Project Summary, Milestones, Payment Schedule and Quarterly Update Reports

Report Date (Q2 2023/2024)
(Due on the 15th of the month following the end of the Quarter)

Project Summary

Project Title:	Understanding and Mitigating Leakage Pathways in Oil and Gas Well Cements
Lead Investigator:	Ergun Kuru
PTAC Committee Sponsor:	
AUPRF Funds Awarded:	$85,800.00
Other Funds Received and Sources:	$28,400.00 (In-kind) Sanjel Energy Services
Start Date:	10/18/2023
Completion Date:	10/17/2024

Provide a brief description of the challenges this project aims to address and how industry and others will benefit including what the anticipated impact is. (i.e., cost savings, improved environmental performance, regulatory outcomes, enhanced access to resources). (Maximum 150 words)

Understanding the mechanisms of failure and debonding and how these impact fluid movements through the cement is crucial for the assessment of cement performance in wells, whether these wells are producing or plugged and abandoned. Most studies focus on overly simplified cement systems (neat Class G) that are not directly comparable to modern wells. Further to this, the existing literature is limited to understanding the cement stability on its own, and often neglects contributions of interfaces between materials or contributions of downhole stresses. These research gaps limit the value of the research and leave uncertainty as to the suitability of various cement designs for real world deployment. Therefore, it is critical that industry-relevant blends and downhole conditions, such as those included in this proposal, are evaluated to ensure confidence in the cement barrier system. Direct imaging and subsequent modeling of the cement pore structure and cement/casing interfaces as proposed in this project, will help to develop new, more reliable barrier technologies, which will result in more efficient, secure primary cement and P&A jobs for the lifetime wellbore integrity and more cost-effective mitigation of fugitive methane emissions.

Total Project: List of milestones/quarterly payments schedule.

Milestone	Quarterly Budgets[1]	Quarter[2] Q2 2023/24
Milestone-1: Direct porosity, permeability measurement and Nano-CT -ESEM analyses of small-scale samples **Deliverable-1:** Quantitative assessment of the effect of cement alternatives (A1, A2, A3) on the permeability of cement plugs and cement plugs inside the pipe **Deliverable-2:** Digital twins of cement plugs and cement plugs inside the pipe		55242.73
Milestone-2: Measurement of the shear bond strength and permeability of the cement plug inside the pipe **Deliverable:** Quantitative assessment of cement alternatives (A1 to A3) on the shear bond strength and permeability of the cement plug inside the pipe		
1	6035.34	
2	55242.73	
3		
Total	61278.73	

Current Quarter Report: (Q2 2023/24)

Exception Highlight for this Quarter:

1.
2.
3.

Update on scope, key questions, gap analysis and work performed this quarter (include any requests for assistance to PTAC or the committees)

Preface

The actual starting date of the project was October 18th, 2023. This 2nd quarterly (Q2 2023/24) project progress report provides the results of the research activities conducted during time period between January 19th 2024 and March 18th, 2024.

In the first part of this report, we present porosity, bulk and pipe cement permeability (using core flow tests), Micro-CT and ESEM test results of the cement sample A3. We also provide comparison of the

[1] All expenditures in Canadian dollars and must include GST and other applicable taxes
[2] Quarters are based on fiscal calendar e.g., Q3 2021/22 is Oct-Dec 2021

all these test results for cement samples A1, A2 and A3. This completes the Milestone-1 and the associated deliverables as proposed in the research contract.
In the second part of the report, we present results of the Nitrogen/Water permeability experiments and shear bond strength measurements for cement samples A1, A2 and A3, all conducted by using Wellbore Simulator. This completes the Milestone-2 and the associated deliverables as proposed in the research contract.

1. Results

1.1 Cement porosity

Porosities of cement blends measured by both drying & weighing and Nitrogen porosimetry methods are summarized in Table 1. For sample A3, only the porosity from the drying and weighing method is presented. Since the sample was cracked during the drying period, we could not measure the porosity using Nitrogen porosimetry method. Results indicated that cement sample has the highest porosity, followed by sample A2 and sample A2 has the lowest porosity. A linear correlation was found between the porosity and water/cement (w/c) ratio as shown in Figure 1, i.e., higher w/c ratio leads to higher porosity.

Table 1. Porosity of the Cement Blends

	Porosity (%)	
Sample	Drying & Weighing Method	Nitrogen Porosimeter
A1	43.74	46.34
A2	49.90	51.69
A3	52.49	cracked

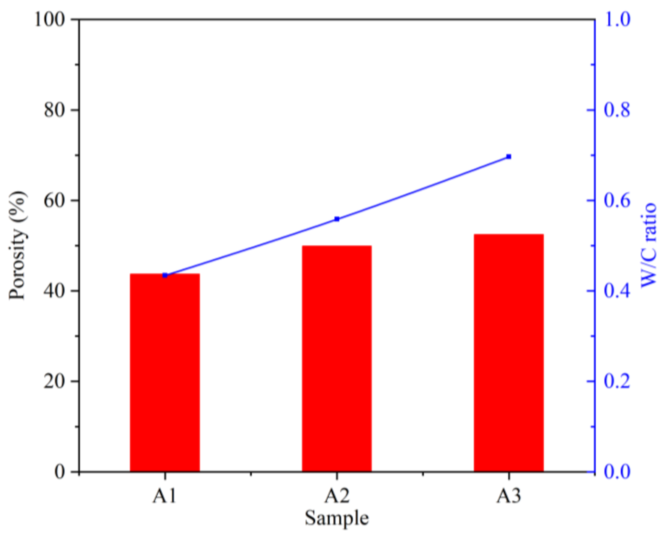

Figure 1. Correlation between porosity and w/c ratio

1.2 Micro-CT Scan results

For the given size of the bulk and pipe cement samples, Micro-CT scanning tests would allow to detect pore sizes larger than 10 microns. Figures 2 and 3 show the CT scan results of bulk cement (Fig.2a) and pipe cement (Fig.3) of A3 blend, respectively. The CT scan results clearly demonstrate the ability of micro-CT scan to detect pores larger than 10 micrometers in the cement samples. As shown by the 3-D reconstruction models, porosity distributions in the bulk cement (Fig.2) and in the pipe cement (Fig.3b) samples are non-uniform. All pores are isolated (i.e. there is no pore connectivity for any sizable flow through the cement matrix) and there are no fractures observed.

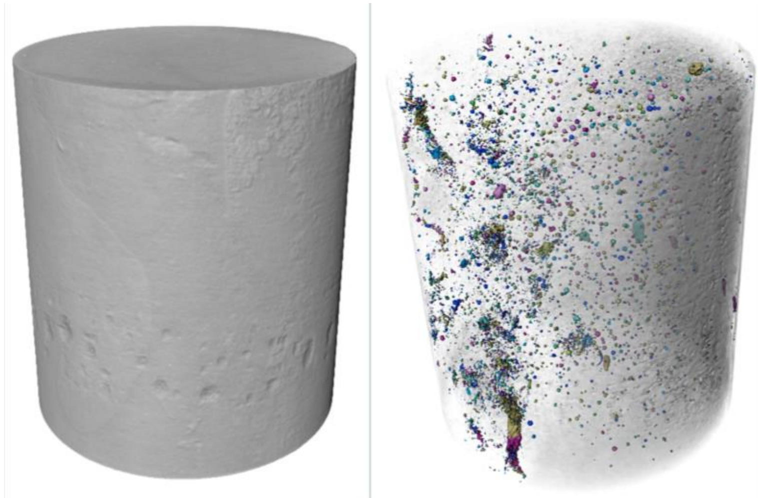

Figure 2. (left) the reconstructed CT image of A3 bulk cement; (right) the pore distribution in A3 bulk cement.

