Some observations on the effects of polymer degradation on geotechnical behaviour

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Abstract

The addition of polymers at or near to the point of deposition, perhaps most commonly referred to as inline flocculation and herein referred to as polymer treatment (PT), is an increasingly popular technique to improve the dewatering rates and consolidation behaviour of clay-rich tailings. Previous research has demonstrated that this process can significantly alter the geotechnical behaviour of some soils, including their consolidation characteristics and the critical state line. It has also been shown that shearing of slurries after the inline flocculation process can reduce some of the changes induced by the polymers. However, insufficient research has been conducted on the effects of long-term degradation of the polymers on geotechnical behaviour – something of particular importance if the strength of tailings in question are relied upon for stability. To investigate the effect of long-term polymer degradation, PT specimens were prepared for triaxial compression testing on a well-characterised clayey slurry. Attempts were then made to degrade the polymers through the flushing of hydrogen peroxide solution for specimens within the triaxial apparatus, followed by shearing to examine the response of the specimen. Results obtained have not been able to demonstrate appreciable changes to the mechanical behaviour of the PT specimens as a result of hydrogen peroxide flushing. However, the amount of flushing carried out was seen in a separate test to be insufficient to completely degrade the polymers.

Keywords: *polymer treatment, polymer degradation, element testing, critical state line*

1   Introduction

The addition of flocculants at or near to the point of discharge has become a useful technique to improve the dewatering rates of some clayey tailings in some applications. This process has been labelled variously 'secondary flocculation', 'inline flocculation', and 'polymer treatment', with the term polymer treatment (PT) adopted herein. Owing to the increased application of forms of PT, studies to characterise the effects PT may have on post-deposition geotechnical behaviour of the treated slurry have been undertaken. Broadly, these studies have indicated the following effects of PT: lower consolidated densities at a given vertical effective stress and higher permeability at a given density (Jeeravipoolvarn et al. 2009; Yao 2012; Manzotti et al. 2014; Beveridge et al. 2015; Gholami & Simms 2015; Reid & Boshoff 2015; Reid et al. 2015; Riley et al. 2015; Znidarcic et al. 2015; Reid & Fourie 2016, 2018a; Abdulnabi et al. 2022), higher undrained shear strengths at a given density (Jeeravipoolvarn et al. 2009; Yao 2012; Beier et al. 2013; Manzotti et al. 2014; Cooling & Beveridge 2015; Gholami & Simms 2015; Reid & Fourie 2016, 2017; Abdulnabi et al. 2022), higher critical state line (CSL) elevation (Reid & Fourie 2016), and various contrasting outcomes when examining the effects of PT on brittleness in undrained monotonic or post-cyclic shearing (Beier et al. 2013; Gholami & Simms 2015; Reid & Fourie 2017), and drastically different penetrometer response (Reid & Fourie 2018a), with a summary of recent studies provided by Reid & Fourie (2018b).

The effect of PT on the CSL elevation and undrained strength at a given density has potentially important applications given the prevalence of methods based on the CSL to characterise the state and liquefaction susceptibility of tailings (Robertson et al. 2000, 2019; Jefferies & Been 2015; Morgenstern et al. 2016; Jefferies et al. 2019; Arroyo & Gens 2021; Reid et al. 2021).

To investigate the longevity of the CSL changes seen from PT, specimens prepared using PT were flushed with hydrogen peroxide to degrade the polymers, followed by conventional triaxial shearing. This enabled an examination of whether the density and shearing response of the PT specimens was a result of the initial fabric created from the PT process or whether it was dependent on the presence and contribution of the polymers to soil behaviour.

2 Background

To provide context to the current study, a brief review of the relevant outcomes of the work of Reid & Fourie (2015, 2016, 2017, 2018a) is provided. These studies were carried out on a slurry comprising a wide gradation of sand, silt, and clay-sized particles, prepared through the combination by dry mass of 31% silica fine sand (e.g. Fanni et al. 2022), 43% silica 200 G, and 26% kaolin, the resulting composite soil had a liquid limit of 24%, plastic limit of 15%, and plasticity index of 9%, with 69% < 75µm, 22% < 2µm, D50 of 13µm, and a specific gravity of 2.64. Sample preparation was carried out as follows:

1. The slurry was mixed to a gravimetric water content (GWC) of 122% using Perth tap water, with sodium chloride salt added to the mixture to produce a fluid salt concentration of 2.6 g/Kg.

