

# Lab scale experiments to support large-scale test

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## Summary

The effect of core length on mechanical degradation of synthetic EOR polymers has been investigated. It has been concluded that there is a length effect; after injection through 29 cm of porous media the degradation is higher than through 3 cm. However, the degradation gradient is steepest close to the core inlet. Recycling polymer at constant flow rate revealed that polymer continue to degrade, even at effective core length of 19 meter. However, the degradation is associated with high pressure gradients, on the order of 100 bar/meter, which at field scale is not realistic.

In experiments mimicking radial flow the mechanical degradation was found to depend on the maximum velocity and not on invasion depth.

We confirmed previous findings that mechanical degradation decreases by decreasing molecular weight and decreasing the brine salinity. In terms of filterability, the tested polymers have excellent filtration properties and permeability reduction after injection of more than 10 L of polymer was low.

## Introduction

The main reasons for doing large scale tests is to validate test results obtained at different scales, e.g. how to scale core flood experiments to field scale. In such a perspective the large scale test on polymer degradation in choke-valves was successful and we were able to match degradation in experiments performed at turbulent flow with small scale experiments at laminar flow.

Phase 2 of this project is planned to focus on mechanical degradation of polymer in porous media, e.g. can mechanical polymer degradation be quantified by critical stretch rate or does degradation also depend on some length effects. Large scale experiments will be duplicated at cm-scale core flood experiments as well as at large scale.

The results from the lab-scale experiments will be used in designing the large-scale experiments.

## Experimental design

Polymer was flooded through multiple Bentheimer core sections. A 30 cm long core, diameter is 3.8 cm, was divided into 4 core segments, with lengths of 3, 5, 8, and 13 cm. We mounted each core segment into a core holder with over burden pressure of at least 130 bar.

The cores were evacuated and then flooded with brine. Pore volume and porosity were measured. Then, the cores were mounted into the flooding rig, shown in Fig. 1. The experimental set-up allows for injection through or bypass the core segments. It also allow us to reinject the effluent, where the effective core length is proportional to the number of cycles. Thereby the effective length can be increased substantially.

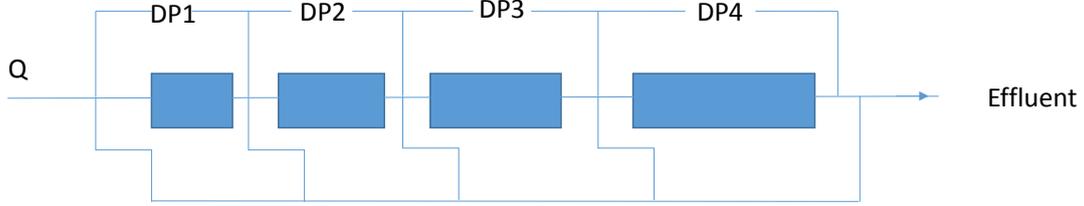


Fig. 1 Experimental set-up.

Two different polymers, high molecular weight HPAM (FP3630) and medium weight ATBS (AN125) were used. Both polymers are commercial SNF polymers and are the same polymers as used in the large-scale test on polymer degradation in choke valves.

We used two different brine compositions, 0.5 wt% NaCl (NaCl) and synthetic seawater (SSW) filtered through 0.45  $\mu\text{m}$  filters. First, a mother solution at 10000 ppm in NaCl was prepared using standard SNF mixing procedure. Thereafter, we diluted the mother solution to 1000 ppm by either NaCl or SSW and measured the viscosity of the 1000 ppm polymer solution vs. shear rate on a Physica Anton Paar Rheometer. In Exps. 3 to 5 we also reduced oxygen content, first by bubbling brine with  $\text{N}_2$  gas and then added 50 ppm of  $\text{Na}_2\text{SO}_3$  and 50 ppm NaSCN scavengers.

The polymer solution was injected at constant rate through the pumps (2 Quizix QX pumps in parallel with maximum flow rate of 2 x 50 ml/min) and cores while monitoring the differential pressure across the cores, by the use of Fuji 0-30 bar pressure transmitters. At stable differential pressure along each core, effluent samples were taken and the viscosity was compared with the injected one.

Polymer degradation is reported as  $\eta_D = 1 - \eta_R = 1 - \frac{\eta - \eta_w}{\eta_0 - \eta_w}$ . Here  $\eta_w$  is the brine viscosity,  $\eta_0$  is the injected polymer viscosity. The viscosities are taken as the average viscosity over 5 viscosity measurements in the shear range from 5.6 to 0.9  $\text{s}^{-1}$ .

Mobility reduction,  $RF$ , defined as the ratio between differential pressure of polymer injection and brine injection was derived for flow rates varying from 0.5 to 98 ml/min. We report  $RF_i$  and  $\eta_{Ri}$  vs. flow rate. In terms of shear rate we used  $\dot{\gamma} = \frac{4\alpha q}{A\sqrt{8k\phi}}$ , where  $q/A$  is the Darcy velocity,  $k$  is the absolute permeability,  $\phi$  is the porosity and  $\alpha$  is factor set equal to 2.5. In some of the experiments we reported the pressure gradient, which from Darcy's law is  $\nabla P = \frac{\Delta P}{l} = \frac{1}{k} v\eta$ .

In total, we injected approximately 10 L of polymer through the cores, followed by equal amount of brine. Endpoint permeability reduction, *RRF*, was derived as the ratio between initial and final permeability. All the experiments were performed at 20°C.

### Main results

Fig. 1 shows bulk viscosity vs. shear rate at 20°C for the four different polymer combinations. Not surprisingly, viscosity increases by increasing molecular weight and decreasing salinity.