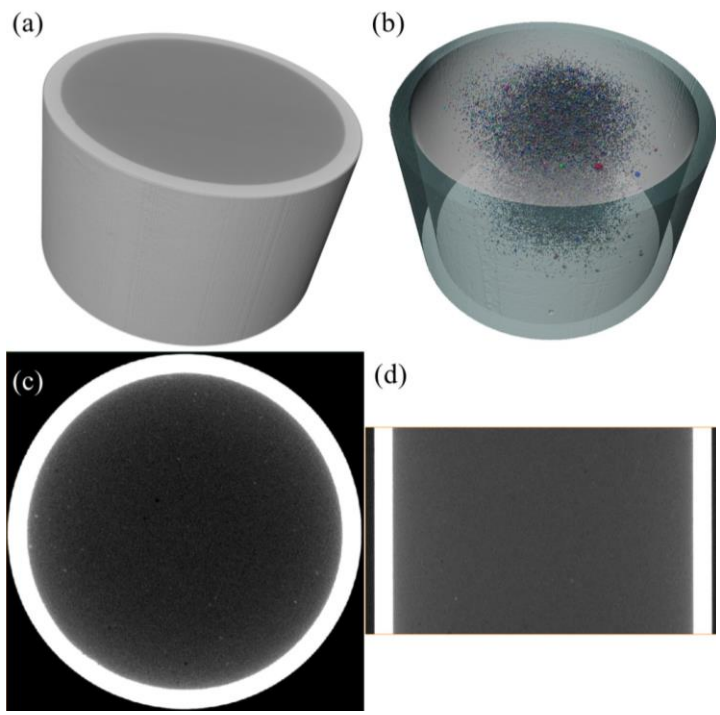

Figure 3 (a) the reconstructed A3 cement in metal pipe; (b) the pore distribution in A3 cement in metal pipe; (c) radial cross section of the CT images; (d) the vertical section of the CT images.

Cross sectional views of the pipe cement sample (Figs. 3c&d) indicate that there are no visible gaps (i.e. microchannels) at the cement/casing interface within the range of 10-micron size. Based on the CT-scan results, porosity of the bulk and pipe cement samples of A3 blend are 0.138% and 0.341%, respectively. Bulk cement porosity is two orders of magnitude lower than the actual porosity value obtained by direct measurement methods. Note that these porosity values are based on the pores, which have size larger than 10 microns (i.e. maximum micro-CT scan resolution). These results indicate that majority of the pores contributing to the total porosity of the cement matrix has size smaller than 10 microns. As long as there is no connectivity among these pores, there will not be any flow (i.e. effective permeability will be extremely low, Nano-Darcy level). 3-D reconstructed images of both bulk (Fig.2 Right) and pipe cement (Fig.3b) confirms that majority of the pores are isolated and there is no connectivity among these pores. Therefore, no significant flow through the matrix of all the cements samples (A1-A3) is anticipated. We did not observe any Nitrogen/Water flow breakthrough during the Core flow experiments (results are presented later in the report) also confirming these conclusions from pore size analyses of micro-CT investigations.

Table 2 summarizes the porosity of all A1-A3 cement blend samples obtained by micro-CT scanning.

Table 2. The porosity measured by the CT scan

Sample	Porosity (%)
A1 small bulk	0.243
A1 in metal pipe	0.216
A2 small bulk	0.154
A2 in metal pipe	0.222
A3 small bulk	0.138
A3 in metal pipe	0.341

Radial porosity distribution across the bulk and the pipe cement samples are shown in Fig.4. For the small bulk cement samples, the pores are more evenly distributed in space (Figs. 4 a&b), however, for the samples in the metal pipe, the larger pores are more concentrated in the center of the cement samples (Figs.4 a&c), which is rather unexpected. Micro-CT technique measurements are prone to be influenced by the presence of steel pipe. Due to beam hardening of X-rays near the pipe wall, CT results show higher density near the pipe wall , which translates into the low porosity values (lower than the porosity of the cement located at the center of the pipe). Previous literature suggests that porosity of the cement close to the pipe/cement interface is higher than that of the ones in the center due to the fact that larger cement particles preferably positioned at the interface. This transition zone is identified as “Interfacial Transition Zone (ITZ)” (Scrivener et al., 2004). Porosity distribution across the radial direction of bulk cement A3 (Fig.4a) shows that matrix porosities close to the edge (outer surface) of the cement sample are higher than at the center of the samples, which is within line of ITZ theory.

Comparison of the bulk cement porosity distribution of A1, A2 and A3 blends indicate that A3 blend bulk cement samples has lowest overall porosity values among all the blends (Fig.4b). Comparison of the pipe cement porosity distribution of A1, A2 and A3 blends, however, indicates that the A3 blend has the highest porosity values, particularly at the middle section of the pipe cement (Fig.4c).

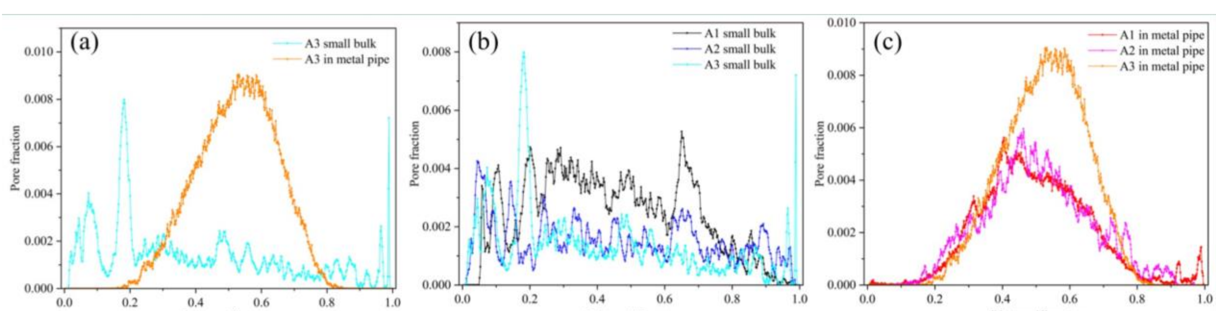

Figure 4. (a) Radial distribution of porosity (fraction) of A3; (b) the comparison of radial distribution of porosity of A1, A2 and A3 small bulk; (c) the comparison of radial distribution of porosity of A1, A2 and A3 in metal pipe

1.3 ESEM Test Results

The cement samples are rotated 360° inside the ESEM chamber to observe the cement-metal pipe interfaces throughout the sample. Figure 5 shows the ESEM images (left) and localized magnification (right) of the cement-pipe interface samples of cement blend A3. Upon inspection of the ESEM images of the full cement/pipe interface, some isolated gaps were observed as shown in Figure 5. The total length of the section where we observed the gap in Figure 5a is 0.4 mm, which corresponds to only 2.3% of the total circumference of the cement/pipe sample. As shown in Fig.5 (right), we measured the gap width at several locations. Average gap width was estimated as 2.7 µm. Further investigation of ESEM images revealed that some of the microcracks were actually located within the cement matrix. It is possible that these minor gaps were formed during the sample preparation process rather than resulting from the cement hydration.

