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Evaluating the Effectiveness of Potassium Ferricyanide for Salt Precipitation Inhibition during CO_2 Storage in Deep Saline Aquifers

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Salt precipitation in the near-wellbore region of deep saline aquifer during CO_2 injection can alter permeability, reduce injectivity, and increase operational costs, thereby reducing the effective storage capacity and raising remediation costs. While previous studies have explored the potential of polymers, acids, and complex synthetics to address this issue, yet few have directly focused on inhibiting salt precipitation. Inhibiting salt precipitation can help maintain storage efficiency and support long-term climate objectives. This study evaluates the effectiveness of potassium ferricyanide (PFC) as a chemical inhibitor for salt precipitation under typical storage conditions. A static test was conducted to assess the interaction between CO_2, PFC, and brine at salinities of 30,000 and 100,000 ppm, and at PFC concentrations of 200 and 500 ppm. Inhibition efficiency, determined through weight difference measurements, showed that at 30,000 ppm salinity, PFC achieved 63.75% and 87.5% inhibition at 200 and 500 ppm, respectively. In contrast, at 100,000 ppm salinity, efficiencies declined to 0% and 19.76%. These results were further supported by NaCl atomic percentage trends and salt mapping. Dynamic core flooding tests showed that the treated core experienced only a 5% reduction in permeability, compared to a 69.3% reduction in the base case. This work highlights PFC as a promising and effective salt inhibitor for enhancing CO_2 injectivity, especially in low-to-moderate salinity conditions.

Keywords: Salt precipitation, CO_2 injection, permeability, potassium ferricyanide, saline aquifers

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Nowadays, anthropogenic carbon dioxide (CO_2) emissions continue to rise, posing significant risks to the global climate system. Elevated atmospheric CO_2 levels, primarily from fossil fuel combustion and industrialization, severe risks to the global climate system [1, 2]. Carbon capture and Storage (CCS) is among the most promising mitigation strategies, involving the injection of captured CO_2 into deep geological formations such as saline aquifers for long-term storage [3, 4]. Saline aquifers are widely regarded as suitable formations for geological CO_2 storage capacity, deep confinement, and widespread distribution [5, 6]. Their global storage potential estimated to range 400 to 10,000 gigatonnes (Gt) of CO_2 enables long-term, large sequestration beyond the capacity of depleted hydrocarbon reservoirs [6-8]. In addition, deep confinement enhances storage security through residual, solubility, and mineral trapping. This depth also facilitates effective pressure management during injection, thereby reducing the risks of formation fracturing and brine migration into overlying formations [6, 9]. In addition, the successful demonstration of CCS in various large-scale field projects, such as the Sleipner Project in the North Sea and the In Salah Project in Krechba, Algeria, have successfully demonstrated CO_2 injection into saline aquifers [10, 11].

However, one of the major technical challenges during CO_2 injection in carbon capture and storage (CCS) projects is salt precipitation. When dry or supercritical CO_2 displaces brine, it causes water evaporation and deposition of halite (NaCl) within pore spaces. This process reduces permeability and injectivity near the wellbore, threatening storage efficiency [12, 13]. Recent laboratory studies show that salt precipitation in sandstones can reduce Young's and shear moduli by up to 50% and double Poisson's ratio [14]. In addition, Sun, et al. [15] reported that porosity decreased by more than 20%

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at 1 mL/min. This indicates that salt precipitation not only impairs injectivity but also poses geomechanically integrity risks in CO_2 storage reservoirs. Moreover, Hauber [16] reported well blockage at Tubaen due to mineral buildup at the perforation-stratum interface, limiting effective storage to just 11% 91.4 Mt out of 12.6 Mt). tis highlights the practical challenges in large-scale CCS operations. Salt precipitation is a common issue during storage monitoring, which leads to halite (NaCl) precipitation and scale deposition, reducing both static and dynamic properties of the porous medium [4, 17, 18].

Salt precipitation is well known in the chemical industry but remains challenging under geological storage conditions due to high temperature, pressure and variable brine compositions. While freshwater pre-flushing has been used to reduce brine salinity and minimize precipitation [19, 20]. This method faces practical challenges due to operational limitations. For example, recent research by He, et al. [4] focused on halite prevention under production settings, but similar issues also arise during CO_2 injection in CCS. Several studies and laboratories have been conducted for halite precipitation and potential chemical inhibitors under varying conditions. Several researchers have conducted static and dynamic tests, reporting that static tests tend to produce smaller salt crystals, while the presence of inhibitors in dynamic tests results in reduced permeability deterioration [21-25].

