

***BaSO₄* Precipitation Impact on the Permeability of a Pressure Porous Medium**

***BaSO₄* Impacto da Precipitação sobre a Permeabilidade de um Meio Poroso de Pressão**

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ABSTRACT

Currently, the petroleum sector is facing new challenges like ultra-profound waters and thick pre salt layers. One of the main concerns is the inorganic scale in production equipment. Scales can occur throughout the chain of petroleum production, from the reservoir until the surface facilities. Its occurrence in the near well region of the reservoir generates a greater impact on the fluids production, where the removal or inhibition of the salt became more complex. The most common scale in the near well region is barium sulfate $BaSO_4$ because its low solubility. This work proposes an experimental study that aims to monitor the effect of $BaSO_4$ precipitation on sandstone porous medium pressure drop using a core holder. Distinctly from others works in the area, this study used a simpler solution to observe the impact of precipitation reaction. The porous medium was saturated with a solution of barium chloride ($BaCl_2$) at 250ppm and afterwards was injected a solution of sodium sulfate (Na_2SO_4) at 2750 ppm. The injection rate was $5 \times 10^{-8} \text{ m}^3/\text{s}$ for two hours under a pressure of 6.985MPa. During the precipitation reaction was observed an increase in the pressure of the system. This increase of pressure is a consequence of permeability reduction occasioned by the deposition of barium sulfate. The effect of precipitation can be observed more significantly at the first twenty minutes of reaction. At the end of experiment was observed a permeability reduction of more than 60% in the porous medium. The properties of the porous medium was measure, however this methodology could not estimate the rate of barium sulfate scale

Keywords: Inorganic scale, Barium sulphate, Permeability, Porosity.

RESUMO

Atualmente, o setor petrolífero está enfrentando novos desafios como águas ultraprofundas e espessas camadas pré-salinas. Uma das principais preocupações é a escala inorgânica nos equipamentos de produção. As escalas podem ocorrer em toda a cadeia de produção de petróleo, desde o reservatório até as instalações de superfície. Sua ocorrência na região próxima ao poço do reservatório gera um impacto maior na produção de fluidos, onde a remoção ou inibição do sal se tornou mais complexa. A escala mais comum na região do poço próximo é o sulfato de bário $BaSO_4$ porque sua baixa solubilidade. Este trabalho propõe um estudo experimental que visa monitorar o efeito da precipitação do $BaSO_4$ sobre a queda de pressão do meio poroso de arenito utilizando um suporte de núcleo. Distintamente de outros trabalhos na área, este estudo utilizou uma

solução mais simples para observar o impacto da reação de precipitação. O meio poroso foi saturado com uma solução de cloreto de bário (BaCl_2) a 250ppm e depois foi injetada uma solução de sulfato de sódio (Na_2SO_4) a 2750 ppm. A taxa de injeção foi de $5 \times 10^{-8} \text{ m}^3/\text{s}$ durante duas horas sob uma pressão de 6,985MPa. Durante a reação de precipitação, foi observado um aumento na pressão do sistema. Este aumento de pressão é uma consequência da redução da permeabilidade ocasionada pela deposição de sulfato de bário. O efeito da precipitação pode ser observado de forma mais significativa nos primeiros vinte minutos de reação. No final da experiência foi observada uma redução da permeabilidade de mais de 60% no meio poroso. As propriedades do meio poroso foram medidas, porém esta metodologia não pôde estimar a taxa de escala de sulfato de bário.

Palavras-chave: Escala inorgânica, Sulfato de bário, Permeabilidade, Porosidade.

1 INTRODUCTION

On the time of the discovery, petroleum accumulations have a primary energy. However, many reservoirs have this primary mechanism inefficient for long term production, as a consequence a large quantity of hydrocarbons stay inside the reservoir after its exhaustion.

Those reservoirs became good candidates for a secondary recovery method for obtain a larger oil production [1]. Water flooding and gas injection are the most common secondary recovery methods. Being the water injection the most used in the world [2]. The injected water in the reservoir has the objective to move the oil out of the porous medium and at the same time occupied the porous maintaining the reservoir pressure.

