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Enhancing Chalk Formation Integrity by Diammonium Phosphate Treatment: A Study on Downhole Cores

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Abstract

Chalk formations in the North Sea (NS) often experience particle migration, leading to production issues like formation damage and reduced production. This study aimed to improve chalk's strength and prevent particle migration using chemical consolidation technique. While different chemicals proved effective on standard Austin chalk (AC) outcrop samples, laboratory tests on NS downhole samples were conducted to assess their effectiveness in real-world conditions.

Two cylindrical AC plugs and three downhole cores from the NS, each measuring 1.5 inches in diameter, were treated with a 1M diammonium phosphate ($(NH_4)_2HPO_4$) solution at elevated temperatures (75°C). The samples were placed in a vacuum-sealed steel cell, pressurized to 1000 psi, and aged for 72 hours to transform calcite ($CaCO_3$) into the harder hydroxyapatite ($Ca_5(PO_4)_3OH$). Rock strength was assessed before and after treatment using a non-destructive impulse hammer. Mineralogical changes were examined via Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction (XRD). Permeability and porosity were also measured pre-and post-treatment. In addition, Total Inorganic Carbon (TIC) analysis was performed on the solution, and Gas Chromatography (GC) was utilized to analyze the gas phase, providing insights into the interactions between calcite and diammonium phosphate (DAP).

DAP treatment significantly increased rock hardness (100-300%) by forming a hydroxyapatite (HAP) layer. SEM, EDS, and XRD confirmed HAP formation, leading to hardening by altering calcite. While effective porosity remained unchanged, permeability decreased after treatment. The treatment was markedly more effective on AC samples due to their higher permeability and lower initial strength than downhole samples.

The paper used representative downhole samples with very positive results for eliminating fine migration through consolidation and making the chalk more cohesive and stronger. The innovative idea can help mitigate particle migration issues, improving operational efficiency and reducing production challenges.

Keywords

Fine Migration, Chalk, Diammonium Phosphate, Consolidation

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1 Introduction

Fine-grained North Sea (NS) chalk, characterized by high porosity and low permeability, constitutes economically important hydrocarbon reservoirs across much of the Danish and Norwegian sectors (Bramwell et al., 1999; Surlyk et al., 2003). Despite a good understanding of the factors controlling reservoir quality and the distribution of reservoir heterogeneities (e.g., Anderskouv & Surlyk, 2012; Blinkenberg et al., 2021; Brasher & Vagle, 1996; Fabricius, 2007; Maliva & Dickson, 1992), oil recovery from fine-grained chalks in the Danish NS is less than 30% of the in-place oil (Puntervold et al., 2009). Production from some chalk fields is further hampered by production-related phenomena that include particle migration of liquefied chalk caused by high drawdowns and depletion of the friable chalk matrix (Kristiansen & Meling, 1996). This process eventually fills the well, and tubing and ultimately destroys the flow near the wellbore.

Various methods have been utilized to strengthen chalk and mitigate particle migration, with chemical treatments being a primary focus. As reviewed by Samarkin et al., (2022), these treatments have proven effective specially on standard Austin chalk (AC) samples (Desouky et al., 2024). Research into chemical consolidation of chalk formations aims to improve rock strength while maintaining permeability, a critical factor for optimizing hydrocarbon recovery. Laboratory studies have identified optimal chemical systems and conditions, highlighting the importance of factors such as reaction kinetics, downhole temperature, and glass-system concentrations in achieving efficient chalk consolidation. Other notable approaches include glass-based chemical consolidation (Hoq et al., 2024), and the use of controlled dissolution glasses to form permeable or impermeable minerals within the formation (Olsen et al., 2021). However, despite these advancements, the application of these treatments to actual NS downhole samples remains largely unexplored, revealing a key gap in current research.

Diammonium phosphate (DAP) has emerged as a promising treatment for chalk consolidation (Desouky et al., 2024a). The chemical process involves the transformation of calcite into hydroxyapatite (HAP), a mineral that significantly enhances rock strength. This reaction not only binds the calcite grains together but also increases the overall stiffness and resistance to deformation of the chalk (Desouky et al., 2023; Desouky et al., 2024b). Studies have shown that DAP treatment can improve the mechanical strength of chalk, making it a viable solution for enhancing the durability and stability of chalk formations (Samarkin et al., 2023).