2. Batches to be used for untreated (UT) specimen preparation were then allowed to settle, with free water decanted such that the remaining slurry would be at non-segregating GWC of 45%.

3. Batches to be used for PT specimen preparation were treated in two stages using the polymer Rheomax ETD DPW 1687 produced by BASF. A total dosage of 500 g of polymer per dry tonne of tailings was used, with the polymer applied at a 0.25% concentration. A series of six 'bucket pours' between two 20 L buckets was seen to produce significant PT effects and was therefore used for all specimens.

4. To prepare specimens for testing, the slurry consolidation approach of Sheeran & Krizek (1971) was used where the slurry was poured into 72 mm diameter columns and vertically loaded to approximately 75 kPa, sufficient to enable free-standing specimens.

5. Triaxial test procedures on the prepared specimens followed standard procedures including back pressure saturation, isotropic or K_0 consolidation, then drained or undrained shearing to measure the CSL across a wide range of stresses.

6. Monotonic and cyclic/post-cyclic direct simple shear (DSS) tests were also carried out in a parallel study, while the effect of additional shearing of the PT material (specimens referred to as PT-S) was also investigated.

7. Shearing PT specimens to high strains using either multi-stage DSS or constant volume ring shear does not appear to cause a breakdown of polymers and/or the fabric created through the PT process (Reid et al. 2023).

While clear evidence has been seen in multiple studies for significant effects on mechanical response of soils from PT, an unanswered question remains whether the effects are likely to diminish over time as a result of polymer degradation. For example, should degradation of the polymers lead the relevant CSL for the in situ soil to shift from the PT the (lower) UT CSL, this could dramatically increase the potential brittleness of such materials. Indeed, in some ways this could be conceptually similar to the processes that form quick clays (e.g. Torrance 1983). Therefore, further research is necessary to examine the mechanical response of PT specimens after polymer degradation has occurred.

3 Experimental methods

3.1 Initial sample preparation

Initial specimen preparation techniques used in the current study were consistent with those of Reid & Fourie (2016) previously outlined. This involved (a) mixing kaolin, silica fine sand, and silica silt; (b) making these up

to a slurry at 122% GWC; (c) carrying out PT on the slurry with polymer added in two increments while pouring the material between two 20 L buckets; and (d) one-dimensionally consolidating the slurry to 75 kPa to produce sufficiently stiff samples as to be free-standing after extrusion. As the constituents of the slurry are commercially available standard soils, it was possible to closely reproduce the mixture previously used in the 2011–2014 period.

After initial preparation as described, the specimen was extruded and trimmed from the slurry consolidometer column and placed on a triaxial base platen. The cell was then placed on the triaxial device, filled with tap water, and an initial confining pressure of 100 kPa was applied and the specimen allowed to consolidate with drainage at both ends to the atmosphere.

