~3 s, reported to be shown by low saline, high Mw HPAM 3630, could result in a Sor of 0.31 at a flux rate of 1 ft/day. A break-up time of ~5 s was reported to be shown by high Mw HPAM 3630 polymers at a higher salinity. These polymers could result in a Sor of 0.24 and 0.08 at 1 and 4 ft/day, respectively. The viscoelastic onset rate is the rate at which the viscoelastic polymer begins to shear thickening characteristic in porous media. A polymer possessing the relaxation time of less than 0.1 s fails to show any viscoelastic onset in the porous media. Low Mw polymers such as HPAM 3130 with the break-up time of ~0.5 s were reported to show the viscoelastic onset at the very high shear rate of 100 s⁻¹. A break-up time of ~3 s is shown by HPAM 3430, the polymer with medium Mw. This polymer was reported to show the onset rate of ~50 s⁻¹. A break-up time of ~5 s was shown by very high Mw polymers such as HPAM 6035 could translate into the very low onset rate of 2 s⁻¹. Generally, the higher the Mw, the higher the break up time. The break-up time gives a measure of a polymer solution's elasticity. The role of a polymer’s Mw and elasticity on the oil recovery applications has been reported.

The ratio of initial filament diameter to filament diameter at the particular time is plotted with respect to time (Figure 2b). Extensional relaxation time of HPAM is determined by fitting the UCM model (eq 1) to the filament diameter versus time data for HPAM (Figure 2c). Maximum extensional viscosity for HPAM is observed in the extensional viscosity versus strain rate plot (Figure 2c). Extensional viscosity as a function of Hencky strain is shown in Figure 2d. Strain hardening index is determined by using power law fit to extensional viscosity versus Hencky strain values around the critical Deborah number. Extensional relaxation time, maximum extensional viscosity, and strain hardening index for HPAM solution in deionized water are reported in Table 4.
The effect of adding SDBS at various concentrations to HPAM on the extensional properties are investigated with deionized water and 1% NaCl. The reproducibility of measured filament diameter versus time is shown (Figures 3 and 4a−f).

The extensional properties such as break-up time, extensional relaxation time, maximum extensional viscosity, and strain hardening index for all the systems with respect to surfactant concentrations are shown in Figure 5a−d. It is clear from Figure 5a that the break-up time of HPAM solution increases in the presence of SDBS at deionized and 1% NaCl conditions as long as its concentration ≤0.1 wt %. Filament breakup is driven by the capillary force and SFT of solution. As the SP solution has lower SFT than the HPAM solution, break-up time is delayed owing to both the surfactant's surface activity and the polymer's elasticity property. However, further increase (>0.1 wt %) in the SDBS concentration reduces the break-up time and it decreases to 3.4 and 2.8 s at 0.2 and 0.3 wt %, respectively, as shown in Figure 5a. The reason for improved extension in HPAM might be attributed to SDBS surface activity that showed synergistic performance around 0.1 wt % of SDBS, which indicates that cmc is around 0.1 wt %. The measured SFT values of SDBS—HPAM combination indicate that the cmc value is around 0.1 wt % (Figure 1). Similar observations were reported in literature. The cmc of SDBS measured using SFT is around 0.075%.

Hence, 0.1 wt % is considered as the cmc value of SDBS for SP solution throughout the study. At 0.2 and 0.3 wt %, HPAM aqueous phase is significantly saturated by SDBS micelles, resulting in extensional property of SP solution being dominated by the surfactant than mixed SP junctions. This significantly affects the extensional rheological behavior of SP solution and consequently, resistance to filament breakup decreased. As the SFT of the SP system in the presence of salts is lower when compared to no-salt conditions (Figure 1), it can be expected that SP system should give relatively higher resistance to breakup in the presence of salts. However, the effect of salts decrease the resistance to filament breakup when compared to the no-salt conditions at all the concentrations of SDBS. This implies that extensional properties of the SP system got affected because of the salt addition.

