



# Journal of Applied Sciences

ISSN 1812-5654

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## Scale Formation Due to Water Injection in Berea Sandstone Cores

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**Abstract:** This study was conducted to investigate the permeability reduction caused by deposition of calcium, strontium and barium sulfates in Berea sandstone cores from mixing of injected Malaysian sea waters (Angsi and Barton) and formation water that contained high concentration of calcium, barium and strontium ions at various temperatures (60-90°C) and differential pressures (75-100 psig). The solubility of common oil field scales formed and how their solubilities were affected by changes in salinity and temperatures (40-90°C) were also studied. The morphology and particle size of scaling crystals formed as shown by Scanning Electron Microscopy (SEM) were also presented. The results showed that a large extent of permeability damage caused by calcium, strontium and barium sulfates that deposited on the rock pore surface. The rock permeability decline indicates the influence of the concentration of calcium, barium and strontium ions. At higher temperatures, the deposition of CaSO<sub>4</sub> and SrSO<sub>4</sub> scales increases and the deposition of BaSO<sub>4</sub> scale decreases, since the solubilities of CaSO<sub>4</sub> and SrSO<sub>4</sub> scales decreases and the solubility of BaSO<sub>4</sub> increases with increasing temperature. The deposition of CaSO<sub>4</sub>, SrSO<sub>4</sub> and BaSO<sub>4</sub> scales during flow of injection waters into porous media was shown by Scanning Electron Microscopy (SEM) micrographs.

**Key words:** Scale deposition, scale solubility, scale concentration, temperature, pressure

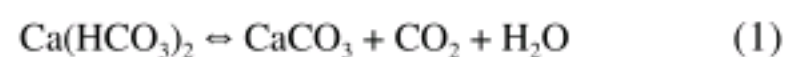
### INTRODUCTION

The injection of seawater into oil field reservoirs to maintain reservoir pressure and improve secondary recovery is a well-established and matured operation. The main objective of water-flooding is to place water into a rock formation at desired rate and pressure with minimal expense and trouble. This objective, however, cannot be achieved unless this water has certain characteristics. The water, therefore, should be treated and conditioned before injection. This treatment should solve problems associated with the individual injection waters, including suspended matter, corrosivity of water scale deposition and microbiological fouling and corrosion. Moreover, the degree of risk posed by deposition of mineral scales to the injection and production wells during such operations has been studied extensively. Scale formation in surface and subsurface oil and gas production equipment has been recognized to be a major operational problem. It has been also recognized as a major cause of formation damage either in injection or producing wells. Scale contributes to equipment wear and corrosion and flow restriction, thus resulting in a decrease in oil and gas production. The most common oil field scales deposited are calcium carbonate, calcium sulfate, strontium sulfate

and barium sulfate. Scale also can deposit when two incompatible waters are mixed and super-saturation is reached (Nassivera and Essel, 1979; Read and Ringen, 1982; Vetter *et al.*, 1982; Todd and Yuan, 1992; Moghadasi *et al.*, 2003b; Moghadasi *et al.*, 2004). In cases where disposal water is mixed with seawater for re-injection, scale deposition is possible (Bayona, 1993; Andersen *et al.*, 2000; Bedrikovitsky *et al.*, 2001; Stalker *et al.*, 2003; Paulo *et al.*, 2001).

There are three principal mechanisms by which scales form in both offshore and onshore oil field system (Mackay, 2005; Jordan and Mackay, 2005):

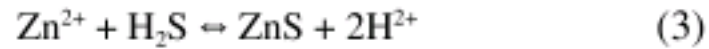
- Decrease in pressure and/or increase in temperature of a brine, goes to a reduction in the solubility of the salt (most commonly these lead to precipitation of carbonate scales, such as CaCO<sub>3</sub>)



- Mixing of two incompatible brines (most commonly formation water rich in cations such as barium, calcium and/or strontium, mixing with sulfate rich seawater, goes to the precipitation of sulfate scales, such as BaSO<sub>4</sub>)



Other fluid incompatibilities include sulfide scale where hydrogen sulfide gas mixes with iron, zinc or lead rich formation waters:



- Brine evaporation, resulting in salt concentration increasing above the solubility limit and goes to salt precipitation (as may occur in HP/HT gas wells where a dry gas stream may mix with a low rate brine stream resulting in dehydration and most commonly the precipitation of NaCl)

Pressure and temperature decrease along the flow string up to the surface in the production well and further changes in thermodynamic conditions occur in the surface equipment. This may again result in scale formation. Normally, these scales do the most damage in the well-bore when there are major falls in pressure but hardly any temperature changes (Khelil *et al.*, 1979).

The objective of this study was to investigate permeability reduction by deposition of common oil field scales in porous media at various temperatures and differential pressures and knowledge of solubility of common oil field scales formed and how their solubilities were affected by changes in salinity and temperature.

### MATERIALS AND METHODS

The experimental apparatus and procedures used in the present study were similar to that used in other investigations (Todd and Yuan, 1990, 1992; Moghadasi *et al.*, 2002; Moghadasi *et al.*, 2003b). The general purpose of the laboratory test was to investigate permeability reduction by deposition of calcium, strontium and barium sulfates in a porous medium and knowledge of solubility of calcium, strontium and barium sulfates and how their solubility are affected by changes in salinity and temperature.

**Core material:** The rock cores used in the testes were Berea cores of 3 inch length and of diameter 1 inch with average porosity of 23.50% and of initial permeability varied from 105.10 to 162.70 md. No oil was present in the cores. All the cores were cleaned using methanol in Soxhlet extractor and dried in a Memmert Universal Oven at 100°C for overnight before use.

**Brines:** The ionic compositions of synthetic formation water and water injection (Angsi and Barton seawater) are

given in Table 1. Note the formation water has calcium, strontium and barium ions and the sea water contains sulfate ions. It is clear that the mixing of these waters can lead to calcium, strontium and barium sulfates precipitation.

Seven salts used for the preparation of synthetic formation water and water injections were given in Table 2. The description of these salts is as follow:

- Sodium Chloride grade (AR) NaCl (M.Wt. = 58.44 g mol<sup>-1</sup>, 99.8% purity) supplied by QReCTM
- Potassium Sulfate K<sub>2</sub>SO<sub>4</sub> (M.Wt. = 174.25 g mol<sup>-1</sup>, 99% purity) supplied by BHD chemicals Ltd Pool England
- Magnesium Chloride MgCl<sub>2</sub>.6H<sub>2</sub>O (M.Wt. = 203.30 g mol<sup>-1</sup>, 98% purity) supplied by R and M Chemicals.
- Calcium Chloride (dihydrate) grade (AR) CaCl<sub>2</sub>.2H<sub>2</sub>O (M.Wt. = 147.02 g mol<sup>-1</sup>, 78% purity) supplied by QReCTM
- Sodium Bicarbonate NaHCO<sub>3</sub> (M.Wt. = 84.01 g mol<sup>-1</sup>, 99.5% purity) supplied by GCE Laboratory Chemicals
- Strontium Chloride (6-hydrate) SrCl<sub>2</sub>.6H<sub>2</sub>O (M.Wt. = 266.62 g mol<sup>-1</sup>, 99% purity) supplied by GCE Laboratory Chemicals
- Barium Chloride (dihydrate) grade (AR) BaCl<sub>2</sub>.2H<sub>2</sub>O (M.Wt. = 244.28 g mol<sup>-1</sup>, 99% purity) supplied by QreCTM

**Scaling test rig:** Experiments were carried out using a test rig, which is schematically shown in Fig. 1. The core test equipment consists of five parts: constant pressure pump, transfer cell, oven, pressure transducer and core holder. There follows a brief description of each part.