The water injection can come of four distinct origins: subsurface, aquifer, surface (from rivers or lakes) or seawater. In offshore camps, because the availability, the seawater is more commonly used. In seawater, sulfate is the most common ion present [3]. Inside the reservoir, the seawater will get in contact with the formation water. This formation water refers to the water saturation of the reservoir at the time of discovery. The formation water is commonly rich in ions of calcium, barium and strontium. To exemplified, the usual composition of the formation water and injection water from a Gulf of Mexico camp are shown in Table 1 [4].

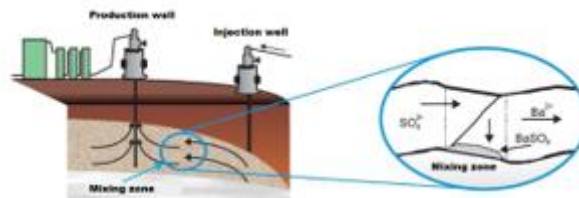
Table 1. Formation and injected water composition

Ion	Formation Water	Injected Water
Na ⁺	13.537	10.556
K ⁺	78,9	380
Mg ²⁺	90	127
Ca ²⁺	277	400
Sr ²⁺	38	13
Ba ²⁺	22	0,02
Fe ²⁺	0,9	0,00
Zn ²⁺	2	0,01
Cl ⁻	20.522	18.980
SO ₄ ²⁻	23	2649
F ⁻	1	1
Alkalinity	84.546	140
Carboxylic Acids	0	0
Total dissolved solids (TDS)	35.439	34.391

In general, when the injection water get inside the reservoir, occurs the following phenomenon [5], as shown in Fig. 1:

- The temperature increases along the curse of injection flow, because the heat exchange between reservoir and formation fluids.
- The pressure drops along the way.
- There are mixing of injection water and the formation water.

Figure 1. Schematic of incompatible waters mixture [6]



The mixing of injection and formation water is inevitable and, as consequence, it will occur inorganic mineral precipitation like barium sulfate (BaSO₄), calcium carbonate (CaCO₃), strontium sulfate (SrSO₄) and calcium sulfate (CaSO₄) [4], [5] e [7].

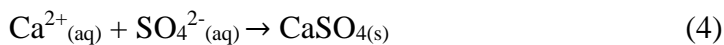
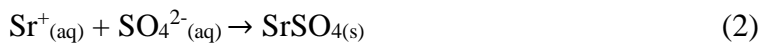
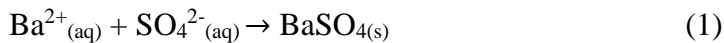
Its occurrence in the near well region of the reservoir creates an impact on the porosity and permeability, especially around the well in the porous medium.

The salt precipitation typically become an inorganic scale on the reservoir. The scales restrict the open area to flow, reducing the production flow rate and productivity of the well. The main problems related to the salt precipitation are porous closing on the formation, perforation packed with gravel and reducing of the lifetime of equipment. The

gravel pack is placed on the annular space, between the formation and the tubing [8] and facilitates the communication between formation and well. Thus, it is important to prevent depositions in this area.

2 BARIUM SULFATE PRECIPITATION

Precipitation is the mass transfer of a solute from a supersaturated solution by the formation of a solid element, for a chemical reaction [9], as shown in Eq. 1 - 4 [10].



Studies like [11], [12], [13] and [14] shows that barium sulfate scale is most dangerous for the flow assurance in the porous medium, being the most insoluble among the common scales, as can be seen in Table 2 [15]:

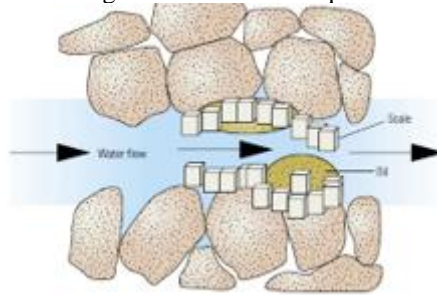
Table 2. Solubility of the most common scales at 25° C

Scale	mg/L
BaSO ₄	2.3
CaCO ₃	53
SrSO ₄	114
CaSO ₄	2000

As it can be formed inside the reservoir, the barium sulfate presents a challenge to be removed or inhibited, causing elevated costs to maintain and workover production flow assurance issues, especially when it occurs in the near well region or completion equipment like gravel packs.

