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Article

Polymer Injectivity: Investigation of Mechanical Degradation of EOR Polymers Using In-Situ Rheology

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Abstract: Water soluble polymers have gained an increasing interest in enhanced oil recovery (EOR) processes, especially as polymer flooding. Despite the fact that the flow of polymer in porous medium has been a research subject for many decades with numerous publications, there are still some research areas that need progress. The prediction of polymer injectivity remains elusive. Polymers with similar shear viscosity might have different in-situ rheological behaviors and may be exposed to different extent of mechanical degradation. Hence, determining polymer in-situ rheological behavior is of great significance for defining its utility. In this study, an investigation of rheological properties and mechanical degradation of different HPAM (partially hydrolyzed polyacrylamide) polymers was performed using Bentheimer sandstone outcrop cores. Results show that, HPAM in-situ rheology is different from bulk rheology measured in rheometer. Specifically, shear thickening behavior occurs at high rates, and near-Newtonian behavior is measured at low rates in porous media. This deviates strongly from measurements in the rheometer. Polymer molecular weight and concentration influence its viscoelasticity and subsequently its flow characteristics in porous media. Exposure to mechanical degradation by flow at high rate through porous media leads to significant reduction in shear thickening and thereby improved injectivity. More importantly, the degraded polymer maintained in-situ viscosity at low flow rates indicating that improved injectivity can be achieved without compromising viscosity at reservoir flow rates. This is explained by reduction in viscoelasticity. Mechanical degradation also leads to reduced residual resistance factor (RRF), especially for high polymer concentrations. For some of the polymer injections, successive degradation (increased degradation with transport length in porous media) was observed. The results presented here may be used to optimize polymer injectivity.

Keywords: Enhanced Oil Recovery (EOR); Polymer flooding; Injectivity; Rheology; Viscoelasticity; Non-Newtonian flow; Mechanical degradation; HPAM

1. Introduction

In today's oil industry, chemical enhanced oil recovery such as polymer flooding plays a substantial role to promote oil production. This is attributed to the achieved improvement on sweep efficiency that boosts oil production over conventional waterflooding. In such a process, water-soluble polymers are added to viscosify injected water in order to achieve lower viscosity contrast between injected water and displaced oil and therefore favorable mobility ratio [1]. Besides mobility control, high viscosity polymers are required for better conformance control relevant to heterogeneous reservoirs with high permeability variations such as presence of thief zones [2]. There are two types of polymers that suit enhanced oil recovery (EOR) applications which are: biopolymers e.g., xanthan and synthetic polymers e.g., partially hydrolyzed polyacrylamide (HPAM). Regardless the nature and difference in molecular structure of these two polymers, polymer viscosity is the main physical property in the context of polymer flooding. Polymer viscosity depends on polymer molecular

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structure, molecular weight [3], polymer concentration [4], salinity [5,6], temperature [7,8], degree of hydrolysis [9], pH [10], flow model and type of forces dominating the flow [11].

While xanthan is well-known to be viscous dominated, HPAM is strongly influenced by both viscous and elastic properties[12]. It is important to understand the significance and consequences when HPAM fluids become elastic dominated. HPAM viscoelasticity is important for many applications in oil industry in general (e.g., drag reducer, drilling, etc.) and specifically in EOR applications such as polymer flooding [1,13-16], Low Salinity Polymer (LSP) flooding [17,18] and Alkaline–Surfactant–Polymer (ASP) flooding [19,20]. In polymer flooding HPAM viscoelasticity is believed to contribute to higher oil recovery in general and some claim it might reduce residual oil saturation due to promoting pulling effect mechanisms [21-24]. HPAM shear thickening behavior may in some cases contribute to improve front stability and oil recovery [25]. On the other hand, the significant pressure gradient associated with shear thickening phenomena can limit polymer injection, cause wellbore damage or fracturing. The influence of mechanical degradation on shear rheology will be discussed in this paper.

1.1. *Polymer Injectivity*

Polymer injectivity is a measure of how easily polymer solution can be delivered into reservoir formation [26]. It is also a measure of how fast polymer solution can be injected and propagate through the reservoir. It is a critical task and a decline in injectivity can negatively turn the predicted cashflow of polymer flooding project [27,28]. This basically is due to the delay of oil production or high pumping cost. Both aforementioned polymers (xanthan and HPAM) may suffer from injectivity problems for different reasons. For instance, the presence of microgels and impurities in xanthan may limit its injectivity [29]. However, HPAM viscoelasticity and retention are the main factors that restrict its injectivity. The design of polymer flooding projects has to cover some key aspects such as reservoir formation, oil saturation, injection strategy, polymer rheology, degradation, compatibility with other chemicals, economy, etc. [30]. This paper intents to investigate some of these aspects such the link between polymer rheology and degradation.

The theories and observations associated with characterization of flow of biopolymers such as xanthan are typically united in that xanthan has pseudoplastic rheological behavior in porous media similar to that predicated in pure shear flow such in rheometer [31-35]. However, the situation is more complicated for HPAM due to it viscoelastic nature and complexity of porous media. Despite the rich literature of polymer flow in porous media, the theoretical interpretations are still conflicting on analyzing the observed HPAM in-situ flow behaviors. HPAM polymers are well-known that have high polydispersity index [36] and also possess long relaxation time. HPAM has a flexible molecular structure and highly sensitive to shear environments. When HPAM flows in porous media, it is exposed to both shear and elongational deformations as it is transported through converging-diverging (C↔D) flow channels [36,37]. This results in successive expansion and contraction (E↔C) of polymer conformation through porous media. Figure **1** illustrates a schematic representation of a typical flow regions that are exhibited by HPAM with respect to shear rate. The polymer exhibits near Newtonian behavior at which its apparent viscosity is independent of imposed shear rates ($\dot{\gamma} < \dot{\gamma}_{c1}$). As the shear rate increases further, apparent viscosity decreases at which polymer solution is exhibiting shear thinning behavior. During shear thinning, polymer molecules start to disentangle with increasing shear rate until approaching another Newtonian plateau at which the state of disentanglement is very high. However, above $\dot{\gamma}_{c2}$, extensional flow becomes predominant at which polymer chains have insufficient time to recoil and align with flow which causes coil-stretch (C↔S) transition that yields in gradual increase of apparent viscosity with shear rate. The normal stresses that responsible for chain stretch cause a rise in the extensional viscosity which consequently cause pressure buildup and high apparent viscosity (shear thickening behavior). If the stretch rates that are associated with shear thickening behavior are high enough, chain stretch might evolve into chain fragmentation. Chain scissions due to mechanical degradation yields in viscosity loss as can be seen at high shear rates displayed in Figure 1. These flow phenomena are detailed elsewhere [11,37-40].

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The large strain forces cause large molecules to shear preferentially. Literature reviews on polymer mechanical degradation [41,42] show that the assessment of mechanical degradation is complex particularly at the presence of entanglements and concentrated conformational regimes. It is very important to understand how HPAM macromolecules contribute on changing its flow phenomena and increase apparent viscosity at high flow rates that scales several folds higher than predicted in simple shear flow such as in rheometer.

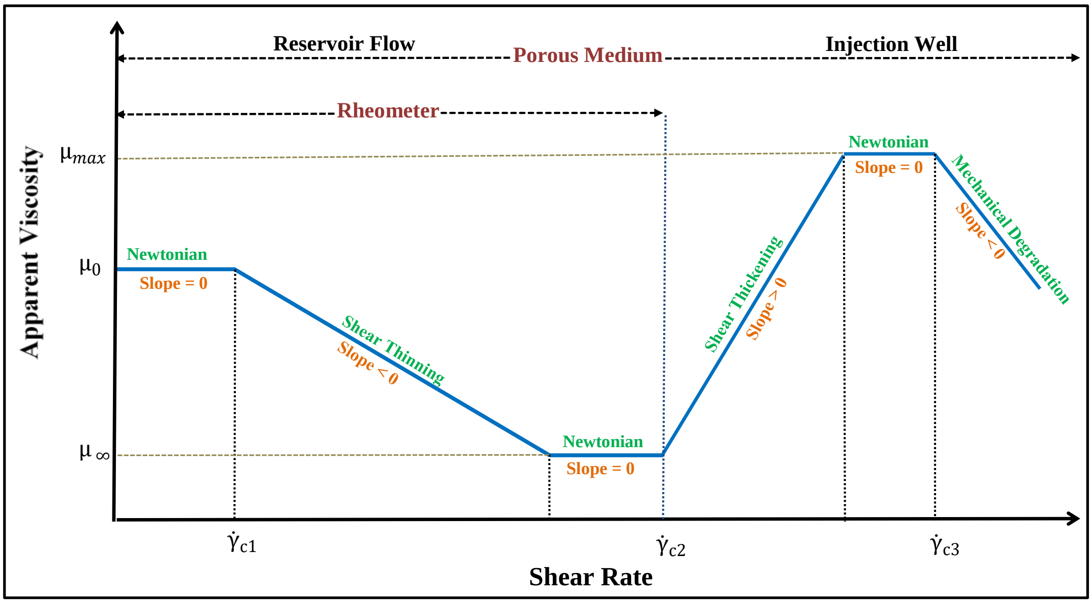

Figure 1 Schematic diagram of HPAM apparent viscosity vs. shear rate.

In addition to HPAM shear stability discussed above, the dimensions and conformation of HPAM molecules strongly depend on salt concentration and types of TDS existing in solution. HPAM is a negatively charged polymer, therefore at the presence of salts, the repulsion forces among polymer chains reduces due to shielding negative charges which cause coiling-up phenomena [43]. In some cases, the presence of salts in high concentration might lead to phase separation (e.g., gel-formation) [44]. The reduction in viscosity due to salinity is more pronounced at the presence of divalent cations (e.g., Ca^{2+}, Mg^{2+}, etc.) compared to that of monovalent cations like Na^{+}.

Previous study by Skauge, *et al.* [45] demonstrated combination of experiments for investigating the contributions of polymer molecular weight and concentration conformational regimes to its rheological properties. The measurements included shear viscosity, dynamic viscosity (small amplitude oscillatory shear (SAOS) and in-situ rheology (Bentheimer cores). The study classified the investigated polymer solutions into different conformational regimes; dilute, semi-dilute, concentrated semi-dilute and gel solutions, based on critical overlap concentration (C^*). SAOS measurements indicated whether the polymer solution is viscous or elastic dominated at certain concentration. The initial studies showed correlation between bulk elastic modulus G' and apparent shear thickening. The more elastic polymer exhibited higher resistance factor in porous media. The study also revealed that, polymer conformation regime has high influence on its in-situ rheological behavior. Shear viscosity data showed that mechanical degradation was high for high Mw polymer dissolved in high salinity brine. Also mechanical degradation was lower for concentrated solutions. Recent review with current knowledge on HPAM polymers flow in porous media concerning theoretical and experimental aspects given by Skauge, *et al.* [46].

