Journal of Petroleum Science and Engineering 144 (2016) 113–120

Contents lists available at ScienceDirect

Journal of Petroleum Science and Engineering

journal homepage: www.elsevier.com/locate/petrol

# Analysis of subterranean Pre-salt carbonate reservoir by X-ray computed microtomography

A.P. Teles [a], A.C. Machado [a], A. Pepin [c], N. Bize-Forest [b], R.T. Lopes [a], I. Lima [a,*]

[a] Nuclear Engineering Program, Federal University of Rio de Janeiro, Rio de Janeiro, P.O. Box 68.509, 21941-972, Brazil
[b] Schlumberger BRGC, Rio de Janeiro, Brazil
[c] Naurex, Rio de Janeiro, Brazil

ARTICLE INFO

*Article history:*
Received 5 March 2015
Received in revised form
4 August 2015
Accepted 14 March 2016
Available online 16 March 2016

*Keywords:*
X-ray
Microfocus
Tomography
Carbonate
Pre-Salt
Porosity

ABSTRACT

The purpose of this study is to evaluate the pore space evolution of a rock sample from the coquina region of the subterranean Pre-Salt carbonate reservoir formation (later referred simply as coquina sample) subjected to hydrochloric acid flow through the whole sample. For that purpose, X-ray computed tomography with a microfocus source was used in order to evaluate the porous media space. The results show that the branches of the wormhole divert away from the areas near the main channel and the quartz volume decreases after acidification. From the porous space 3D model it could be verified that, although the acid reacts with the most porous part of the medium, the wormhole also crosses regions of lower porosity. Such a process leads to an increase in the total porosity of the carbonate rock.


## 1. Introduction

X-ray computed tomography (XCT) was originally developed for applications in the medical field. However, this technique was quickly incorporated into other non-destructive areas, such as geology (Deng et al., 2015). The great XCT contribution in this area is to provide volumetric assessment for entire rock microstructures, which leads to a better understanding of the transport and reaction of fluids in 3D porous media.

The main principle of XCT with a microfocus radiation source (μXCT) is the same as for XCT, which consists in obtaining image reconstructions through an object based on the attenuation of X-rays, following Beer's law (Webb, 1988). The photoelectric effect is a dominant attenuation mechanism at low X-ray energies, up to approximately 50–100 keV, while Compton scattering is dominant at higher energies, up to 5–10 MeV. In general, for geological samples, these two processes need to be considered unless higher energy sources are applied. In this sense, μXCT images are presented in a gray scale where low attenuation corresponds to darker gray tones.

In the oilfield industry, acid stimulation is the second oldest subterranean reservoir production enhancement process, preceded only by nitro-shooting (Williams et al., 1979). For subterranean carbonate reservoirs, hydrochloric acid (HCl) has the highest dissolution capacity compared to other acidizing systems such as organic acids or chelating agents. Additionally, it is easily available and economically feasible (Bradley, 1987). The acidification procedure in carbonate reservoirs consists in bypassing the immediate wellbore damage resulting from drilling or production and connecting with the reservoir through high conductivity channels. A large body of previous research led to the adoption of a model of porosity enhancement after acid flow based on a pattern consisting of a channel starting from the point of contact between the acid and the wellbore face and evolving according to a law of least resistance, following the regions of highest permeability of the rock standing before the flow of fresh acid. The resulting channel is known as a wormhole, as a reference to the stochastic nature of its evolution through the reservoir. The size and pattern of the wormhole also depend on factors like acid concentration and pumping rate. Previous studies defined four types of wormholes: conical, dominant, ramified and face dissolution. Among these, dominant wormholes are found to be the most efficient, as they need the least volume of pumped acid for their formation, therefore reaching farther within the reservoir and resulting in the best porosity/permeability increase (Fredd and Miller, 2000; Gouze and Luquot, 2011). Wormhole formation remains however partially unexplained, due to the interplay between transport and

*Abbreviations:* XCT, X-ray computed tomography; μXCT, X-ray computed tomography with a microfocus radiation source; VES, Viscoelastic surfactant; FRT, Formation Response Tester; PVBT, Pore volume to break through; ROI, Region of interest; VOI, Volume of interest

* Corresponding author.
*E-mail address:* inaya@lin.ufrj.br (I. Lima).

http://dx.doi.org/10.1016/j.petrol.2016.03.008
0920-4105/

reaction at the pore scale and the stochastic nature of wormhole development (Fredd and Fogler, 1998).

The acid propagates wormholes through heterogeneous carbonates much more rapidly than in homogeneous rocks. This observation suggests that local pressure drops created by vugs are more dominant in establishing the wormhole flow path than chemical reactions occurring at the smaller pore level. This demonstrates that the total injection volume to breakthrough is affected by spatial distribution, and the amount and connectivity of vuggy pore space (Izgec et al., 2010).

In this study, acid was flowed through the coquina sample at controlled temperature, pressure and flow rate in order to investigate the effect of HCl on porosity evolution and the distribution of the quartz volume.

## 2. Materials and methods

### 2.1. Sampling

The rock sample used in this study was collected at a quarry in North East Brazil, belonging to the “morro do chave” formation, an Albian Pre-Salt carbonate formation mainly consisting of coquina sedimentary rocks (Azambuja Filho et al., 1998). This quarry is the only Pre-Salt outcrop analog officially recognized in Brazil (position: 9° 45′ 33.93″ S – 36° 9′ 17.67″ W). The coquina formation belongs to the Pre-Salt reservoir at the bottom of the stratigraphic organization.