Scales deposits are driven by two mechanisms: deposition and adhesion process. The deposition process refers to nucleation of the solids (or crystals) out of solution and its growth. The adhesion process refers to the sticking of crystals on the surface and the building up a layer [16]. A representation of the adhesion process is depicted in Fig. 2

Figure 2. Representation of inorganic scale adhesion process in the porous medium [6]



In an earlier work [17], it was proposed an experimental study about inhibition of barium sulfate scale. The goal was to investigate the efficiency of three different inhibitors to prevent the formation of the salt, this work show the extent of permeability damage caused by barium sulfate, the influence of high temperature in elevate the solubility of the salt and that at temperature of 60°C the Phosphino Carboxylic Acid (PPCA) showed a very efficient $BaSO_4$ inhibition effect. Using a similar methodology, the present study proposes to reproduce how the scale affects the permeability of the porous medium under high pressure at a bench scale core holder.

3 MATERIALS AND METHODS

3.1 CORE MATERIAL CHARACTERIZATION

The rock core used in the test were sandstone Berea Buff, acquired at Kocurek Industries – Hard Rock Division, with 0,0635m length and 0,0383m in diameter (Fig. 3). The core was oil free and dry. The Table 3 shows the manufactured details for Berea Buff sandstone.

Figure 3. Core sample - sandstone Berea Buff



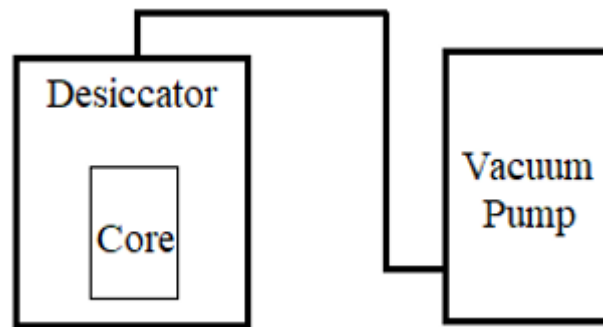
Table 3. Manufactured details for Berea Buff sandstone

Properties	
Formation	Upper Devonian
Permeability	150 - 350 mD KCL 400 - 500 mD N2
Porosity	20 - 22 %
UCS	3800 - 4500 psi
Homogeneous	YES
Perm By	KCL/N2

3.1.1 Porosity Determination

The average porosity of a sandstone Berea Buff is measured as shown in Fig. 4.

Figure 4. Schematic of the average porosity determination apparatus.



Adapting the methodology proposed by [18], it was measured the dry mass of the core. The core was cover with the distilled water and placed inside a glass desiccator connected to a vacuum pump. Next, it was executed six cycles of 20 minutes each at 500 mmHg to achieve complete saturation of the core. At each cycle, the mass of the sample was measured. The end of the process was determined by the same mass measured in the previous cycle. This is called saturated mass. The Eq. 5 is used to determine the average porosity of the core.

$$\phi = \frac{m_{saturated} - m_{dry}}{V \cdot \rho_{fluid}} \quad (5)$$

Where: $m_{saturated}$ is the saturated mass of the sample; m_{dry} is the dry mass of the core; V is the volume of the core; ρ_{fluid} is the density of the fluid used for saturation.

3.1.2 Permeability Determination

It was calculated two permeabilities: absolute permeability and instantaneous permeability. The first one concerns the rock's physical property determined from

Darcy's Law. The second one is a measure of apparent permeability due to scale deposition in porous medium from clean sample permeability.