1.2. *Mechanical Degradation*

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One of the most important aspects of HPAM polymer is mechanical degradation. Such an effect directly influences polymer viscosifying efficiency as well as alteration of its rheological properties. Both HPAM shear thickening behavior and mechanical degradation are well reported [12,47-50]. Mechanical degradation might occur along with the onset of shear thickening [51]. Onset of shear thickening has deserved high attention in the literature as it is an indication of viscoelasticity in porous medium [50,52-55]. Any alteration of molecular structure of HPAM through exposing HPAM to shear rate above or below the onset of shear thickening may change its apparent shear thickening behavior [56]. Preshearing polymer by exposing HPAM to wellbore mechanical degradation is a suggested approach to improve its viscoelastic properties which promotes its injectivity[57]. Despite the efforts that have been made on understanding and modeling polymer mechanical degradation, the dependence of polymer mechanical degradation on polymer Mw, MWD, concentration, and polymer transport distance in porous media requires more investigation [12,26,49].

The impact of mechanical degradation on polymer average Mw and molecular weight distribution (MWD) were examined through different studies. For example Seright, *et al.* [58] investigated mechanical degradation effect on polymer Mw and MWD by using gel permeation liquid chromatography (GPC). The degraded solutions showed narrower MWD compared to that of undegraded one. Hence, degraded polymer solution has lower polydispersity. This concept was also confirmed by size exclusion chromatography (SEC) [59] and asymmetric flow field-flow fractionation (AF4) [30,60-62]. Reduction of Mw or change in MWD is the reason of observed reduction in screen factor [12,63] that correlates with reduction of resistance factor as well.

Noïk, *et al.* [36] investigated the effect of Mw, concentration and different types of solvents on mechanical degradation of HPAM in short glass cylinders packed with sand particles. The high Mw polymers were subjected to the wellbore mechanical degradation through successively reinjection into porous media. Reinjection process represents the evolution of degradation as a function of residence time or the length of porous media. Degradation was assessed by observing the change in intrinsic viscosity of solutions before and after degradation. They found that the degree of degradation is independent of concentration for dilute solutions and only depends on Mw. However, for concentrated solutions, the degradation increases with concentration and has less dependency on average Mw.

Several studies attributed HPAM mechanical degradation to the polymer degradation in sandface, and therefore, understate the effect of polymer residence time or transported distance in porous media [12,38,49,64,65]. For example, Maerker [12] attributed mechanical degradation to the first 0.5 inch of porous media while Warner [64] attributed it to the first inch of unperforated wellbore based on studies performed in Berea rock. Müller, et al. [51] reported that HPAM polymers are progressively degraded until reach asymptotic value. This was based on experiments performed on beds packed with particles of different size by reinjecting the polymer solution at certain Reynold's numbers. The extent of degradation increased with particle size. The particles' size affect extensional thickening behavior mainly because it controls the characteristic length of elongational flow that occurs in pores. Lower particle size implies a larger average strain rate for a given velocity. Lower extensional thickening was found with decrease in particles size. A recent study given by Jouenne, *et al.* [66] highlighted the observation of mechanical degradation at entry face and limited it for the first 6mm of porous media based on studies performed in ceramic disk. However, Al-Shakry, *et al.* [67] conducted experimental studies using HPAM polymers that showed high Mw polymer underwent successive degradation as reinjected into the porous media. This suggests that the degree of degradation might depend on exposure time and number of exposures to high strain beside polymer Mw and concentration. The findings are also in line with Åsen, *et al.* [68]. The dependence of mechanical degradation on travelled distance in porous media has a significant practical consequences specifically when considering the effect on large scale medium such as field conditions. These observations were also supported by other studies based on analyzing shear viscosity data alone [69-71].

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Despite of current efforts made both experimentally and theoretically to clarify the problem of mechanical degradation, the current understanding is not complete and further analysis are required. This paper extends our previous work [67] that provides a basis for this study on experimentally investigation of the impact of mechanical degradation on polymer in-situ rheology. This study extends the analysis to address the influence of polymer physicochemical properties, particularly molecular weight and concentration on polymer mechanical degradation and its in-situ behavior. Particular attention was given for the impact of preconditioning the polymer solution prior to injection into the porous media on polymer in-situ rheology. The study was performed in a realistic porous medium using high preamble linear Bentheimer core plugs. The results from this study give an insight into in-situ rheological behavior of commercial HPAM polymers, which may be beneficial in polymer screening and designing of polymer flooding EOR operations. The results from this study also may serve as useful input for simulation models.

2. Material and Methods

2.1. Synthetic Brine

Synthetic brine of 1wt.% NaCl was prepared and filtered by using a 0.45 μm cellulose nitrate filter. The filtered brine was employed in the preparation of polymer solutions, core saturation and permeability measurements.

2.2. Polymer Preparation

Three types of partially hydrolyzed polyacrylamide (HPAM) were used in this study with different concentrations as shown in Table 1. These polymers are Flopaam 3330s, 3430s and 3630s that are donated as polymer A, B and C, respectively. Each polymer was prepared with low and high concentration within semi-dilute region which provide low and high degree of entanglements, respectively. The selected concentration for each polymer was based on overlap concentration (C^*) determined in earlier studies performed by Skauge, *et al.* [45].

The polymer stock solution of 5000 ppm were prepared by gradually dissolving 3.0 g of polymer powder into the vortex of 1 wt.%NaCl brine under vigorous stirring until the vortex disappeared. Then the stirring speed was decreased to 150 rpm. The polymer solution left under slow mixing for at least 24 hours before dilution into required concentration. The polymer was thoroughly sealed during the preparation. The prepared aqueous polymer was stored at 5 °C inside a fridge and used within two weeks of preparation to avoid any chance of chemical degradation.

Table 1 Molecular weights and concentrations of polymers

Polymer	Polymer (Flopaam) type	Molecular weight (10^6g/mol = MDa)	Polymer concentration (mg/L = ppm)
A	3330s	8	1000 4000
B	3430s	12	1000 3000
C	3630s	18	500 1000

2.3. Shear Viscosity Measurements

Shear viscosity measurements were performed at 22 °C by using Malvern Kinexus Pro Rheometer. The rheometer is equipped with different geometries that make the measurements more accurate and convenient based on fluid types and viscosity. Hence, double-gap geometry was used during the measurements of viscosities lower than 10 cP and cone-plate geometry was used for measuring viscosities higher than 10 cP. The viscosity measurements were fitted by power law model given below:

$$\eta = K\,\dot{\gamma}^{n-1} \qquad \text{Equation 1}$$

where, η is shear viscosity (cP), K is the consistency index, n is the flow behavior index and $\dot{\gamma}$ is shear rate (s^{-1}).

2.4. Porous Medium

The experiments were conducted in Bentheimer cores with an average length and diameter of 10 and 3.8 cm, respectively. Similar cores of Bentheimer with 5 cm length were used for prefiltering and preshearing processes. Details of each core are given in results and discussion section.

2.5. Experimental Procedures

The experimental setup is displayed in Figure 2 mainly consists of dual piston Quizix pump, transfer cylinder, core holder, pressure transducers, back pressure regulator and effluent collector. Note that, backpressure regulator was used during permeability measurements to dissolve any air in the setup and removed during polymer injection to avoid polymer degradation.

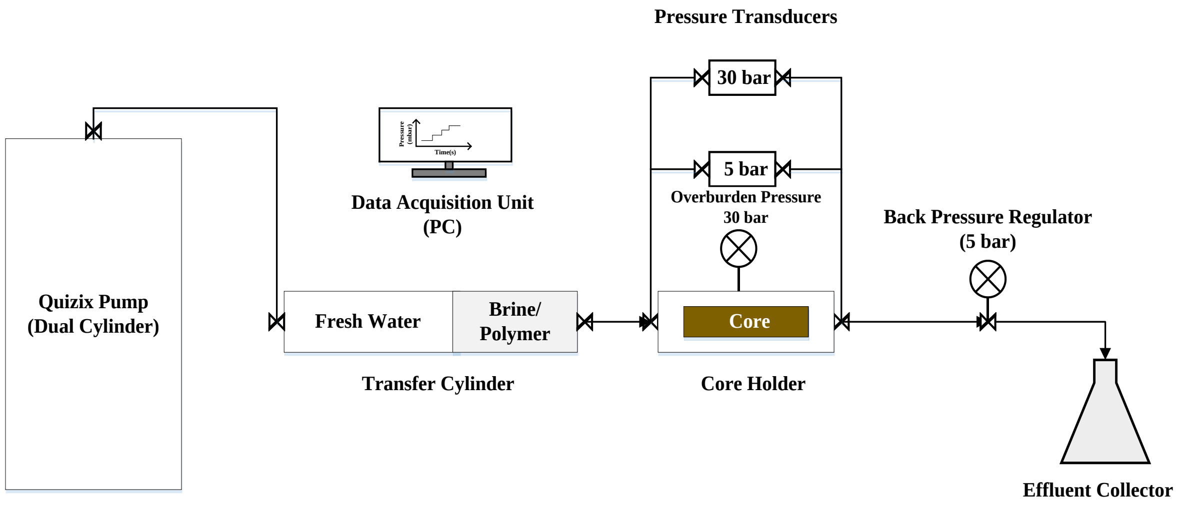

Figure 2 Schematic diagram of core flooding apparatus.

Core flood experiments were conducted at room temperature (22 ºC) and consist of three stages as detailed in the previous study [67]. The core flood procedure was performed as following:

Brine Pre-Flush

Before injecting the brine, the core plugs were vacuumed and saturated with brine for at least two days to ensure achieving ionic equilibrium between the core plug and brine followed by porosity measurements. Then, the core plug was mounted in the core holder and brine was injected at different flow rates to measure absolute permeability. Absolute permeability ($\mathrm{K_{abs}}$) was obtained by Darcy's law (Eq. 2):

$$\mathrm{K_{abs}} = \frac{\mathrm{Q}\,.\eta\,.\mathrm{L}}{\Delta\mathrm{P.A}} \qquad \text{Equation 2}$$

where, Q is injection flow rate, η is fluid viscosity, ΔP is pressure drop across the core, L and A are core length and cross-sectional area, respectively.