The primary objective of this research is to evaluate the efficacy of DAP treatment on both AC and NS downhole samples under simulated reservoir conditions. By conducting this assessment, we aim to address the critical issue of particle migration in chalk formations, particularly in the NS region. This study seeks to quantify the improvements in rock strength and formation integrity resulting from DAP treatment, comparing its effects on standard AC samples with those on real NS downhole cores. The significance of this research lies in its potential to enhance operational efficiency and mitigate production challenges in NS chalk formations. This research not only contributes to the scientific understanding of chalk consolidation processes but also offers practical implications for improving hydrocarbon recovery and extending the productive life of chalk reservoirs in the NS and potentially other similar geological settings.

2 Materials

The study utilized two types of chalk samples: AC and NS chalk. The AC samples were cylindrical plugs, 1.5 inches in diameter. The NS chalk samples were obtained from downhole cores, also 1.5 inches in diameter. For the treatment process, a 1M DAP ($(NH_4)_2HPO_4$) solution was prepared using reagent-grade chemicals sourced from Sigma Aldrich Company. This solution was chosen for its potential to transform calcite ($CaCO_3$) into HAP ($Ca_5(PO_4)_3OH$), thereby enhancing the structural integrity of the chalk samples.

3 Methodology

The study investigated the effectiveness of DAP treatment on chalk samples from two sources: outcrop AC and downhole NS chalk. Experiments were carried out using plugs, chips, and powders from both chalk types. In the first set of experiments, cylindrical plugs (1.5 inches in diameter) were prepared from both sources. Initial characterization included measuring petrophysical parameters (porosity and permeability) and conducting mineralogical and elemental analyses on sacrificial samples from each group.

Before treatment, the plugs' mechanical properties were evaluated non-destructively using impulse hammering technique. Measurements were taken on both the top and bottom surfaces of the samples, using a grid pattern to ensure uniform coverage. A total of 32 data points was collected for each sample. The plugs were then treated in a vacuum-sealed steel cell at 75°C for 72 hours with 1M DAP under 1000 psi pressure. Post-treatment, samples were cooled, thoroughly washed with deionized (DI) water, and dried in an oven at 60°C for 24 hours. A second round of mechanical assessments was conducted after the treatment, following the same procedures as the initial evaluation. A comparative analysis of the pre- and post-treatment results was performed to assess the impact of the treatment on the mechanical stiffness of the chalk.

In the second set of experiments, small chips extracted from the sacrificial samples were treated with 1M DAP solution in bakers at 75°C for 72 hours, intended for Scanning Electron Microscopy (SEM) imaging and Energy Dispersive Spectroscopy (EDS) analysis. The third set of tests was designed to evaluate the rate and efficiency of DAP reactivity using powdered samples, which increase the surface area-to-volume ratio, thereby facilitating more extensive reactions. Therefore, powder samples were prepared for treatment in the OLT-HP-500 reactor, a high-pressure, high-temperature apparatus capable of operating up to 773 K and 34 MPa. The particle size distribution of the powder was determined using the HELOS/KR QUIXEL instrument. A 50 g sample of powdered chalk was combined with 200 mL of 1M DAP solution and subjected to stirring at 1000 rpm for 72 hours. The reaction was conducted at a temperature of 75°C and an initial pressure of 1000 psi. The pressure was initially applied by injecting pure nitrogen into the reactor cell until the target pressure was reached. After the treatment period, the gas was sampled. Then, the reactor cell was disassembled and the powder was filtered using a 0.4-micron filter, and the solution was collected. The filtered powder was thoroughly rinsed with DI water and filtered multiple times to eliminate any residual solution. Subsequently, the powder was dried in an oven at 60°C for 24 hours.

A comprehensive set of analytical techniques was employed to assess treatment effectiveness. These included X-ray Diffraction (XRD) for mineralogical changes, SEM for microstructural alterations, and EDS to confirm HAP formation. The ammonium (NH_4^+) concentration in the solution was measured both before and after the addition of chalk using the MANTECH PC-BOD multiplier analyzer. This analyzer provided accurate detection of ammonium levels, enabling a detailed comparison of the changes in concentration after chalk treatment. Also, A UV-Vis Spectrometer (Analytik Jena SPECORD-50) was employed for Total Inorganic Carbon (TIC) measurements of the DAP solution before and after treatment quantified carbon in the solution, while Gas Chromatography (GC) analyzed gaseous byproducts.