Table 1: The ionic compositions of synthetic formation and injection waters

Ionic	High salinity formation water (ppm)	High barium formation water (ppm)	Barton seawater (ppm)	Angsi seawater (ppm)
Sodium	52,132	42,707	9,749	10,804.50
potassium	1,967	1,972	340	375.05
Magnesium	4,260	102	1,060	1,295.25
Calcium	30,000	780	384	429.20
Strontium	1,100	370	5.4	6.577
Barium	10	2,200	<0.2	-
Chloride	146,385	67,713	17,218	19,307.45
Sulfate	108	5	2,960	2,750
Bicarbonate	350	2,140	136	158.80

Table 2: Compounds of synthetic formation and injection waters

Compound	High salinity formation water (ppm)	High barium formation water (ppm)	Average between Barton and Angsi seawater (ppm)
Sodium Chloride	132,000	106,500	26,100
Potassium Sulfate	-	-	5,180
Magnesium Chloride	35,625	853	9,846
Calcium Chloride	110,045	-	-
Sodium Bicarbonate	482	-	-
Strontium Chloride	3,347	-	-
Barium Chloride	-	3,914	-

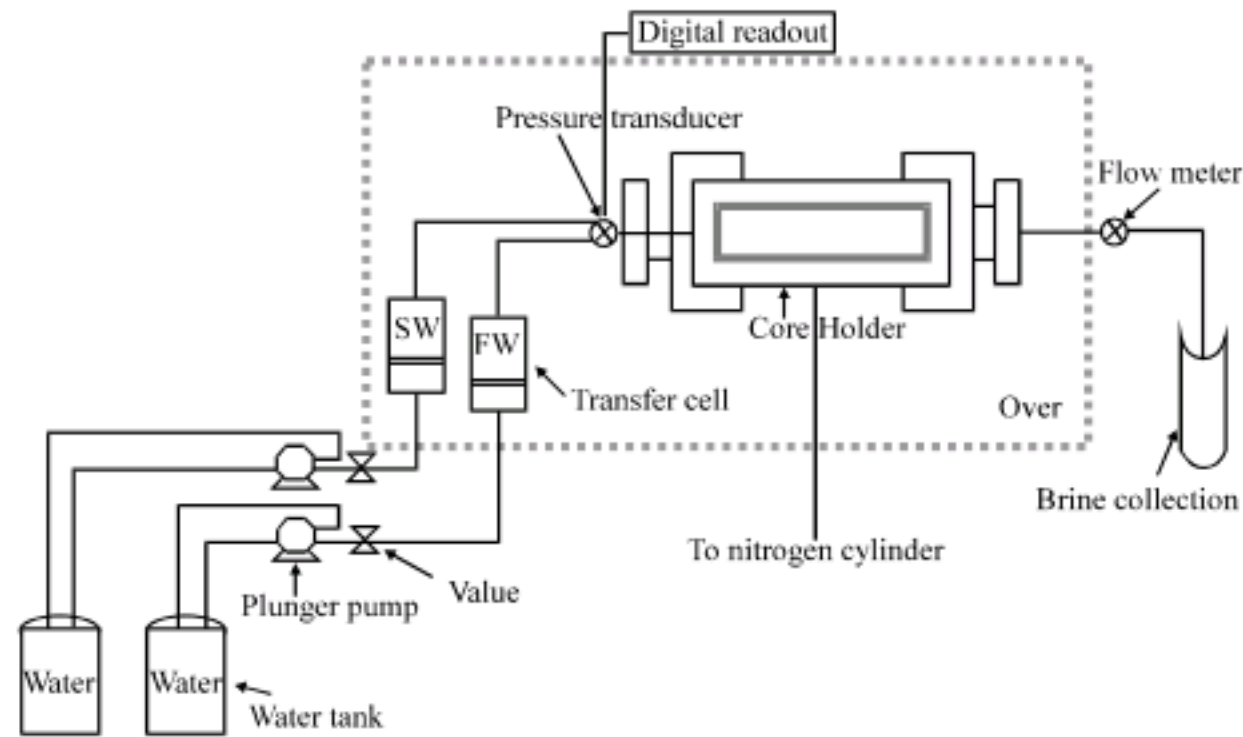


Fig. 1: Schematic diagram of the core flooding apparatus

**Constant pressure pump:** Two Double-piston plunger pumps manufactured by Lushyong Machiney Industry Limited, with 1.5 horse power motor, maximum design pressure of 35 bars and approximate flow rate of  $20 \text{ L min}^{-1}$  are used to inject the brines during flooding at different pressures. These pumps operate on pressure and hence the required pressure for the experiment is of 75 to 100 psig. The required pressure is set on the pump with the help of regulator. On opening the valve, the pump will deliver the set amount of pressure to the experimental rig and the extra fluid will be send back to the tank by the pump.

**Transfer cell:** Two Stainless steel transfer cells manufactured by TEMCO, Inc., USA which can withstand pressures up to 10,000 psig is used to store and pump the injected brine to the core holder. Each cell with a capacity of 1000 mL has a free-floating piston, which separates the pump fluid (distilled water) from the injection brine. The pump fluid is pumped into a transfer cell to displace the brine into the core.

**Oven:** During all flooding runs, the core holder is placed inside a temperature controlled oven.

**Pressure transducer:** The differential pressure across the core during flooding runs was measured by using a pressure transducer (model E-913 033-B29) manufactured by Lushyong Machiney Industry Limited, with a digital display.

**Core holder:** A Hassler type, stainless steel core holder designed for consolidated core samples, 3 inch length and 1 inch diameter, was used. The holder was manufactured by TEMCO, Inc., USA and could withstand pressures up

to 10,000 psig. A rubber sleeved core holder, subjected to an external confining pressure, into which a sandstone core is placed.