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Note that, interstitial velocity (v) was obtained from Darcy velocity (v_D) where ϕ is the porosity of porous media.

$$v = \frac{\mathrm{Q}}{\mathrm{A}.\phi} = \frac{v_D}{\phi} \qquad \text{Equation 3}$$

Darcy or Superficial velocity v_D also applied to calculate reservoir shear rate $\dot{\gamma}$. A conventional formula was used to estimate reservoir shear rate as below [65]:

$$\dot{\gamma} = \alpha \frac{4\, v_\mathrm{D}}{\sqrt{8\, \mathrm{K_w} \phi}} \qquad \text{Equation 4}$$

where v_D is Darcy fluid velocity, ϕ porosity, $\mathrm{K_w}$ absolute permeability, α shape factor which is assumed 2.5 for Bentheimer [1,38].

Polymer Injection

The investigated polymers were pretreated first before injection into the main cores. Pretreatment processes consist of prefiltering, re-injecting and preshearing as illustrated Figure 3. Pre-filtering and preshearing processes were performed on short cores (L = 5 cm) at low and high flow rates, respectively. Reinjected polymer has been prefiltered first in short core then sheared at high flow rate in long core (L = 10 cm). The flow rate used in prefiltering process was (Q = 0.5 cc/min, v_D = 0.6 m/day) whereas the flow rates applied in preshearing and reinjecting are given in Table 2. Recall that the purpose of prefiltering was to remove any microgel in the solution and filter out any possible large Mw species. This step represents commercial polymers which are used in field applications. It is also considered as a baseline for comparison with presheared and reinjected solutions. Preshearing was carried out to induce partial degradation in which large Mw species in the solution are possibly filtered and mechanically sheared to lower Mw species. While re-injection was designed to evaluate the evolution of polymer degradation with respect to residence time of polymer solution, core characteristic length, number of passes and multi entry effects.

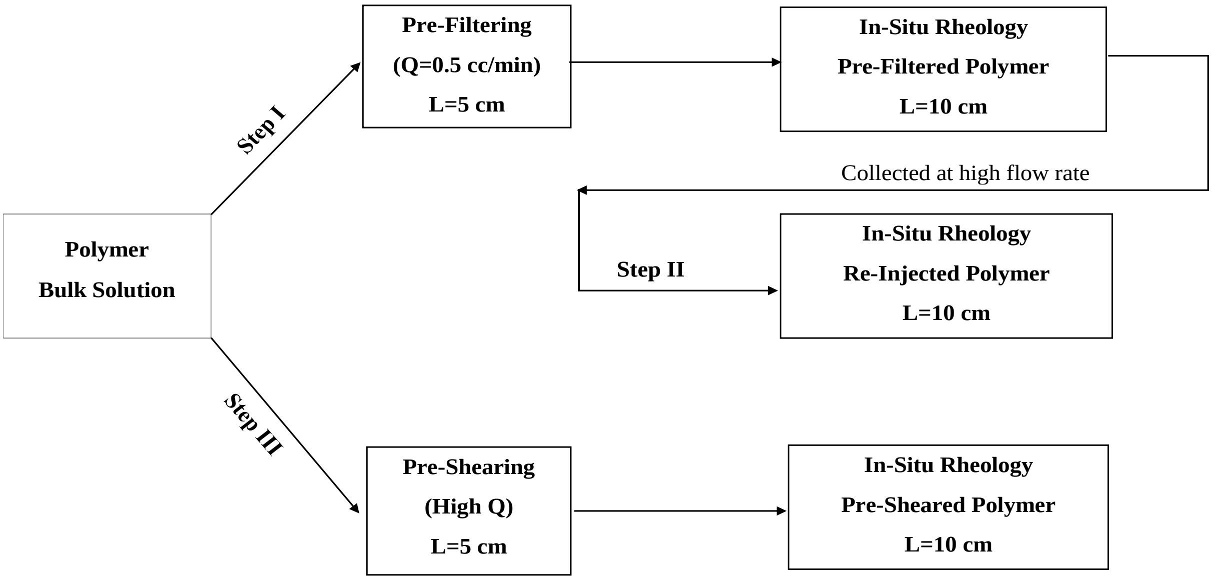

Figure 3 Polymer pretreatment processes.

Table 2 Applied flow rates for presheared and reinjected polymer solutions.

Polymer	A		B		C	
Concentration (ppm)	1000	4000	1000	3000	500	1000
Q (cc/min)	26	22	15	7	12	6

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v_D (m/day)	33.5	28.4	19.4	9.0	15.5	7.7

Pretreated polymer solution was injected into the main core at low flow rate (Q=0.5 cc/min, v_D = 0.6 m/day) for at least 2 PV to satisfy porous medium polymer adsorption level and achieving stable differential pressure. Then, flow rate was increased gradually to achieve highest flow rate given in Table 2 and then varied in a stepwise manner from highest to lowest flow rate. Each rate step was continued until stabilized pressure drop across the core was achieved. Resistance factor (RF) was calculated as following [72]:

$$\mathrm{RF} = \frac{\Delta \mathrm{P_p}}{\Delta \mathrm{P_w}} \qquad \text{Equation 5}$$

where, $\Delta\mathrm{P_p}$ is the pressure drop of polymer during polymer flow and $\Delta\mathrm{P_w}$ is the pressure drop of brine before polymer flow in porous medium.

Samples of effluents were collected at different flow rates to measure their shear viscosity by rheometer and compare with initial solution viscosity. The following equation was used to express the change in shear viscosity (shear degradation) [66]:

$$\mathrm{Deg}(\%) = \frac{\eta_i - \eta_e}{\eta_i - \eta_w} \times 100 \qquad \text{Equation 6}$$

where, η_i is injected solution viscosity, η_e is effluent viscosity and η_w is brine viscosity which was measured to be 1.04 cP. The viscosity data used in this equation were measured at shear rate of 10 s^{-1}.

Brine Post-Flush

After terminating the polymer injection, tapering was performed by injecting 5 PV of diluted polymer effluent with 50 and 25 % of initial effluent concentration. During tapering the injection of diluted polymer was performed at low flow rate (Q = 1.0 cc/min, v_D = 1.3 m/day) for 1 PV then gradually increased to high flow rates. After tapering with polymer, brine was injected at low flow rate (Q=0.5 cc/min, v_D = 0.6 m/day) for 1 PV then the injection rate was increased in stepwise manner. Core's permeability to brine was re-measured after injecting 5 PV of brine at high flow rates proceeded by two steps of tapering. Tapering was performed in an effort to approach 'true' residual resistance factor (RRF) which was calculated using Eq.7 [72]:

$$\mathrm{RRF} = \frac{\mathrm{K_{wi}}}{\mathrm{K_{wf}}} \qquad \text{Equation 7}$$

where, $\mathrm{K_{wi}}$ is the absolute permeability to brine before polymer flow and $\mathrm{K_{wf}}$ is the absolute permeability to brine after polymer flow.

3. Results and Discussion

3.1. Shear Viscosity

The shear viscosity of polymer solutions were measured in rheometer at wide range of shear rates as shown in Figure 4. At the mid-range of shear rates, all the solutions exhibited a predominant shear thinning behavior. The measurements were showed a good fit to the power law model (Eq.1) using the fitting parameters given in Table 3. Concentrated solutions show higher slope of shear thinning behavior compared to the solutions with lower concentration as seen in Figure 4. Accordingly, the flow behavior index n decreases as polymer concentration increases (see Table 3) and vice versa for the consistency index K. This is due to the high degree of entanglements present in concentrated solutions. Hence polymer molecules are more sensitive to imposed shear rate that reduces the degree of entanglements which results in lower viscosity with increasing flow rates [73].

Shear viscosity increases with increasing polymer molecular weight or concentration. For a given polymer concentration of 1000 ppm, the shear viscosity of polymer A, B and C were 8.4, 13.6 and 19.0 cP, respectively measured at $\dot{\gamma} = 10\ s^{-1}$. We are using shear rate of $10\ s^{-1}$ in this study as a reference of reservoir relevant shear rate. The increase of viscosity with Mw is ascribed to increase in hydrodynamic volume and charge density per molecule. While the increase of viscosity with respect to concentration is ascribed to the increase of the number of molecules that increases the interaction and repulsion forces among negatively charge polymer molecules [6].

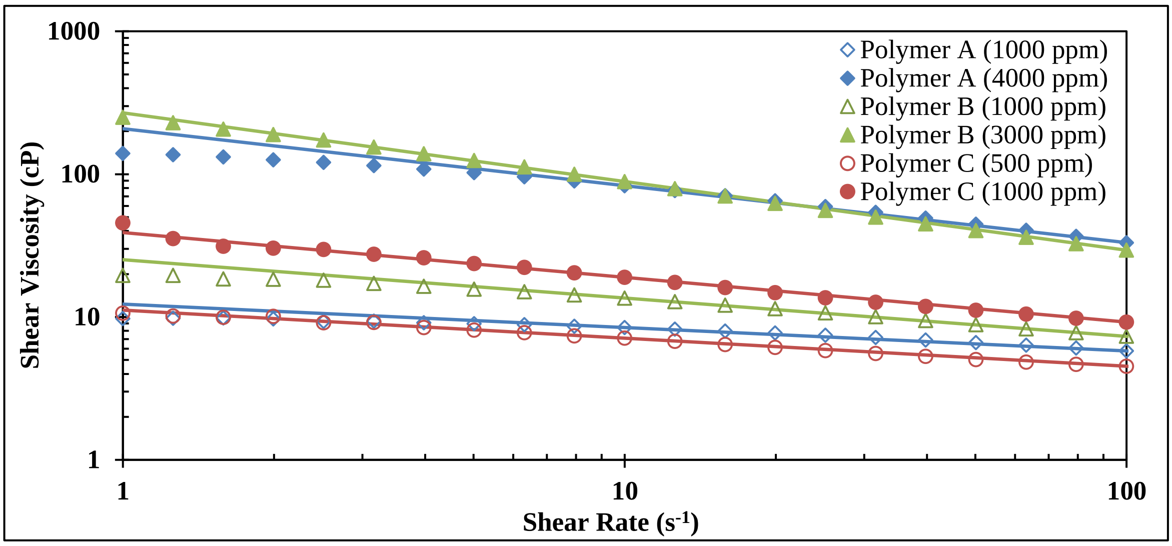

Figure 4 Shear viscosity of prefiltered bulk solutions A, B and C in 1 wt. % NaCl at 22 °C. Solid lines represent power law model.