The absolute permeability was define using a core holder unit. In this procedure was observed the permanent pressure for each flow rate applied and the absolute permeability is calculated by Darcy's Law, Eq. 6:

$$\kappa = \frac{q \cdot \mu \cdot L}{\Delta P \cdot A} \quad (6)$$

where: k , is the absolute permeability; q , is the flow rate; μ , is the fluid viscosity; L , the length of the core; ΔP , the pressure drop and the A , is the cross-section area of the core.

For the definition of the absolute permeability was used distilled water and the pressure was monitored at the flow rates of 5.0001×10^{-08} , 6.6668×10^{-08} , 8.3335×10^{-08} , 1.0000×10^{-07} e 1.16669×10^{-07} m³/s for about nine hours.

To calculate the instantaneous permeability the Darcy's Law (Eq. 6) was applied for each point of pressure measure over time considering the pressure drop as the difference of pressure at each instant to reference pressure.

3.2 BRINE PREPARATION

It was prepared two solutions for test the barite precipitation. The first one at 250 ppm of BaCl₂ (barium chloride) and the second one at 2750 ppm of Na₂SO₄ (sodium sulfata). The solution of Na₂SO₄ was put on the transfer cell on the experimental unit, for experimental procedure.

The porous medium was saturated with the BaCl₂ solution (using the same procedure of saturation described before for average porosity definition – section 3.1.1). After the saturation process is completed, the porous medium was placed on the core holder unit.

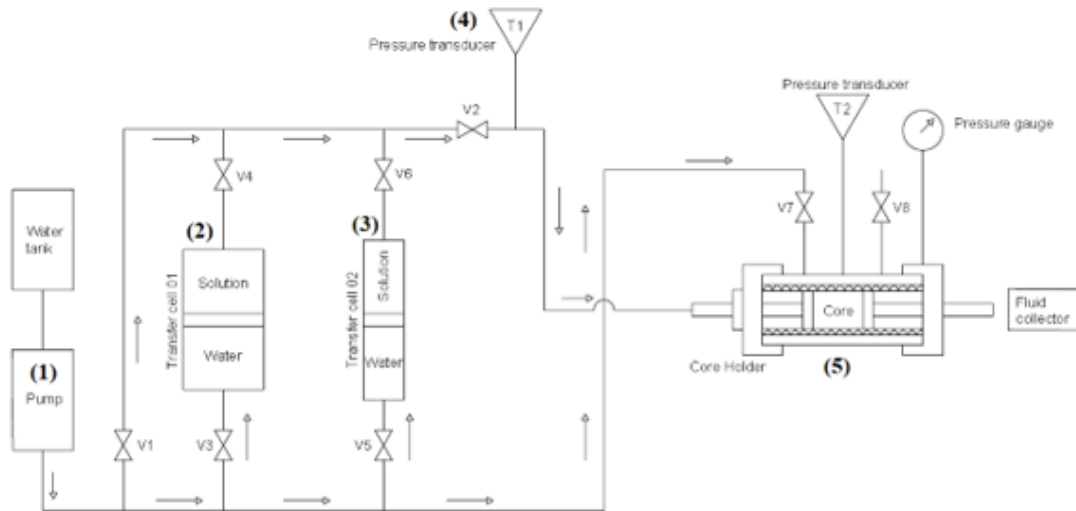
The experiment was conducted at 25°C temperature and at 6.895 MPa confinement pressure. The precipitation reaction under high pressure were monitored for almost two hours.

3.3 EXPERIMENTAL PRECIPITATION TEST

The precipitations test is divided into three steps: confinement pressure achievement; absolute permeability determination; effect of precipitation reaction inside the porous medium.

Firstly, the details of the equipment and experimental setup are presented with the help of Fig. 5. The core holder unit consists of five main parts: (1) High pressure pump; (2, 3) Transfer cell; (4) Pressure transducer and (5) Core holder.

Figure 5. Experimental unit



The experimental unit was manufactured by Lab Conte, the high-pressure pump model is Waters 515 HPLC Pump. The pressure transducer is the Yokogawa, model EJA 110a. The high-pressure pump (1) function is to pump distilled water into the system through the line until the transfer cell (3). The transfer cell holds the solution that will be carried through the line until the core holder. The pressure transducer (4) register the pressure during the flooding injection on the core holder entry. The core holder (5) simulate the confinement pressure for the saturated core using a rubber jacket around the sample.