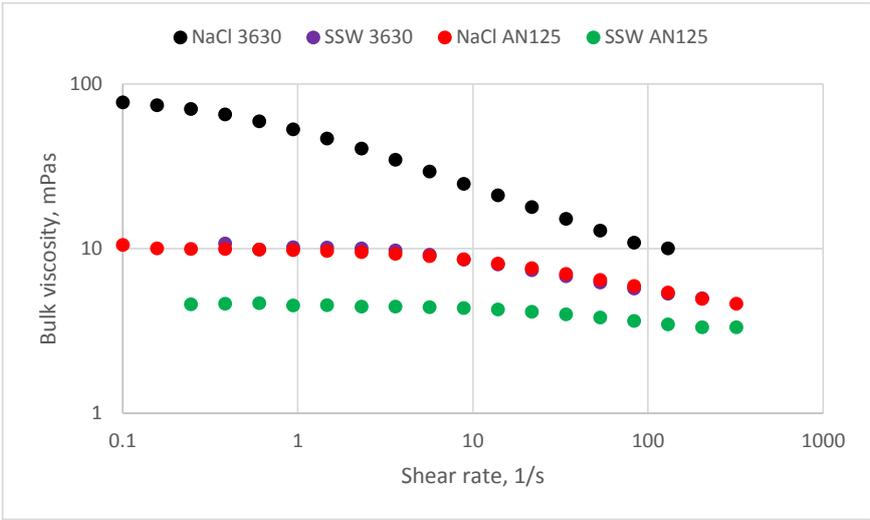


Fig. 1 Bulk viscosity at 20° for the different polymer combinations.

As reported in Table 1, there are some permeability variations for the different core segments. However, the average permeabilities for the entire cores are similar, in the range of 1.8 Darcy. Porosity for the different core segments were 0.210.

Table 2 Core properties.

Core segment	1	2	3	4	Total
Length, cm	3	5	8	13	29
Exp.	Permeability, Darcy				
1	1.31	1.14	2.31	2.56	1.80
2	2.17	1.83	2.04	1.94	1.96
3	2.32	1.37	1.58	1.88	1.71
4	1.83	1.21	1.85	2.15	1.80

In Exp. 1 we injected 1000 ppm 3630 in 0.5% NaCl. Fig. 2 indicates the flood sequence. The following observations can be made: (1) Differential pressures are extremely high, 50 to 80 bar per core segment, (2) No plugging tendency, even though more than 1000 pore volumes of polymer were injected. From the stable differential pressure data, we derive  $RF$ , see Fig. 3. At flow rates below 10 ml/min  $RF$ s are similar, while at  $q > 10$  ml/min  $RF_1$  is significantly higher than the subsequent  $RF$ s. Note that  $q = 10$  ml/min corresponds to flow velocity,  $v = 61$  m/d or shear rate,  $\dot{\gamma} = 850$  s<sup>-1</sup>.

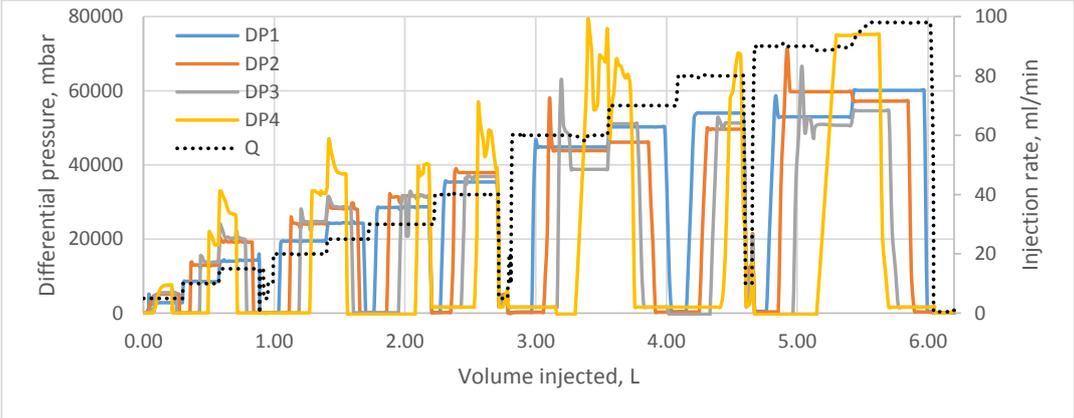


Fig. 2 Differential pressure across the core segment during injection of 1000 ppm 3630 in 0.5% NaCl.

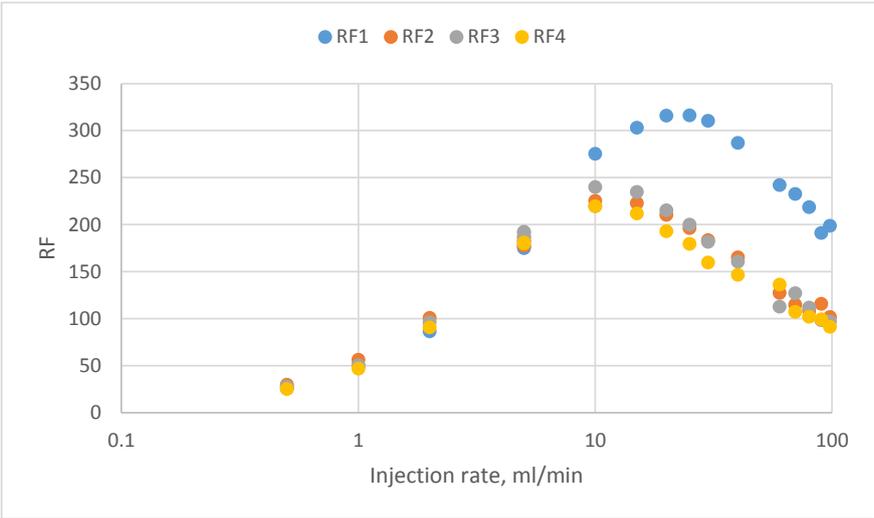


Fig. 3 Mobility reduction vs. injection rate, 1000 ppm 3630 in 0.5% NaCl.

Polymers were sampled at each flow rate. Fig. 4 shows the normalized remaining viscosity,  $\eta_{Ri}$ , for the different core segments. Note that the polymer reported  $\eta_2$  has been flooded through core segments 1 and 2. As can be seen, the polymer was not degraded in the pump system or in flow lines;  $\eta_{R0}$  is constant. We observed some degradation in the core segments, and viscosity degradation increases by increasing the flow rate.