3.2   Triaxial hydrogen peroxide flushing

Once the specimen had completed primary consolidation under 100 kPa, a toxic interface apparatus was connected to the base of the triaxial and the back pressure pump. This apparatus features a flexible membrane inside a cylinder, therefore allowing application of pressure through the moving of the membrane and displacement of the fluid. With such an arrangement, one side of the membrane contains water (and is connected to the back pressure pump), while the other side has the toxic or aggressive fluid being used in testing. This allows the use of fluids in testing while avoiding their coming into contact with pressure/volume controlling pumps which are typically not suited to directly operate with such fluids. In the current program the toxic interface cylinder was filled with 10% solution of hydrogen peroxide on one side of the membrane, then forced through the sample by means of pressure applied with the back pressure pump. Hydrogen peroxide was selected for availability and it's high oxidation potential which should be able to degrade the organic polymer, and its usage in clay science literature indicating that it does not alter the structure or properties of clays. The arrangement used in shown in Figure 1.
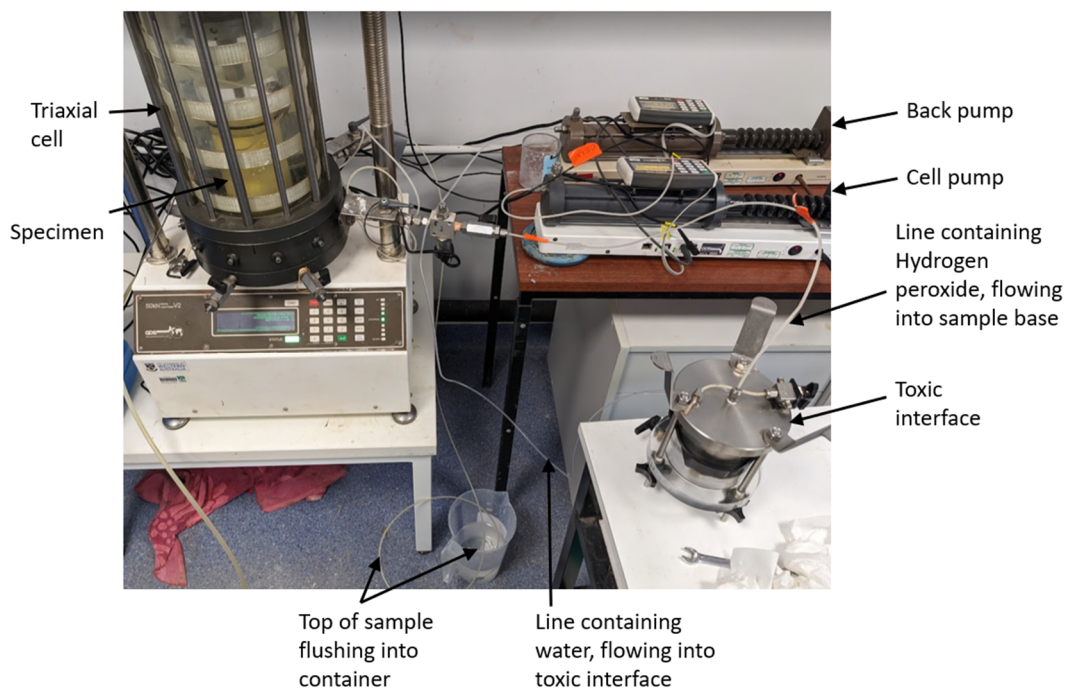

Figure 1   Setup for hydrogen peroxide flushing of specimen

Upon commencement of flushing by applying pressure to the back pump, it was found that it was difficult to maintain flow into the sample. Rather, after some entry of hydrogen peroxide into the sample, reaction with the organics in the system (assumed to be the polymer) led to pressure build-up, thus causing fluid to exit rather than enter the specimen. Therefore, a cyclic flow pattern was followed, wherein the hydrogen peroxide would be pushed into the specimen for a period (thus advancing farther 'up' the specimen) then reverse flow would occasionally occur as the reaction between polymer and the hydrogen peroxide occurred.

To better promote flow, increased cell pressures (250 and 400 kPa, respectively) and base flushing pressures 20 kPa lower than the cell pressure were used. Approximately 400cc (~2x void space) of hydrogen peroxide was flushed through the specimens. After this was complete, approximately 200cc of deionised water was then flushed through the Test 1 specimen to remove much of the residual hydrogen peroxide, while Test 2 transitioned immediately to back pressure saturation without removal of residual hydrogen peroxide. It is acknowledged that the high gradient used across the specimen could lead to piping. However, owing to time constraints, such an approach was required.

3.3   Shearing

After completion of flushing the hydrogen peroxide and then water, back pressure saturation was carried out by ramping the cell and back pressure together slowly while maintaining the same isotropic effective stress throughout (250 or 400 kPa). Upon completion of ramping and observation of negligible subsequent volume change, undrained shearing was carried out at a rate of 0.05 mm/min. After completion of shearing the specimens were removed to obtain end of test GWC.