4 Results and Discussion

4.1 Petrophysical Characterization

Sample NS 120 was designated as the sacrificial sample due to its initial cracked condition. Detailed porosity and permeability measurements for both chalk types are presented in Table 1. The permeability of AC samples is significantly higher than that of the downhole samples, ranging from one to two orders of magnitude greater. This discrepancy can be attributed to the downhole samples being much more compacted in comparison to the AC. Despite this difference in permeability, the porosity values for both chalk types fall within a similar range. Following the treatment, porosity remained unchanged, while permeability experienced significant impairment. For the highly permeable AC, permeability reductions reached up to 90%, with the extent of impairment decreasing as the initial permeability lowered. In contrast, the tight NS samples exhibited no measurable reduction in permeability, likely due to their already low permeability restricting further changes.

Table 1: Petrophysical Properties for Both Chalk Types

Plug Number/Sample Id	Source	Depth (m)	Porosity (%)	Permeability (mD)
NS 11	North Sea	2112.82	36.89	0.86
NS 57		2130.6	29.07	0.07

NS 120		2159.79	30.31	1.11
NS 143		2168.58	33.81	1.88
AC 11	Austin	-	32.7	34.47
AC 13		-	28.6	19.62

4.2 Impulse Hammering

The AC samples demonstrated significant increases in elastic stiffness after DAP treatment, while the NS samples showed minimal changes. For instance, AC 11 and AC 13 exhibited increases of 115% and 312% in elastic stiffness, respectively, following the treatment, as illustrated in Fig. 1. In contrast, NS 11, NS 57, and NS 143 displayed similar boxplots before and after treatment, with a higher variance i.e. standard deviation (Std) in the collected data points compared to their pre-treatment measurements (see Table 1).

Table 2: Statistical Summary of the Mechanical Assessment by Impulse Hammering

Statistic	AC 11		AC 13		NS 11		NS 57		NS 143	
	Before	**After**	**Before**	**After**	**Before**	**After**	**Before**	**After**	**Before**	**After**
Average (GPa)	3.05	6.57	2.26	9.32	14.87	14.96	16.58	16.37	18.16	18.63
Std (GPa)	0.67	1.35	0.52	2.30	0.91	1.77	1.62	3.39	1.18	1.68
Change (%)	115.55		312.38		0.62		-1.31		2.62	

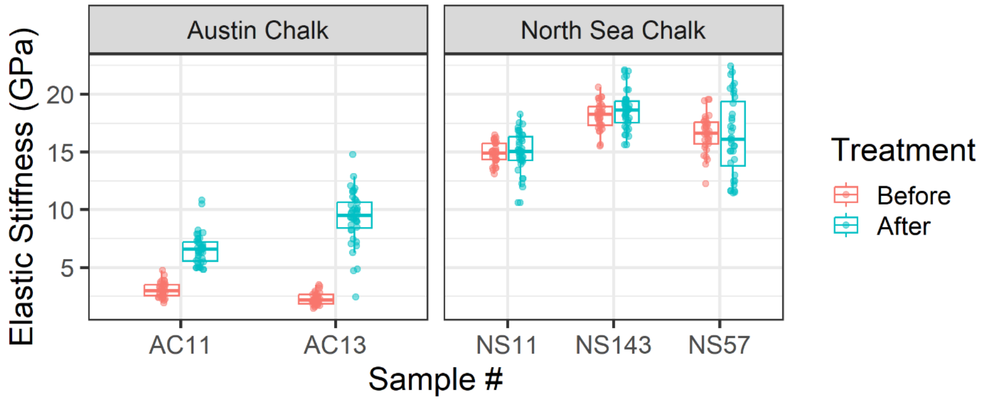

Fig. 1: Comparison of Chalk Elastic Stiffness Before and After DAP Treatment

For instance, when evaluating the highest change in elastic stiffness observed in the NS downhole samples—specifically sample NS 143—statistical analysis was conducted. The resulting F-statistic was 0.6074, indicating a relatively low value. This suggests that the difference between the "Before" and "After" treatment groups is not statistically significant. Therefore, the treatment did not induce a statistically meaningful change in the measured elastic stiffness.