The valves V1, V2, V3, V4, V5, V6, V7 e V8 correspond to needles valves, that are open or closed depending on which circuit will flow.

The details of the core holder can be seen in Fig 6.

Figure 6. Core holder

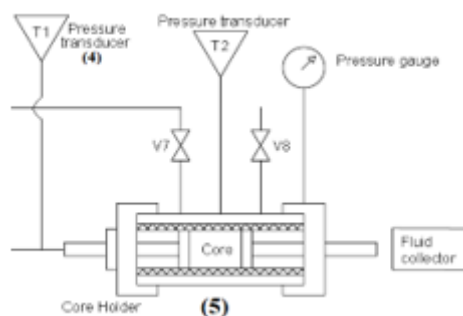


Fig. 6 presents a scheme of a stainless-steel core holder designed for consolidated core samples with 0.0635 m in length and 0.0383m in diameter. The holder could withstand pressures up to 15.858 MPa. A rubber jacket core holder is subjected to an external confining pressure, into which the sandstone core is placed.

The first step is to achieve the confinement pressure around the sample. For this step, only the valve V7 is open. The starts operating and systems rises its pressure until the operation pressure of 6.895 MPa. This first step takes around two hours to assemble the equipment until achieve the confinement pressure.

The second step is the absolute permeability determination. This procedure is done in two parts, one is to clean the porous media and the other permeability determination. The cleaning procedure initiate after the system is pressurized by closing valve V7 and opening the valves V1 and V2 to start the flow. Its circulated distilled water for about one hour at flow rate of $1,67 \times 10^{-8} \text{ m}^3/\text{s}$ and $3,34 \times 10^{-8} \text{ m}^3/\text{s}$ each. This procedure has the intent to make sure that unconsolidated grains are carried out from the core. The equipment is then disassembled and assemble again for measure of permeability. The first step is repeated to initiate the second part that is to determine absolute permeability, valve V7 is again maintain closed and the valves V1 and V2 are open. The pump operates at minimum flow rate of $5 \times 10^{-8} \text{ m}^3/\text{s}$ the data are register for this rate until pressure stabilizes. Then, the pump is set to next flow rate reported in section 3.1.2. This procedure continues up to the maximum flow rate. The averaged time for each flow rate was around one and a half hour each. The total time for the cleaning (first part) was around four hours and to absolute permeability determination (second part), was around nine hours. The total time of execution of the second step takes about thirteen hours. At the end, the system pressure is relief, by opening valve V8, to exchange the sample saturated with water. The sample is taken to a kiln, manufactured by Lucadema, for 24 hours at 105°C , after this period she achieves standard mass then she is saturated with barium chloride following the same procedure described in section 3.1.1 for porosity determination.

The third step is the observation of the precipitation reaction. It starts with the barium chloride saturated sample being inserted in the core holder and the placement of the supersaturate solution of sodium sulfate into the transfer cell 02 (3 – in Fig. 5). The same procedure of the first step is employed to achieve operation pressure of 6.985 MPa. Then the valves V5, V6 and V2 are open as the others are maintain closed. The solution of sodium sulfate in the transfer cell 2 is pushed into the sample core saturated with

barium chloride. The pump flow rate is set to $5 \times 10^{-8} \text{ m}^3/\text{s}$ and the pressure was monitored with time for two hours

To finish the experiment the valve V8 is open to relief the pressure and disassemble the holder

It was observed that the flow through the system take about fifteen seconds to achieve the inlet of the porous medium and it takes around twenty five minutes to be sensed for all the porous medium. After the precipitation procedure, the core sample is put on a kiln at 105°C for 24 hours until achieve standard mass. Then the average porosity and the absolute permeability are measure again.

4 RESULTS AND DISCUSSION

Porosity

The average parameters of the sample are listed in Table 4. They were used to estimated experimentally the porosity of the porous medium.