Figure 3. (a−f) Filament diameter as a function of time for various surfactant concentrations in SP systems prepared with deionized water.
The results of relaxation time for HPAM and SP solutions are provided in Figure 5b. The linear data representing the decrease in filament diameter versus time for all the cases are extracted and fitted with the UCM model (eq 1). The slope of the fitted data represents the longest relaxation time.7−10,59,60 The determined extensional relaxation time of all the solutions is reported in Table 4. Extensional relaxation time determined using UCM fit is also higher for SP solution of 0.1 wt % SDBS than other solutions (Table 4) at both deionized and 1% NaCl conditions. Addition of 1% NaCl to SP systems contracts the polymer chain, which is reflected in the reduced relaxation time of SP systems.

3.1.2. Effect of SDBS on Extensional Viscosity of HPAM. The effect of SDBS on extensional viscosity of HPAM solution is shown in Figure 5c. SDBS is added in increasing concentration from 0.01, 0.05, 0.1, 0.2, and 0.3 wt % and the addition of surfactant typically reduces the SFT of fluid (Figure 1).76 SDBS is anionic and possess negative charge. Surfactant powder when added to the water results in the formation of monomers up to a concentration called CMC.76 After CMC, micelles form. These surfactant micelles may interact with polymer chains and increase the solution viscosity as reported.77 Therefore, maximum extensional viscosity of HPAM solution increases with the inclusion of SDBS; HPAM solution extensional viscosity of 75 Pa·s increases to 91 and 140 Pa·s with the inclusion of 0.01 and 0.05 wt % SDBS, respectively. This extensional viscosity further increases to 220 Pa·s at 0.1 wt %. However, further increase in SDBS concentration to 0.2 and 0.3 wt % showed reverse behavior and reduced the extensional viscosity to 166 and 80 Pa·s, respectively. The reason of increased extensional viscosity lies in the interaction of SDBS micelles with HPAM chains; the interaction at 0.1 wt % have developed cross-links of HPAM−SDBS those create a transient network in solution, consistent with the study explaining effect of anionic surfactant on polymer viscosity.78 At 0.01 wt % of SDBS, the interaction between the surfactant and HPAM did not reach the level where cross-links reached to enough strength; thus, extensional viscosity increased slightly from 75 to 91 and 140 Pa·s, respectively. This is in accordance with filament break-up results of HPAM solution with 0.1 wt % SDBS (Figure 1). At 0.2 wt % of SDBS, the amount of surfactant became significantly higher and therefore, the surface charges of SDBS−HPAM cross-links get screened out due to the excessive shielding of SDBS micelles. Therefore, the extensional viscosity of HPAM−SDBS decreased at surfactant concentrations higher than cmc. Although in general, the behavior of SP system at 1% NaCl is similar to behavior observed in deionized water, there are few interesting points to note. Maximum extensional viscosity of HPAM in the presence
of 1% NaCl is 41 Pa·s which is higher than 35 Pa·s, the maximum extensional viscosity of HPAM in the presence of 0.01% SDBS. This could be attributed to the fact that the SFT value of the 0.01% SDBS–HPAM system in 1% NaCl is 28 mN/m, whereas the SFT value of HPAM at this condition is 49 mN/m. As per eq 6, the higher the SFT value, the higher the extensional viscosity unless the added surfactant leads to additional network formation which is strong enough to induce significant extensional resistance. As the concentration of 0.01% is an order lower than the cmc value of these SP systems, significant network might have not been formed. Also, as expected, charge shielding effects lead to the relatively lower extensional viscosity of the SP system at various concentrations of the surfactant in 1% NaCl when compared to deionized conditions.