Table 3 Power Law fitting parameters

Polymer	A		B		C	
Concentration (ppm)	1000	4000	1000	3000	500	1000
n	0.84	0.60	0.73	0.52	0.80	0.69
K	12.30	207.98	25.17	268.52	11.17	38.99

3.2. *In-Situ Polymer Rheology*

3.2.1. Apparent Viscosity in Porous Medium vs. Shear Viscosity in Rheometer

In this study, polymer apparent viscosity in porous medium is represented by resistance factor (RF) as a function of interstitial velocity. However, translating polymer flow velocity in porous medium to shear rate is required to correlate flow velocity in porous medium with shear rate in rheometer. Determining shear rate in porous medium is challenging due to many factors such as wide pore size distributions, tortuosity and complexity of porous media. A conventional formula given in Eq.4 was used to estimate reservoir shear rate. Figure 5 shows the viscosity profiles of polymer B (3000ppm) in porous medium versus bulk viscosity in rheometer. Resistance factor and apparent viscosity profiles of polymer in porous media were consistent. The polymer exhibits a predominantly shear thinning behavior in rheometer while different flow behaviors in porous media. At shear rates $\dot{\gamma} < 30\ s^{-1}$, the polymer exhibited shear thinning behavior in porous media while shear thickening behavior was observed at shear rates $\dot{\gamma} > 52\ s^{-1}$. A near-Newtonian behavior was observed during the transition between shear thinning to shear thickening behaviors. Both in-situ behaviors (near-Newtonian and shear thickening) in porous medium were not predicted by bulk rheology. This is expected due to different nature of flow exists in porous medium which is not purely shear flow as in rheometer [37].

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Figure 5 shows shear viscosity decreases while the apparent viscosity increases. For example, at $\dot{\gamma} \approx 400\ s^{-1}$, shear viscosity was 16.7 cP while apparent viscosity was ~300 cP which is more than 10 times higher than shear viscosity. This indicates the contribution of extensional viscosity to apparent viscosity at high flow rates. Polymer apparent viscosity is a combination of shear and extensional viscosity of viscoelastic polymers [1].

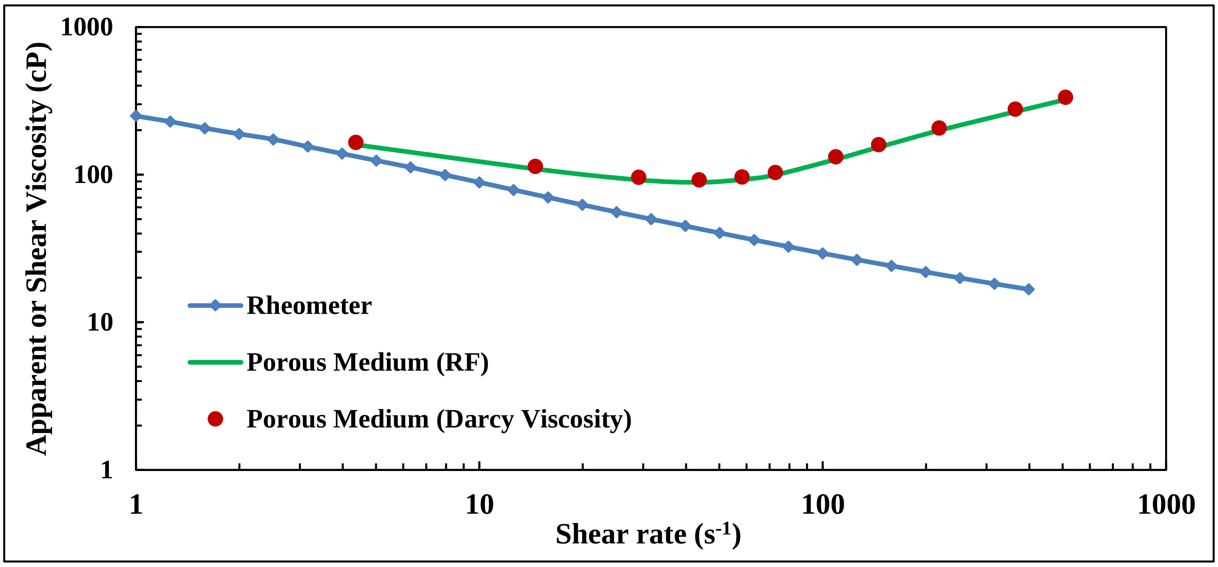


Figure 5 Viscosity profiles of prefiltered Polymer B (3000 ppm) as measured in porous medium and in bulk.

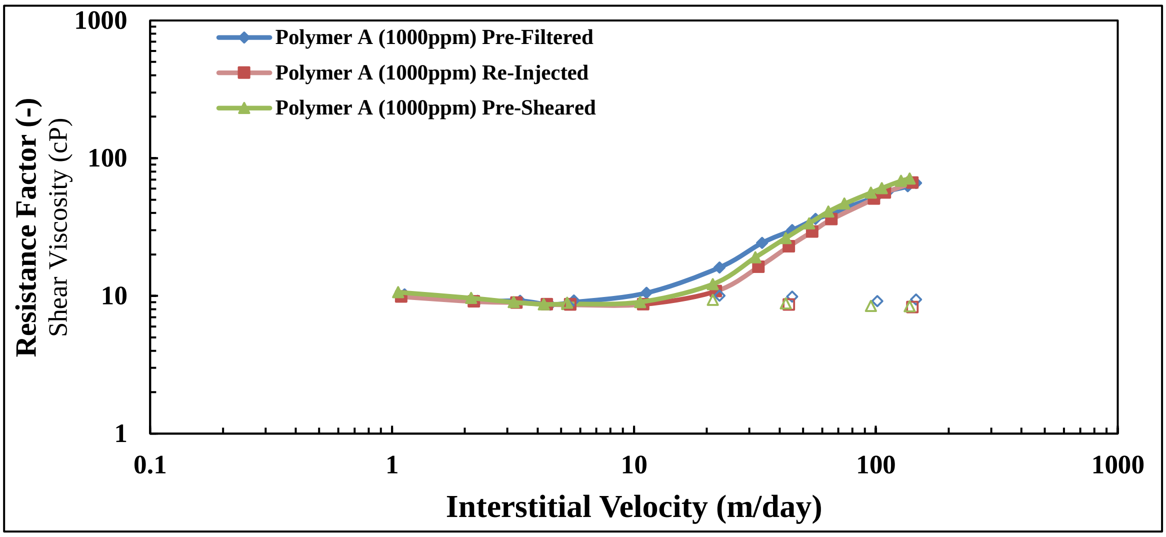
3.2.2. Flow of Semi-Dilute Polymer Solutions

Figure 6 depicts resistance factor of polymer A, B and C versus interstitial velocity. The concentration of polymer A and B was 1000ppm while for polymer C was 500ppm. Shear viscosity data of prefiltered solutions A, B and C were 8.4, 13.5 and 7.1 cP, respectively as tabulated in Table 4. At low velocities all the polymers exhibited near-Newtonian behavior followed by shear thickening at high velocities. This represents the general behavior of polymer flow in porous medium for semi-dilute solutions. Similar observations have been reported elsewhere [11,12,49,50,74]. The RF curves are strongly dependent on polymer molecular weight. For example, RF at reservoir velocities of polymer A was 2 times lower than that of polymer B which was ~ 18.4. Similarly, shear thickening behavior was more dramatic for polymer C with high Mw. This could be observed from the earlier onset of shear thickening for polymer C (v_c= 2.5 m/day) to that of polymer B and A (v_c = 4.1 and 7.0 m/day), respectively. Moreover, the stronger viscoelastic properties of high Mw polymer C can be observed from the slope of apparent shear thickening 7.7 $(\mathrm{m/day})^{-1}$ compared to 3.5 and 0.5 $(\mathrm{m/day})^{-1}$ for polymer B and A, respectively. This yields higher RF values for polymer C (RF ~196) at high flow rate compared to that of lower Mw polymers.

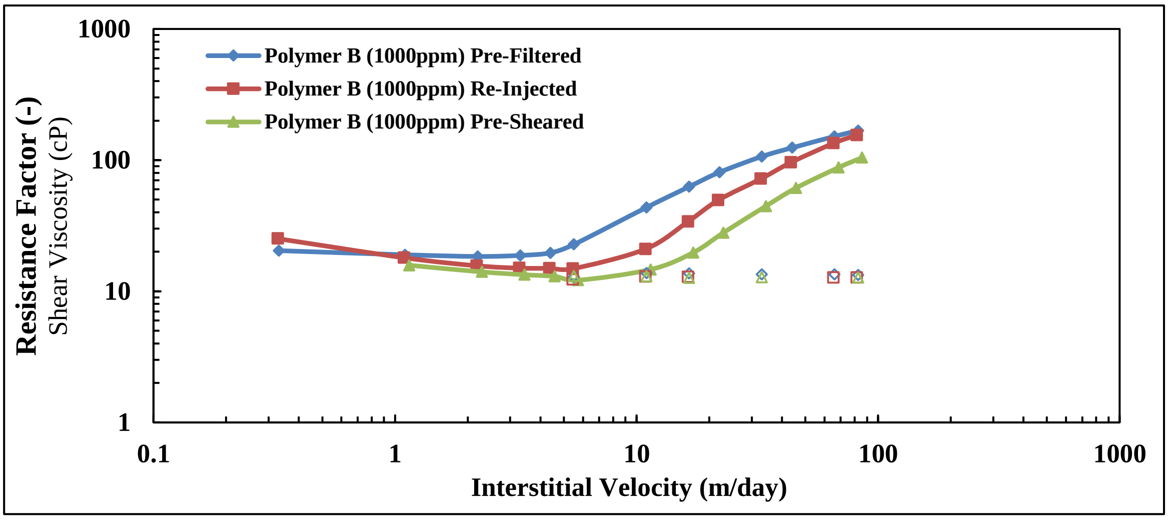
It is worth noting that, effluents shear viscosity of prefiltered solutions for polymers A and B did not show significant mechanical degradation at the investigated flow velocities. However, prefiltered polymer C showed degradation at high velocities (Deg = 21.7%). This could be the reason for lower RF values for reinjected polymer C in Figure 6c.

Reinjection process was carried out to simulate the polymer flow deep in the reservoir (radially distant from the wellbore). This process also demonstrates the effect of exposure time at high shear on polymer degradation. In this process the polymer solution passed two cores at different flow rates before measuring in-situ rheology in the main core. The first core (5 cm length) was used for pre-filtering process at low flow rate (Q=0.5cc/min). Where, the second core (10 cm length) which was used as shearing media. Hence, this process differs from the presheared polymer process in which the polymer solution was sheared in a short core and at high flow velocity before the measurement of in-situ rheology in the main core (see Figure 3 for details). Therefore, the results will be compared with pre-shearing process. Pretreatment methods (reinjection and preshearing) caused a reduction of RF values at high velocities while RF values were similar to prefiltered solution at low velocities. This could be clearly seen from the shift of the onset to higher velocities and reduction on the degree of shear thickening. In Figure 6a, RF profile of reinjected polymer A indicates more degradation occurred compared to presheared solution. This is analogous to the observation shown in Figure 6c for polymer C at low concentration. This confirm the occurrence of successive degradation as the polymer reinjected in porous media which is inline with some other studies [68,75]. This was not observed for polymer B. The successive polymer degradation observed in this study in contrast to the current understanding of mechanical degradation which is mainly confined to sand face degradation and is believed to be independent of travelled distance in porous media [38,49,66].