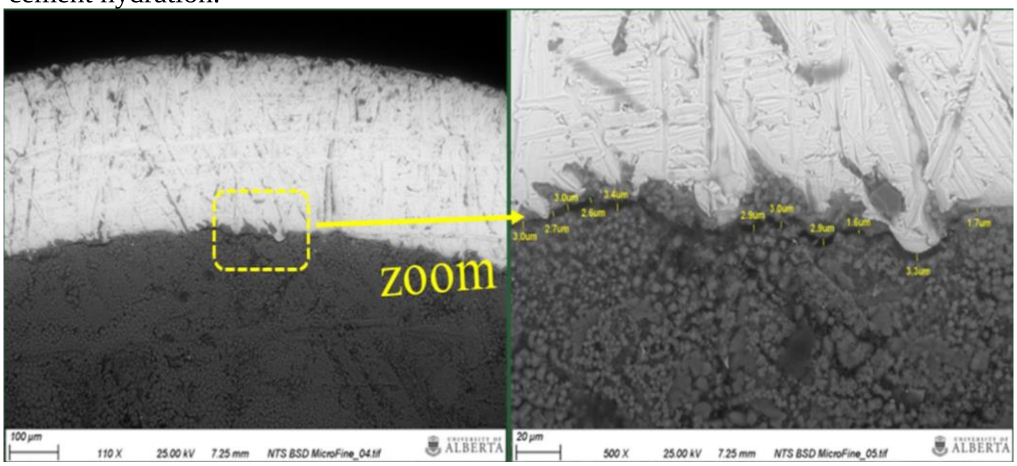

Figure 5 ESEM images of the cement-pipe interface of the sample prepared by using A3 blend.

Summary of the isolated gap width measured for samples of all cement blends is shown in Table 3. Although some difference in gap width is observed among the cement blends A1-A3, the actual size of these gaps is too small to lead any measurable flow for all practical purposes. A recent study (Moghadam et al. 2022) reported that if the gap can be maintained below 20 µm leakage rates are below ~100 kg/yr (equivalent to methane emissions of a dairy cow) regardless of the depth of the gas source.

Table 3. The average gap width at the cement-pipe interface (ESEM tests)

Cement Blend	Average gap width (µm)
A1	2.3
A2	3.7
A3	2.7

1.4 Core flow test results

Water and Nitrogen permeability tests (of the cement plugs prepared by using cement blend A3) were conducted using core flow set-up described in Section 2.2.1 of report Q1. Both bulk cement and pipe cement samples were used in the water flow tests with 900 psi fluid injection pressure and 1000 psi confining pressure. Note that the pressure difference in these permeability tests were as high as 900 psi over a short section (2 in) of the cement plug. These are very severe tests that will allow even very small differences in system sealability to be probed. Such high-pressure differentials would not be experienced in the wellbore owing to the much longer plug length relative to the tests performed here.

Despite these severe test scenarios with high-pressure differentials, no Water or Nitrogen flow breakthrough were observed in all the experiments. A high precision pump (Teledyne ISCO syringe pump 260DX - capable of injecting and recording fluid flow rate as low as 16.6 Nano-L/step) has been used for fluid injection into the cement matrix. Although we did not observe any fluid breakthrough during the flow tests, we were able to measure the change in fluid volume in the pump storage with an accuracy of Nano-liter/step level. Assuming that the change in fluid volume in the injection pump could be used as an indicator of the amount of fluid intake of the different cement samples, we had conducted series of flow tests. The results of tests showing how the instantaneous water/Nitrogen intake flow rates and cumulative water/Nitrogen intake volume change versus time are presented in Figure 6 (Cement Blend A3 only) and Figure 7 (comparison of the results for cement blends A1-A3).

Figure 6 illustrates variations of the average water intake flow rate (calculated from the volume change in the ISCO pump over every 10 minutes) into the bulk and pipe cement A3 samples (Fig.6a) and the cumulative volume of water injected into the bulk and pipe cement A3 samples (Fig.6b) over the time. As shown in Figure 6a, during the first several hours of water injection, the flow rate fluctuated significantly due to the instability of the system. As the injection time increases, the average water flow rate into the two samples stabilized. The average water intake flow rate of A3 bulk cement was slightly higher than that of A3 in metal pipe. The cumulative volume of water injected was very small, less than 0.08 ml over 25 hrs. of the test duration, equivalent of 0.62% of the total pore volume of the bulk cement sample A3.

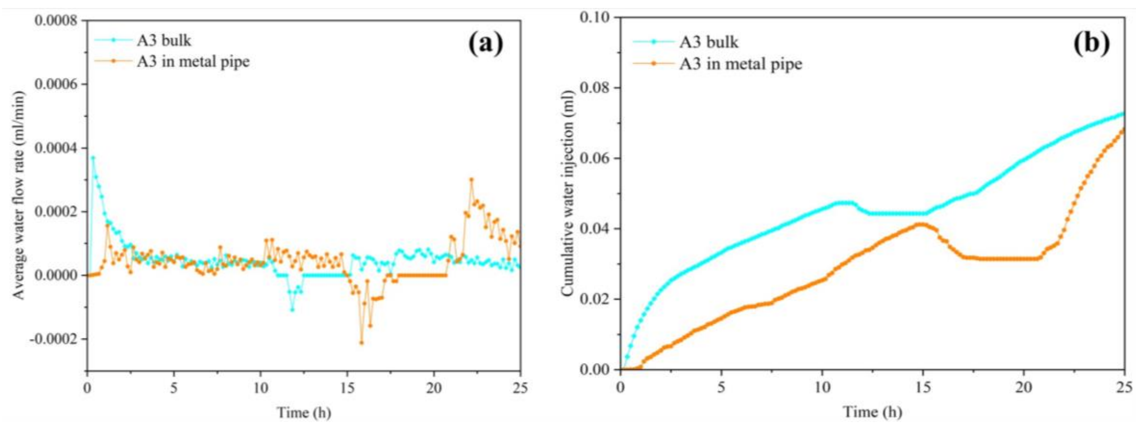

Figure 6. (a) comparison of average water flow rate intake of A3 bulk cement and A3 in metal pipe and (b) the comparison of cumulative water volume intake of A3 bulk cement and A3 in metal pipe.

Figure 7 shows comparison of the average water flow rate into the A1, A2 and A3 bulk cement (Fig. 7a) and the comparison of the average flow rate into the A1, A2, and A3 pipe cement (Fig. 7b). Figure 8 illustrates comparison of the cumulative volume of water intake of A1, A2 and A3 bulk cement (Fig. 8a) and comparison of cumulative volume of water intake of A1, A2 and A3 pipe cement (Fig. 8b).

As shown in Figures 7a and 8a, A3 bulk cement has relatively higher flowability for water than A1 and A2 bulk cement. The cumulative volume of water injected into A1 bulk cement in 17 hours is 0.037ml, which is 0.66% of the total pore volume of the A1 bulk cement. The cumulative volume of water injected into the A2 bulk cement in 25 hours was 0.057 ml, which is 0.89% of the total pore volume of the A2 bulk cement. The cumulative volume of water injected into the A3 bulk cement in 25 hours was 0.073 ml, which is 1.08% of the total pore volume of A3 bulk cement.

As shown in Figures 7b and 8b, A2 cement in pipe has relatively higher flowability for water than A1 and A3 cements in pipe. The cumulative volume of water injected into the A1 cement in pipe in 25 hours was 0.05 ml, which is 0.98% of the total pore volume of A1 pipe cement. The cumulative volume of water injected into A2 cement in pipe in 25 hours was 0.17ml, which is 2.84% of the total pore volume of A2 pipe cement. The cumulative volume of water injected into the A3 cement in pipe in 25 hours was 0.068 ml, which is 1.17% of the total pore volume of A3 pipe cement.

Slight difference in flowability values might be the indication of the variations in microstructures of the cement blends A1-A3. However, from the practical point of view, all the recorded injected water volumes are very low and there was no water breakthrough observed in any of these tests. The fact that there was no water break through observed in any of these experiments suggest that for these optimally placed cements, there should not be any anticipated wellbore integrity issues.