In this context, potassium ferricyanide (K3[Fe (CN)6]) called PFC emerges as a potential inhibitor for salt precipitation. It possesses strong ionic complexation properties that may help sequester free Na+ and Cl- ions in solution, thereby delaying or preventing the crystallization of NaCl [26]. Research by Le [23] conducted a jar test using PFC, confirming the effectiveness of PFC that achieved 100% inhibition efficiency, effectively preventing halite scale precipitation. In addition, Ruan, et al. [27] studied under different temperatures and brine conditions. It was found that the halite saturation index (SI) increased from -0.028 to +0.025 as temperature increased, reaching SI reaching 0 at 55.3°C. Ferrocyanide (FCN) effectively prevented halite precipitation at 25°C. However, it is used in CO_2 injection scenarios and has not been thoroughly investigated, presenting a novel avenue for research into its effectiveness and environmental compatibility as a salt precipitation inhibitor.

These findings highlight that although PFC shows excellent performance in controlled experiments, its effectiveness and environmental compatibility under real CCS conditions remain unexplored. This gap in research highlights the need to evaluate PFC' performance and compatibility under controlled experiments that mimic CCS reservoir conditions. This study aims to evaluate Potassium Ferricyanide (PFC) as a potential salt inhibitor under CO_2 injection, with its effectiveness assessed through morphological and elemental analyses under static and dynamic reservoir conditions.

EXPERIMENTAL

Chemicals and Materials Preparation

Berea sandstone cores were selected as the representative porous medium for both static aging cell and dynamic core flood experiments, with the range of porosity and permeability is 20 ± 2% and 145 ± 20 mD respectively. Each core sample has a length and diameter within 76.2 ± 2 mm and 38.1 ± 2 mm respectively. For the dynamic core flooding test, core sample BR3 was designated as the base case specimen to establish a reference for permeability changes, while core BR2 was treated with potassium ferricyanide (PFC) to inhibit salt precipitation. BR2 (porosity and permeability is 7.36% and 201.177 mD respectively) and BR3 (porosity and permeability is 20.91 % and 148.926 mD respectively).

Sodium chloride (NaCl) solution was selected for synthetic brine preparation (all 99% purity; R&M Chemicals, London, UK). The NaCl have been prepared using different salinities of 30,000 ppm and 100,000 ppm.

Purified CO_2 with about 99.5% purity from Linde was used in the experiments. For dynamic core flooding experiments, all injections were done in supercritical phase (temperature of 60˚C, injection pressure of 1,600 psi, and confining pressure 2500 psi).

Potassium Ferricyanide (PFC) (CAS: 13746-66-2; Sigma-Aldrich), was selected for this study using different concentrations of 200 ppm and 500 ppm due to it allowing the study to both extend into underexplored concentration ranges and evaluating PFC's proven ability to inhibit NaCl crystallization. PFC have been prepared by dissolved in 30,000 ppm of NaCl and 100,000 ppm of NaCl.

Methods

For static aging cell test, six Berea sandstone cubes ($1cm^3$) were used to investigate the CO_2/brine/rock interactions. For the quantitative analysis, the mass was taken at three conditions: before saturation, after saturation, and after CO_2 exposure. Additionally, elemental composition and surface morphology of the rock samples were examined at the Centralized Analytical Laboratory (CAL), Universiti Teknologi PETRONAS, Malaysia. The samples were saturated with brine in a vacuum desiccator for 24 hours. Four samples were then saturated with PFC inhibitor for an additional 24 hours, while two samples were used as a base case.

Firstly, the samples were placed in the 500-mL aging cells filled with 400 mL of brine. Secondly, the cells were tightly closed to prevent atmospheric interference. Subsequently, supercritical CO_2 was injected through the one-way inlet until the pressure reached 1600 psi with the aid of a gas booster. Safety precautions were taken, ensuring no pressure leakage through the inlet and lid. Lastly, all cells were stored in an oven at 60 °C for 24 hours, after which the samples were removed and dried in the oven for an additional 24 hours and weighed. The mineralogy and surface morphology were ascertained to understand the role of PFC in inhibiting salt precipitation. Noted that sample 1 and sample 2 would be as recorded base case in this test.

Dynamic core flooding test were conducted, where all test fluids–including brine solution, carbonated brine (20% of CO_2 and 80% of brine), supercritical CO_2, and the PFC inhibitor solution–were loaded into the injection accumulators. The experiment was performed under controlled conditions with a confining pressure of 2500 psi, an injection pressure of 1600 psi, and a temperature maintained at 60 °C.

For the first test, core sample BR2 was used to evaluate potassium ferricyanide for inhibiting salt precipitation. Initially, brine was injected at the designated rate (0.5, 1, and 2 cc/min), within 10 pore volume (PV) for the measurement of the initial permeability. This was followed by the injection of the chemical inhibitor, PFC solution, at the flow rate of 0.5 cc/min for two hours to prevent salt precipitation. Then, carbonated brine was introduced at 0.5 cc/min within 4 PV to simulate dissolution in the reservoir. Supercritical CO_2 was then injected at a rate of 2 cc/min within 4 PV to evaluate the permeability reduction due to salt precipitation. Brine was reintroduced for the determination of the final permeability. The same procedure was repeated using a second test core (BR3), but without PFC injection, to serve as base case.