**Test procedures**

**Beaker test:** For each experiment of common oil field scales, 100 mL of each filtered opposite waters are heated in the oven at the test temperature and poured simultaneously into a beaker. This solution in beaker is heated on hot plate with continuous stirring by magnetic stirrer for one hour. After this the solution is filtered through  $0.45 \mu\text{m}$  filter paper. After filtration, 5 mL of the filtrate is taken into a 50 mL volumetric flask and is diluted with distilled water to make up 50 mL of solution. This instantaneous dilution is performed in order to prevent  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  precipitation between filtering and analytical determination of the Ca, Ba and Sr concentration. The calcium, barium and strontium determinations are calibrated by measuring five standard solutions. Standard solutions are prepared from  $\text{CaCl}_2$ ,  $\text{BaCl}_2$  and  $\text{SrCl}_2$  solutions. Calcium, barium and strontium concentrations in the diluted filtrates are determined by Atomic Absorption Spectrometry. After multiplying with the dilution factor, the exact concentrations of calcium, barium and strontium are computed.

**Core test**

**Core saturation:** A schematic diagram of core saturation used in this study was shown in Fig. 2. Before each run, the core sample was dried in a Memmert Universal Oven at  $100^\circ\text{C}$  for overnight. The core sample was prepared for installation in the core-holder. A vacuum was drawn on the core sample for 5 h to remove all air from the core. The core was saturated with formation water at room temperature. The formation water is then injected by hand pump into the core-holder to saturate the core until the

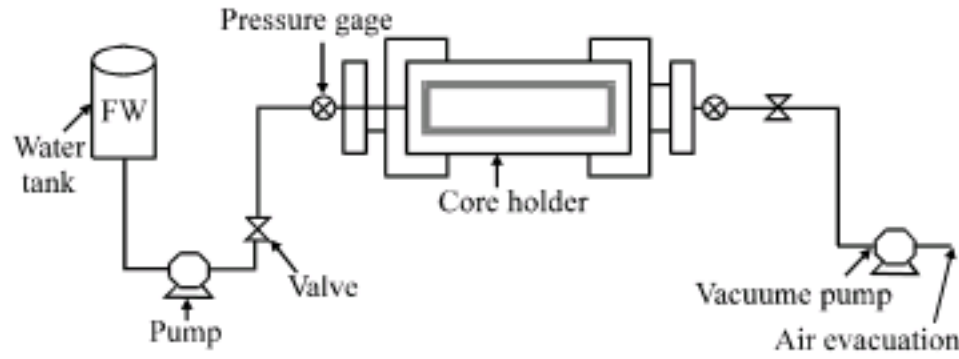


Fig. 2: Schematic diagram of the core saturation apparatus

pressure reach 1400 psig. The system is left overnight to ensure 100% saturation.

**Flooding experiment:** As shown in Fig. 1, the system consisting of the core holder assembly with the saturated core sample and transfer cells containing the two incompatible waters (SW and FW) are placed inside the oven and heated to the desired temperature of the run. The system is left three hours for temperature equilibrium to be attained. The required confining pressure is then adjusted to be approximately at double inlet pressure. A flooding run is started by setting both plunger pumps at the same pressure (ranging from 75 to 100 psig), then turning them on. Thus, the two waters (SW and FW) are always injected into the core sample at a mixing ratio of 50:50. The inlet pressure is measured by pressure transducer while the outlet pressure is atmospheric pressure. During each run, the flow rate across the core is recorded continuously and the permeability of core is calculated using Darcy’s linear-flow equation before and after scale deposition. For selected runs, the core sample is removed at the end of flooding and cut into sections for Scanning Electron Microscopy (SEM).

**RESULTS AND DISCUSSION**

**Beaker test:** The calcium, strontium and barium concentrations in the diluted filtrates were determined by atomic absorption spectrometry. The solubility of CaSO<sub>4</sub>, SrSO<sub>4</sub> and BaSO<sub>4</sub> at various temperatures of this study were calculated. Graphical presentations are given in Fig. 3.

The expected trend in this temperature range is a decrease in CaSO<sub>4</sub> and SrSO<sub>4</sub> solubilities, because the dissociation of CaSO<sub>4</sub> and SrSO<sub>4</sub> is exothermic reaction. But this phenomenal was different for the BaSO<sub>4</sub>. The solubility of BaSO<sub>4</sub> increases with the increase of temperature due to its endothermic reaction.

A graphical presentation of the experimental results is shown in Fig. 3. The sulfate ion content in the sea water brine reacts with the barium ions in the formation water instantaneously but it reacts with both calcium and

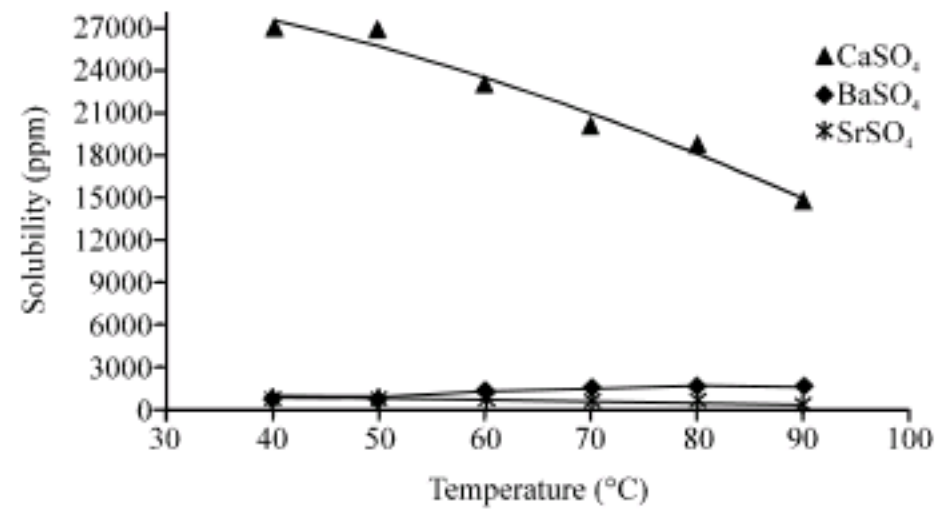


Fig. 3: Solubility of scale formation is dependent on temperature

strontium ions only with heating. The more precipitation of CaSO<sub>4</sub>, SrSO<sub>4</sub> and BaSO<sub>4</sub> results from the presence of a large concentration of calcium, strontium and barium ions. The experimental results confirm the general trend of solubility dependency for common oil field scales on temperature which is obvious and is similar to that observed in the earlier work (Jacques and Bourland, 1983; Lindlof and Stoffer, 1983; Oddo *et al.*, 1991; Moghadasi *et al.*, 2003a; Mackay, 2003; Rousseau *et al.*, 2003; Mackay and Jordan, 2005).

**Core test:** The main objective of this part of the investigation is to study permeability reduction caused by common oil field scales deposition in porous media. The coreflood experiments were designed to investigate the effect of temperature (60-90°C), differential pressure (75-100 psig) and concentrations of calcium, strontium and barium ions on the scaling tendency of brines (Table 1).