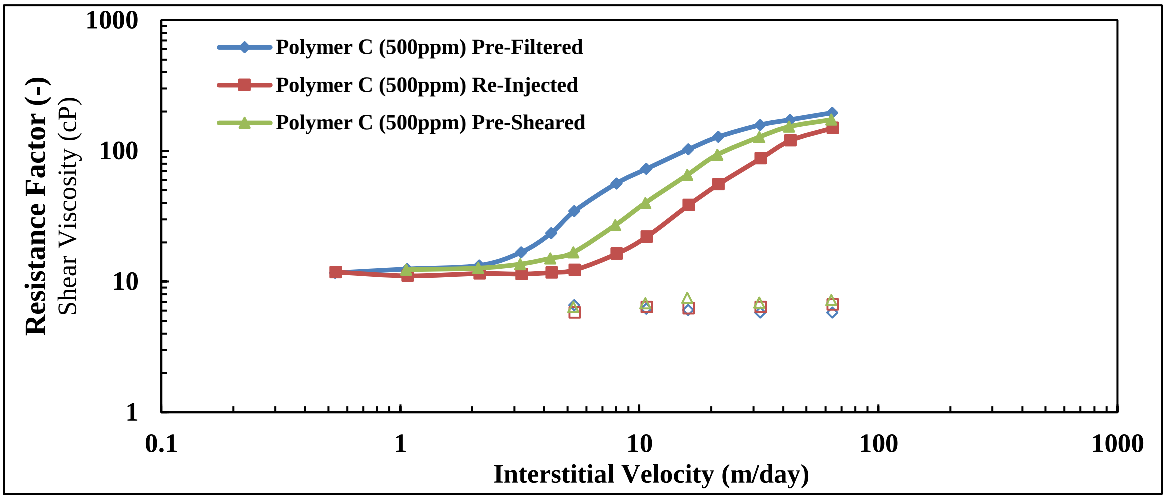
The difference between shear viscosity of effluents and resistance factor values at low velocities is varied depending on polymer type. This indicates that the difference in polymer retention correlates with the increase of polymer molecular weight. That is, the difference between RF values and effluent viscosity at similar velocities increases with increases of polymer molecular weight. For instance, Newtonian RF values were ~ 9, 18, 12 with bulk shear viscosity of 8.4, 13.5 and 7.1 cP for prefiltered polymers A, B and C, respectively.



a) Polymer A (Mw ≈ 8 MDa, Concentration = 1000 ppm)



b) Polymer B (Mw ≈ 12 MDa, Concentration = 1000 ppm)



c) Polymer C (Mw ≈ 18 MDa, Concentration = 500 ppm)

Figure 6 Resistance factor versus interstitial velocity for semi-dilute polymers A, B and C. Open markers indicate effluent shear viscosity for the given velocity measured at $\dot{\gamma} = 10\ s^{-1}$.

Table 4 Core and viscoelastic properties of semi-dilute polymers at concentration of 1000ppm for polymer A, B and 500ppm for polymer C.

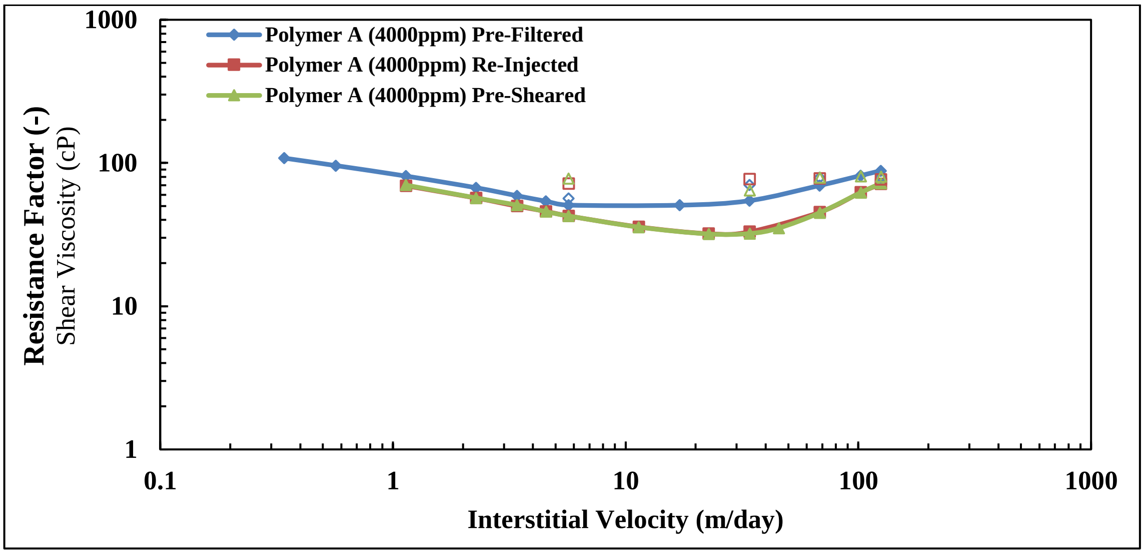
	L (cm)	D (cm)	ϕ (-)	K_{wi} (Darcy)	K_{wf} (Darcy)	RRF (-)	η_i (cP)	η_e (cP)	v_c (m/day)	m $(m/day)^{-1}$
Polymer A Pre-Filtered	9.28	3.79	0.23	1.81	1.12	1.61	8.44	8.19	6.98	0.54
Polymer A Re-Injected	9.74	3.79	0.23	2.26	1.16	1.95	8.19	8.28	16.92	0.53
Polymer A Pre-Sheared	10.22	3.77	0.24	2.40	1.48	1.62	8.52	8.42	15.79	0.66
Polymer B Pre-Filtered	9.82	3.79	0.23	2.16	0.96	2.24	13.57	13.31	4.06	3.50
Polymer B Re-Injected	9.57	3.79	0.23	2.08	1.24	1.68	13.31	12.75	7.69	2.28
Polymer B Pre-Sheared	10.27	3.77	0.23	2.80	1.54	1.82	13.54	12.75	11.99	1.46
Polymer C Pre-Filtered	9.82	3.77	0.24	2.57	1.40	1.84	7.11	5.79	2.51	7.68
Polymer C Re-Injected	9.78	3.77	0.24	2.39	1.28	1.86	5.79	-	6.71	3.00
Polymer C Pre-Sheared	9.72	3.77	0.24	2.25	0.82	2.75	7.13	7.21	4.00	4.32

3.2.3. Flow of Concentrated Polymer Solutions

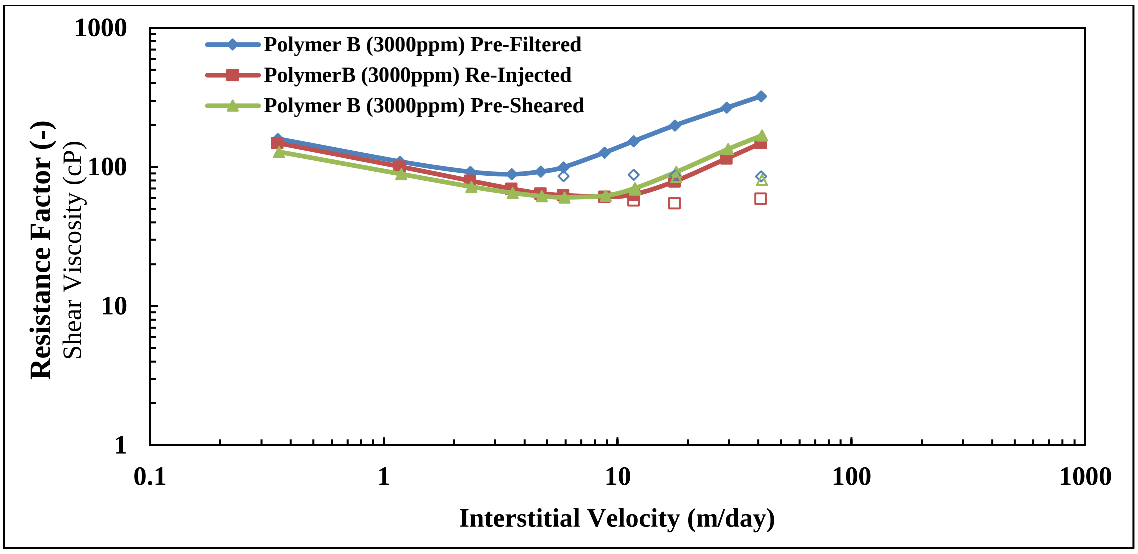
The behavior of concentrated polymer solutions ($C \gg C^{*}$) in porous media seems to be dominated by shear viscosity that overrides their elastic properties. In such solutions, the interaction between polymer molecules is dominant and polymer chains are entangled [11]. Figure 7 shows apparent shear thinning, near-Newtonian and shear thickening behaviors which become very important flow aspects for high concentrated polymer solutions with high shear viscosity. For instance, Figure 7a depicts the flow of polymer A at concentration of 4000ppm in porous media, in particular parlance, it exhibits pseudo-gel behavior. As this figure shows for prefiltered polymer A, at high velocities (v > 31.0 m/day) weak shear thickening behavior is observed with slight increase in RF values compared to near-Newtonian plateau at flow velocities 5.7 < v < 31.0 m/day. RF values corresponding with shear thickening behavior are lower than bulk shear viscosity. This suggests that the contribution of extensional viscosity is lower than that of shear viscosity that dominates the polymer flow behavior. Below v < 5.7 m/day, the RF values increase with decreasing flow velocity that indicates strong shear thinning behavior. Similar trend was observed for relatively higher Mw polymer B except the shear thickening becomes stronger (see Figure 7b). This confirms that the concentration or in another term solution conformational regime has an obvious influence on polymer in-situ rheology. However, concentrated high Mw polymer C (Figure 7c) exhibits near-Newtonian behavior at lower shear rates that is analogous to semi-dilute polymers discussed earlier without the presence of shear thinning behavior. This could be ascribed to lower shear viscosity of polymer C at 1000ppm compared to the other two polymers A and B. Higher molecular weight polymers possess higher RF at high velocity than the low Mw polymers with higher concentration. This indicates also the contribution of higher elastic properties such as elastic viscosity compared to that of lower Mw solutions.