4   Results and discussion

The shearing behaviour of the flushed PT specimens is presented in Figure 2 as deviator and mean effective stress against axial strain. Consistent with all previous normally consolidated tests on this soil mixture, a contractive response was seen with mean effective stress decreasing monotonically throughout shear. Similarly, an initial peak deviator stress was seen followed by modest strain softening at larger strains. While deviator stress did not reach a consistent value at high strains, this is likely owing to the significant parabolic deformation and thus non-uniformity of stresses within the specimen at high strains, rather than some ongoing strain softening process. The negligible change seen in mean effective stress near the end of the shearing process is such that the end point of the test could plausibly represent critical state conditions.
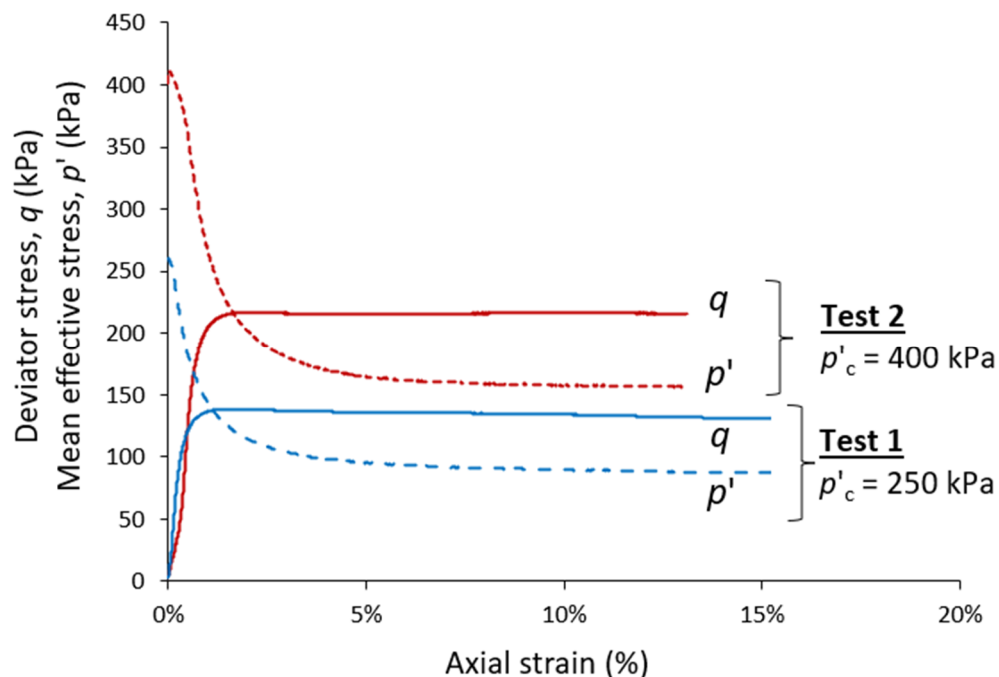

Figure 2   Shearing response tests after flushing

The results of the tests are then presented in a state diagram format in Figure 3, with the UT and PT CSLs previously measured by Reid & Fourie (2016) shown for comparison. Both are seen to shear to a final state close to the PT CSL, with the final state seen within typical experimental variability in CSL determination both generally (e.g. Reid et al. 2021) and for the previous test program on PT specimens (Reid & Fourie 2016).
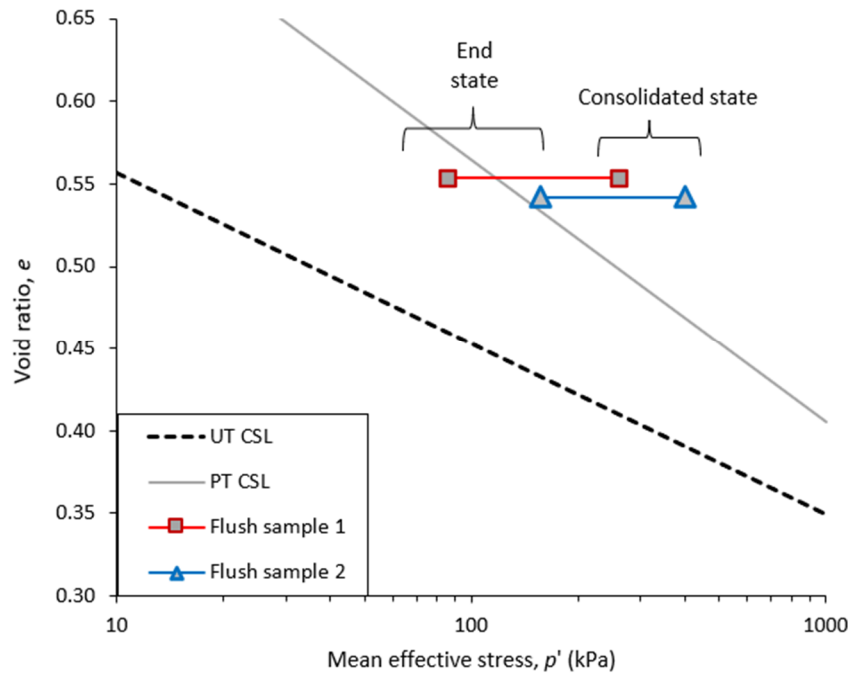
Figure 3   State diagram comparing Flushing Test 1 to the untreated (UT) and polymer treated (PT) critical state lines (CSL) obtained by Reid & Fourie (2016)