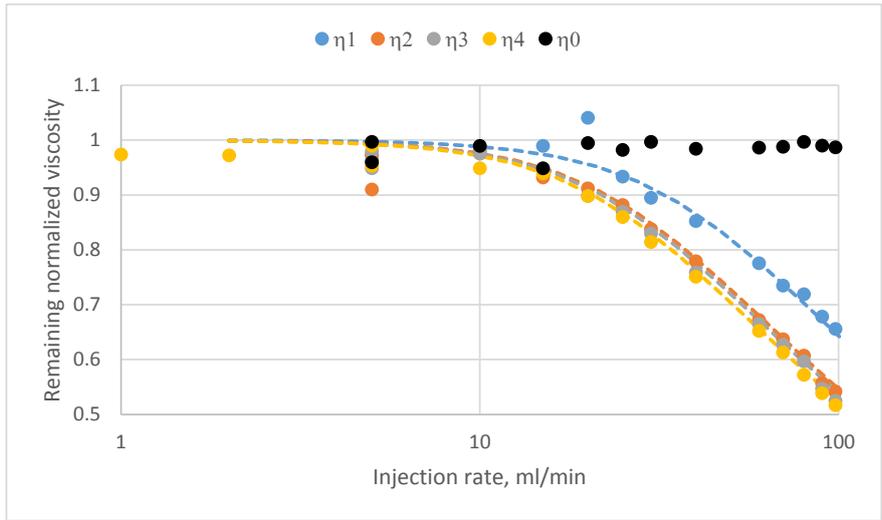


Fig. 4 Viscosity degradation vs flow rate, 1000 ppm 3630 in 0.5% NaCl.

Viscosity, shown in Fig. 4, is well matched with the following form  $\eta_{Ri} = \left[1 + \left(\frac{q}{q_c}\right)^2\right]^{-m/2}$ . Here  $m = -\frac{1}{2}$  and  $q_c$  is the critical flow rate for onset of degradation, i.e.,  $q_c$  is here proportional to shear rate. It can be seen that polymer is degraded in core segment 1, further degraded in core segment 2, thereafter, marginal degradation in core segments 3 and 4. The significant degradation in core segment 2 may be because of the low permeability in core segment 2 compared with core segments 3 and 4, see Table 1. However, there are some length effect on polymer degradation, as indicated in Fig. 5. Here, at each flow rate, approximately 60 to 70% of the total degradation takes place across the first 3 cm of the core.

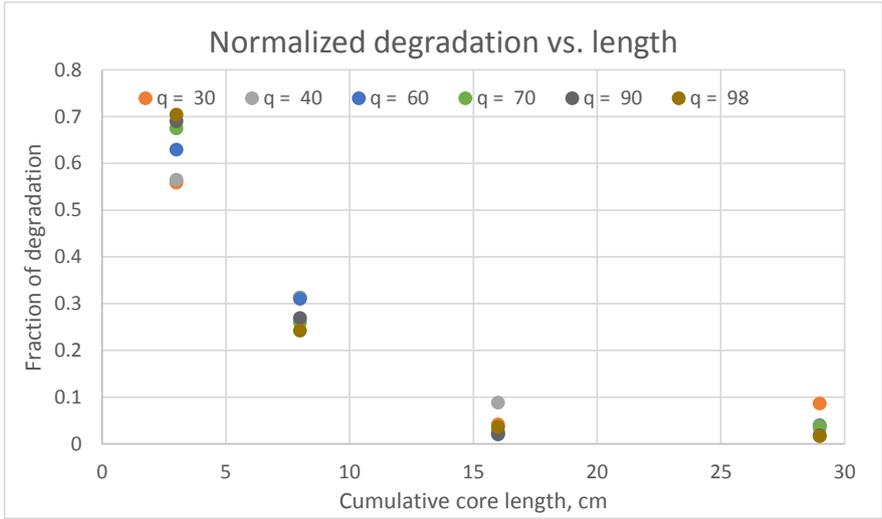


Fig. 5 Polymer degradation per core segment, 1000 ppm 3630 in 0.5% NaCl.

In Exp. 2 we injected 1000 ppm 3630, but diluted in SSW. Fig. 6 shows mobility reduction vs. flow rate for the different core segments and Fig. 7 shows the normalized remaining

viscosity. Also in this experiment, degradation depends on length, however, less than in Exp. 1. As seen in Fig. 8, more than 80% of the total degradation takes places in the first 3 cm. Fig. 8 also indicates a length effect that may depend on flow rate; the fraction degraded in first core segment decreases by decreasing flow rate. However, it should be noted that the accuracy in determining the fraction of degradation decreases by decreasing total degradation (e.g., at  $q = 5$  ml/min total degradation is 4.6% in first core segment and 5.6% for the entire core). Also note the degradation of 1000 ppm 3630 diluted in SSW at high rates in the pump system.

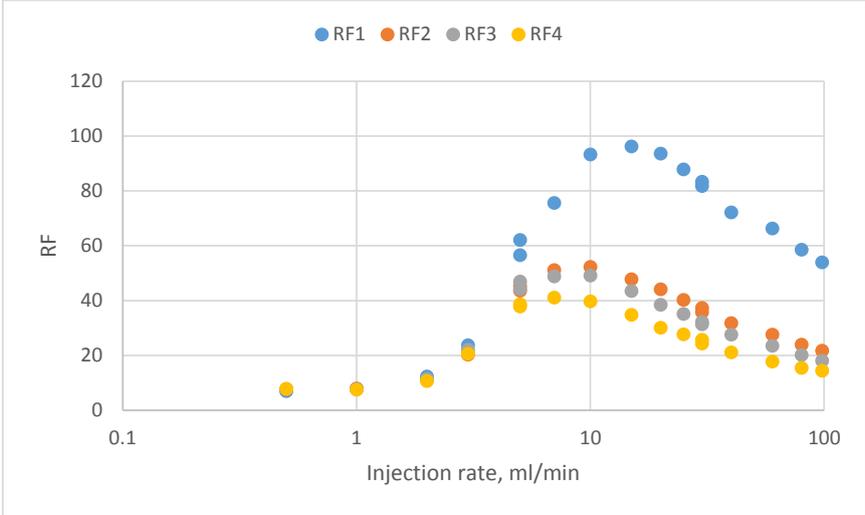


Fig. 6 Mobility reduction vs. injection rate, 1000 ppm 3630 in SSW.