Prior to Field Emission Scanning Electron Microscope- Energy Dispersive X-Ray Spectroscopy (FESEM-EDS), was employed to characterize pore-scale morphological and elemental changes associated with salt precipitation. Rock samples obtained after core-flooding experiments were dried under controlled conditions prior to analysis. FESEM imaging was conducted to examine surface morphology and salt deposition within the pore structure, while EDS was used for semi-quantitative elemental analysis to identify the presence and spatial distribution of sodium (Na) and chlorine (Cl). All analyses were performed using FESEM system (TESCAN Clara) at the Centralized Analytical Laboratory (CAL), Universiti Teknologi PETRONAS, Malaysia. This integrated FESEM-EDS approach was applied to compare elemental signatures and surface features between the base case, and samples treated with 200 ppm and 500 ppm PFC.

RESULTS AND DISCUSSION

Figure 1 shows the FESEM images at 30,000 ppm (left) and 100,000 ppm (right) before and after the static aging cell test. As observed in Figure 1, at lower salinity (30,000 ppm, left), for the base case (1a), minor structural changes were observed, particularly in the grain matrices (blue arrow), which were covered by salt layers with crystal fragments present in the pore spaces (red arrow). For the 200-ppm concentration of PFC (2a), small foreign particles (green arrow) were observed attached to the grain matrices by salt crystals. At 500 ppm (3a), these small particles appeared to have been washed away (orange circle). The microstructural evidence indicates that higher PFC concentration effectively suppresses polycrystalline growth and modifies nucleation behaviour, thereby limiting pore-scale blockage. Similar observations have been reported in previous studies, where PFC increased critical supersaturation required for halite nucleation and reduced crystal attachment within pore networks [23, 27]. These findings support the role of PFC as a salt inhibitor to improved pore connectivity under CO_2 injection conditions.

At higher salinity (100,000 ppm, right), more severe salt precipitation occurred due to the increased NaCl concentration in the brine. For the base case (1b), salt mainly formed as large foreign fragments on the grain matrices (red arrow), with minor pore-throat narrowing also observed due to thicker deposits (orange circle). These FESEM observations are consistent with those reported by Yusof, et al. [28], who similarly identified severe halite precipitation and pore-scale blockage under high-salinity conditions in the absence of chemical inhibition.

For the 200 ppm PFC sample (2b), the grain matrices were coated with fine salt crystals. In contrast 500 ppm PFC (3b), severe coating was observed on top of the aggregates (blue arrow), and the disappearance of fracture lines between the grain matrices indicated precipitation of fine salt. Overall, 500 ppm PFC at 30,000 ppm salinity appeared to inhibit nucleation and growth of larger polycrystals, promoting smaller deposits such as monocrystals and fine salts at higher concentrations [23, 29].