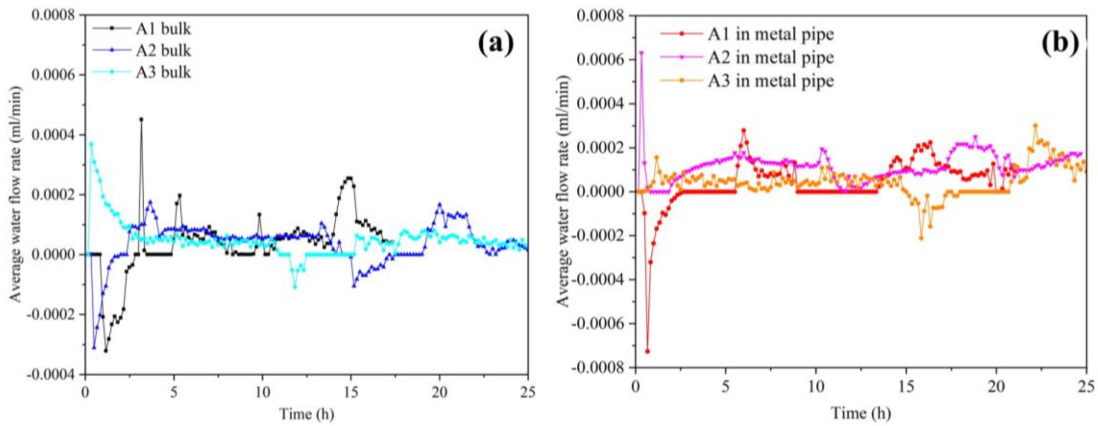

Figure 7. (a) comparison of average water flow rate intake of A1, A2 and A3 bulk cement (b) comparison of average water flow rate intake of A1, A2 and A3 in metal pipe.

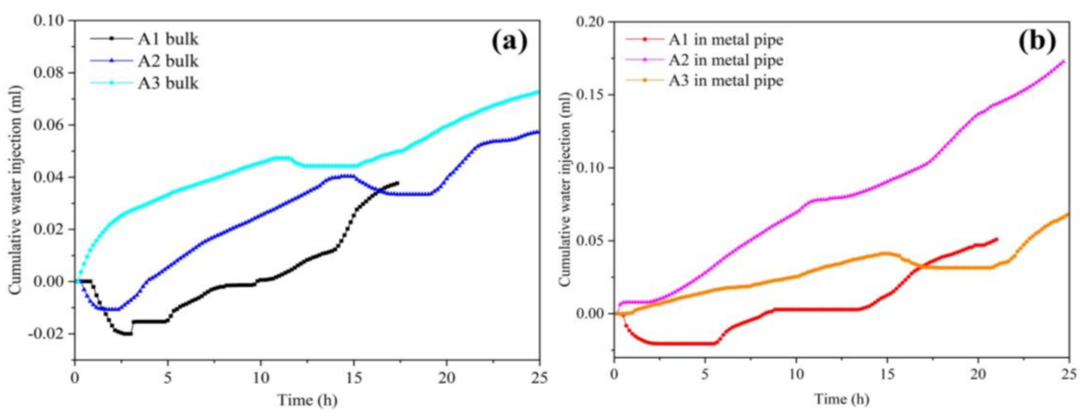

Figure 8 (a) comparison of cumulative water intake of A1, A2 and A3 bulk cement; (b) comparison of cumulative water intake of A1, A2 and A3 in metal pipe.

Figure 9 illustrates variations of the average Nitrogen intake flow rate into the A3 bulk and pipe cement (Fig.9a) and the cumulative volume of N_2 injected into the A3 bulk and pipe cement (Fig.9b) over the time. The average Nitrogen intake flow rate fluctuated significantly. The average Nitrogen intake flow rate into A3 cement in pipe was generally higher than that of A3 bulk cement. The cumulative volume of Nitrogen injected into the A3 bulk cement and pipe cement was 0.2 ml and 1.4 ml over 35 hrs. of the test duration, respectively. The higher cumulative injected Nitrogen volume observed in A3 cement in the metal pipe might be because of the fact that the cement dehydrating effect potentially causes additional gap width between the cement and the pipe.

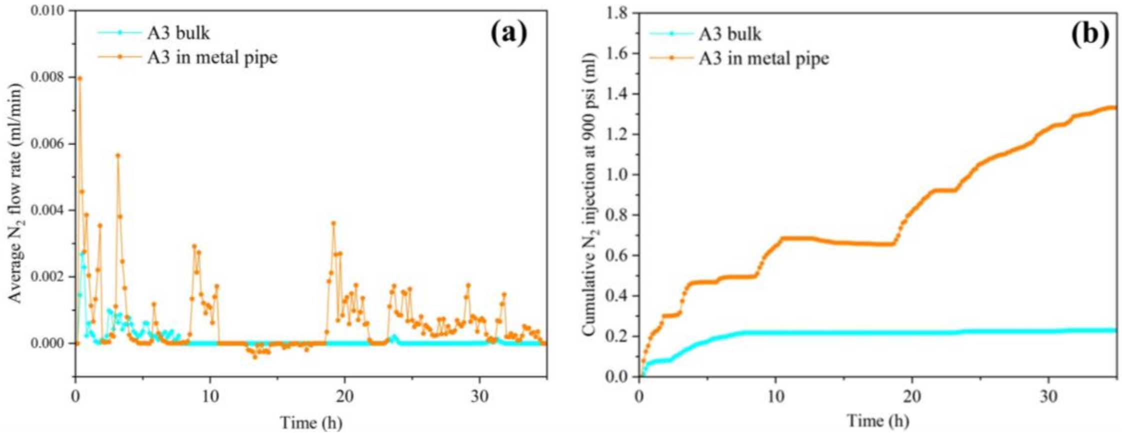

Figure 9. (a) comparison of average N_2 flow rate intake of A3 bulk cement and A3 in metal pipe and (b) the comparison of cumulative N_2 volume intake of A3 bulk cement and A3 in metal pipe.

Figure 10 shows comparison of the average Nitrogen flow rates into the A1, A2 and A3 bulk cement (Fig. 10a) and pipe cement (Fig. 10b). As shown in Figure 10a, the Nitrogen flow rate into A2 bulk cement showed some larger fluctuations throughout the test and it was relatively higher than Nitrogen flow rates into A1 and A3 bulk cement. Similarly, the average Nitrogen flow rate into the A2 cement in metal pipe was higher than the Nitrogen flow rates into A1 and A3 cement in metal pipe (Figure 10b).

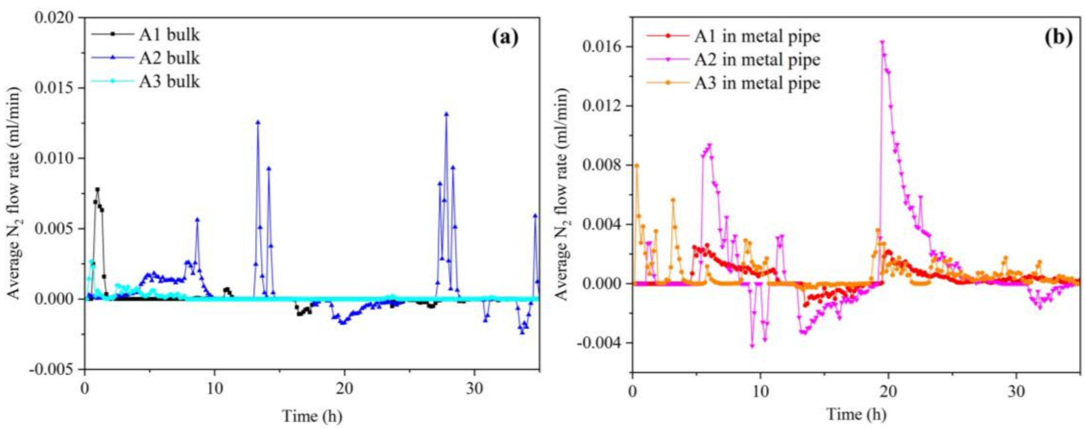

Figure 10. (a) comparison of average N_2 flow rate through A1, A2 and A3 bulk cement (b) comparison of average N_2 flow rate through A1, A2 and A3 cement in metal pipe.