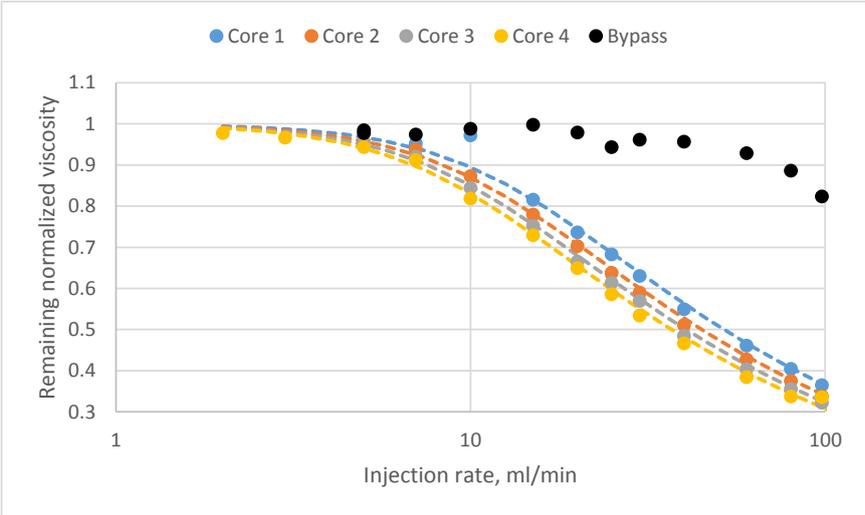


Fig. 7 Viscosity degradation vs flow rate, 1000 ppm 3630 in SSW.

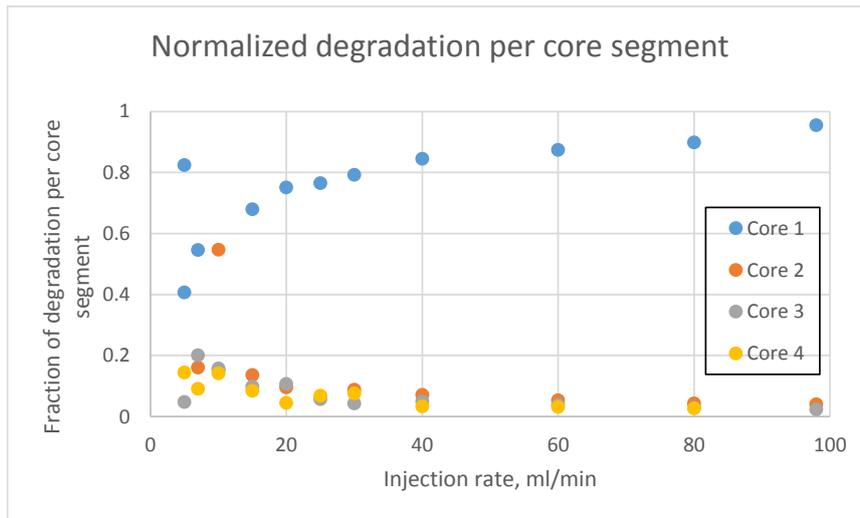


Fig. 8 Degradation per core segment, 1000 ppm 3630 in SSW.

During the flow sequence we also compared the differential pressure along the core segments by flooding through core segments 1 (3 cm) and 2 (5 cm) with flow only through core segment 3 (8 cm). As shown in Fig. 9 the differential pressures are similar and so is the polymer degradation (at  $q = 30$  ml/min,  $\eta_{R1,2} = 0.590$ , while  $\eta_{R3} = 0.586$ ). A similar observation was made at  $q = 25$  ml/min.

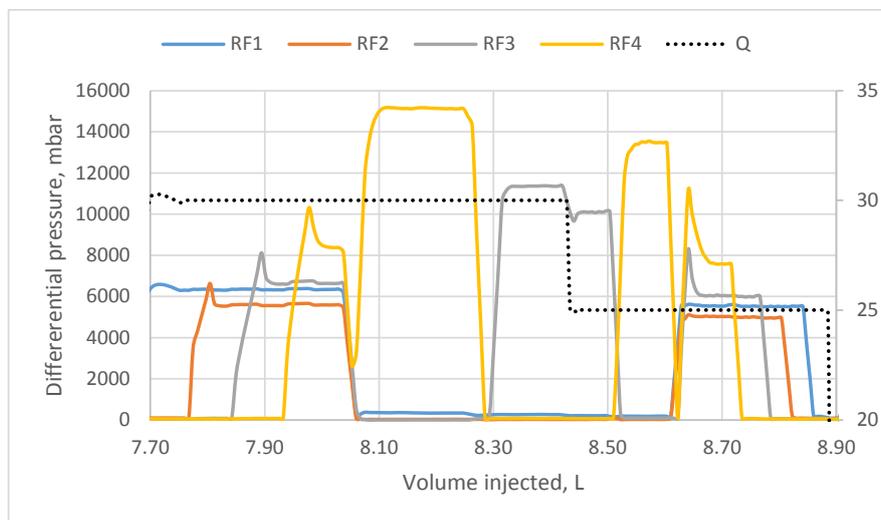


Fig. 9 Differential pressure across cores, 1000 ppm 3630 in SSW.

In Exp. 3 we injected 1000 ppm AN125 diluted in 0.5% NaCl. This polymer is known to be more mechanically stable than the high molecular weight HPAM polymers. It is therefore anticipated to observe less degradation. Fig. 10 shows the mobility reduction and Fig. 11 shows the degradation. As seen in Fig. 11, AN 125 polymer is more shear stable than 3630. The same observation can be made from Fig. 10; compared with Fig. 6 there is a shift in onset of elongation. However, as in Fig. 4, a significant fraction of the polymer degradation takes

part in core segment 2, which from Table 1 has the lowest permeability. The open circles in Fig. 10 denotes the flood experiments bypassing core segment 2 and Fig. 12 shows a separation between degradation in core segment 3 and 4.

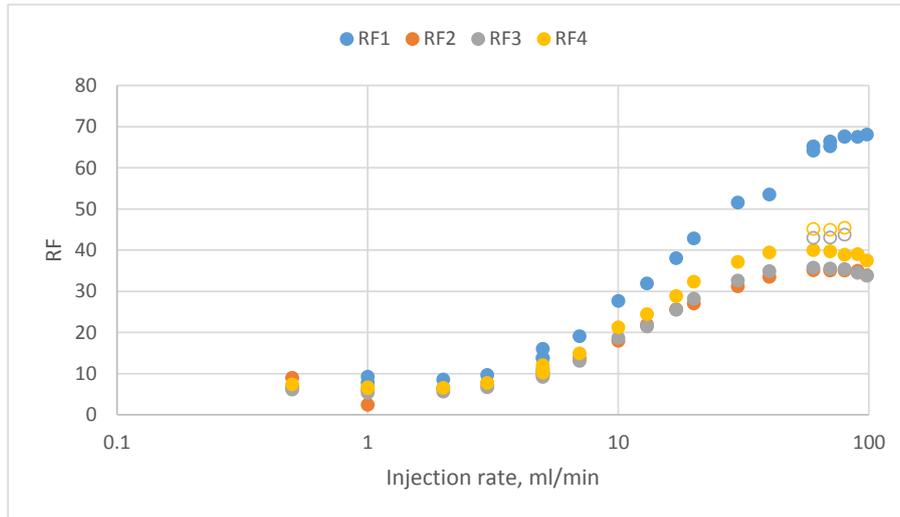


Fig. 10 Mobility reduction vs. injection rate, 1000 ppm AN125 in 0.5% NaCl.