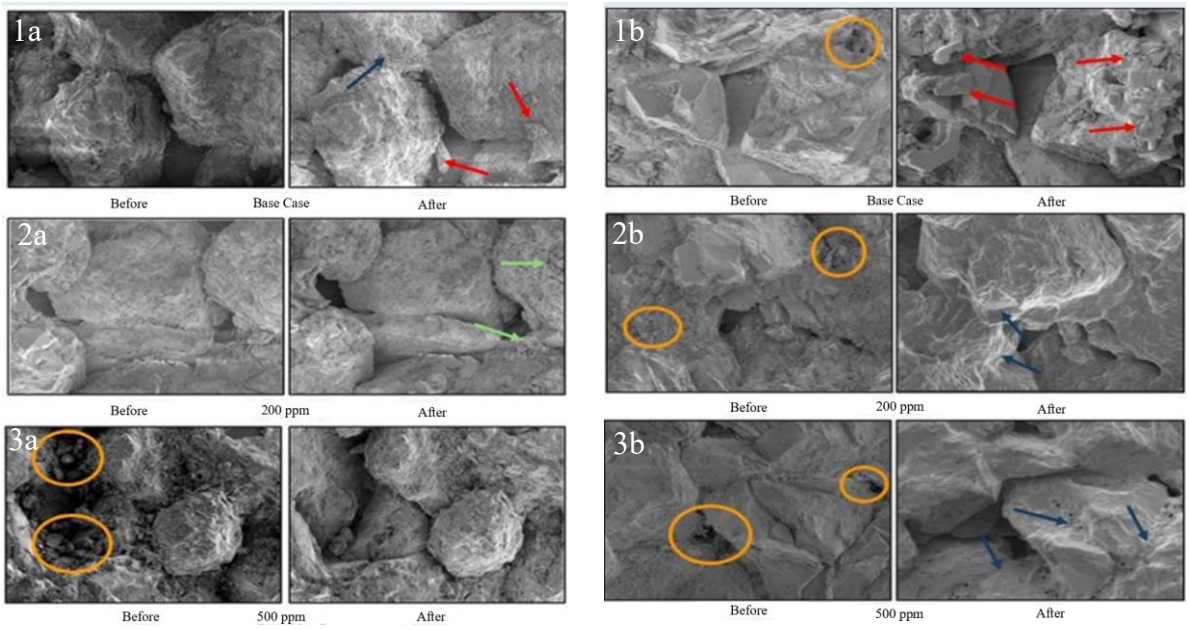

Figure 1. FESEM Imaging- 30,000 ppm (left) and 100,000 ppm (right) od NaCl Brine for (1) Base Case, (2) 200 ppm of PFC, and (3) 500 ppm of PFC.

Table 1 summarizes the EDX-measured atomic percentages of Na and Cl to quantify salt content under different PFC concentrations. At 30,000 ppm salinity, the 500-ppm PFC effectively prevented salt deposition, with Na and Cl levels below 1%, indicating effective suppression of salt precipitation. In contrast, the base case showed the highest total atomic percentage of Na and Cl (7.36%), followed by 200 ppm (2.67%) and 500 ppm (0.92%). It is clearly demonstrated to have a concentration-dependent inhibition effect. This trend aligns with previous reports indicating reduced halite nucleation and crystal retention at higher inhibitor concentration [30] confirming that increasing PFC loading mitigates salt accumulation even at low salinity.

Table 1 Table w 3further presents weight measurements before and after testing. After drying at 60 ˚C for 24 hours, all samples exhibited increased mass due to NaCl deposition. The lowest weight difference of 4.28% was observed at a PFC concentration of 500 ppm at low salinity conditions (30,000 ppm), indicating minimal salt precipitation. These findings are consistent with the static bottle tests of Le [23], where the addition of PFC (50-500 ppm) completely prevented precipitate formation. The percentage weight gain ranked in ascending order as samples 5, 1, 3, 2, 4, and 6, indicate varying degrees of salt deposition.

Table 1. Permeability Change of BR2 and BR3.

Sample	Condition		Total Atomic	Weight Difference (%) $\frac{\text{After (g)} - \text{Before (g)}}{\text{Before (g)}}$
	Concentration (ppm)	Salinity (ppm)	Na + Cl	
1	Base Case	30,000	7.36	5.36
2		100,000	39.67	8.70
3	200	30,000	2.67	8.02
4		100,000	54.03	11.32
5	500	30,000	0.92	4.28
6		100,000	31.83	12.54

Table 2. Permeability Change of BR2 and BR3.

Liquid Permeability (mD)	Base Case (BR3)	Treated Core (BR2)
Initial	137.78	93.02
Final	42.25	87.97
Difference	-69.3%	-5%

Table 2 compares the liquid permeability between BR3 (base case) and BR2 (treated with PFC). A significant permeability impairment was observed, with liquid permeability, BR3 experienced a significant 69.3% decline. This behaviour is consistent with prior core-flooding studies, which reported significant permeability loss due to salt precipitation and pore-scale blockage [28, 31, 32]. In contrast, BR2 only saw a 5% reduction. This demonstrates PFC's success in inhibiting salt crystallization and preventing severe pore blockage which effectively reduces salt precipitation during core flooding, minimizing formation damage and preserving injectivity. However, the observed porosity reduction in BR2 suggests that further research is needed to understand long-term chemical interactions and optimize PFC performance for field applications.

CONCLUSION

This study demonstrated the effectiveness of potassium ferricyanide (PFC) in mitigating salt precipitation during CO_2 injection under both static and dynamic conditions. In static tests, PFC effectively inhibited the formation of large polycrystalline salt crystals, showing high efficiency at 30,000 ppm salinity but reduced performance at 100,000 ppm. FESEM-EDX analyses further confirmed that PFC treatment altered the spatial distribution and accumulation of Na and Cl, indicating inhibition of crystal growth and surface attachment [27].

More importantly, dynamic core flooding experiments further validated its performance of PFC under flow conditions representative of CO_2 storage applications. The 500 ppm PFC treated core exhibited only a 5% permeability reduction compared to 69.3% observed in the base case. The substantial improvement can be attributed to the ability of PFC to hinder salt nucleation and crystal aggregation, thereby preventing pore-throat blockage and preserving effective flow pathways [23, 27]. Overall, the findings highlight PFC as a promising salt precipitation inhibitor effectively preserved permeability and injectivity under CO_2 exposure, emphasizing the importance of dynamic evaluation for realistic CCS conditions and salt mitigation strategies.

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