During each run, the flow rate across the core was recorded continuously and the permeability of core was calculated using Darcy’s linear flow equation. The flow rate decreased during the experiments only when a super-saturated solution was flowing through the cores. This confirms that the decrease of flow rate is due to precipitation of the calcium, strontium and barium sulfates inside the core with the consequent reduction in its permeability and porosity. In the following, extend of permeability damage, decline trend of permeability ratio and the results for various temperatures, concentrations and differential pressure are discussed.

**Extend of permeability damage:** Extend of permeability loss caused by CaSO<sub>4</sub>, SrSO<sub>4</sub> and BaSO<sub>4</sub> scaling in the rock pores varied in different situations. Figure 4a and 5a show the permeability change of a less damaged core at a differential pressure 75 psig and 60°C (Fig. 4a) and 90°C (Fig. 5a). Figure 4b and 5b show that of a severely

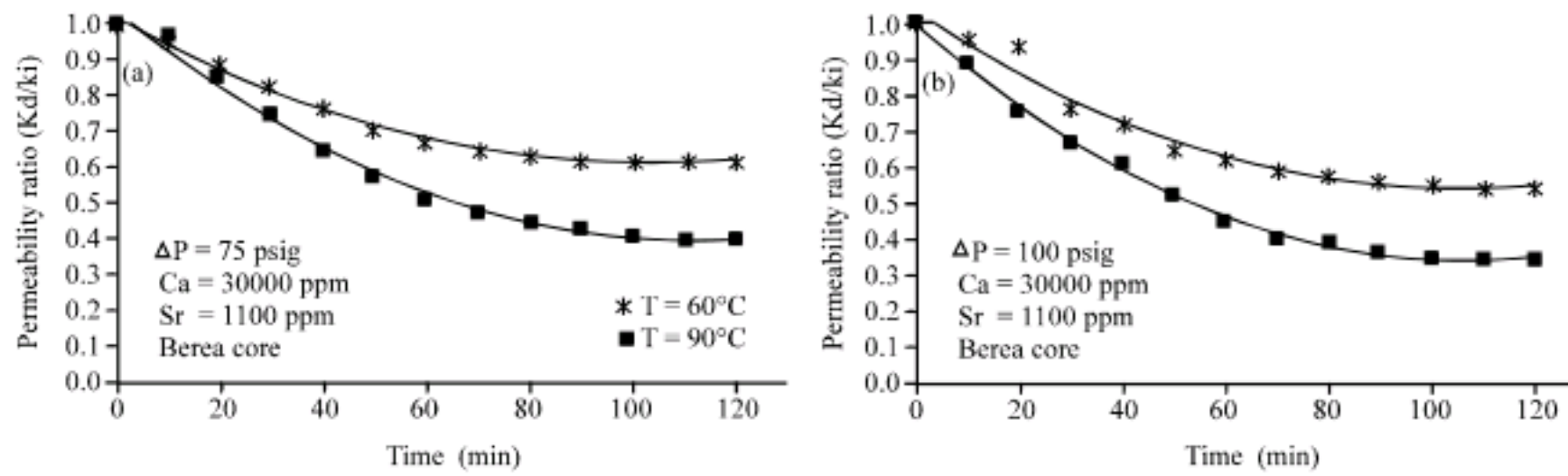


Fig. 4: Variation of permeability ratio as a function of time showing the effect of temperature at (a) 75 and (b) 100 psig

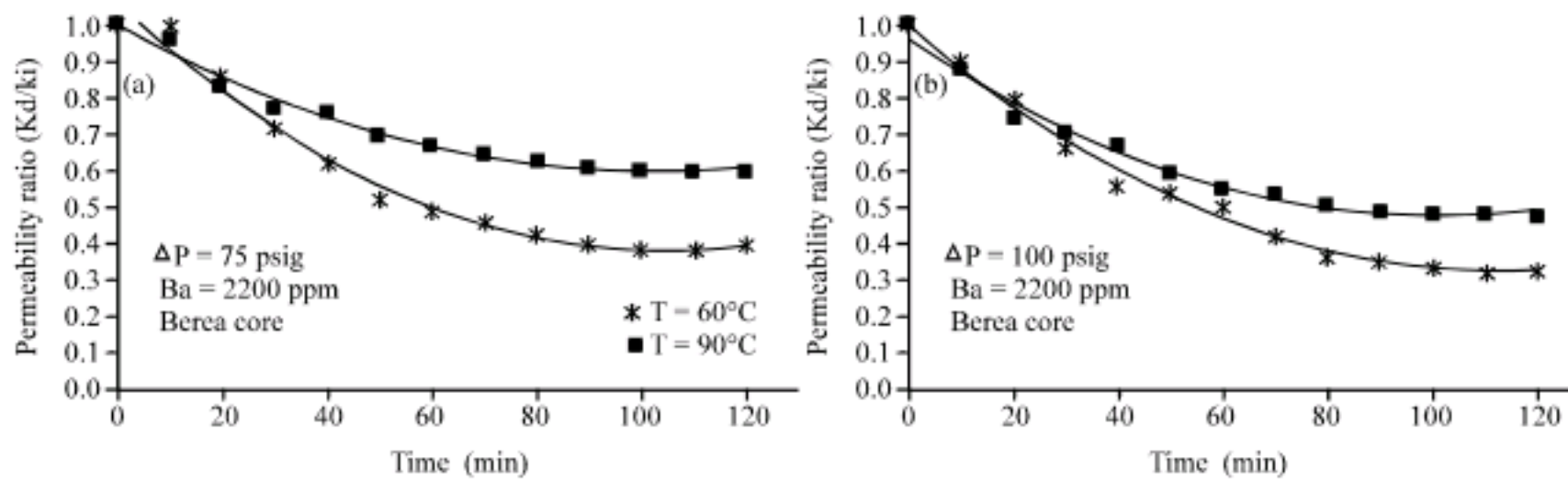


Fig. 5: Variation of permeability ratio as a function of time showing the effect of temperature at (a) 75 and (b) 100 psig