Table 4. Average parameters of the sample Berea Buff

Diameter [m]	Length [m]	Dry mass [kg]	Saturated mass [kg]
0.0383	0.0635	0.1484876	0.1623930

The fluid used to saturate the medium porous was distilled water of specific mass of $1000 \text{ kg}/\text{m}^3$.

The calculated porosity was 19%. It was close to the value reported by the Kocurek Industries – Hard Rock Division that was reported between 20% to 22% (Table 3). This reduction on porosity can be explain by the different methods used to porosity determination and in this case for the presence of the unconsolidated grains inside the porous medium. In the next step – permeability determination - it was observed that a little amount of thin grains left the porous medium, and the presence of this solids during the saturation procedure can affect the porosity. It is noteworthy that the solids where too thin and we were not able to estimate its amount at that moment.

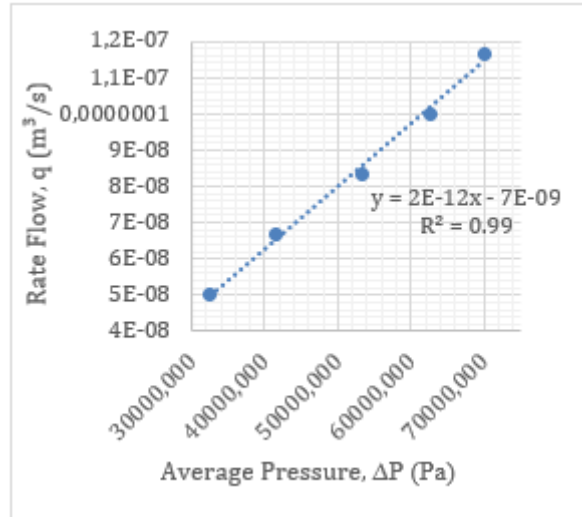
Permeability

The fluid used to estimate the permeability of the core sample was distilled water, and the results are shown in Table 5 and Figure 7.

Table 5. Medium pressure measure

Rate Flow (m ³ /s)	Average Pressure (Pa)
5.0001E-08	32558.078
6.6668E-08	41609.616
8.3335E-08	53377.596
1.00002E-07	62664.494
1.16669E-07	70029.288

Figure 7. Flow versus pressure curve



The Eq. 6 was used to calculate the permeability. The estimated permeability at distilled water was 88.92 mD.

The value obtained for permeability was shorter than the presented by Kocurek Industries – Hard Rock Division that was 150-350 mD (Table 3). However, we stress that it was used a different fluid for the permeability determination, and the method used for determination of permeability was different.

The Table 6 shown the physical properties of Berea Buff sandstone used in this study.

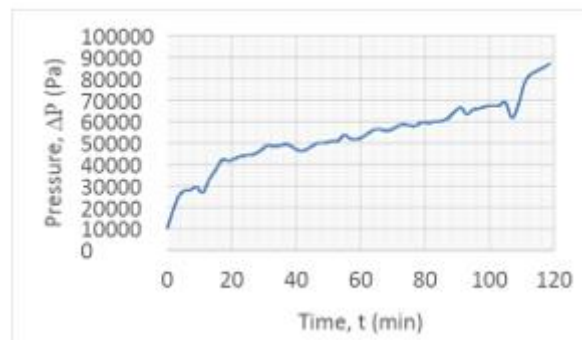
Table 6. Properties of the sample

Sample	Sandstone Berea Buff
Dry mass (kg)	0.1484876
Length (m)	0.0635
Diameter (m)	0.0383
Average porosity (%)	19
Absolute permeability (mD)	88.92

Barium Scale Pressure Test

The dynamics of pressure observed in this experiment is plotted in Fig. 8.