Also, a Q-Q plot with an R^2=0.95 indicates a very high degree of similarity between the quantiles of two before and after being compared (See Fig. 2).

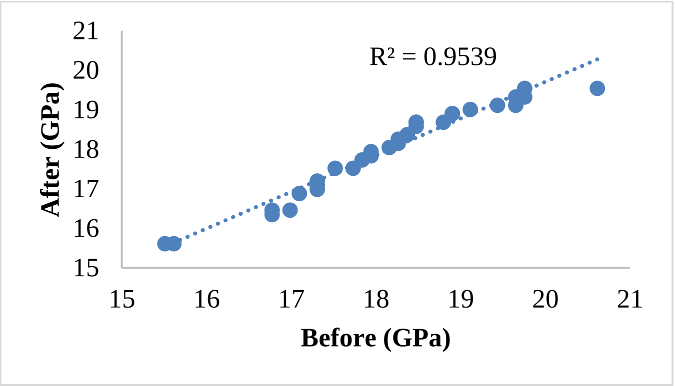

Fig. 2: Q-Q Plot of Sample NS 143 Elastic Stiffness Before and After DAP Treatment

AC samples, characterized by higher permeability (one to two orders of magnitude greater than NS downhole samples), showed significant elastic stiffness improvements (115–312%) after DAP treatment. This mirrors the natural hardening seen in the Isle of Purbeck, where the presence of permeable pathways allowed fluids to flow and facilitated cementation. In contrast, the NS samples, with their compacted and low-permeability nature, exhibited minimal changes in stiffness, akin to how restricted permeability in natural settings limits pressure solution-cementation. The Isle of Purbeck serves as a valuable analog, demonstrating that maintaining or inducing permeable pathways is essential for effective hardening. The effect of permeability on the artificial hardening of chalk is evident in this study and aligns with natural processes observed in formations like the Isle of Purbeck (Jones et al., 1984).

4.3 Calcite-DAP Reaction Analysis

The reaction between chalk and DAP was thoroughly investigated using various analytical techniques for both chalk types. Small chips and components of the powder treatment specifically the solution, the powder itself, and the gas were examined to elucidate the reaction pathways and their distinct outcomes for each chalk type. The reaction between calcite and DAP is well-documented in the literature and is commonly represented as Eq. 1:

$$\mathrm{10CaCO_3 + 6(NH_4)_2HPO_4 + \xrightarrow{2H_2O} Ca_{10}(PO_4)_6(OH)_2 \downarrow + 6(NH_4)_2CO_3 + 4H_2CO_3} \tag{1}$$

This reaction showcases the transformation of calcite into HAP in the presence of DAP, with ammonium carbonate and carbonic acid as byproducts.

To analyze the mechanical strength improvement resulting from DAP treatment in chalk, the cementation characteristics at the particle contact level were examined using SEM. This investigation focused on observing how the DAP treatment influences the bonding between chalk particles and how it enhances the overall cohesion of the material. Initial SEM imaging of the untreated small chips revealed typical calcite grains for both chalk types, with the primary distinction being the abundance of coccolith structures in the NS chalk samples. Images A and C in Fig. 3 depict untreated chalk composed of calcite grains, which appear relatively uniform in size and shape, with clean and distinct surfaces. There is no evidence of HAP crystal formation or inter-particle cementation. In contrast, Images B and D in Fig. 3 illustrate the effects of DAP treatment, showing a clear formation of HAP crystals with a distinct rosette-like or flower-like morphology growing on the surface of the calcite grains. This transformation highlights the successful reaction between calcite and DAP, leading to the development of a cemented structure. The presence of HAP enhances cohesion among the grains, indicating that DAP treatment effectively improves the mechanical strength of chalk by facilitating inter-particle binding. In the treated samples, SEM images demonstrated the formation of HAP, which appeared to bind the calcite grains together, indicating a successful reaction with the DAP solution as shown. These observations are not due to the masking of surface details by conductive coatings but rather indicate the growth of non-carbonate minerals on calcite particles, a phenomenon also documented by Hjuler et al. (2018).

