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Polymer Resin for Squeezing
Revision: 1

Organisation name of lead contractor for this deliverable:
SINTEF Petroleum AS

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Work package: WP 9.5 Polymer resin for squeezing
Lead contractor: SINTEF Petroleum

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Public abstract

This report is part of the research project MiReCOL (Mitigation and Remediation of CO2 leakage) funded by the EU FP7 programme. Research activities aim at developing a handbook of corrective measures that can be considered in the event of undesired migration of CO2 in the deep subsurface reservoirs. MiReCOL results support CO2 storage project operators in assessing the value of specific corrective measures if the CO2 in the storage reservoir does not behave as expected. MiReCOL focuses on corrective measures that can be taken while the CO2 is in the deep subsurface. The general scenarios considered in MiReCOL are 1) loss of conformance in the reservoir (undesired migration of CO2 within the reservoir), 2) natural barrier breach (CO2 migration through faults or fractures), and 3) well barrier breach (CO2 migration along the well bore).

The objective of this work was to test the sealing ability of a commercially available temperature-activated polymer resin with respect to cement failure at laboratory scale. Two different well leakage scenarios were considered: (a) failure of the cement sheath (via micro-annuli, cracks, voids) and (b) debonding at the cement-casing interface. Cement core samples with artificially created leak paths were prepared: (1) a cement core cut in half axially and reassembled with a thin steel plate, (2) a cement core in two halves with moulded vertical leak paths in the centre. Permeability of the samples and average fracture thickness of the leak paths were measured before and after the squeeze procedure. The polymer resin was then squeezed into the core samples along the leak paths, and left to cure. After repeated permeability measurements, the samples were disassembled and the affected surfaces were studied by optical microscopy. The polymer resin proved to be fairly successful in plugging the designed leak paths: permeability and the average fracture thickness were significantly reduced after the treatment for both samples.
Public introduction (*)

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Long term success of CO2 storage is heavily dependent on maintaining well integrity. Wells have been identified as the most important potential leakage pathways in a CO2 storage project. Complex well construction, in general consisting of casings/liners of different diameters, cement in the A, B and C annuli between the casings, packers, and other well barrier elements, creates numerous possibilities for leakage pathways. Specifically, if we consider cement failure as the cause of CO2 leakage, possible leakage pathways can occur at the casing-cement or cement-formation interfaces, or through fractures or mud channels in the bulk cement.

Thus prevention and remediation options for any potential leakage through the wellbore system play a crucial role in implementation of large scale CO2 storage. Squeeze cementing is the most common remediation practice in oil and gas industry used for various well leakage scenarios. Squeeze cementing is generally performed by perforating the casing and squeezing a sealant behind the casing. The sealant is most commonly Portland G cement slurry with appropriate additives, but a temperature-activated or pressure-activated polymer resin may be used instead. There are a couple of important advantages of polymer resin materials compared to cement. Viscosity and curing time can be tailored to enable placement into the target interval, and prevent premature setting at an undesired location.

The objective of this work was to test the sealing ability of a commercially available temperature-activated polymer resin with respect to cement failure at laboratory scale. Two different well leakage scenarios were considered: (a) failure of the cement sheath (via micro-annuli, cracks, voids) and (b) debonding at the cement-casing interface. Cement core samples with artificially created leak paths were prepared: (1) a cement core cut in half axially and reassembled with a thin steel plate in between, (2) a cement core in two halves with moulded vertical leak paths in the centre. A standard core-flooding setup with addition of an injection coil was used in this work. Permeability of the samples and average fracture thickness of the leak paths were measured before and after the squeeze procedure. The polymer resin was then squeezed into the core samples along the leak paths, and left to cure. After repeated permeability measurements, the samples were disassembled and the affected surfaces were studied by optical microscopy. The polymer resin proved to be fairly successful in plugging the designed leak paths: permeability and the average fracture thickness were significantly reduced after the treatment for both samples. No flow was established through sample 1 after the treatment, whereas the permeability of sample 2 was reduced by a factor of 42, to the order of the initial permeability of sample 1. Successful performance of the polymer resin in the large leak paths is of special interest since such large fractures may cause severe leakage in a real well.
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INTRODUCTION