Extensional viscosity of SDBS-HPAM combinations measured with respect to the Hencky strain around the critical Deborah number is shown in Figure 5d. In general, the strain hardening index determined using power law for SDBS-HPAM in 1% NaCl solution is higher when compared to solution prepared in deionized water (Table 4). Walter et al. also reported that polyelectrolyte solutions in the presence of ions tend to strain at higher values when compared to the base case nonionized conditions. Ions cause the polymer chain to coil and more energy needs to be expended in stretching the coiled polymer chains during elongation flow.11,15,79

3.2. Effect of SDBS on Shear Viscosity of HPAM. Shear viscosity measured for SDBS-HPAM systems show the monotonic decrease with respect to surfactant concentration (Figure 6). Similar behaviors for other SP systems in shear field were reported.50–54 Upon comparing the shear rheological results with extensional rheological results, it is understood that the elasticity, break-up resistance and extensional viscosity of SP solution, prepared with 0.1 wt % SDBS in 1000 ppm HPAM, is higher than in the HPAM solution. Therefore, it can be said polymer resistance to extensional deformation can be increased by adding anionic surfactant of cmc value. However, HPAM’s shear resistance cannot be increased by adding anionic surfactants.

3.3. Effect of Monovalent/Divalent Salts on Extensional Properties of HPAM. The effect of adding NaCl and CaCl₂ at various concentrations to HPAM on the filament break-up time is shown in Figure 7a.

3.3.1. Effect of Monovalent and Divalent Ions on the Break-up and Extensional Relaxation Time on HPAM. Adding monovalent NaCl to HPAM results in the drastic decrease of filament break-up time. From the results in
7a and Table 4, it can be observed that the break-up time of HPAM decreases to 1.86 and 1.0 s in the presence of 1 and 5 wt % NaCl, respectively. Finally, it reaches a minimum value of 0.75 s for 10 wt % NaCl. In general, higher the concentration of NaCl, higher the loss of extensional resistance to breakup. Typically, NaCl tends to dissociate into Na\(^+\) and Cl\(^-\) ions in solution where Na\(^+\) deposits on negatively charged polymer chains.\(^{38}\) With increasing concentration, this increasing deposition progressively screens the charge present on the polymer chains. A complete screening of charges is expected at high NaCl concentration, which results in a significant loss of extensional resistance to breakup. Mechanistically, it causes the filament of HPAM (in the presence of salt) to break up faster than the filament of HPAM (prepared in sole DI water). For 1.0 wt % NaCl, the increased relaxation time for the HPAM solution is also confirmed by UCM fit results as shown in Figure 7b and Table 4. It is evident that relaxation time with 5 and 10 wt % was lesser when compared with 1.0 wt % NaCl (Table 4); CaCl\(_2\) salts have much significant effect on the break-up time of HPAM solutions (Figure 7a). For 1.0 wt % NaCl, the increased relaxation time for the HPAM solution is also confirmed by UCM fit results as shown in Figure 7b and Table 4. It is evident that relaxation time with 5 and 10 wt % was lesser when compared with 1.0 wt % NaCl (Table 4); CaCl\(_2\) salts have much significant effect on the break-up time of HPAM solutions (Figure 7a). As calcium concentration increases, the break-up time decreases significantly from 3.12 (HPAM solution) to 0.51 s at 1.0 wt % CaCl\(_2\) concentration. Its value for 5 and 10 wt % CaCl\(_2\) further decreased to 0.22 and 0.08 s, respectively, as shown in Figure 7a. UCM fit (Figure 7b) also showed that the extensional relaxation time of the HPAM solution in the presence of CaCl\(_2\) is lower, when compared to the case where HPAM is prepared with monovalent, NaCl brine (Table 4). This can be attributed to the fact that the divalent ions undergo manning condensation with polymer chains and form cation and polymer chains to have neutral local electrical charge.\(^{38,0}\)