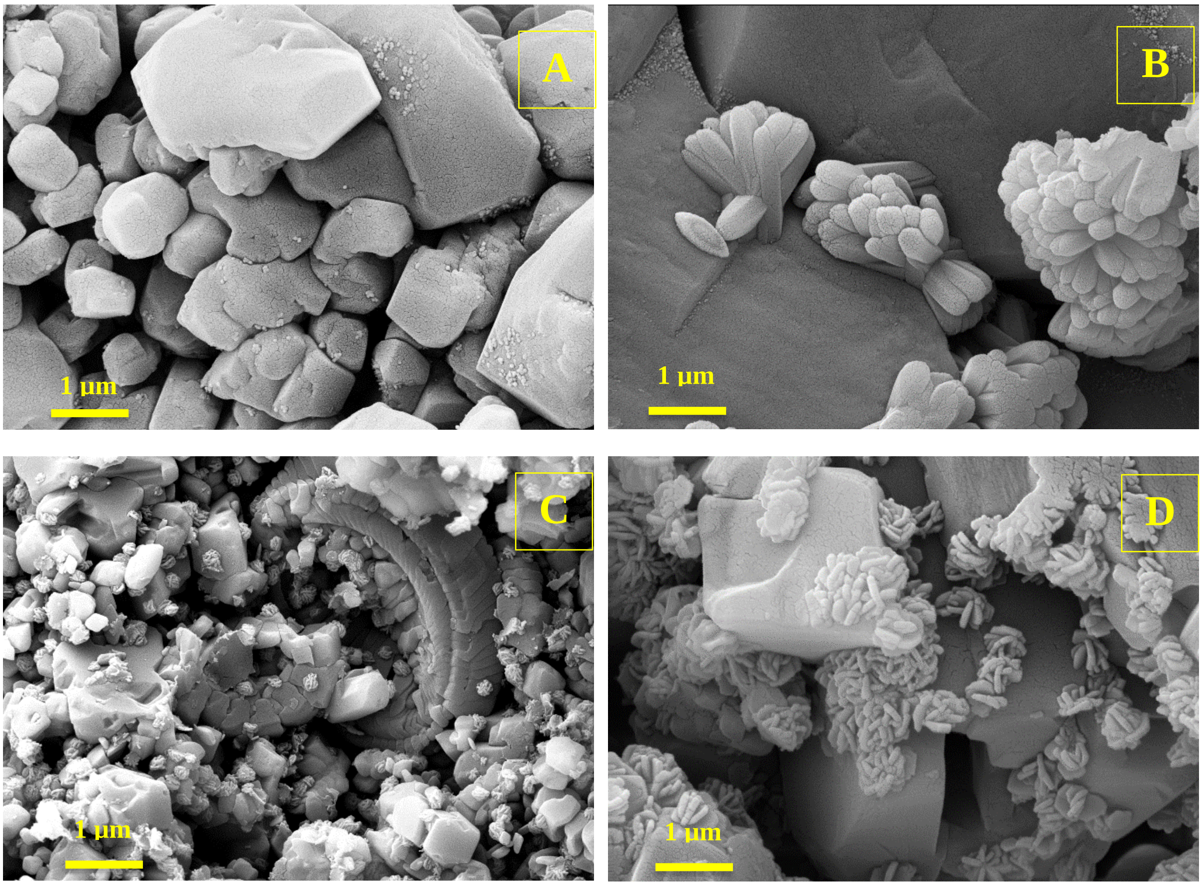

Fig. 3: SEM of A) Fresh AC sample prior to treatment B) Treated AC and C) Fresh NS sample prior to treatment D) Treated NS Chalk

Additionally, the EDS analysis of the untreated samples revealed the presence of only calcium (Ca) and oxygen (O). In contrast, analysis of treated samples identified phosphorus (P) in addition to Ca and O at points corresponding to the HAP phase like Spectrum 9, Spectrum 10, and Spectrum 11. At points associated with calcite grains e.g. Spectrum 14, only Ca and O were detected, consistent with the untreated samples. This pattern was observed for both chalk types, as illustrated in the Fig. 4.