This report is part of the research project MiReCOL (Mitigation and Remediation of CO₂ leakage) funded by the EU FP7 programme. Research activities aim at developing a handbook of corrective measures that can be considered in the event of undesired migration of CO₂ in the deep subsurface reservoirs. MiReCOL results support CO₂ storage project operators in assessing the value of specific corrective measures if the CO₂ in the storage reservoir does not behave as expected. MiReCOL focuses on corrective measures that can be taken while the CO₂ is in the deep subsurface. The general scenarios considered in MiReCOL are 1) loss of conformance in the reservoir (undesired migration of CO₂ within the reservoir), 2) natural barrier breach (CO₂ migration through faults or fractures), and 3) well barrier breach (CO₂ migration along the well bore).

Long term success of CO₂ storage is heavily dependent on maintaining well integrity. Wells have been identified as the most important potential leakage pathways in a CO₂ storage project [1-3]. Complex well construction, in general consisting of casings/liners of different diameters, cement in the A, B and C annuli between the casings, packers, and other well barrier elements, creates numerous possibilities for leakage pathways. Well leakage scenarios have been thoroughly examined in MiReCOL report D8.1 "Description of CO₂ well failure modes, causes and consequences", 2015. Specifically, if we consider cement failure as the cause of CO₂ leakage, possible leakage pathways can occur at the casing-cement or cement-formation interfaces, or through fractures or mud channels in the bulk cement [2].

Decades of CO₂-flooding operations in enhanced oil recovery provide a valuable insight into the medium-term consequences of CO₂ leakage through the wellbore system [4]. Cement zones that have reacted heavily with CO₂ observed at casing-cement and cement-shale interfaces gave clear evidence of CO₂ migration along both of these interfaces. The extensive study by Carey et al. [4] inspired a number of laboratory-scale studies of the cement and casing integrity in contact with carbonated brine, where references [5-8] are examples of more recent studies. These studies [4-8] have consistently proven the effects of prolonged reaction of cement with CO₂-brine, i.e. calcium migration, dissolution and formation of distinct zones in the cement matrix – such as depleted portlandite, CaCO₃ precipitation and a silica-rich amorphous zone. Although partial plugging and permeability reduction of the leak paths at the casing-cement or cement-formation interfaces, or of the cement bulk may be achieved by carbonate deposition [4,5,7], in case of larger initial leak paths degradation of the cement after prolonged CO₂-brine flow becomes a prominent process [7,8].

Thus prevention and remediation options for any potential leakage through the wellbore system play a crucial role in the implementation of large scale CO₂ storage. A remediation operation can be described as an attempt to repair a leak in a well barrier element, such as the annular cement, tubing or casing. Squeeze cementing is the most common remediation practice in oil and gas industry used for various well leakage...
scenarios [9,10]. Squeeze cementing is typically used to solve lost circulation during drilling or to repair poorly cemented sections or leaks in the casing/liner, or to prevent migration of fluids within the wellbore system. This operation is usually performed at the time of running the casing, i.e. during well construction if the primary cement job failed. However, it can also be used for remediation of leakage later in the life-cycle of a well.

Squeeze cementing is generally performed by perforating the casing and squeezing a sealant behind the casing. The sealant is most commonly Portland G cement slurry with appropriate additives, but a temperature-activated or pressure-activated polymer resin may be used instead. An important disadvantage of the cement slurry is that the relatively large particle size limits placement efficiency into micro-cracks and small void spaces [9]. On the other hand, polymer resin materials can be designed to have viscosity close to water prior to curing, which makes them very suitable for squeezing into micro-cracks.

Temperature-activated sealants can be in principle used for squeeze cementing operations, yet this is not a common practice in the oil and gas industry. Temperature-activated sealants are polymer resin systems designed to cure at a specific temperature. This allows placement, pumping or squeezing of the resin while in the liquid state into the desired interval, and subsequent curing when the resin reaches the appropriate temperature. Curing temperature, density, viscosity and curing time can be accurately designed for a particular application. In general polymer resins tolerate some degree of contamination and are compatible with most wellbore fluids and cements. In addition, treatments with polymer resin systems can be reversible (via milling or acid treatment). There have been some reports of successful field application of temperature activated sealants, for example in the Middle East as a lost circulation material while drilling gas wells offshore [11], and for plugging and abandonment operations [12]. Temperature activated sealants can be in principle used in squeeze cementing operations for remediation of casing/liner and annular cement integrity loss.