The existence of shear thinning in porous media is conditional and argued by Seright, *et al.* [76] at which they attribute shear thinning to presence of micro-gels in polymer solution. However, the results in Figure 7 show a contradictory observation and confirm the existence of shear thinning phenomena for concentrated solution in porous media even if the polymer has been pre-filtered or presheared (microgels-free).

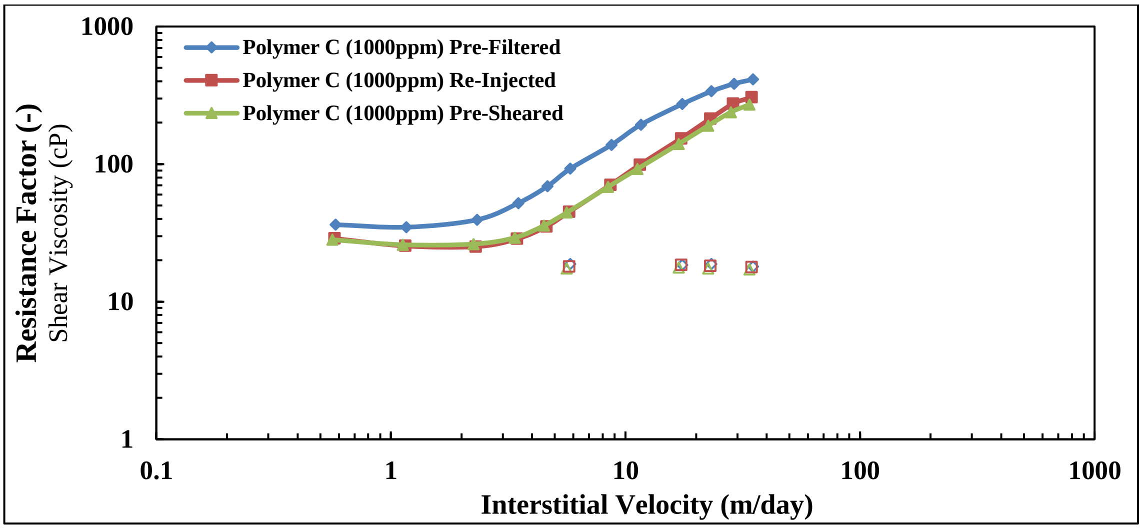
Exposing the polymer to wellbore mechanical degradation alters its viscoelastic behavior through the shift of Newtonian plateau and onset of shear thickening to higher velocities as is seen in Figure 7 for both reinjected and presheared solutions A and B while insignificant alteration occurs on shear thinning part. Effluent viscosity of Reinjected solution B suffers more degradation compared to presheared sample which could be due to attachment of backpressure device that might induce additional degradation to polymer solution [67]. RF curves of degraded solutions C (reinjected and presheared) are coincide which indicate the solution tolerates wellbore mechanical degradation compared to semi-dilute solution with same polymer C discussed above (see Figure 6c). However the RF curves of degraded solution C were lower than prefiltered solution which could be due to filtration effect and also lower RRF (see Table 5.).



a) Polymer A (Mw ≈ 8 MDa, Concentration = 4000 ppm)



b) Polymer B (Mw ≈ 12 MDa, Concentration = 3000 ppm)



c) Polymer C (Mw ≈ 18 MDa, Concentration = 1000 ppm)

Figure 7 Resistance factor versus interstitial velocity for concentrated polymers A B and C at concentration of 4000 3000 and 1000 ppm, respectively. Open markers indicate effluent shear viscosity for the given velocity measured at $\dot{\gamma} = 10\ s^{-1}$.

Table 5 Core and viscoelastic properties of concentrated polymers A, B and C at concentration of 4000, 3000 and 1000 ppm, respectively.

	L (cm)	D (cm)	ϕ (-)	Kwi (Darcy)	Kwf (Darcy)	RRF (-)	η_i (cP)	η_e (cP)	v_c (m/day)	m $(m/day)^{-1}$
Polymer A Pre-Filtered	9.69	3.77	0.23	2.75	1.48	1.86	83.02	79.31	31.00	0.37
Polymer A Re-Injected	10.05	3.74	0.23	2.53	1.58	1.60	79.31	76.74	43.00	0.43
Polymer A Pre-Sheared	9.95	3.77	0.23	2.50	1.84	1.36	77.91	-	42.00	0.45
Polymer B Pre-Filtered	9.85	3.79	0.22	2.40	0.73	3.31	88.76	85.90	4.37	8.45
Polymer B	9.52	3.78	0.22	2.64	1.04	2.53	85.90	66.12	11.21	2.96

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Re-Injected										
Polymer B Pre-Sheared	9.93	3.78	0.22	2.35	0.85	2.77	83.79	80.79	9.08	3.59
Polymer C Pre-Filtered	10.04	3.78	0.22	2.12	0.23	9.27	18.95	17.98	2.31	16.07
Polymer C Re-Injected	9.81	3.78	0.22	2.01	0.33	6.09	17.98	17.46	3.78	9.71
Polymer C Pre-Sheared	9.68	3.78	0.23	2.37	0.80	2.96	17.86	17.14	3.40	8.47

3.2.4. Onset and Slope of Shear Thickening Behavior

Figure 8 presents the resistance factor change for prefiltered polymers A, B and C at different concentrations. As this figure shows, RF is influenced by both polymer molecular weight and concentration. That is, RF gains strength with increasing Mw and concentration. This is inline with the increase in shear viscosity shown in Figure 4 where shear viscosity increases with increase in Mw and concentration. However, the impact of Mw and concentration on RF values in the porous media is a function of velocity. RF is dominated by molecular weight at high velocities to greater extent than concentration and vice versa at low velocities below the onset of shear thickening. For instance, RF curves of polymer A converge to similar values at high velocities regardless of the significant difference in concentration while the concentration differentiates the RF at low velocities. This indicates the contribution of both shear and elastic viscosity in polymer flow through porous media, although shear viscosity reaches its minimum at high velocities [74]. The degree and magnitude of shear thickening increase with increase in molecular weight and concentration. This highlights the influence of shear and elastic viscosities on the slope of apparent shear thickening [37].

The onset of shear thickening and the flow behavior of polymer are more important than the extensional magnitude itself in determining the suitability of polymer for EOR applications [77]. Hence, onset of shear thickening has received an extensive attention in the literature [50,52,53,78]. Figure 8 shows polymer molecular weight has an obvious influence on the onset of shear thickening. A solution with higher molecular weight experience earlier onset of shear thickening. With increasing polymer Mw, the apparent shear thickening increases, conversely, the onset of shear thickening shifts to lower velocities [79].

Moreover, Figure 8 shows that onset of shear thickening is independent of polymer concentration. This observation excludes 4000ppm polymer A which exhibits a gel-like behavior and yields shift of the onset to higher velocities. This confirms that the conformational state of polymer solution has more influence on the onset of shear thickening compared to the concentration value itself. This could be one of the reasons that cause a controversial observations in the literature regarding the correlation between the onset and polymer concentration. For example, Chauveteau and Moan [52] reported the onset of shear thickening decreases with increasing polymer concentration for investigated wide concentration range (21-1360 ppm). However, a close look on the reported data, we could see the onset was almost similar for a certain concentration range (e.g., 170-680 ppm). This again indicates the conformational state influences the onset of shear thickening. For instance, within a semi-dilute regime, the onset of shear thickening decreases with molecular weight increase regardless of concentration [79,80]. The apparent viscosity from resistance factor is gradually increases as polymer concentration increases [81].

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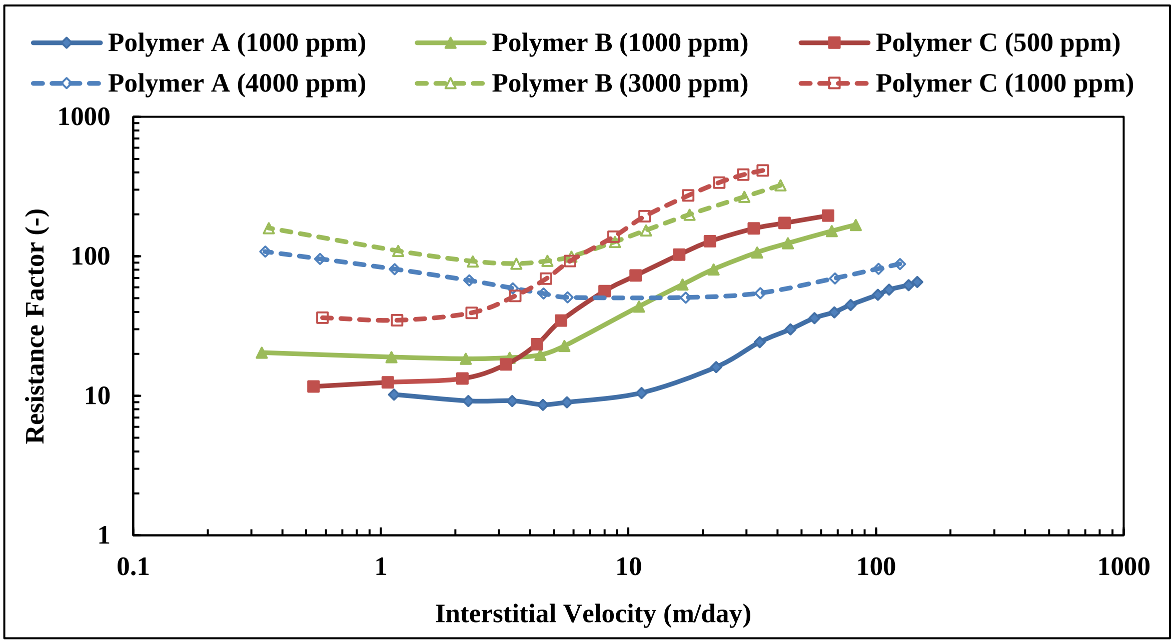

Figure 8 Resistance factor versus interstitial velocity for pre-filtered Polymer A, B and C at different concentrations

3.3. *Polymer Mechanical Degradation*

The presented results showed the impact of wellbore mechanical degradation on both bulk shear viscosity and in-situ rheology. Despite insignificant impact of mechanical degradation on shear viscosity of effluents, a considerable alteration of in-situ rheology behavior was occurred. The significant alteration was found with reduction of apparent shear thickening behavior by shifting its onset to higher velocity and reduction of the slope while maintaining in-situ viscosity. The amount of alteration was influenced by polymer conformational regime. For instance, the change of the onset of shear thickening by comparing presheared solution A with a prefiltered sample was 126.2% at concentration of 1000 ppm, while this percentage drops to 35.5% when polymer concentration increases to 4000 ppm. This is also valid for polymer B and C. This elucidates that increasing polymer concentration is beneficial for polymer shear stability [36,82]. However the impact of mechanical degradation on the slope of shear thickening was independent of polymer conformation regime (concentration). For example, the change on the slope of shear thickening of presheared solution B compared to prefiltered sample was 58.6% at concentration of 1000 where it was 57.5% when polymer concentration increased to 3000 ppm. Similar observations were found for the other two polymers. This indicates that the impact of wellbore mechanical degradation on shifting onset of shear thickening to higher velocities was lower for concentrated solutions compared to that of semi-dilute polymer solutions. While the change in the slope of shear thickening due to mechanical degradation seems independent of concentration.