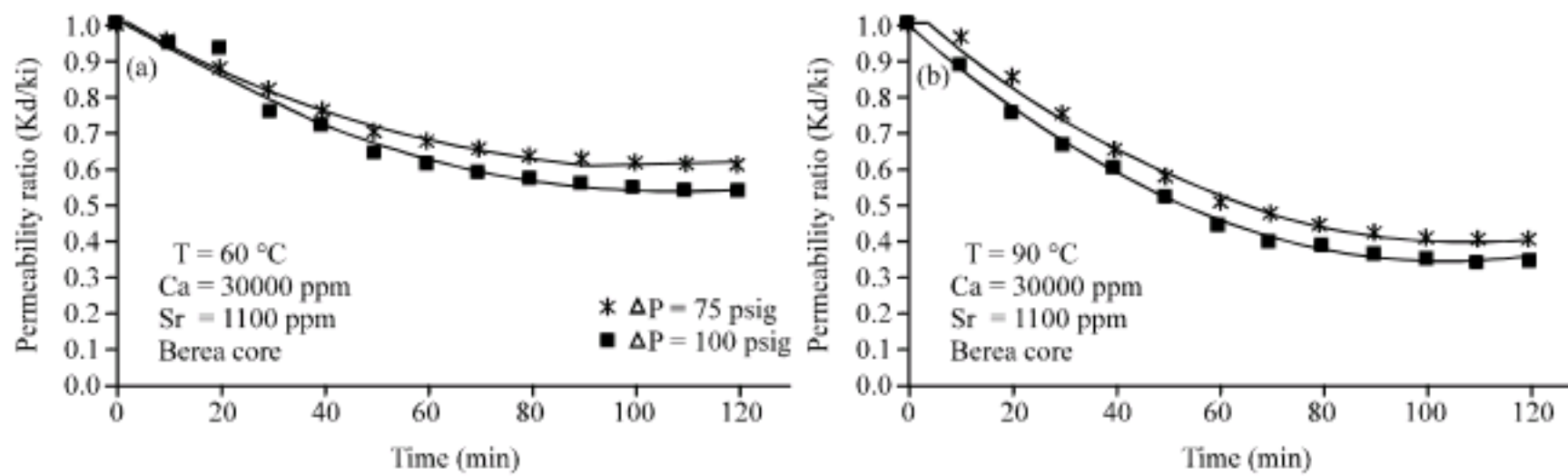


Fig. 6: Variation of permeability ratio as a function of time showing the effect of differential pressure at (a) 60 and (b) 90°C

damaged core after  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  scaling at a differential pressure of 100 psig and 60°C (Fig. 5b) and 90°C (Fig. 4b). About 38.57-65.43% permeability losses were observed in Fig. 4, but 41.15-66.98% initial permeability reduction could occur in a heavily scaled core, as shown in Fig. 5. The reduction in permeability is possibly caused by crystals blocking the pore throats as shown later in the SEM view (Fig. 9, 11). The amount of precipitation varied within the Berea sandstone cores, there being more scale near the formation water inlets and least scale was observed furthest from the inlet parts.

**Decline trend of permeability ratio:** Figure 4-7 show the permeability decline trend changes with brine-injection time. During the initial flow period, the permeability declined sharply soon after the two waters mixed in the pores. The permeability decline then slowed and gradually leveled out after the permeability decreased greatly. This phenomenon was observed in all the core tests in which the scaling damage was severe.

**Effect of temperature:** Temperature has a significant influence on solubility and crystal growth of calcium, strontium and barium sulfates. To study its effect on the

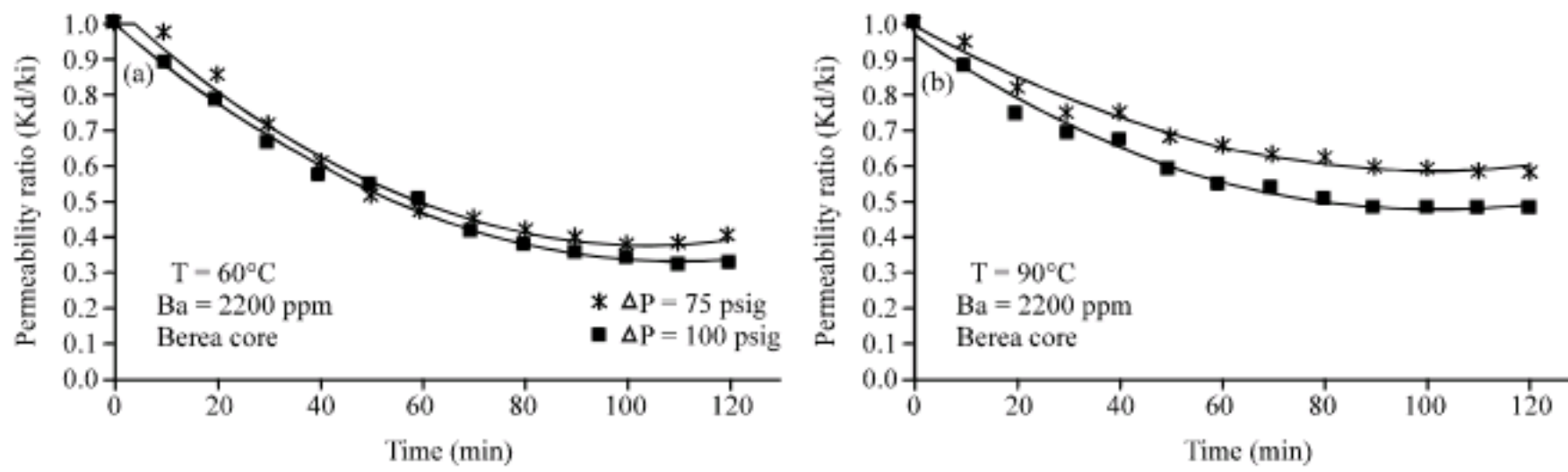


Fig. 7: Variation of permeability ratio as a function of time showing the effect of differential pressure at (a) 60 and (b) 90°C