The objective of this work was to test the sealing ability of a commercially available temperature-activated polymer resin with respect to cement failure at laboratory scale. Two different well leakage scenarios were considered: (a) failure of the cement sheath (via micro-annuli, cracks, voids) and (b) debonding at the cement-casing interface. For simplicity casing perforation was avoided in our experimental setup and we started with cement core samples with artificially created leak paths at the cement-cement and cement-steel interfaces. The samples were: (1) a cement core cut in half axially and reassembled with a thin steel plate in between, (2) a cement core in two halves with moulded vertical leak paths in the centre. A standard core-flooding setup with addition of an injection coil was used in this work. Permeability of the samples and average fracture thickness of the leak paths were measured before and after the squeeze procedure. The polymer resin was then squeezed into the core samples along the leak paths, and left to cure. After repeated permeability measurements, the samples were disassembled and the affected surfaces were studied by optical microscopy.
2 ENERGY PROCEDIA PUBLICATION: REMEDIATION OF LEAKAGE THROUGH ANNULAR CEMENT USING A POLYMER RESIN: A LABORATORY STUDY

The results of this work were presented at 8\textsuperscript{th} Trondheim Conference on CO\textsubscript{2} Capture, Transport and Storage (TCCS-8), Trondheim, Norway, June 16-18, 2015. The following manuscript will be published in Energy Procedia.
Remediation of Leakage through Annular Cement Using a Polymer Resin: a Laboratory Study

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Abstract

Long term success of CO\textsubscript{2} storage is heavily dependent on maintaining well integrity. Prevention and remediation of leakage through wells plays a crucial role in large scale implementation of CO\textsubscript{2} storage. Squeeze cementing is the most common remediation practice in the oil and gas industry used for various well leakage scenarios. The objective of this work was to test the sealing ability of a commercially available temperature-activated polymer resin in a laboratory-scale squeeze cementing operation. Two well leakage scenarios were selected: micro-annuli or cracks in cement and debonding at cement-casing interface. Cement (with or without steel) core samples with designed vertical leak paths were prepared. Permeability of the samples was measured both before and after the squeeze procedure. Then the samples were disassembled and studied by optical microscopy. The squeeze procedure proved to be successful for plugging the designed leak paths.

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Keywords: *micro-annuli; CO2 leakage; squeeze cementing; temperature-activated polymer resin; permeability*

Introduction

Long term success of CO\textsubscript{2} storage is heavily dependent on maintaining well integrity. Wells have been identified as the most important potential leakage pathways in a CO\textsubscript{2} storage project [1-3]. Complex well construction, in general consisting of casings/liners of different diameters, cement in the annuli A, B and C between the casings, packers, and other well barrier elements, creates numerous possibilities for leakage pathways. Specifically, if we consider cement failure as the cause of CO\textsubscript{2} leakage, possible leakage pathways can be at casing-cement or cement-formation interfaces, or through fractures or mud channels in the cement bulk [2]. Decades of CO\textsubscript{2}-flooding operations in enhanced oil recovery provide a valuable insight into medium-term consequences of CO\textsubscript{2} leakage through the wellbore system [4]. Cement zones heavily reacted with CO\textsubscript{2} observed at casing-cement and cement-shale interfaces were a clear evidence of CO\textsubscript{2} migration along both of these interfaces. The extensive study by Carey et al. [4] inspired a number of laboratory-scale studies of the cement (and casing) integrity in contact with carbonated brine, where references [5-8] are examples of more recent studies. These studies [4-8] have consistently proved effects of prolonged reaction of cement with CO\textsubscript{2}-brine: calcium migration, dissolution and formation of distinct zones in the cement matrix – such as depleted portlandite, carbonated (CaCO\textsubscript{3} precipitation) and amorphous zone (silica rich). Although partial plugging and reduction of permeability of the leak paths at casing-cement or cement-formation interfaces, or through cement bulk, may be achieved by carbonate deposition [4,5,7], in case of larger initial leak paths dissolution and degradation of the cement upon prolonged CO\textsubscript{2}-brine flow become prominent processes [7,8].