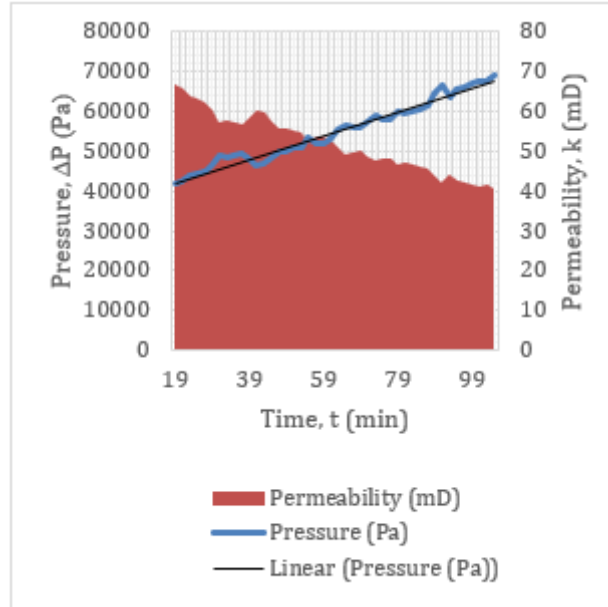
Figure 8. Instantaneous pressure versus time



As mentioned in section 3.3, the system takes twenty five minutes to produce the injection fluid, through the outlet of the unit. And the injection fluid takes about fifteen seconds to achieve the inlet of the core. Knowing that the barium sulfate has a very low solubility and that the solutions used react as soon as they get in contact with which other, we can see an elevated growth rate in the pressure during the first fifteen minutes of experiment. It observed that during this initial time of experiment the precipitation occur more intensely, increasing the mass quantities inside the porous medium. As the flow continues, the solids deposits are carried through the sample. The presence of the barium sulfate difficult the flow, it results in a pressure increase. With the progress of the injection, the sodium sulfate solution keep consuming the barium present in the core, elevating the quantities of deposits. The experiment was kept for two hours. This time was necessary for the all limiting reagent to be consumed. Considering the time of experiment and the flow rate used, was not sufficient for the deposits to adhere and form the scale in our sample.

However, if we idealize a scenario where after those fifteen minutes occur deposition and initial scale formation, we can estimate the instantaneous permeability, using Darcy's Law, for which point about one hour after those fifteen initial minutes. The result of the variation of permeability with the pressure is shown in Fig. 9.

Figure 9. Instantaneous permeability drop and instantaneous pressure with time



The Table 7 presents the instantaneous permeability and the instantaneous injection pressure at the times: 0, 19, 105 and 119 minutes of experiment.

Table 7. Instantaneous permeability and average pressure over the time of experiment

Time (min)	Average Pressure (Pa)	Instantaneous Permeability (mD)
0	0	88.92
19	41727	66.92
105	68843	40.56
119	87181	32.03

We can see that the precipitation reaction occurs immediately at the contact of the injection solution with the solution contained in the porous medium. Also, can be noted that at 19 minutes the instantaneous permeability reduces almost 25% compared to the original permeability and at 105 minutes of the procedure the instantaneous reduction was 55% from the original.

This instantaneous permeability reduction was observed with the increase of the pressure during the test. This phenomenon occurs as a result the reduction of the space for the fluid displacement, as a consequence the precipitation of the barium sulfate in the porous medium. From the start of the precipitation reaction until the end (at 119 minutes) the porous medium instantaneous permeability, reduces in more than 60% from the original.

That way we can see that barium sulfate creates significant damage on the porous medium permeability.

5 CONCLUSION

The impact of the precipitation of barium sulfate in a porous medium confined under high pressure, 6.895MPa at 25°C, has been investigated. The following conclusions are obtained from the study:

- The barium sulfate precipitation was observed meaningfully at the first 19 minutes of the experiment. However, the rate of scale or salt deposition could not be estimated by the present work.
- After the 19 min the precipitation continue until the limiting reagent is exhausted. This is noted by the increase of the pressure on the injection line. The reason for that is the injection fluid encounters restrictions to pass through the porous medium. As the deposition of barium sulfate proceeds it reduces the porous volume of the sample.
- The instantaneous damage on the permeability due the deposition of sodium sulfate is significant: more than 60% of reduction in approximately 2 hours. This implicates on the need to implement control measures to remove or inhibit the formation of barium sulfate scales.

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