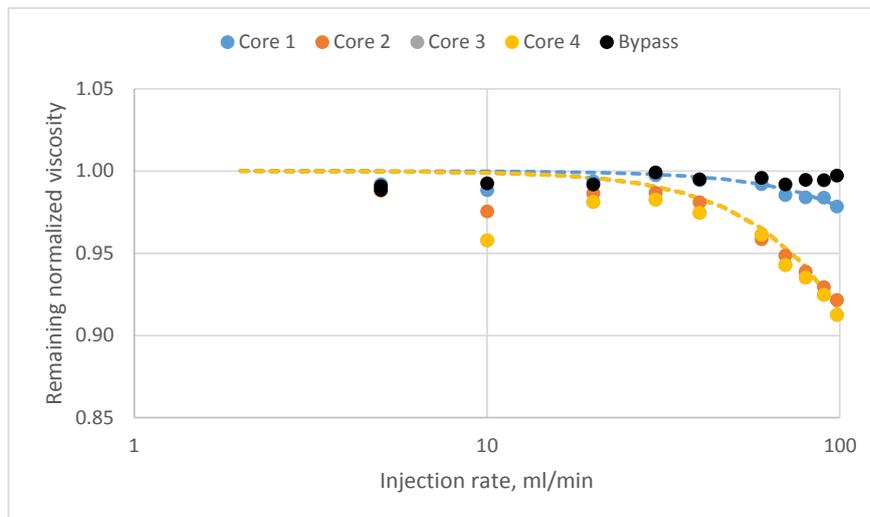


Fig. 11 Viscosity degradation vs flow rate, 1000 ppm AN125 in 0.5% NaCl.

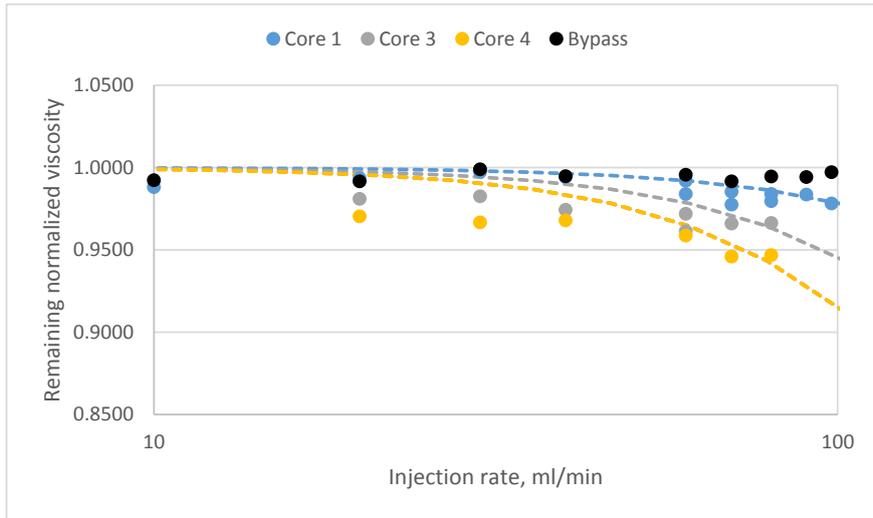


Fig. 12 Viscosity degradation vs flow rate, while bypassing core segment 2, 1000 ppm AN125 in 0.5% NaCl.

For this polymer, we also performed recycling. First, we injected polymer at  $q = 98$  ml/min through the core segments 3 and 4. At stable differential pressure, we collect 250 ml, which together with core pore volume of 49.3 ml and pump volume is 50 ml is the new reservoir, in total 350 ml. We injected for 1.5 hours, equal to 26 cycles or an effective length of 550 cm. On regular basis we collected samples from the reservoir and measured the viscosity. In a parallel experiment we cycled bypass the core segments.

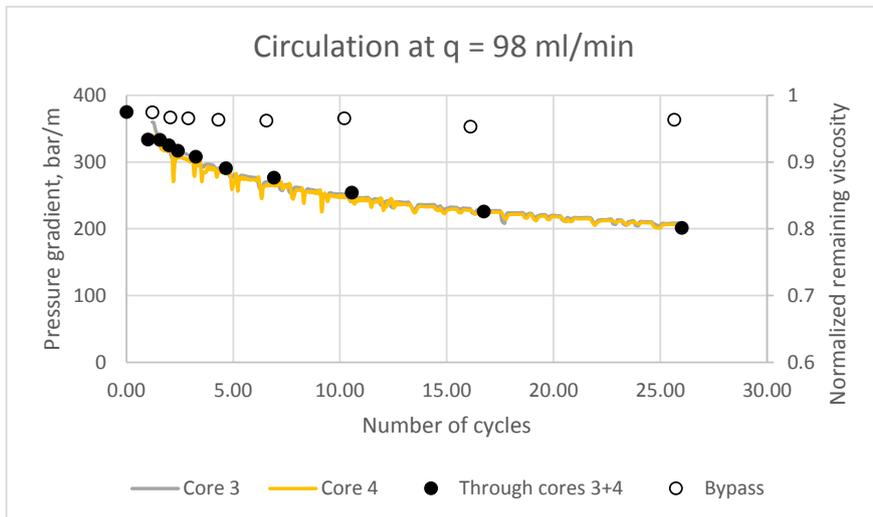


Fig. 13 Viscosity degradation during recycling of the 1000 ppm AN125 polymer in 0.5% NaCl.