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attempt to repair a leak in a well barrier element, such as the annular cement, tubing or casing. Squeeze cementing is the most common remediation practice in oil and gas industry used for various well leakage scenarios [9,10]. Squeeze cementing is generally performed by perforating the casing and squeezing a sealant behind the casing. The sealant is most commonly Portland G cement slurry with appropriate additives, but a temperature-activated or pressure-activated polymer resin may be used instead. Squeeze cementing is typically used to solve lost circulation during drilling, or to repair poorly cemented sections, or leaks in the casing or liner or to prevent migration of fluids within the wellbore system. This operation is usually performed at the time of running the casing, i.e. during well construction if the primary cement job failed. However, it can be used for remediation of leakage later on in the life-cycle of a well.

Temperature-activated sealants can be in principle used for squeeze cementing and repair of casing leaks and annular cement failure. Temperature-activated sealants are polymer resin systems designed to cure at a specific temperature. This allows placement, pumping or squeezing of the cement slurry while in the liquid state into the desired interval, and subsequent curing when the resin reaches the appropriate temperature. Curing temperature, density, viscosity and curing time can be accurately designed for a particular application. In general polymer resins tolerate some degree of contamination and are compatible with most wellbore fluids and cements. In addition, treatments with polymer resin systems can be reversible (via milling, acid treatment). There have been some reports of successful field applications of temperature-activated sealants, for example as lost circulation material [11] or for plugging and abandonment operations [12].

The objective of this work was to test the sealing ability of a commercially available polymer resin with respect to cement failure at laboratory scale. Two different well leakage scenarios were considered: (a) failure of the cement sheath (via micro-annuli, cracks, voids) and (b) debonding at the cement-casing interface. For simplicity casing perforation was avoided in our experimental setup and the starting points were cement core samples with artificially created leak paths at the cement-cement and cement-steel interfaces. Permeability of the samples and average fracture thickness of the leak paths were measured before and after the squeeze procedure. Temperature-activated polymer resin was then squeezed into tempered core samples along the leak paths, and left to cure. After repeated permeability measurements, the samples were disassembled and affected surfaces were studied by optical microscopy.

Experimental

Sample Preparation

Two core samples were prepared as shown in Figs.1 and 2. In both cases, Portland G cement slurry was prepared according to API specification 10A. Cement slurry was poured into a rubber sleeve for core flooding (1.5” inner diameter, about 25 cm in length) plugged at the bottom side, to obtain the correct sample dimensions. The cement cores were initially curing in an oven at 66 C° at atmospheric pressure for several days and afterwards taken out to cure further at room temperature.

In case of sample 1, shown in Fig. 1, the cement core was pulled out of the rubber sleeve and vertically sliced in halves. The core was further cut to 14.7 cm in length and the end faces were grinded. Cement halves were then assembled with 0.5 mm thick stainless steel plate, as shown in Fig. 1(a). Rough edges were filled with plaster and the surface was polished, see Fig. 1(b). Such an assembled sample was mounted back into a rubber sleeve.

In case of sample 2, shown in Fig. 2, a plastic (PEEK) plate mould was designed to create vertical leak paths. Drawing of the cross section of the mould is shown in Fig. 2(d). The cement slurry cured against the plastic mould which was set in the center of a rubber sleeve. The excess cement at the ends of the core was cut off together with the rubber sleeve, giving the final core length of 17.2 cm. The final cement core with the plastic mould was then removed from the sleeve and easily disassembled, as shown in Figs. 2(a,b). Both cement interfaces were rather smooth, apart from some voids and one fracture running along the interface with the mould, as seen in Fig. 2(a). In this case, the edges of the cement halves remained smooth, which made reassembling of the sample straightforward as seen in Figs. 2(c,e).
Fig. 1. Sample 1 - cement sample with a steel plate: (a) Half-cylinders reassembled with a steel plate; (b) Rough edges of the cement halves were filled with plaster and surface was polished.

Fig. 2. Sample 2 - cement sample with designed vertical leak paths: (a) Half-cylinder with a smooth surface; (b) Half-cylinder with a patterned surface; (c) Cement halves reassembled; (d) Technical drawing of the cross section of the PEEK mould, numbers are in mm. (e) Side view of the leak paths.