The samples were taken from massive blocks which allowed the drilling of several core plugs corresponding to dimensions fitting the Formation Response Tester (see Section 2.2 for explanations). The blocks were divided into halves with a reduced height of 10 cm for drilling purposes. Cutting was conducted with a buzz saw with a diamond-tipped blade. Further coring was performed using a diamond coring drill bit with a 2.54 cm inner diameter and a fine grained diamond distribution for soft rocks.

The resulting coquina sample had the following dimensions: $25.4 \pm 0.2$ mm diameter, $87.9 \pm 0.2$ mm height and $154135.03 \pm 0.02$ mm$^3$ volume.

### 2.2. Acidizing procedure

HCl acid reacts easily with carbonates according to Eqs. (2.1) and (2.2) (Economides and Nolte, 1989; Williams et al., 1979).

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O \quad (2.1)$$

$$CaMgCO_3 + 4HCl \rightarrow CaCl_2 + MgCl_2 + 2CO_2 + 2H_2O \quad (2.2)$$

The reaction products, such as $CaCl_2$, are compatible with most reservoir fluids and are more soluble than reaction products from organic acids such as Ca-acetate or Ca-formate.

The acid system used for the fluid flow experiments is a viscoelastic surfactant acid (known as VES Acid (Al-Mutawa et al., 2005)). Ever since its introduction in the market (Samuel et al., 1997), the non-damaging viscoelastic diverter (Chang et al., 2001) and its reactive version, VES Acid, have proven to offer better diversion and cause less damage when compared to conventional gelled acid, due to their non-polymeric nature. Unlike foam, VES Acid is continuously pumped as a single fluid for stimulation and diversion, limiting the need for multiple steps. The detailed diversion mechanism of VES Acid is described in the literature and is primarily based on the salt-induced phase transformation of vesicular micelles into large worm-like micelles from 10 nm to 100 nm (Fig. 1), while the elasticity and viscosity of the system are related to the degree of micellar entanglement (Chang et al., 2001; Lungwitz et al., 2007). The salt (i.e.: $CaCl_2$, $MgCl_2$) which causes the transition into wormlike micelles is produced by the reaction of HCl with limestone and/or dolomite according to Eq. (1). The resulting viscosified acid creates higher flow resistance and the subsequent acid is diverted into lower permeability zones (Al-Mutawa et al., 2005; Lungwitz et al., 2004). This acidification system has been used in most carbonate environments for the past 10 years. In Brazil, records of pumped VES Acid can already be found (Lungwitz et al., 2004; Villanueva et al., 2011), but only in the Post-Salt region. As this technology has only recently been introduced for Pre-Salt carbonates, no records of its application in this region have been published so far.

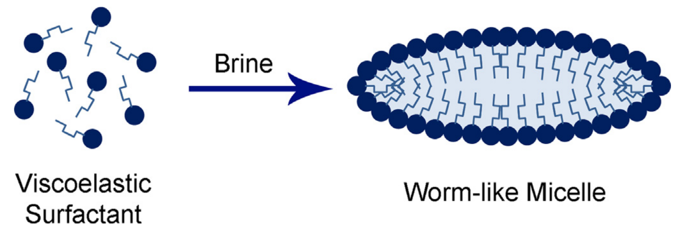


**Fig. 1.** Addition of salt to a viscoelastic surfactant producing a worm-like micelle.

The equipment used for flow measurement is called Formation Response Tester (FRT). The FRT can hold cores with a diameter of either 2.54 cm or 3.81 cm and up to 30.48 cm long. Experiments can be simulated at desired pressure and temperature, up to 34.47 MPa and 149 °C. The experimental procedure for acid flow is outlined in the steps below. The core-flow apparatus used is similar to the one shown in the schematic representations of the equipment in Figs. 2 and 3.

1. Use 3.81 cm diameter coquina outcrop core.
2. Dry core overnight in an oven at 60 °C.
3. Take core out of the oven, measure dry weight.
4. Saturate core in brine (5 wt% $NH_4Cl$).
5. Measure wet weight. The weight difference between dry and wet rock is the volume of brine that fills the rock's pores. Pore volume is calculated by dividing this weight difference by the density of the brine used.
6. Place saturated core in a Hassler-type core holder under a confining (overburden) pressure of 13.79 MPa and back pressure of 7.58 MPa. Core holder temperature is ramped up to the operating temperature of 45 °C.
7. Fluids are pumped at constant flow rates through the cores using a Quizix pump. For our experiments, we tried four rates: 16.7 nm$^3$/s, 50.0 nm$^3$/s, 83.3 nm$^3$/s and 167.0 nm$^3$/s.
8. Low range and high range Rosemount differential pressure transducers are used to measure the pressure drop across the length of the core.
9. Initial, stable permeability measured with 5 wt% $NH_4Cl$ at the equivalent flow rate in production direction until a stable permeability is obtained.
10. Inject Viscoelastic Self-diverting Acid in injection direction until rapid decrease in differential pressure attributable to wormhole breakthrough is observed. Record the number of pore-volumes of acid necessary to breakthrough the core as PVBT (pore volume to break through).
11. Flush the core with 5 wt% $NH_4Cl$ to remove the Viscoelastic Self-diverting Acid from the core.
12. Measure final, stable permeability with 5 wt% $NH_4Cl$ at the equivalent flow rate in production direction until a stable permeability is obtained.