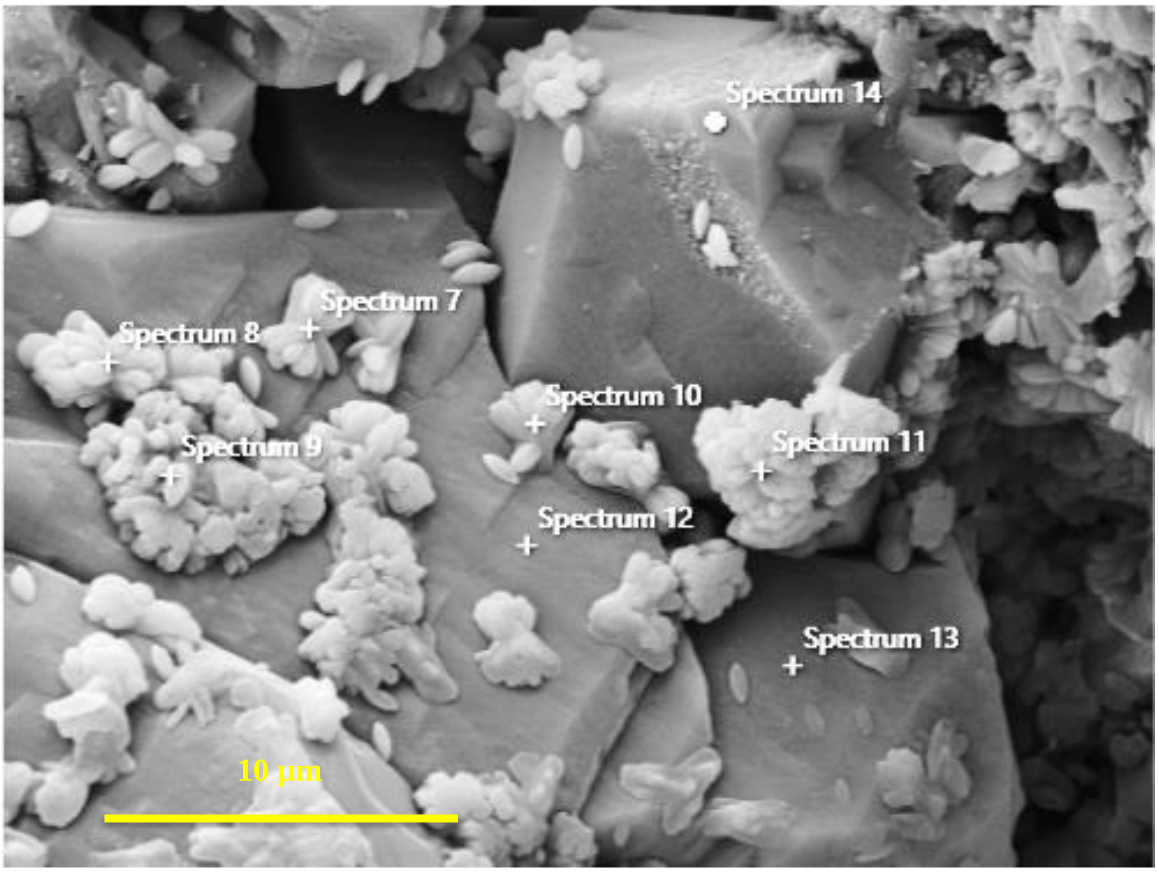

Fig. 4: EDS Analysis at Different Spots Revealing Calcite and HAP Crystals

The formation of HAP likely diminishes the quantity of mobile fines available for migration, which typically occur at the micron and submicron scale (Muecke, 1979). This process not only enhances the structural integrity of the chalk but also potentially mitigates particle migration by anchoring fines to the chalk fabric. Consequently, this may reduce the likelihood of bridging and subsequent productivity decline. Also, it is likely to counter the effect of improved oil recovery from chalk reservoirs by water-flooding that might cause mechanical weakening and change in elasticity as reported by Gram et al. (2021). However, it is crucial to balance these benefits against the potential reduction in permeability that may result from the HAP treatment itself. The overall effectiveness of the treatment should be evaluated by considering both the improved stability and any associated decrease in fluid flow capacity.

Grain size distribution was assessed following an identical grinding process for both chalk types. For AC, the median grain size (x50) was 6.26 µm, whereas the NS chalk had a median value of x50 = 6.48 µm. Additionally, the specific surface area (Sm) of the AC powder was 6465.15 cm^2/g, while the NS powder exhibited a lower specific surface area of Sm = 4555.20 cm^2/g.