Injection Setup and Procedure

The setup used for these experiments is based on a general core flooding scheme, as shown in Fig. 3(a). It contains most of the common equipment to perform a core flooding, such as a pump, back pressure valve and a differential pressure gauge. The core holder is illustrated in Fig. 3(b). As an addition to this setup we have added an injection coil, made out of 1/8 inch Teflon tubing, illustrated in more detail in Fig. 3(c). The injection coil is filled with polymer resin by using a syringe. The polymer resin was injected into the core sample by flooding brine as a displacing fluid into the bottom of the coil. By using the injection coil, only a small volume of the polymer resin was injected into the core flooding system and unnecessary contamination was avoided. The polymer resin was designed to cure at 60°C, well above ambient temperature, in order to avoid plugging of the core flooding system.

The following core-flooding procedure was used for both samples. The confining pressure was 50 bars throughout the whole experiment. The initial fracture permeability was measured with 1% NaCl solution at ambient temperature and with a back pressure of 20 bars. Next, the core holder and core sample were heated to 60°C. The polymer resin mixing method was the same for both samples, to achieve the same gelling and curing time. The injection time was the same (8 min) for both samples, but the injection rate...
was increased for sample 2 since the fracture volume was larger, see Table 1. The injection into sample 1 was performed from the top, whereas for sample 2, the bottom-up direction was chosen to ensure better filling of the large leak paths. In both cases, the injected volume was a sum of the system (dead) volume and 80% of the calculated fracture volume. The shut in period was about 200 min during which the core holder was closed. After the setting period, the core holder was dismounted and the injection and outlet ports were cleaned from the residual polymer resin. Then the permeability measurement was repeated. The apparent average fracture thickness was calculated before and after the treatment.

![Fig. 3. Illustration of the injection setup: (a) Flow setup scheme; (b) Core holder; (c) Illustration of polymer resin injection procedure.](image)

**Results and Discussion**

The permeability measured before and after the injection test, plus measured and calculated average fracture thickness for both samples are summarized in Table 1. The apparent average fracture thickness was determined using Darcy’s law and verified by volumetric and geometrical measurements of the leak path. The initial fracture thickness values determined by these two methods are in agreement. The measured fracture thickness (column 4 in Table 1) was slightly larger than fracture thickness calculated using Darcy’s law (column 5 in Table 1) for sample 1. For sample 2, the initially measured fracture thickness was a nominal value of 0.5 mm according to the design of the leak paths, see Fig. 2(d).

**Table 1. Permeability values and average fracture thickness before and after polymer resin injection for samples 1 and 2.**

<table>
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<tr>
<th>Sample</th>
<th>Fracture Permeability before (Darcy)</th>
<th>Fracture Permeability after (Darcy)</th>
<th>Measured Fracture Thickness before (µm)</th>
<th>Calculated Fracture Thickness before (µm)</th>
<th>Calculated Fracture Thickness after (µm)</th>
<th>Injection rate (ml/min)</th>
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<tr>
<td>1</td>
<td>47</td>
<td>~ 0</td>
<td>20</td>
<td>17</td>
<td>0</td>
<td>0.1</td>
</tr>
<tr>
<td>2</td>
<td>1717</td>
<td>41</td>
<td>500</td>
<td>467</td>
<td>25</td>
<td>0.3</td>
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Despite being assembled from loose pieces of cement and steel, and filled with plaster on the edges, sample 1 had a rather small volume of void space between the cement, steel and plaster, resulting in average fracture thickness of 17 µm. After the treatment with the polymer resin, the fracture permeability dropped to nearly 0 Darcy indicating complete plugging of the leak path. In the case of sample 2, the initial leak path volume was much larger, resulting in much higher initial permeability (1717 Darcy) and an average fracture thickness of 467 µm. After the treatment, the permeability of sample 2 reduced to about the same value as the initial permeability of sample 1, still resulting in measurable flow through the
sample. Nevertheless, the reduction of permeability by a factor of 42 was significant and the average fracture thickness dropped to 25 µm.

After the treatment with the polymer resin, the samples were opened and the cement surfaces exposed to the resin were studied. Overview photos of both cement surfaces for samples 1 and 2 are shown in Fig. 4. The injection direction for both samples is indicated by the arrow, and the designed leak paths in sample 2 are numbered. The smooth surface of sample 2 is shown in Fig. 4(c), whereas the patterned surface appears in Fig. 4(d).