Finally, the peak undrained shear strength from both tests is compared to previous results on UT and PT specimens against consolidated void ratio in Figure 4. The results of the flushed tests are indistinguishable from previous PT tests. This strongly suggests that whatever degree/proportion of polymer degradation that occurred in the flushing process was insufficient to affect the mechanical response of the specimen during shearing. However, it is currently unclear as to whether this outcome is a response of insufficient degradation of the polymer, or whether the response of PT specimens seen is largely controlled by the fabric created during formation, thus being unaffected by subsequent polymer degradation.
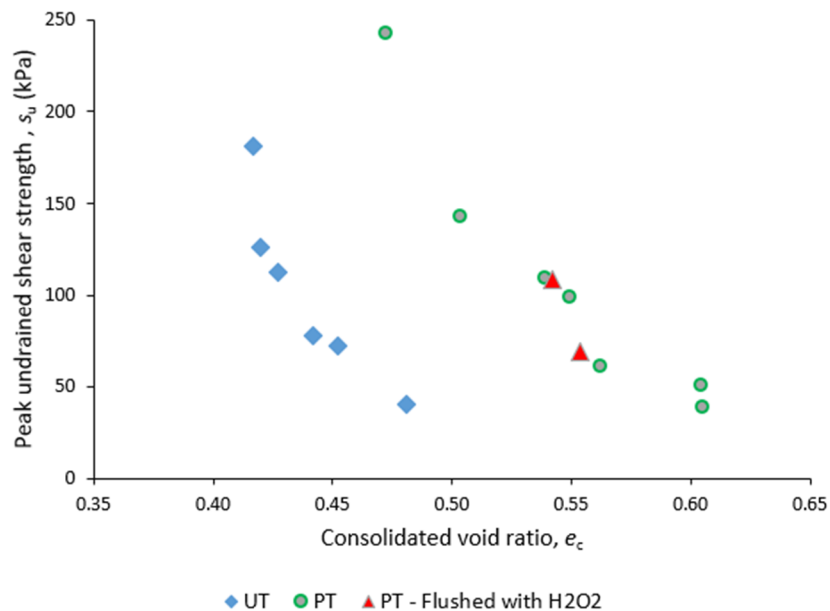

Figure 4   Comparison of peak undrained shear strength for flushing test with untreated and polymer treated results at a range of consolidated void ratios

5 Polymer degradation assessment

Owing to the lack of any observable effect from the flushing of ~2× pore volumes of hydrogen peroxide on the mechanical behaviour of PT samples, a separate assessment was carried out to estimate what quantity of hydrogen peroxide would be required to degrade all of the polymer within the PT material. A sample was prepared by one dimensionally consolidating to 25 kPa, then known quantities of 10% solution of hydrogen peroxide were repeatedly added to the sample in a beaker to visually observe at what point bubble formation (evidence of gas generation in the form of carbon dioxide generation) occurred. It was observed that even after addition of 10 pore volumes, bubble formation continued. This suggests that a significantly larger quantity of flushing than was possible in the triaxial tests would be required to achieve complete polymer degradation.