3.3.1. Influence of Mechanical Degradation on In-Situ Rheology

Figure 9 depicts the impact of mechanical degradation on reduction of RF values of high Mw polymers B and C to that of prefiltered polymer A which has relatively lower Mw. Recall from the discussion above, polymer Mw is a dominating factor on the polymer flow behavior after onset of shear thickening for semi-dilute polymers. Reduction of slope and shift of onset of shear thickening to higher velocities is an indication of reduction of polymer MWD [50]. For example, the degree was reduced and onset of shear thickening of high Mw Polymer C (Mw ≈ 18 MDa) shifted to higher velocities due to preshearing. Therefore, RF curve similar to that of lower Mw prefiltered Polymer B

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(Mw ≈ 12 MDa) was achieved. The similar observation was found for presheared polymer B where preshearing resulted in shifting RF values closer to RF of prefiltered polymer A (Mw ≈ 8 MDa). This was also observed for concentrated solutions as shown on the right Figure 9.

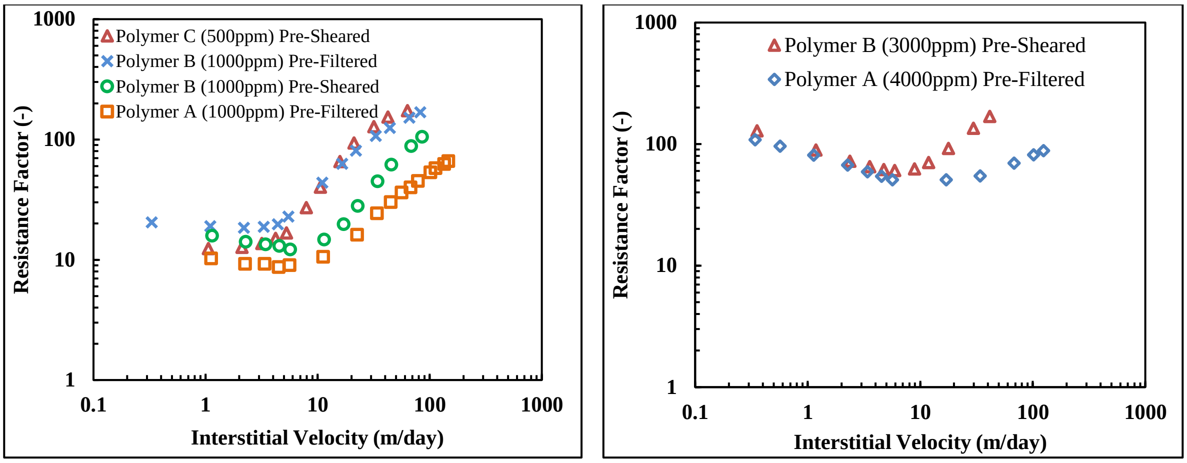

Figure 9 Influence of pre-preshearing on shifting the in-situ behavior of high Mw polymer to that of lower Mw prefiltered solution. On the left, solutions with lower concentration. On the right, solutions with concertation.

3.3.2. Mechanical Degradation at Elevated Velocities

Figure 10 displays effect of mechanical degradation on shear viscosity of effluent polymer solutions at a broad range of shear rates. These experiments were designed to compare the degradation effect in different polymers which have been exposed to comparable shear rate. It is clear that, polymer C with high Mw experienced more shear degradation at similar injection rate applied for all solutions. For instance, degradation at Q=90cc/min was 4.0, 12.0, 20.0% for polymer A, B and C respectively.

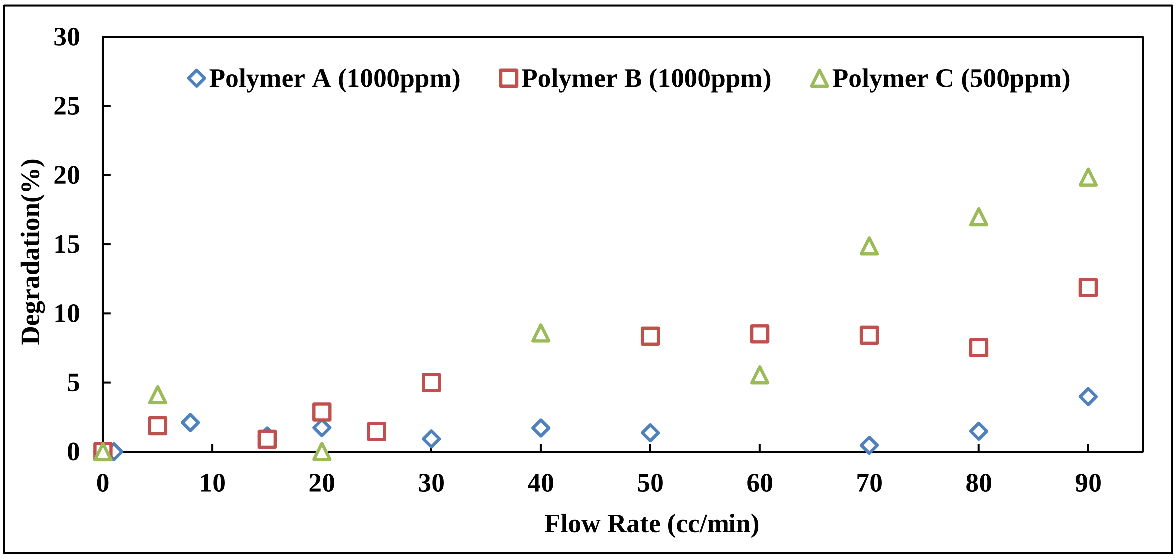

Figure 10 Shear degradation at elevated velocities for polymer A, B and C.

Figure 11 shows the flow of polymer B that has been presheared at different flow rates. Fresh (undegraded) solution is the same solution described in Figure 10 that has a shear viscosity of 13.3 cP and was injected into short Bentheimer core at various injection rates. It can be seen that, the RF profile of fresh solution is identical to RF profile of prefiltered solution at similar velocities. This indicates that prefiltering at low flow rates ($Q \leq 0.5$ cc/min ,$v_D \leq 0.6$ m/day) will not alter RF values.

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However, increasing preshearing rate to (Q = 15 cc/min, v_D = 19.4 m/day) will significantly alter viscoelastic properties such as the onset and degree of shear thickening, while not significantly affect in-situ viscosity and bulk shear viscosity. However preshearing the polymer at very high injection rate (Q = 110 cc/min, v_D = 141.2 m/day) causes a shear degradation of 16% while a considerable reduction(> 50%) on in-situ viscosity by comparing its Newtonian plateau that observed in porous media with prefiltered solution. The reduction of polymer viscoelastic properties such as the onset and degree of shear thickening is extremely high. The maximum RF value of presheared solution at Q=110 m/day was 26.6 which is more than 6 times lower than that of prefiltered solution at comparable velocity. Additionally, RRF of presheared solution at Q=110cc/min was reduced to 1.7 compared to 1.8 and 2.2 for presheared solution at Q=15cc/min and prefiltered solution at Q=0.5cc/min, respectively.

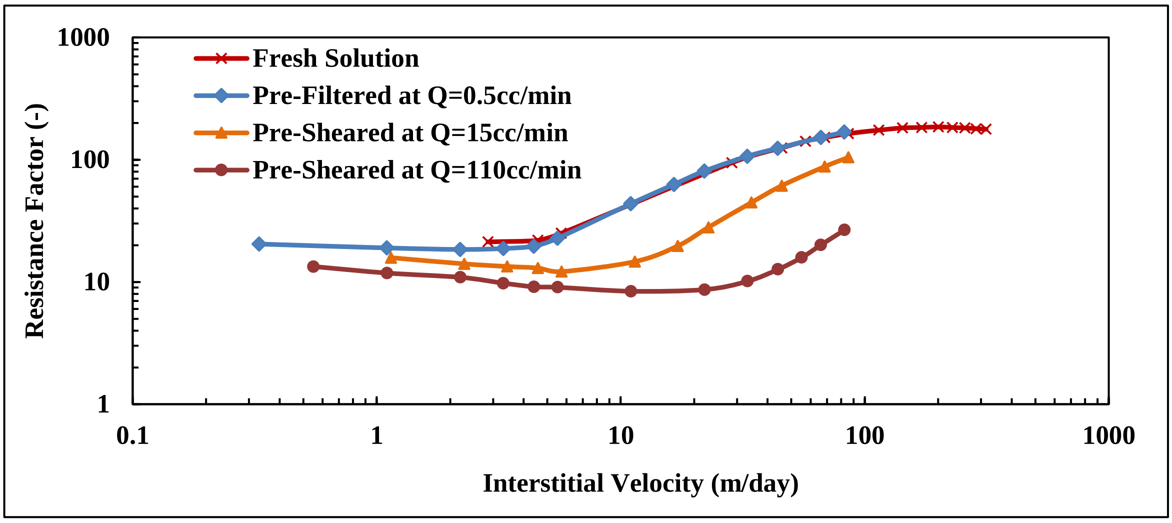

Figure 11 Resistance factor versus interstitial velocity for polymers B presheared at different flow rates.

3.4. *Permeability Reduction*

Polymer retention in porous media has similar importance as its viscoelasticity discussed above. It is a determining parameter in screening EOR polymers. When HPAM transported in porous media, it tends to adsorb on rock surfaces which might also trap within small pores resulting in polymer retention. Polymer retention reflects polymer adsorption, mechanical entrapment and hydrodynamic retention [72]. As a consequence of polymer retention, permeability reduction occurs. Experimentally permeability reduction could be evaluated by residual resistance factor (RRF) [57]. Figure 12 depicts RRF values measured for prefiltered polymers A, B, C at different concentrations. It can be see that, concentrated polymers have higher RRF compared to solutions with lower concentration. Furthermore, RRF appears significantly dependent on polymer Mw to greater extent than concentration. This could be elucidated by looking at similar polymer concentration of 1000ppm, we can see RRF for polymer A, B and C were 1.6, 2.2 and 9.3 respectively. This emphasizes the increase of RRF with Mw [79].