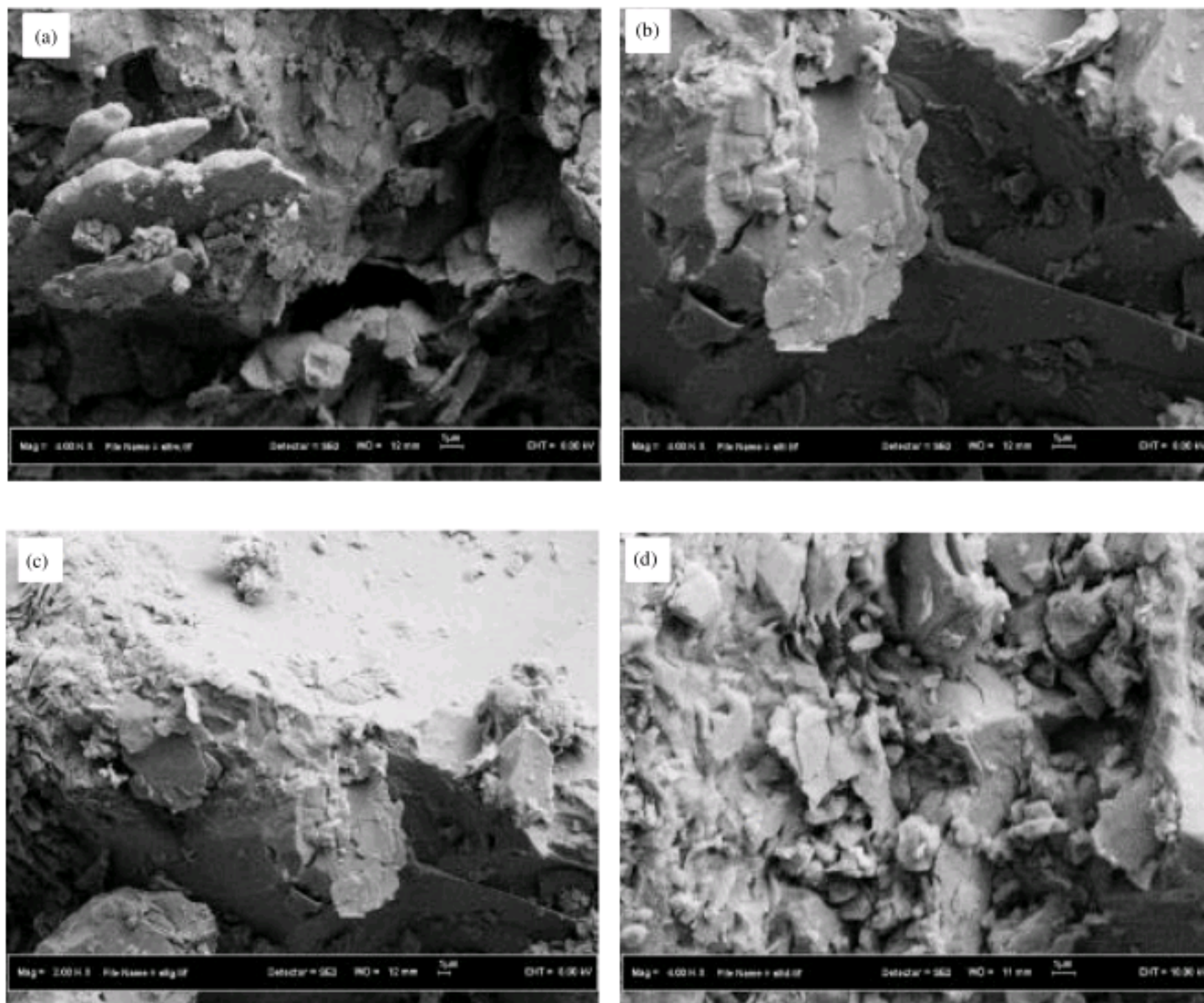


Fig. 8: SEM image of an unscaled Berea sandstone cores

permeability reduction, a number of tests were carried out where concentration of injected brine was kept constant at differential pressure from 75 to 100 psig and temperatures of 60 and 90°C. Figure 4 shows variation of permeability reduction with time at different temperatures. It also shows that at higher temperatures the permeability declines more rapidly. This is because the rate of  $\text{CaSO}_4$  and  $\text{SrSO}_4$  precipitations increases with temperature. The

increase in temperature also causes a raise in supersaturation, because the solubility of  $\text{CaSO}_4$  and  $\text{SrSO}_4$  decrease with temperature. This must have led to an increase of rate of precipitation and consequently a faster permeability decline. Figure 5a and b show the variation of permeability reduction with time at different temperatures. It also shows the effect of temperature on permeability reduction as temperature rises, the rate of

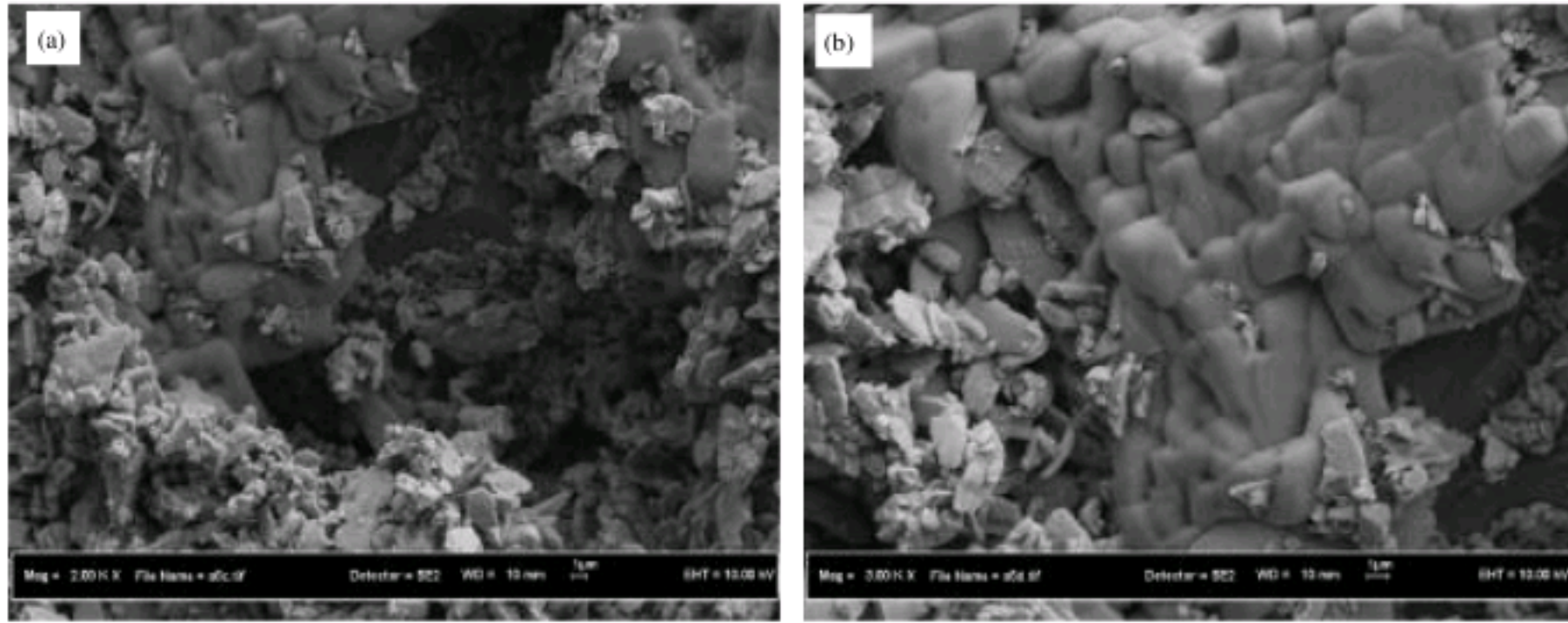


Fig. 9: SEM image of BaSO<sub>4</sub> scale in inlet face of Berea sandstone core at 100 psig and 60°C