Figure 5 presents images of three PT samples exposed to different pore volume ratios, all of which led to bubble formation, further suggesting that the quantities of hydrogen peroxide flushed in the triaxial tests was insufficient to achieve complete polymer breakdown.
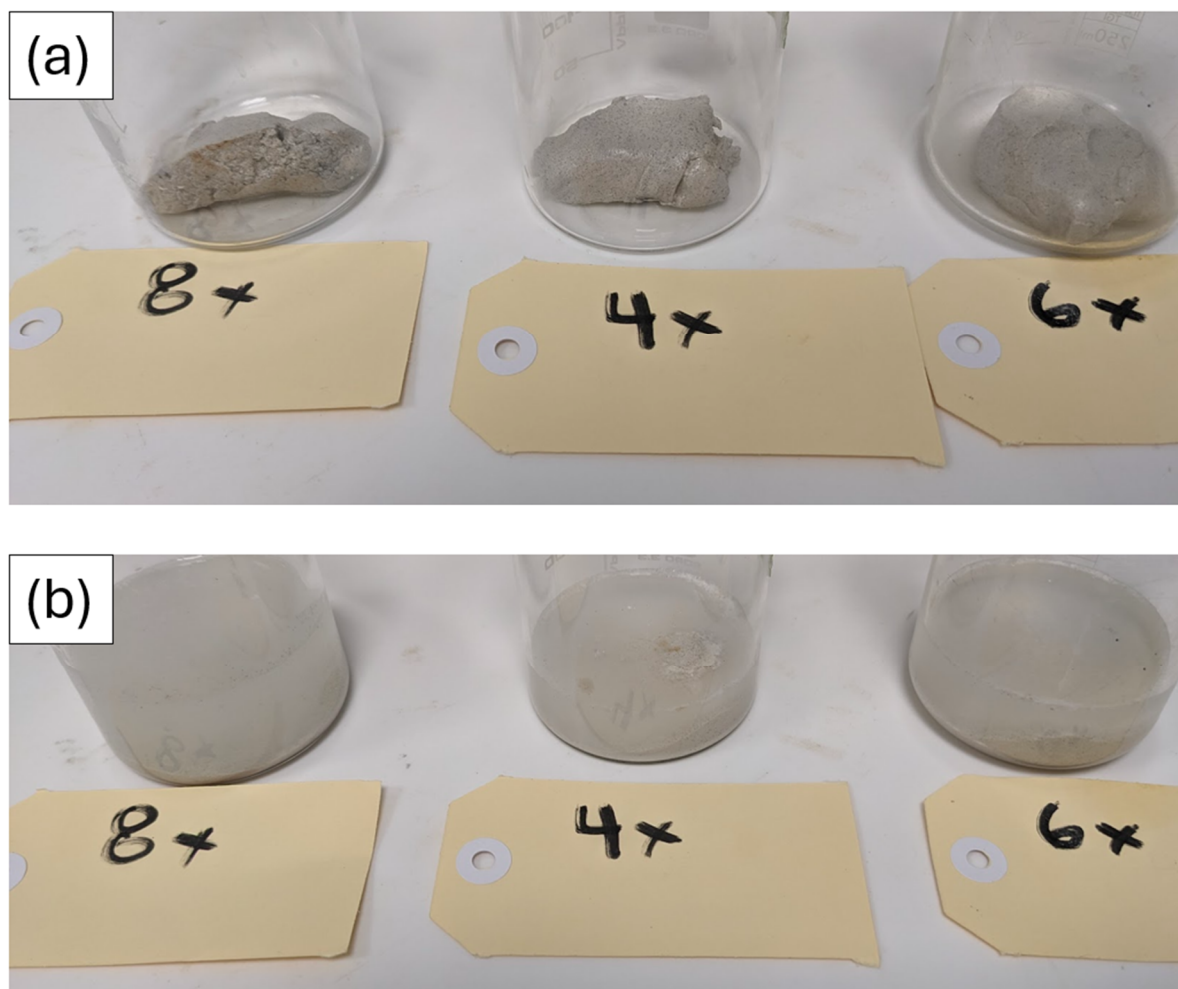

Figure 5 PT samples. (a) Prior to addition of hydrogen peroxide; (b) After approximately addition of various pore volume ratios, as indicated by the label

6 Conclusion

The current study has investigated whether the degradation of polymers of specimens prepared from a slurry, with PT carried out in the preparation, will lead to changes in mechanical properties during subsequent shearing. This work built on previous studies of a mixture of laboratory-standard soil that showed significant

effects from PT including a change to the CSL and higher peak undrained shear strengths at a given consolidated void ratio.

To attempt to degrade the polymer, hydrogen peroxide was flushed through PT specimens within a triaxial cell, followed by undrained shearing. No meaningful change in the mechanical properties was seen compared to previous tests on PT specimens. However, it was also established that far larger quantities of hydrogen peroxide would likely be required to completely degrade the polymer than was achieved in the triaxial flushing experiments carried out in this study.

Acknowledgement

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