Fig. 13 shows that cycling bypass the cores (open circles) resulted in viscosity of 0.975 after first cycle, reducing to 0.964 after 26 cycles. Through the porous media, viscosity was 0.934 after first cycle reducing to 0.802 after 26 cycles. As predicted the differential pressures

across the core segments declined during the injection. When plotting the pressure gradients, as in Fig. 13, the two core segments have the same pressure gradient. This experiment indicate that a polymer, where the degradation after 3 cm is 2.2% will after 550 cm increase to 19.8%. However, this is at the cost of a pressure gradient exceeding 200 bar/m.

In Exp. 4 we injected 1000 ppm AN125 in SSW. Fig. 14 shows the mobility reduction and Fig. 15 shows the viscosity. As for Exp. 3, we did also perform reinjection of the polymer, and in this experiment both at  $q = 80$  ml/min and at 10 ml/min, see Figs. 16 and 17.

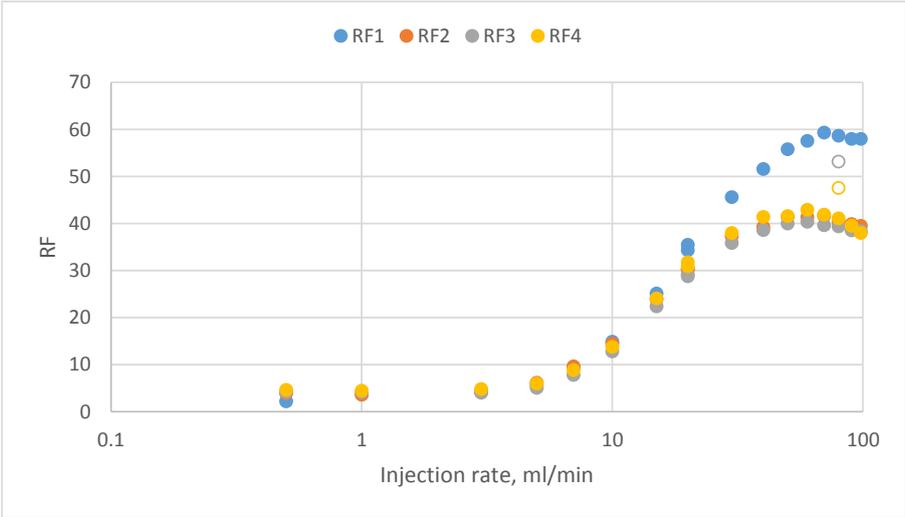


Fig. 14 Mobility reduction vs. injection rate, 1000 ppm AN125 in SSW.

The open circles in Fig. 14 reports mobility reduction through core segments 3 and 4, while bypassing core segments 1 and 2.

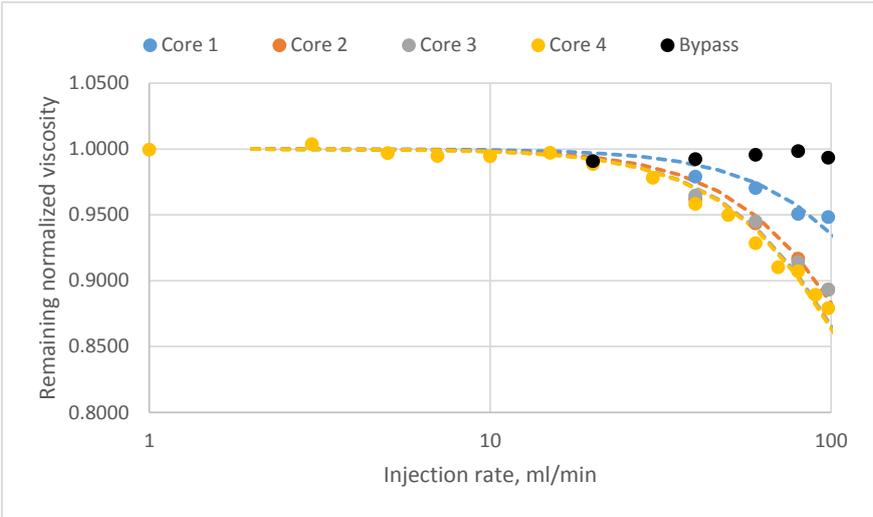


Fig. 15 Viscosity degradation vs. flow 1000 ppm AN125 in SSW.

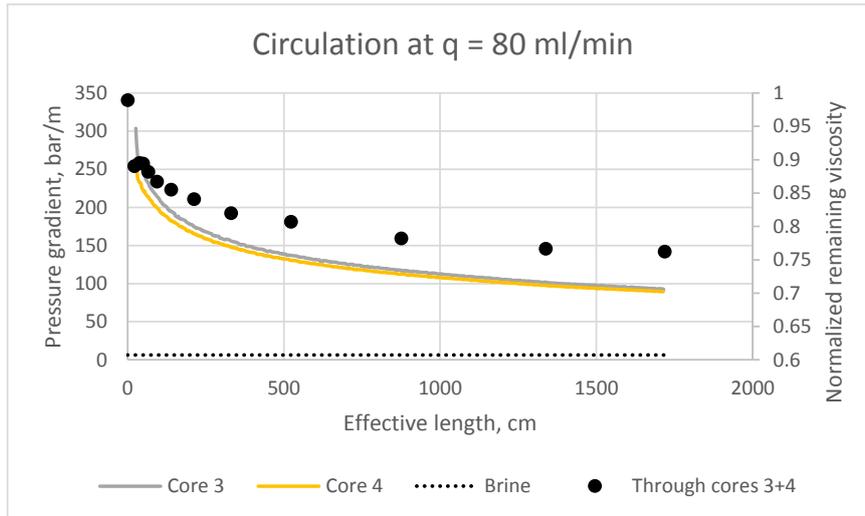


Fig. 16 Viscosity degradation during recycling of the 1000 ppm AN125 polymer in SSW at  $q = 80$  ml/min.

During the recycling of polymer the reservoir volume was 200 ml in addition to pore volume and pump volume. The total number of circulations was 81 at  $q = 80$  ml/min and 42 at  $q = 10$  ml/min. The black dotted line in Figs 16 and 17 represents the pressure gradient for brine injection. Comparison of Figs. 16 and 13 shows that also the AN125 polymer degradation in porous medium, in contrast to choke-type degradation experiments, depends on brine salinity. Diluted in SSW, the polymer at  $q = 80$  ml/min was degraded approximately 5% during the first 3 cm, increasing 10% for the first 21 cm and 24% at 17 meter. Again, the degradation coincides with high pressure gradient, here at approximately 100 bar/meter.