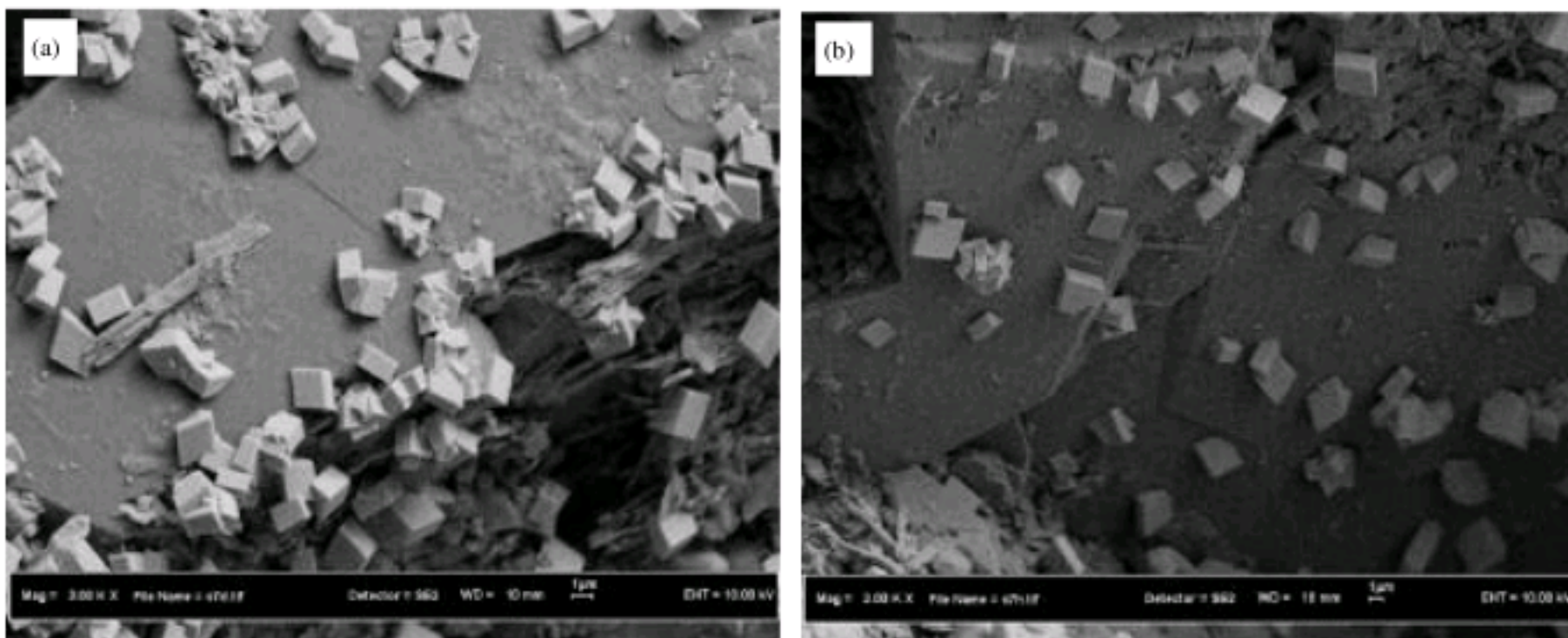


Fig. 10: SEM image of BaSO<sub>4</sub> scale in outlet face of Berea sandstone core at 100 psig and 60°C

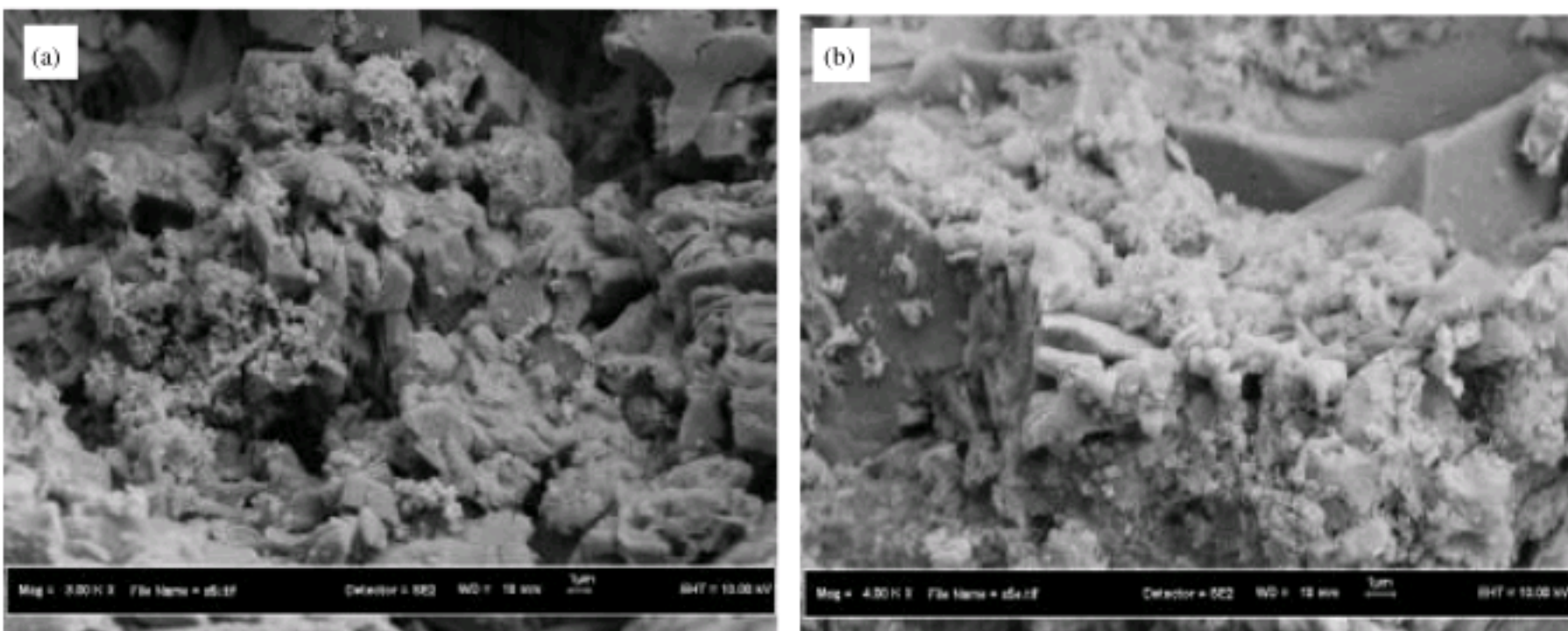


Fig. 11: SEM image of CaSO<sub>4</sub> and SrSO<sub>4</sub> scales in inlet face of Berea sandstone core at 100 psig and 90°C