Figure 11 shows the comparison of cumulative volume of Nitrogen injected into the A1, A2 and A3 bulk cement samples (Fig. 11a) and into the A1, A2 and A3 cements in metal pipe (Fig. 11b). The cumulative volume of Nitrogen injected into the A1, A2 and A3 bulk cement over 35 hrs. of the test duration were 0.21, 1.1 and 0.23 ml, corresponding to 1.63%, 8.55% and 1.79% of the total pore volume, respectively. The cumulative volume of Nitrogen injected into the A1, A2 and A3 pipe cement over 35 hrs. of the test duration were 0.78, 2.3 and 1.3 ml, corresponding to 6.71%, 19.78% and 11.43% of the total pore volume, respectively. Nitrogen flowability of the A2 bulk and pipe cement was relatively higher than that of A1 and A3 cements. Difference in Nitrogen flowability values might be the indication of the variations in microstructures of the cement blends A1-A3. However, from the practical point of view, all the recorded injected Nitrogen volumes were very low and there was no Nitrogen breakthrough observed in any of these tests. The fact that there was no Nitrogen break through observed in any of these experiments suggest that for these optimally placed cements there shouldn't be any anticipated wellbore integrity issues.

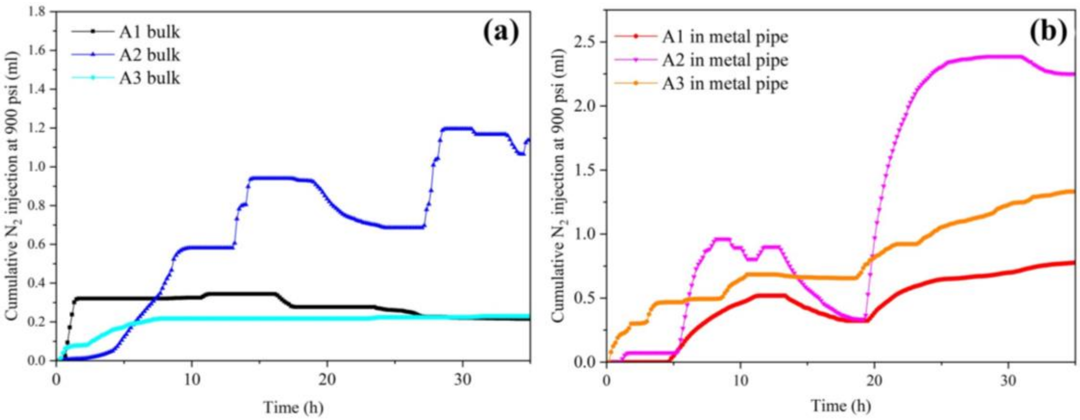

Figure 11. (a) comparison of cumulative N_2 intake of A1, A2 and A3 bulk cement; (b) comparison of cumulative N_2 intake of A1, A2 and A3 in metal pipe.

1.5 Wellbore simulator test results

1.5.1 Experimental procedure

The cement slurry is prepared by the API 10B-2 guidelines. After the cement slurry is poured into the inner casing of the wellbore simulator, the cement is cured at room temperature for 7 days while applying a constant pressure of 1000 psi in the annular space and on top of the cement slurry.

Once curing is finished, we first conduct the water permeability test by using the constant pressure mode of the ISCO pump. During the water permeability test, we keep the annular space pressure constant at 1000 psi while injecting water from the bottom of the cell at constant pressure of 1000 psi. If there is a water breakthrough, the average water flow rate is recorded and then the permeability is calculated. If there is no water breakthrough, the experiment is terminated after 50 hours of water injection.

Nitrogen permeability test is conducted immediately after the water permeability test. During the Nitrogen permeability test, we also keep the annular space pressure constant at 1000 psi while injecting Nitrogen from the bottom of the cell at constant pressure of 1000 psi. If there is no gas breakthrough, the Nitrogen injection is terminated after 100 hours. If a Nitrogen breakthrough is observed, a bubble flow meter is used to measure the Nitrogen flow rate at the outlet of the wellbore simulator at different injection pressures. The Nitrogen permeability is then calculated by using the measured values of gas flow rate and corresponding injection pressures.

1.5.2 Water permeability test results

Figure 12a illustrates the average water flow rate variation into A1 cement blend in the wellbore simulator. As shown in Fig. 12a, the average flow rate is relatively high at the beginning of the injection because of the system instability and the flow rate is gradually stabilized as the injection time increased. Figure 12b illustrates the cumulative volume of water injected into the A1 cement blend in the wellbore simulator. The cumulative volume of water injected into the cement column after 50 hours was only 2.93 ml. No water breakthrough was observed and no water volume remains was observed at the top of the cement column after all tests (i.e. after both water and Nitrogen injection tests are conducted) were finished using the cement blend A1.

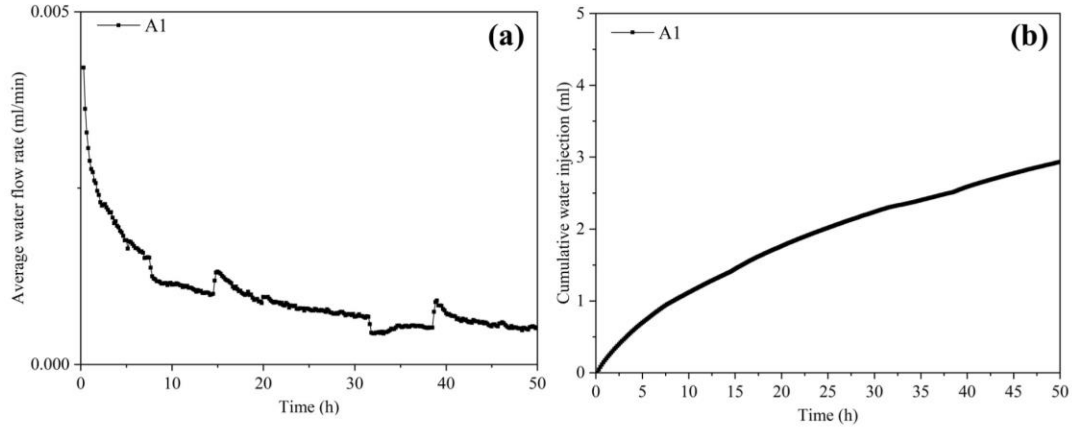

Figure 12 (a) Average water flow rate into the cement blend A1 in the wellbore simulator and (b) Cumulative volume of water intake of cement blend A1 in the wellbore simulator.

Figure 13a illustrates the average water flow rate variation into A2 cement blend in the wellbore simulator. Similar to cement blend A1, the average flow rate was relatively high at the beginning of the injection because of the instability of the system, which gradually leveled off as the water injection time increased. Figure 13b illustrates the cumulative volume of water injected into the A2 cement blend in the wellbore simulator. After 50 hours of injection, the cumulative water volume injected into the A2 cement blend was 6.04 ml, which is about twice as much as that of A1 cement blend. No water breakthrough was observed and no water volume remains was observed at the top of the cement column after all tests (i.e. after both water and Nitrogen injection tests are conducted) were finished using the cement blend A2.