At injection rate of 10 ml/min, the viscosity degradation is marginal, even after effective core length of 9 meter.

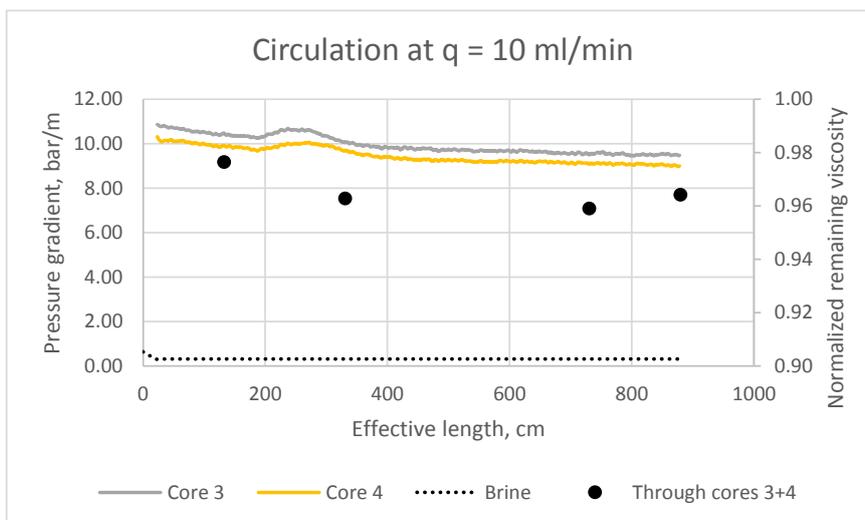


Fig. 17 Viscosity degradation during recycling of the 1000 ppm An125 polymer in SSW at  $q = 10$  ml/min.

The same core plugs, as used for recycling AN125 in SSW, was after water flooding used for recycling 1000 ppm 3630 in SSW, Exp. 5.

First, 1000 ppm 3630 in SSW was recycled through core segments 3 and 4 at  $q = 80 \text{ ml/min}$ , see Fig. 18. In a parallel experiment, the same polymer was recycled, but bypassing the cores at  $q = 80 \text{ ml/min}$ , red triangles in Fig. 18. In a new experiments, 1000 ppm 3630 in SSW was recycled through core segments 3 and 4 at  $q = 10 \text{ ml/min}$ , see Fig. 19. Fig. 20 summarizes the three different experiments.

At  $q = 80 \text{ ml/min}$ , remaining viscosity  $\eta_R$  after 21 cm was 0.414, decreasing to 0.297 after recycling to effective length of 19 meter. At  $q = 10 \text{ ml/min}$  the contrasts are larger; after 21 cm  $\eta_R$  was 0.890, decreasing to 0.592 at 21 meter.

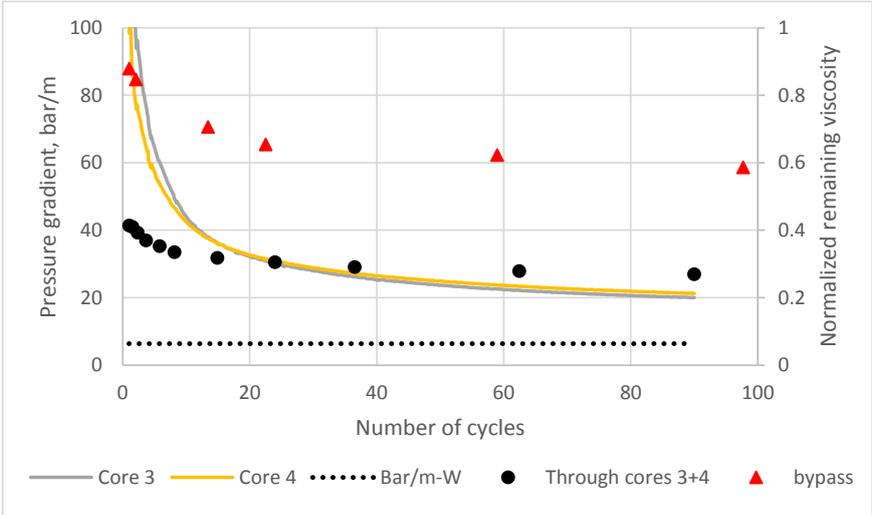


Fig. 18 Viscosity degradation during recycling of the 1000 ppm 3630 polymer in SSW at  $q = 80 \text{ ml/min}$ .

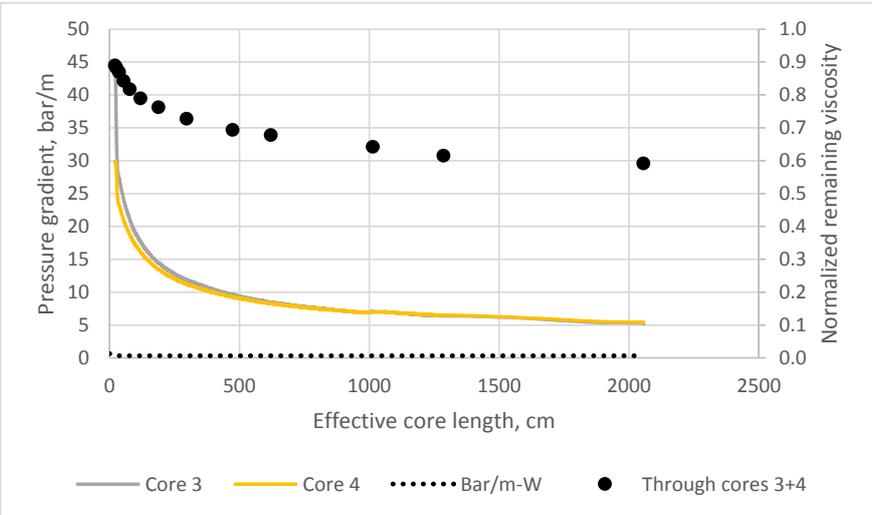


Fig. 19 Viscosity degradation during recycling of the 1000 ppm 3630 polymer in SSW at  $q = 10 \text{ ml/min}$ .