Monitoring the reaction pressure reveals an initial increase followed by a gradual decline. The initial pressure rise of approximately 30–40 psi within the first 5 hours is likely attributed to the generation of gaseous products such as NH_3 and CO_2, stemming from the decomposition of $(NH_4)_2CO_3$ and H_2CO_3 as elaborated in Eq. 1. Over time, a portion of these gases may dissolve into the liquid phase of the reaction mixture, leading to a progressive reduction in pressure as equilibrium between the gas and liquid phases is established. A similar trend was observed for both chalk types, with AC exhibiting a slightly higher initial pressure rise and a gentler decline compared to NS chalk, as depicted in the Fig. 5.

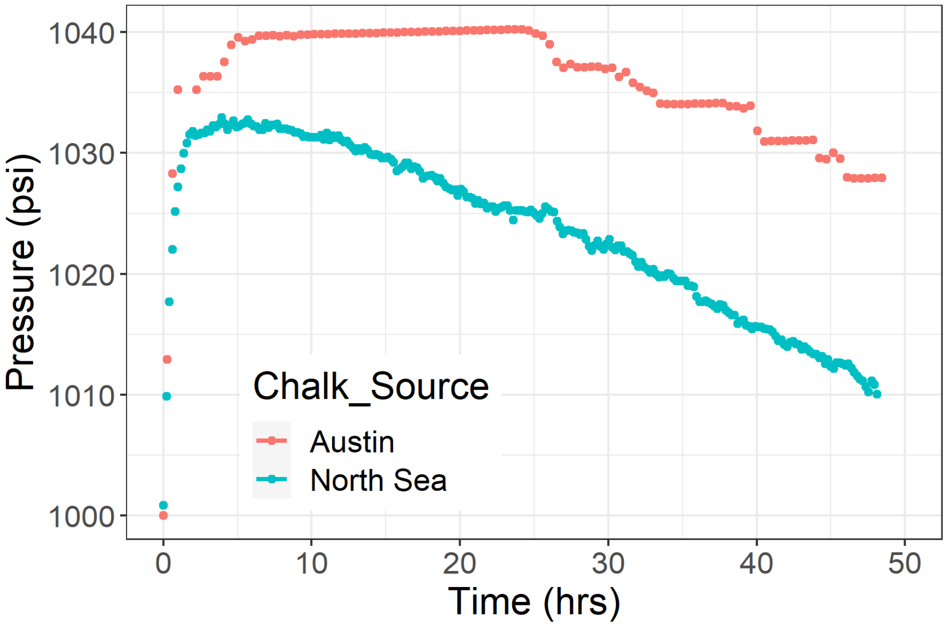

Fig. 5: Reaction Pressure Change with Time

After the treatment period, gas samples collected from the top of the reactor were analyzed, confirming the presence of N_2 and CO_2 in the gas phase, as shown in Fig. 6. Similar chromatograms of gaseous products were obtained from both the AC and NS chalk reactions with DAP. The available GC model lacked a column for NH3 detection; however, the concentration of NH4+ in the solution was measured before and after treatment with the MANTECH system, showing a decrease in NH4+ levels. For instance, the NH4+ concentration in the 1M DAP solution used to treat the NS, after a 10-fold dilution, decreased from 1083 ppm to 835 ppm following the reaction. This suggests that a portion of the NH4+ was released into the gas phase as NH_3 during the reaction between DAP and chalk. Both gas and solution analyses indicated that the reaction progression for both types of chalk followed a highly similar pattern, as described by Eq. 1.