For the sample 1, a thin layer of polymer resin remained unevenly spread across both cement surfaces, and also on the steel plate (not shown). The polymer resin is the light grey layer in Fig. 4(a,b). The resin was mostly concentrated at the top part of the sample where the injection was initiated. The resin penetrated to a great extent through the gap between the cement and plaster (white material on the edges of the sample 1). On the cement-plaster interface to the left in Fig. 4(a) and to the right in Fig. 4(b), the resin reached all the way to the bottom surface, whereas on the other sides the resin has not reached all the way to the bottom.

In the case of sample 2, the polymer resin acted as a binding agent between the cement halves. When the halves were detached, some resin remained glued onto both surfaces. The polymer resin reached the bottom surface of sample 2 as well, and cured within the leak paths. However, none of the leak paths was completely filled with the cured polymer resin. Comparing Figs. 4(a) and (b), it can be seen that leak paths 1, 3 and 4 were filled to a large extent, whereas leak path 2 was partly filled mostly in the upper half of the sample.

More detailed optical images of the cement surfaces exposed to the polymer resin treatment in samples 1 and 2 are presented in Fig. 5. The bright region towards the edge of sample 1 in Fig. 5(a) is plaster also seen in Figs. 4(a,b). The cured polymer resin has light grey colour and is filling the gap between the plaster and the cement, and a thin layer is covering the cement surface. Fig. 5(b) shows some partially filled interface voids, which can be also noticed in the overview images in Fig. 4(a). Some of the interface voids appeared to be completely filled, while others were empty or the resin remained attached to the steel. In sample 2, the polymer resin partially filled some of the interface voids as shown in Fig. 5(c), but the most of the resin neatly cured within the grooves as seen Fig. 5(d).

The use of the same injection procedure as for sample 1, was not the most efficient approach for a large leak path volume in sample 2. To improve filling of such large designed leak paths, the injection procedure needs to be modified. Injecting only 80 % of the fracture volume was a safety measure, but it did not prevent plugging of the injection and outlet ports in either of the tests. Hence, the injected polymer resin volume and injection pressure and rate can be increased, but with expectation that the injection and outlet ports will be plugged upon curing. In addition, back pressure should be introduced during curing in order to contain the injected polymer resin more efficiently within the leak paths. Another option is to prolong the shut in period to ensure better curing of the injected polymer resin.
Fig. 4. Overview images of the cement surfaces exposed to polymer resin, injection direction is indicated by the arrow: (a,b) Sample 1 - surfaces against the steel plate; (c) Sample 2 – smooth surface; (d) Sample 2 – patterned surface. Numbers in (d) mark the grooves in the patterned surface, and in (c) polymer resin remaining attached to the smooth surface from the respective groove.
Fig. 5. Cement surface exposed to polymer resin: (a) Sample 1 - top left corner of the cement half in Fig. 4(a); (b) Sample 1: partially filled interface voids in the central region of the same cement half-piece; (c) Sample 2 - polymer resin partially filling the grooves and an interface void in between the grooves 3 and 4; (d) Sample 2 - polymer resin partially filling one of the grooves.

4. Conclusions

The injection setup was adequate for a laboratory scale squeeze operation into cement(-steel) core samples. The polymer resin properties such as viscosity, gelling and curing time were adjusted with respect to the leak path size and shape. The polymer resin proved to be fairly successful in plugging the designed leak paths for the two selected leakage scenarios: cement-casing debonding and fractures in annular cement. The polymer resin penetrated through the whole sample length in both cases.

The permeability and the average fracture thickness were significantly reduced after the treatment. In case of cement-steel sample, which had a small initial leak path (17 µm thick on average), the average fracture thickness dropped to nearly zero after the treatment and no flow was established. In a practical
situation however, such small fractures (17 µm) pose a smaller problem with respect to the long term CO$_2$ escape. The performance in the large fracture (nominal thickness 0.5 mm) is however, even more important because a fracture of this size can cause severe leakage [13] if it extends through the entire cemented interval. A reduction in permeability in the large fractures by a factor of 42 corresponding to a reduction in apparent fracture thickness from 467 µm to 25 µm is remarkable. A recommendation for future work would be improving the plugging performance of the polymer resin by optimizing the injection and curing procedure.

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