In Fig. 20 we have plotted the viscosity reduction in terms of reduction in molecular weight. We assume that viscosity depends on Mw and use the Mark-Houwink equation  $[\eta] = AM_w^a$ . From previous work  $a$  is found to be  $a = 0.67$ .  $A$  is found by setting the non-degraded molecular weight of 3630 equal to 20E+06 Dalton;  $A = 0.046$ .

Note that recycling the polymer through the core segments at  $q = 10$  ml/min degraded the polymer to the same molecular weight as bypassing the polymer at  $q = 80$  ml/min.

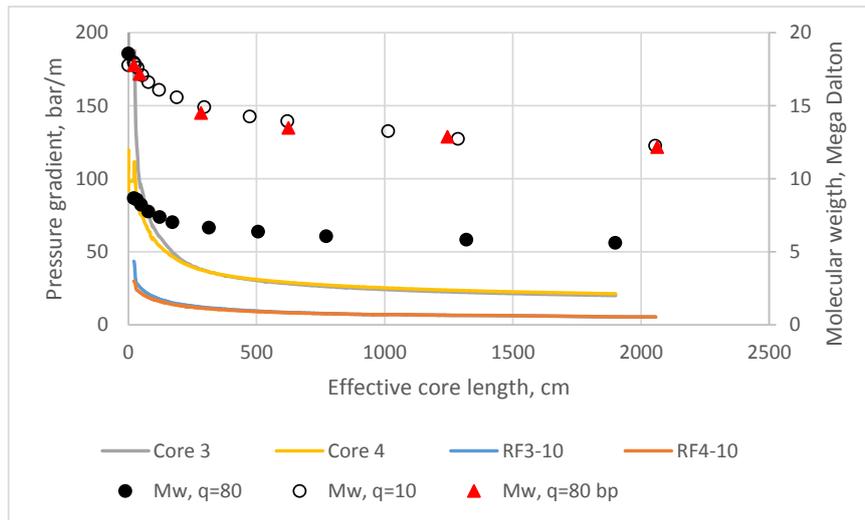


Fig. 20 Viscosity degradation during recycling of the 1000 ppm 3630 polymer in SSW at  $q = 10$  and  $80$  ml/min.

So far we have only focused on linear flood. The final experiment was motivated by development of an experimental procedures that mimic radial flow, in linear cores. We used core segments 1, 3 and 4. Through core segment 1 we injected at constant rate of  $40$  ml/min. The effluent was sampled and used to feed a second pump, which delivered polymer to core segments 3 and 4. Flow rate of second pump was varied from  $3$  to  $50$  ml/min. To put it simple, core segment 2 in Fig. 1 was replaced with an additional pump.

Fig. 21 shows mobility reduction vs. flow rate for  $1000$  ppm  $3630$  in SSW. Open triangles are data taken from Exp. 2, see Fig. 6. As can be seen we reproduced RF1 at  $q = 40$  ml/min. Filled grey and yellow circles shows mobility reduction through core segments 3 and 4 for the degraded polymer. Note the different behavior of degraded and non-degraded polymer. The black circles report the remaining viscosity, reported as the ratio between  $\eta_{R4}$  and  $\eta_{R1}$  ( $q = 40$  ml/min). At  $q = 40$  ml/min  $\eta_{R1} = 0.631$  and,  $\eta_{R4} = 0.555$  ( $\eta_{R4}/\eta_{R1} = 0.88$ ) This is similar to the observation made in Fig. 8; more than  $80\%$  of degradation took place in the  $3$  cm front core. When the flow rate through core segments 3 and 4 was reduced, there was practically no further degradation in core segment 3 and 4. On the contrary, degradation in core segments 3 and 4 was significant when flow rate was increased to  $50$  ml/min.

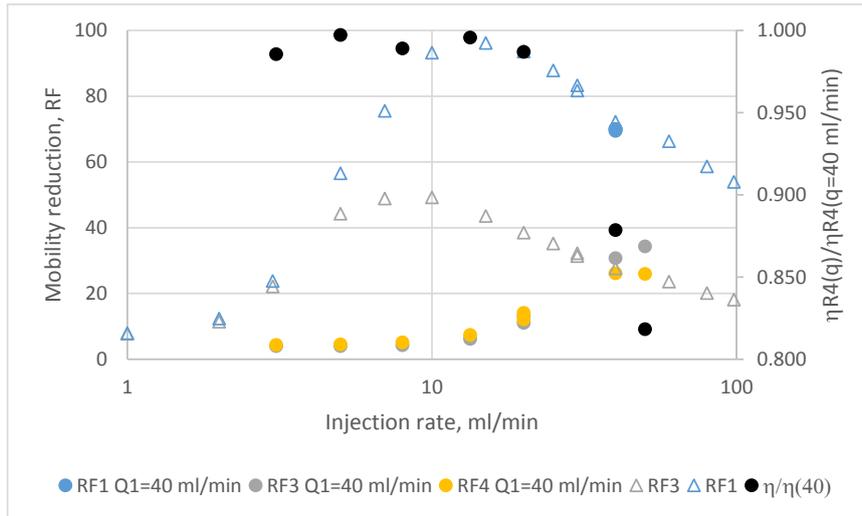


Fig. 21. Viscosity reduction in semi-radial flood experiment, 1000 ppm 3630 in SSW.

Permeability reduction, RRF was measured for the different core segments and are summarized in Table 2. As predicted, RRF increased by increasing molecular weight. In addition RRF is higher in SSW brine than in 0.5% NaCl brine. It should be noted that RRF values are measured after injection of approximately 10 L of 1000 ppm which implicate that the filterability of the polymers are excellent.

Table 2 Permeability reduction, RRF measured at  $q = 5$  ml/min.

Exp.	Polymer	Core 1	Core 2	Core 3	Core 4
1	3630-NaCl	1.4	1.2	1.1	1.2
2	3630-SSW	3.4	2.8	2.6	2.2
3	AN125-NaCl	-	1.0	1.0	1.1
4	AN125-SSW	1.3	1.3	1.5	1.6
5	3630-SSW	3.6	2.2	1.8	1.8

## Conclusions

In linear flow, we conclude that mechanical degradation in porous media depends on core length. However, the degradation gradient is highest over the first few cm. Further degradation is associated with high pressure gradients that exceed realistic pressure gradients. High pressure gradients across narrow intervals may not be critical. Since the degradation strongly depends on velocity, i.e., shear rate, the maximum velocity will in radial flow, in practice, dictate the level of degradation.