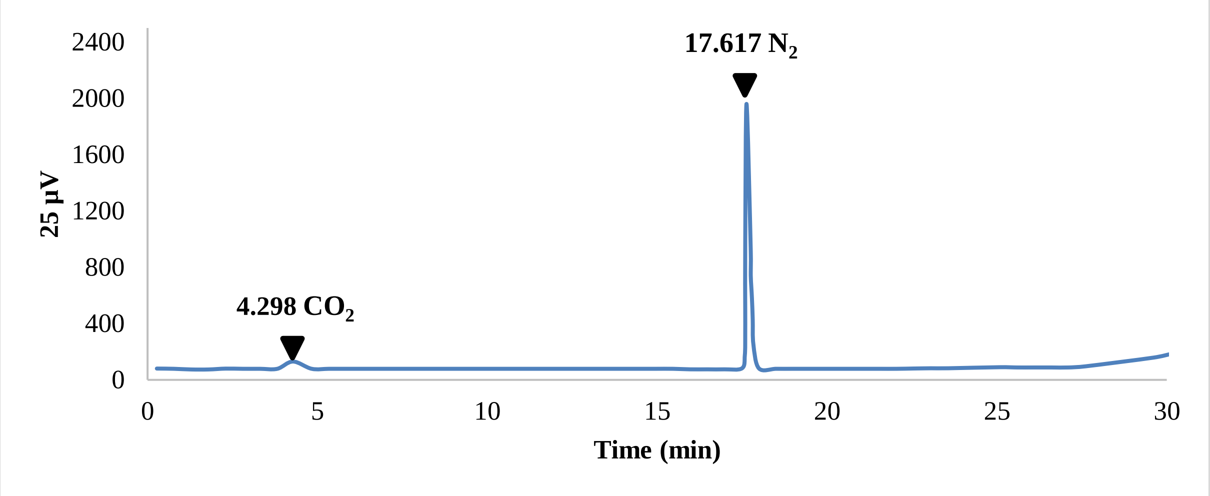

Fig. 6: Chromatogram Output of Gas Sample from AC Reaction with DAP Product Analyzed by GC

The filtered powder XRD analysis revealed that the high-purity calcite present in both chalk types underwent significant transformation into HAP during the reaction. The fresh powder samples, with calcite content exceeding 99% which is common in chalk reservoirs (Frykman et al., 2004), showed substantial conversion after treatment. Specifically, 94% of calcite was transformed into HAP in the NS chalk sample, while 66% conversion was observed in the AC sample suggesting that the first might be more reactive than the later. The results could be supported by the solution carbon intake which is an indication of the calcite dissolution.

Hence, the TIC measurements were utilized to shed light on the reactivity of calcite with DAP in both AC and NS powder samples. Initially, TIC concentrations differed, with AC mixed with 1M DAP showing 1.11 g/L, while the NS sample measured 390.2 mg/L. This difference likely reflects variations in initial carbonate content or calcite reactivity between the materials. These measurements were taken shortly after mixing, prior to loading the reactor cell.

Post-reaction TIC values increased significantly in both cases, with AC reaching 11.54 g/L and NS powder rising to 18.96 g/L. These substantial increases indicate extensive calcite dissolution during the reaction. Notably, despite its lower initial TIC, the NS powder exhibited a greater rise, implying higher reactivity or more readily accessible calcite content compared to AC.

While the DAP treatment proved highly effective for AC samples, its limited impact on intact NS downhole plugs highlights the challenges posed by the tight, low-permeability nature of these formations. However, the high reactivity observed in the powdered state indicates that the treatment could still be beneficial in scenarios where the chalk structure is more accessible to the DAP solution. These findings underscore the importance of considering formation characteristics when developing consolidation strategies for chalk reservoirs. Future research should focus on optimizing treatment methods for low-permeability formations to maximize the potential of DAP in mitigating particle migration and enhancing production efficiency in NS chalk reservoirs.

5 Conclusion

This study demonstrates the potential of DAP treatment for enhancing chalk formation integrity, particularly in addressing particle migration issues in NS reservoirs. The key findings of this research are:

1. DAP treatment significantly increased the elastic stiffness of AC samples, with improvements ranging from 115% to 312%.
2. NS downhole plugs showed minimal changes in elastic stiffness after treatment, likely due to their low permeability and tight structure. Statistical analysis confirmed that the observed changes were not statistically significant.
3. SEM and EDS analyses confirmed the formation of HAP on treated samples, evidenced by the detection of phosphorus post-treatment.
4. The powdered NS chalk exhibited high reactivity with DAP inside the reactor, suggesting that the treatment's effectiveness is influenced by the chalk's physical state and surface area accessibility.

These findings underscore the need for strategies, such as microfracturing, to enhance permeability during treatment and replicate the natural development of cementation pathways observed in geological formations.

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