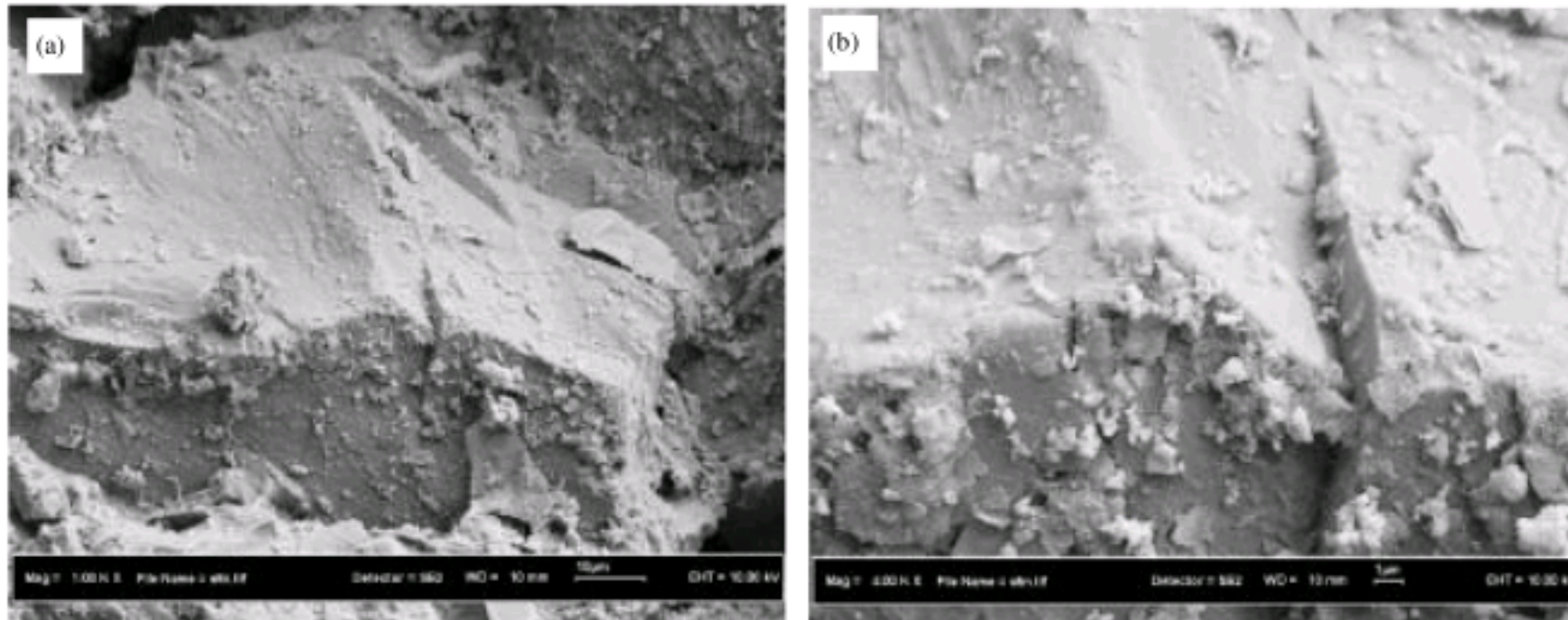


Fig. 12: SEM image of  $\text{CaSO}_4$  and  $\text{SrSO}_4$  scales in outlet face of Berea sandstone core at 100 psig and  $90^\circ\text{C}$

nucleation and crystal growth and plugging were decreased. The permeability decline is less rapid at higher temperature, since the rate of  $\text{BaSO}_4$  precipitation decrease with temperature. This is because the solubility of  $\text{BaSO}_4$  increases with temperature.

**Effect of differential pressure:** To investigate the effect of differential pressure on permeability reduction a number of tests were carried out. In these experiments, the concentration of brine and temperature were kept constant and differential pressure varied from 75 to 100 psig. The variation of permeability reduction with time at different differential pressures is shown in Fig. 6a, b and 7a, b. From these results, the permeability decline of porous medium is evident, even at such low differential pressures. The results illustrate that at low differential pressure, scale formation has already as significant effect on the permeability decline. As, the differential pressure was increased, the rate of permeability decline becomes more rapid. Moreover, at higher differential pressure more sulfate ions will pass through the porous medium in a given interval of time. The super-saturation at the porous medium will therefore increase the rate of precipitation. This increased precipitation rate will produce a larger permeability decline. These results agreed with result by reported by Todd and Yuan (1990, 1992) and Moghadasi *et al.* (2002, 2003b).

**Scanning electron microscopy analysis:** The scaled core samples were examined by SEM to observe the particle size and morphology of the precipitates. The formation of  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  during flow of injection and formation waters in porous media have been observed by Scanning Electron Microscopy (SEM) micrographs which shows  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  crystals formation in porous space. Figure 9-12 show SEM image of the  $\text{CaSO}_4$ ,

$\text{SrSO}_4$  and  $\text{BaSO}_4$  scaling crystals in rock pores precipitated from mixed seawater with formation water inside the cores. Comparison of  $\text{BaSO}_4$  with  $\text{CaSO}_4$  and  $\text{SrSO}_4$  formed in porous media did not show significant differences in crystal external morphology. The differences line in the irregularity of crystals formed in rock pores and the crystal size variations from one location to another in a core. In all core tests, the abundance of scale reduced significantly from the front of the core to the rear indicating that scale formation in porous media was rapid with the observation that the flow rate decreased soon after two incompatible waters were mixed into a core. The observations of scaling sites from previous tests (Todd and Yuan, 1990, 1992) were confirmed by this test results. At the inlet face (Fig. 9a, b, 11a, b), the amount of crystals of  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  is high as compared to outlet face (Fig. 10a, b, 12a, b) indicating more precipitation at the inlet face. The reason the scaling decreased downstream of a core is clear: most of the scaling ions had deposited within the front sections as soon as they were mixed and left few ions to precipitate from the flow stream in the rear sections. Figure 8a-db show an SEM image of an unscaled core samples.

## CONCLUSIONS

- The experimental results confirm the general trend in solubility dependencies for common oil field scales, determined at various temperatures. A temperature rise from  $40$  to  $90^\circ\text{C}$  causes an increase in  $\text{BaSO}_4$  solubility and a decrease of  $\text{CaSO}_4$  and  $\text{SrSO}_4$  solubilities
- Permeability decline caused by  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  scale formation in the porous media ranged from 38.57 to 66.98% of the initial permeability,

depending on brine composition, initial permeability, temperature, differential pressure and brine injection period

- $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  precipitates did not plug the Berea sandstone cores to the same extent. The experiments with  $\text{CaSO}_4$  and  $\text{SrSO}_4$  precipitates produced a 38.57-65.43% reduction in initial permeability. The experiments with  $\text{BaSO}_4$  precipitate produced a 41.15-66.98% reduction in initial permeability
- The pattern of permeability decline in a porous medium due to scaling injection was characterized by a concave curve with a steep initial decline which gradually slowed down to a lower. The initial steepness of these curves generally decreased with increasing distance from the point of mixing of the incompatible brines. The concave shape of the permeability-time curves was common to the majority of the porous medium flow tests
- The formation of  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  during flow of injection and formation waters in porous media have been observed by Scanning Electron Microscopy (SEM) micrographs show  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  crystals formation in porous space. At the inlet face, the amount of crystals of  $\text{CaSO}_4$ ,  $\text{SrSO}_4$  and  $\text{BaSO}_4$  is high as compared to outlet face

#### ACKNOWLEDGMENTS

The authors would like to thank the University Technology Malaysia and Mosti for paid fees to publish this study.

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