### 3.3.2. Effect of Monovalent/Divalent Salts on Extensional Viscosity of HPAM

Extensional viscosity as a function of strain rate and Hencky strain for HPAM in the presence of NaCl and CaCl\(_2\) is shown (Figure 7c,d). It has been established that shear rheology of polyelectrolyte solutions is lower in the presence of CaCl\(_2\) salts.\(^8\) Similar behavior is observed with viscosity results of HPAM solutions consisting of CaCl\(_2\). With CaCl\(_2\), the maximum extensional viscosity of HPAM solutions substantially reduced than the ones of pure HPAM solution and HPAM solutions with NaCl (Figure 7 and Table 4). For 1.0 wt % CaCl\(_2\), the value of extensional viscosity of HPAM solution is 48 Pa s. At 5 and 10 wt % CaCl\(_2\), a further decrease has been observed with 24 and 13 Pa s (Figure 7c). The divalent ions, because of a higher charge and polarizability, may bind more tightly to the polyelectrolytes. Therefore, these ions can cause additional extensional viscosity reduction as compared to monovalent ions. Divalent ions can also cause a drop in the viscosity of polymer solution due to the bridging effect. Thus, compared to monovalent ions (Na\(^+\)), the effect of divalent ions (Ca\(^{2+}\)) will be more detrimental on rheological properties of polymer methods used for oil recovery applications.

Strain hardening index for saline HPAM solution of 1.0 wt % NaCl and CaCl\(_2\) was higher than the pure HPAM solution and also in other NaCl and CaCl\(_2\) solutions at the concentrations 5 and 10 wt % (Table 4). This could be attributed to the fact that, ions tend to coil the polymer chains and more energy needed to expend them during extensional flow.\(^{11,15,79}\) Therefore, the strain hardening, which quantifies the polymer hardening capability, is higher for saline HPAM solutions. Walter et al.\(^8\) also reported the similar observation that polyelectrolyte solutions in the presence of calcium chloride tend to persist at relatively longer strain when compared to the base case.

Generally, with an increase in temperature, decrease in the shear viscosity of HPAM polymer were reported.\(^{53,54,81,82}\) However, with the increase in temperature, an increase in
extensional viscosity was reported for polycarbonate system in air.\(^\text{61}\) For the anionic surfactant—HPAM system, shear viscosity decreases considerably with respect to the increase in temperature\(^\text{62}\) when compared to nonionic surfactant—HPAM system. This signifies that both temperature and surfactant addition can decrease the steady shear resistance of polymer monotonically. However, with the increase in surfactant concentration, monotonic increase or decrease in extensional viscosity is not observed in this work. It would of interest to see how temperature influences the extensional rheological trend for these systems in future.

4. CONCLUSIONS

In this study, the extensional rheological effect upon on adding the anionic surfactant (SDBS) at various concentrations to the HPAM polymer solution is studied at deionized and monovalent salt conditions. Extensional rheological behaviour of the SP system is compared with their shear rheological behavior. Further, the effect of adding monovalent and divalent salts is studied. The following conclusions can be drawn.

1. Adding surfactant to a polymer in deionized conditions enhances its extensional rheological properties especially at 0.1% surfactant, the cmc value of the surfactant. At cmc values, the relaxation time increases from 0.299 to 0.64 s and extensional viscosity increases from 75 to 220 Pa. Improved extension attained at the cmc could be attributed to the optimal cross-links formed between SDBS micelles and the HPAM polymer.

2. The strength of cross-links is affected when the concentration of the SDBS surfactant is above and below the cmc. Therefore, the extensional rheological properties of the SP system are impaired at the concentration other than cmc.

3. Improved extension attained at the cmc values is not reflected in the shear rheology, as it shows a monotonic decrease in resistance for the HPAM polymer with respect to surfactant addition. This is because during shear, the rolling action of the polymer molecules results in the cancellation of stresses.

4. Comparing the performance of SDBS-HPAM at deionized conditions with 1% NaCl, it can be said that all the extensional rheological except the strain hardening index becomes lower in the presence of monovalent salts.

5. It is important to point out that all the experiments were performed at room temperature. Temperature increases the extensional resistance of polycarbonate,\(^\text{61}\) and it may also influence the extensional rheological results of these SP systems.

6. In general, both the monovalent and divalent salts have a negative effect on the extensional rheology of HPAM polymers. With increasing NaCl, viscosity of the HPAM polymer solution first increases and then decreases.

7. The divalent salts are more detrimental to the performance of the HPAM polymer.

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Notes

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