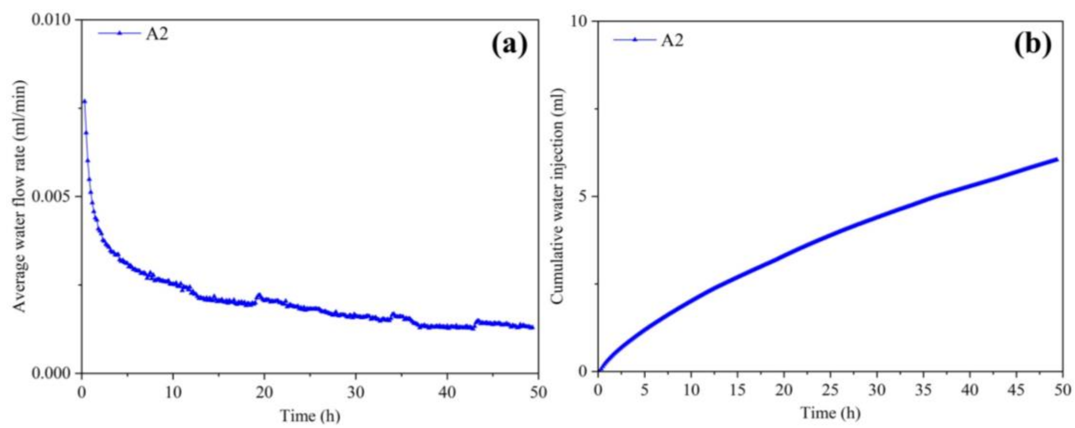

Figure 13 (a) Average water flow rate through the cement blend A2 in the wellbore simulator and (b) Cumulative water volume intake of cement blend A2 in the wellbore simulator.

We conducted two water permeability tests using cement blend A3. Figure 14a illustrates the average water flow rate variation into A3 cement blend in the wellbore simulator. The average water flow rate in both tests was high at the beginning but leveled off during the last 10 hours of injection. Figure 14b illustrates the cumulative volume of water injected into the A3 cement blend in the wellbore simulator. The cumulative water volume injected into the cement blend A3 in the wellbore simulator was 21.2 ml and 14.5 ml during the first and second permeability tests, respectively. These values were both much higher than the results obtained in the wellbore simulator for cement blends A1 and A2. No water flow out of the wellbore simulator was observed during the Test 1 of the cement blend A3. However, when we opened the test cell after conducting both water and Nitrogen permeability tests, we observed that some water was accumulated on the top of the cement column inside the wellbore simulator. To further explore the source of this water, we conducted a second water permeability test using the cement blend A3. This time, we opened the test cell immediately after finishing the water injection test. We observed that about 12 ml of water was accumulated at the top of the cement column, which was very close to the cumulative injected water volume of 14.5ml. We then calculated the permeability of the cement blend A3 using the flow rate data obtained during the last 10 hours of water injection test. The permeability of the cement blend A3 was estimated to be 174.5 Nano Darcy, which is very low for all practical purposes.

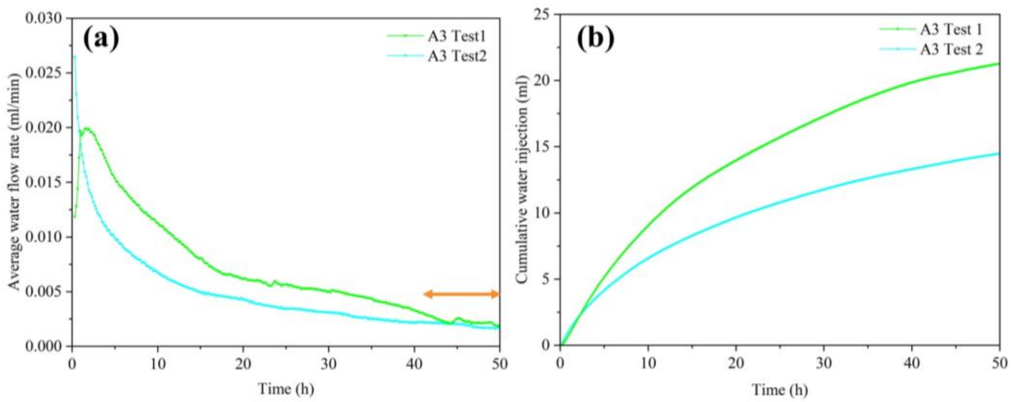

Figure 14 (a) Average water flow rate through the cement blend A3 in the wellbore simulator and (b) Cumulative water volume intake of cement blend A3 in the wellbore simulator.

Figure 15a illustrates the variations of average flow rate into the cement blends A1, A2 and A3. As shown in Fig. 15a, the lowest average water flow rate was observed for the cement blend A1. Average water flow rate observed for the cement blend A2 was slightly higher than that of A1. The highest average water flow rate was observed for the cement blend A3. Again, it can be seen that the average flow rates of A1 and A2 reached steady state within 10 hours, while A3 reached steady state after 40 hours in both tests. This may be due to the relatively small particle size of A3, so that the specific surface area of A3 is larger than that of both A1 and A2, thus making it easier for A3 to come into contact with water and hydrate, and the relatively high average flow rate in the first 40 hours may be a result of the continued hydration of A3. After 40 hours, the average flow rates into A1, A2 and A3 were all very close to each other.

Figure 15b shows the comparison of the cumulative volume of water injected into the cement blend A1, A2 and A3 in the wellbore simulator. Results shown in Fig 15b indicate that the cement blend A3 had the highest cumulative volume of water intake, followed by the cement blend A2 and the cement blend A1 had the lowest volume of water intake.

No water breakthrough out of the test cell and no water collections on the top of cement column were observed after all tests were finished using cement blends A1 and A2.

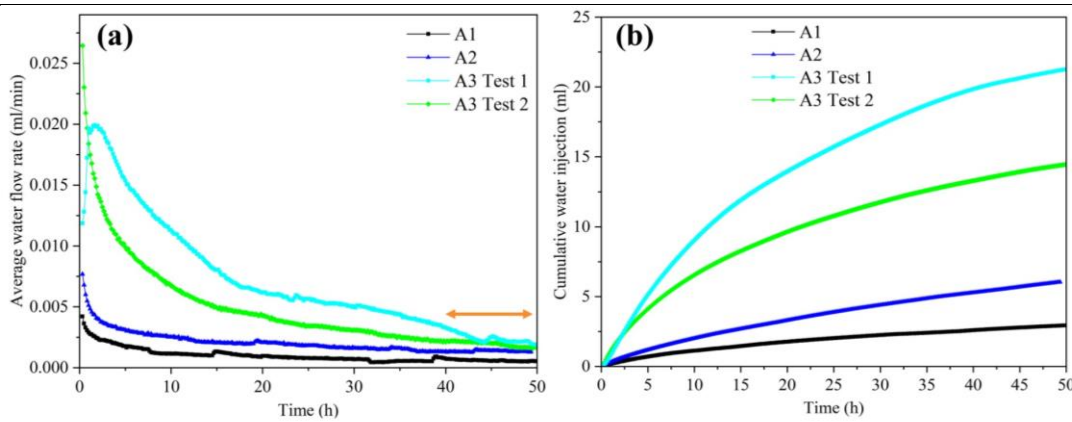

Figure 15. (a) Comparison of average water flow rate through the cement blends A1, A2 and A3 in the wellbore simulator (b) Comparison of the cumulative volume of water intake of cement blends A1, A2 and A3 in the wellbore simulator.

Figure 16a illustrates the average Nitrogen flow rate variation into the cement blend A1 in the wellbore simulator. The average flow rate of Nitrogen injection showed large periodic fluctuations. Figure 16b illustrates the cumulative Nitrogen volume injected into the cement blend A1 in the wellbore simulator. The cumulative Nitrogen volume intake of cement blend A1 over 100 hours was only 1.84 ml. No gas breakthrough was observed for the cement blend A1 tested in the wellbore simulator.