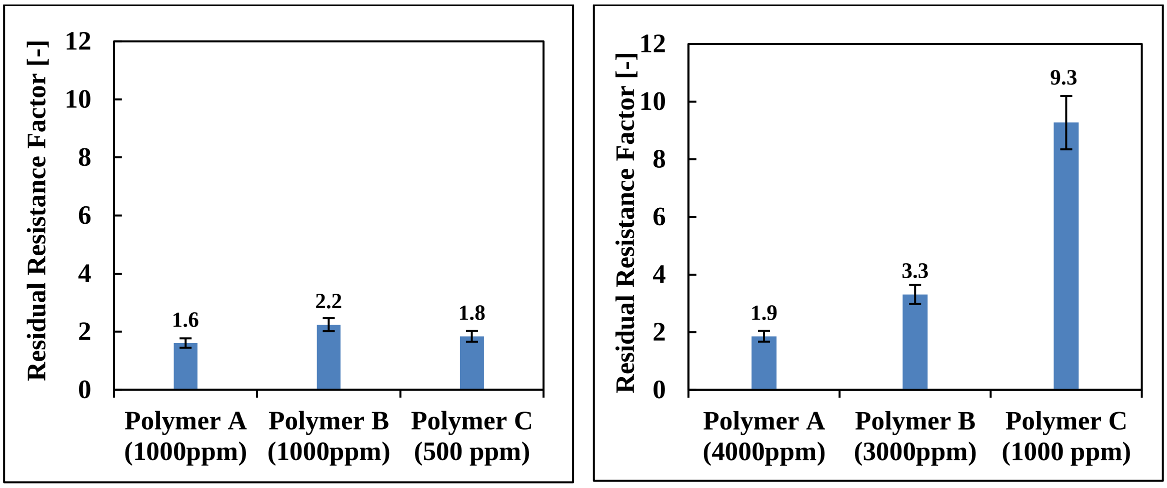

Figure 12 Residual resistance factor (RRF) of prefiltered polymers A, B and C. On the left, polymers with lower concentration. On the right, concentrated polymers.

Figure 13 displays the impact of polymer pretreatment (prefiltering, reinjecting and preshearing) on RRF values. Presented RRF values are quite scattered. This could be due to challenges on measuring 'true' RRF which has been debated in literature [83]. One reason could be due to experimental artifacts ascribed to the amount of brine and strategies applied during brine post-flush such as tapering [67]. Another reason could be due to unapproachable steady-state condition during injecting brine alone after polymer flooding to satisfy Darcy's Law conditions stated in Eq.2. This is suggested due to viscoelasticity of retained molecules [84]. However, in some cases the impact of mechanical degradation on RRF was not significant. This might be due to the reason that high molecular species tend to adsorb first as the polymer transports in porous media. High molecular species could also be found in degraded solutions if degradation is not significant which they are enough to form similar adsorbed layer of non-degraded solutions [70]. Measuring 'true' RRF is an essential task that would certainly improve the estimation of polymer effective viscosity in porous media by using the term RF/RRF.

However, the general trend from the data presented in Figure 13 shows that pretreatment of polymer solutions prior to injection into the porous media results in reduction in RRF vales. That is, RRF of prefiltered solutions generally higher than RRF of presheared and reinjected solutions.

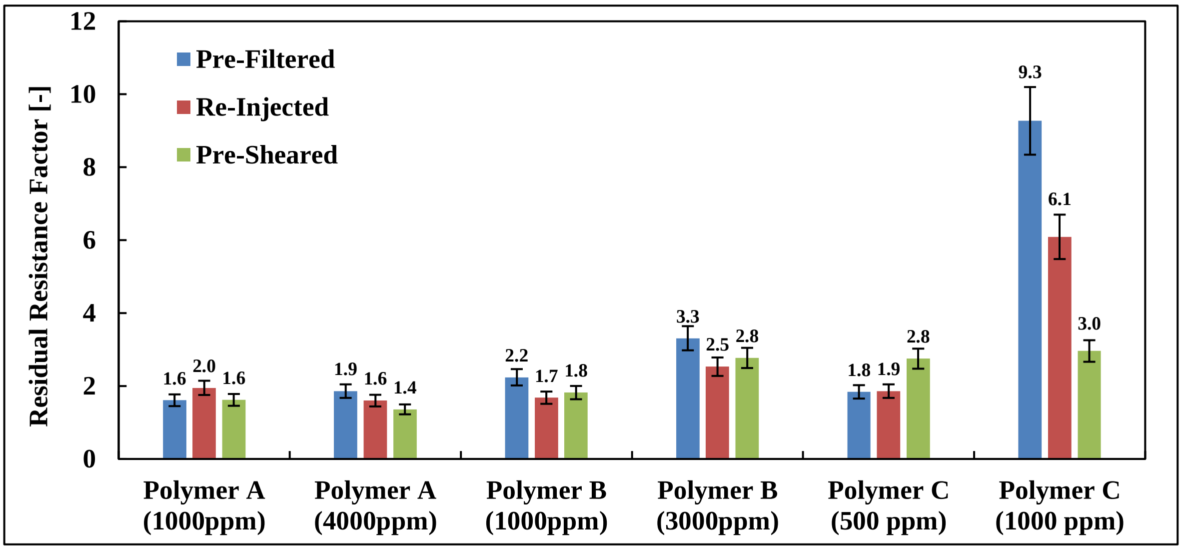

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Figure 13 Residual resistance factor (RRF) of polymer A, B and C at different concentrations.

3.5. *Polymer Injectivity*

As it was stated before, polymer injectivity is a measure of how easily polymer solution can be delivered into reservoir matrix. The shear viscosity measurement alone cannot predict injectability of polymer solutions, as it does not reflect the existence of all flow regimes during polymer flow in porous media. Besides other factors, matrix fracturing is one major concern in polymer flooding projects that restricts polymer injection. Injection under matrix condition that might evolve into fracture formation is only a function of injection pressure, irrespective of polymer bulk viscosity [85]. This means fracture initiation is more attributed to polymer viscoelasticity particularly shear thickening behavior that yields-in significant pressure build-up. Therefore, polymer injectivity could be inferred from RF and RRF measurements. The pressure gradient associated with high RF values that found at wellbore region reduces polymer injectivity. Additionally, the decline in polymer injectivity might occur when retention is high as reflected by high RRF in this study (e.g., RRF > 3 is not recommended for EOR applications). Shear thickening may be dampened for each polymer by two ways either by increasing the polymer concentration or mechanically degrading the polymer solution prior to the injection. From economical prospective, the former requires increasing the dosage of polymer which subsequently demands high cost. Also it might yield in high RRF. Whereas the latter relates to polymer type more specifically polymer Mw at which the cost of manufacturing process of low and high Mw polymer is quite similar [86]. The loss of shear viscosity within 20-30% due to preshearing could be tolerated economically and cause significant reduction in extensional viscosity that results in a reduction of resistance factor [71]. Additionally, preshearing could also be beneficial at which high molecular species are sheared and avoided. These species are mainly responsible for wellbore plugging problems that result in permeability reduction. This will increase that shear rates at wellbore area that promote pressure build-up and eventually mechanically degrade the polymer. This suggests preshearing high Mw polymer is a favorable strategy to optimize its injectivity which is consistent with other studies [87].

4. Conclusions

Series of core flood experiments have been performed to investigate in-situ behavior of partially hydrolyzed polyacrylamide polymers (HPAM) in porous media. The influence of HPAM molecular weight and concentration on polymer in-situ rheology has been investigated. Additionally, the impact of mechanical degradation on polymer rheological behavior has been studied. Based on the results, polymer injectivity can be optimized. More specifically, the following conclusions could be made:

- In-situ rheological behavior of HPAM in porous media is different from bulk rheology observed in rheometer.
    - Shear thickening behavior was observed at high velocities representative of those present in the near wellbore region. Near-Newtonian behavior was observed at low velocities representative of those present deep in the reservoir.
    - Onset of shear thickening is dependent on molecular weight.
    - The degree and magnitude of shear thickening increased for higher polymer Mw and concentration.
    - Shear thinning behavior at low velocities was observed for concentrated solutions while not for semi-dilute solutions.
- Exposing HPAM solutions to mechanical degradation through preshearing process prior to injection facilitates its flow in porous media and enhances its injectivity. This is ascribed to a reduction in viscoelastic properties.
    - Onset of shear thickening shifted to higher velocities.
    - The magnitude and the degree of shear thickening behavior is reduced.
    - In-situ viscosity at low flow rates is maintained.

- RRF appears to be dominated by molecular weight and concentration.
  - High RRF found for high molecular weight polymers with high concentration.
  - Degraded solutions have lower RRF values specifically for concentrated solutions of high Mw polymers. This effect is more pronounced when polymer solution is degraded at very high velocities.
- Improvement (reduction) in polymer viscoelastic properties and RRF through preshearing process can optimize polymer injectivity.

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Conflicts of Interest: The authors declare no conflict of interest.

Nomenclature

A = cross-sectional area (cm^2)
AF4 = Asymmetrical flow field-flow fractionation
C = concentration (ppm)
C^* = critical overlap concentration (ppm)
D = core dimeter (cm)
Deg = degradation (%)
EOR = enhanced oil recovery
GPC = gel permeation chromatography
HPAM = partially hydrolyzed polyacrylamide
k = viscosity constant
K_{abs}= absolute permeability (Darcy)
K_{wf} = absolute permeability after polymer flow (Darcy)
K_{wi} =absolute permeability before polymer flow (Darcy)
L = core length (cm)
Mw = molecular weight (MDa)
MWD = molecular weight distribution , dimensionless
n = power law index, dimensionless
PV = pore volume, dimensionless
Q = flow rate (cc/min)
RF = resistance factor, dimensionless
RRF = residual resistance factor , dimensionless
SAOS = small-amplitude oscillatory shear
SEC = size exclusion chromatography
TDS = total dissolved solids
v = interstitial velocity (m/day)
v_c = onset of shear thickening (m/day)
v_D = Darcy velocity (m/day)
ΔP_p = pressure drop during polymer flow (bar)
ΔP_w = pressure drop during water flow (bar)
η_e= effluent viscosity (cP)

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η_i= injected solution viscosity(cP)
η_w= brine viscosity (cP)
ϕ = porosity, dimensionless
$\dot{\gamma}$ = shear rate (s^{-1})

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