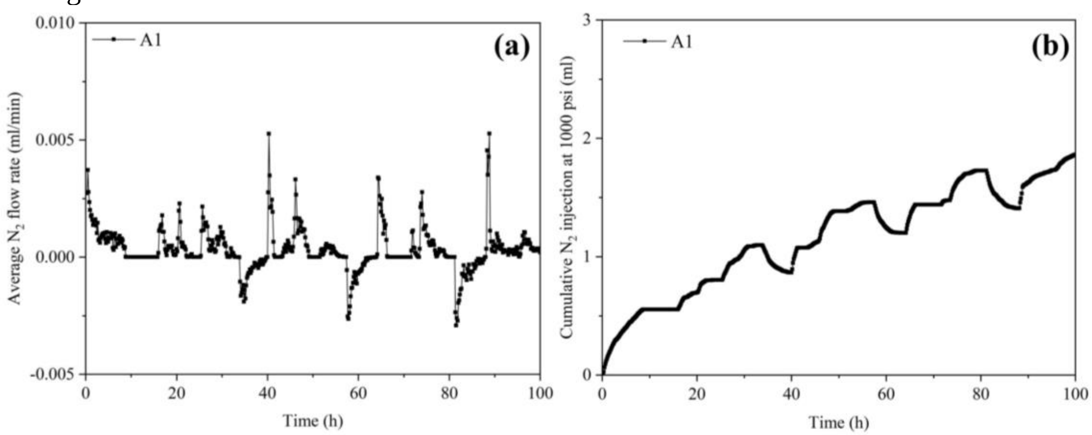

Figure 16. (a) Average Nitrogen flow rate through the cement blend A1 in the wellbore simulator and (b) Cumulative Nitrogen volume intake of cement blend A1 in the wellbore simulator.

Figure 17a illustrates the average Nitrogen flow rate variation into the cement blend A2 in the wellbore simulator. Similar to the cement blend A1, the average flow rate of Nitrogen injection into the cement blend A2 also showed large periodic fluctuations. Figure 17b illustrates the cumulative Nitrogen volume injected into the cement blend A2 in the wellbore simulator. The cumulative Nitrogen volume intake over 100 hours was 6.4 ml. No gas breakthrough was observed for the cement blend A2 in the wellbore simulator.

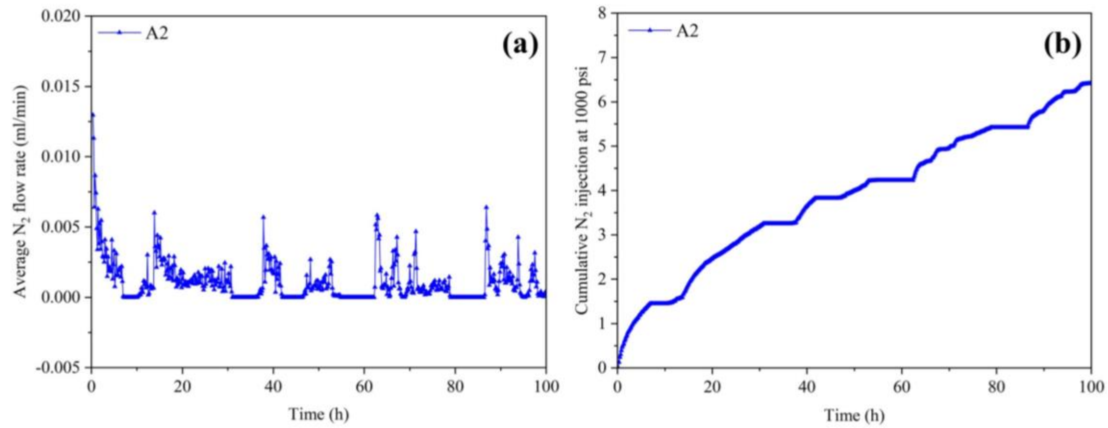

Figure 17. (a) Average Nitrogen flow rate through the cement blend A2 in the wellbore simulator and (b) Cumulative Nitrogen volume intake of cement blend A2 in the wellbore simulator.

Two Nitrogen flow tests were conducted using cement blend A3 in the wellbore simulator. Figure 18a illustrates the average Nitrogen flow rate variation into the cement blend A3 in the wellbore simulator. Similar to the cement blends A1 and A2, the average flow rate of Nitrogen injection into the cement blend A3 also showed large periodic fluctuations. Figure 18b illustrates the cumulative Nitrogen volume injected into the cement blend A3 in the wellbore simulator. In Test 1, the cumulative Nitrogen injection volume was 3.84 ml and no gas breakthrough was observed. In Test 2, however, gas breakthrough was observed after 31 hours. The possible reason for the difference between Nitrogen injection rates in Test 1 and Test 2 is that in Test 1, we did not open the wellbore simulator immediately after the water injection experiment nor did we remove the water from the top of the cement column. In the Test 2, we opened the wellbore simulator immediately after the water injection experiment and removed the water from the top of the cement column before performing the second Nitrogen injection test. This additional stress cycle prior to the N2 flow test might have contributed to the observed gas breakthrough in the second test.

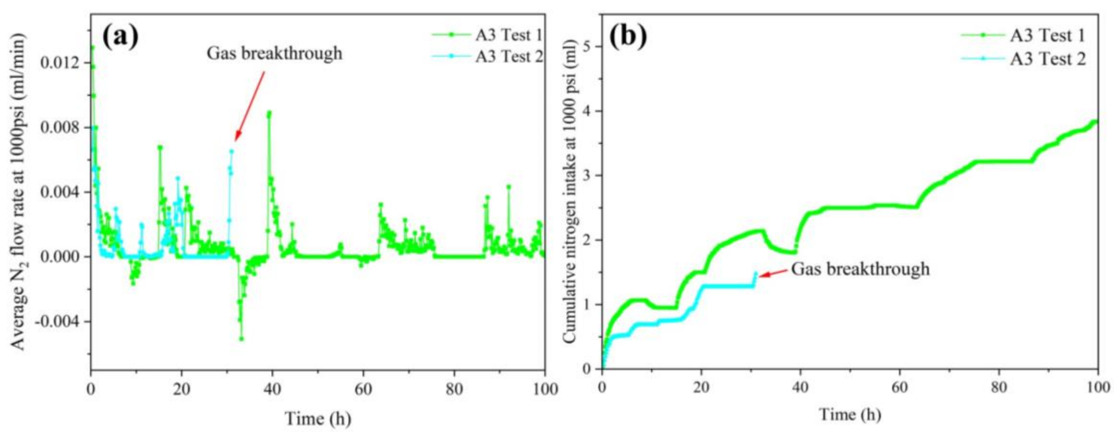

Figure 18. (a) Average Nitrogen flow rate through the cement blend A3 in the wellbore simulator and (b) Cumulative Nitrogen volume intake of cement blend A3 in the wellbore simulator.

Since gas breakthrough was observed in Test 2, we terminated the Nitrogen injection experiment and refilled the ISCO pump and tested the permeability. We adopted the approach of keeping the confining pressure constant (1000 psi) and changing the inlet pressure for permeability testing. Permeability was measured at Nitrogen injection pressures of 1000 psi, 900 psi, 800 psi, 700 psi and 600 psi and the results are shown in Figure 19. Apparent Nitrogen permeability values showed a linear correlation with the gas injection pressure. When the gas injection pressure was reduced from 1000 psi to 600 psi, the apparent permeability decreased from 5.08 micro Darcy to 1.81 micro Darcy. The permeability changed with the increasing Nitrogen injection pressure mainly because the effective stress was not constant in these experiments. The confining pressure was kept constant at 1000 psi level. The higher injection pressure means lower effective stress and, hence, higher permeability. These permeability values are relatively high as compared to cement matrix permeability commonly observed at the Nano-Darcy level, implying that Nitrogen breakthrough might have occurred through the microchannels formed at the cement/casing interface. While the permeability has increased in the tested systems relative to the bulk cement, the permeability is still low and still significantly below the generally accepted 100 microDarcy upper limit for ideal bulk cement permeability.

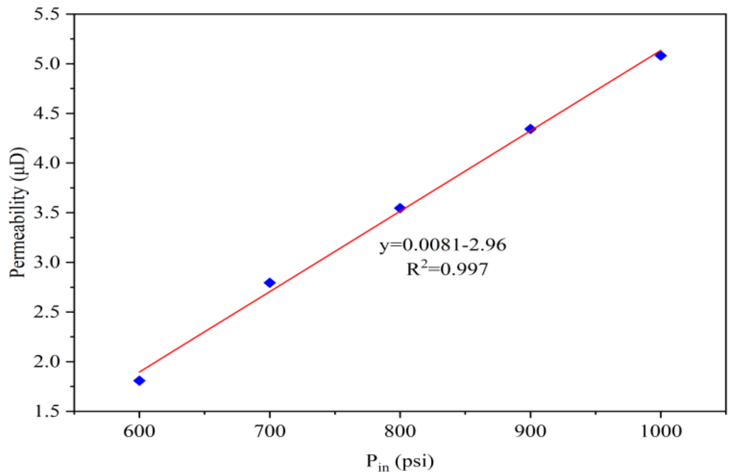

Figure 19 Nitrogen permeability of the cement blend A3 measured by using the data obtained in Test 2 in the wellbore simulator under different Nitrogen injection pressure.

Figure 20a shows comparison of the average Nitrogen flow rates through the cement blend A1, A2 and A3 in the wellbore simulator. As shown in Fig. 20a, there are large fluctuations in the average Nitrogen flow rates into all cement blends. Figure 20b shows the comparison of the cumulative volume of Nitrogen injected into the cement blend A1, A2 and A3 in the wellbore simulator. Results shown in Fig 20b indicate that the cement blend A2 had the highest volume of Nitrogen intake, followed by the cement blend A3 and the cement blend A1 had the lowest volume of Nitrogen intake.

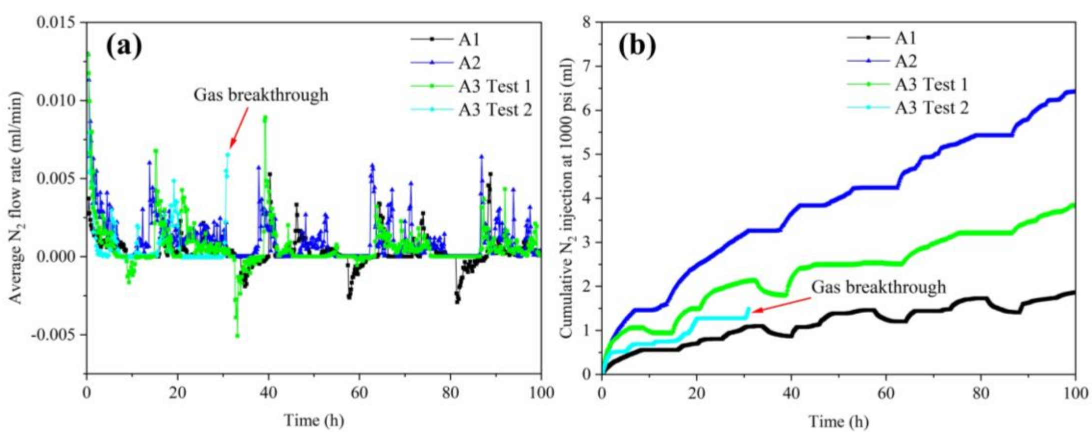

Figure 20. (a) Comparison of average Nitrogen flow rate into the cement blends A1, A2 and A3 in the wellbore simulator (b) Comparison of the cumulative volume of Nitrogen intake of cement blends A1, A2 and A3 in the wellbore simulator.

Table 4 summarizes the results of the shear bond strength measurements for the cement blend A1, A2 and A3, all measured after conducting water and Nitrogen injection tests. Comparison of the results from shear bond strength measurement tests indicates that the cement blend A1 has the highest shear bond strength while the cement blend A3 (Test 1) has the lowest shear bond strength. However, the shear bond strength of cement blend A3 (Test 2) in the second test was measured as the highest among the three blends. Considering the second permeability test of the cement blend A3 has been conducted using slightly different procedure than that of the one used in the previous three tests (i.e. in the 2nd permeability test using cement A3, we opened the cell after the water permeability experiment and collected water from the top of the cement column inside the wellbore simulator, closed the wellbore simulator and conduct the Nitrogen permeability test, where as in all the previous 3 tests water and Nitrogen permeability tests were conducted back to back without opening the cell until all the tests were finished), it is very likely that the shear bond strength test result for cement blend A3 (Test 2) might have been affected by the change in the experimental procedure.

No significant correlation was observed between shear bond strength and flowability.

Table 4. The shear bond strength of A1, A2 and A3

Blend	shear bond strength (MPa)	Cumulative water volume (ml/50 hours)	Cumulative N_2 volume (ml/100 hours)
A1	5.24	2.93	1.84
A2	4.64	6.04	6.4
A3 Test 1	4.06	21.2	3.84
A3 Test 2	5.25	14.5	breakthrough

2. Conclusions

Analyses of microstructural characteristics (using ESEM, micro-CT scan tests) of small-scale bulk and pipe cement samples of A3 blends were completed. Porosity and permeability tests were also conducted using large scale bulk and pipe cement samples of A3 cement blend.

Cement blend A1 has the lowest porosity (43.74%) while the cement blend A3 has the highest porosity value (52.49%).

Micro-CT scan results confirmed that cements have non-uniform pore size distribution and pores were not interconnected for any sizable fluid flow. Micro-CT scan results of pipe cement plugs (at 6-micron resolutions) have shown no presence of any gap at the cement/casing interface.

Isolated gaps were observed in the ESEM analyses of all cement samples in metal pipe. However, these gaps were observed only in the limited sections of the cement/casing interface (i.e. 2.0-2.5% of the total circumference of the cement/pipe sample) and the magnitude of these gaps were too small (i.e. within the range of 2.3-3.7 micron) to lead to any measurable fluid flow for all practical purposes.

No Nitrogen or water breakthrough were observed in all bulk and pipe cement plug flow tests indicating all cements have extremely low permeability, in the order of nano-Darcies.

No water and Nitrogen break through were observed using cement blends A1 and A2 in the wellbore simulator tests.

No water flow was observed out of the wellbore simulator during the 2 permeability tests conducted by using cement blend A3. However, some small volume of water was collected on top of the cement column in the wellbore simulator, indicating possible water breakthrough occurred in the cement blend

A3. After 40 hours of testing, the average water flow rate through all cement samples are leveled off with some small differences in magnitudes.

Nitrogen breakthrough was observed during the second Nitrogen permeability test using the cement blend A3. This gas breakthrough might have been caused by the additional stress cycle prior to the N2 flow test.

5. References

1. Al Moghadam, Koen Castelein, Jan ter Heege, Bogdan Orlic. A study on the hydraulic aperture of micro annuli at the casing–cement interface using a large-scale laboratory setup. Geomechanics for Energy and the Environment, Volume 29, 2022, 100269. https://doi.org/10.1016/j.gete.2021.100269.
2. Scrivener, K. L., Crumbie, A. K., & Laugesen, P. (2004). The interfacial transition zone (ITZ) between cement paste and aggregate in concrete. Interface Science, 12(4), 411–421. https://doi.org/10.1023/B:INTS.0000042339.92990.4c

Budget Summary Update: Comparing original budget to actuals

	Total Project Original Budget (all years)	Budget FY 2023-24	Actual Quarter Expenditures this fiscal year	Actual YTD Expenditures this fiscal year	YTD Variance %
AUPRF Funds Received	85,800	85,800			
Other Funds Received (and Sources)					
Total Revenue	**85,800**				
Project Expenses			55,242.73	61,278.73	
Total Expenditures			55,242.73	61,278.73	
Excess (or Deficiency) of Revenue over Expenditures				23,721.27	

Variance analysis (includes changes to invoicing schedule)
1.