# POLITECNICO DI TORINO

Department of Environment, Land and Infrastructure Engineering.

Final thesis to obtain the Master of Science degree in Petroleum Engineering.

Core flooding tests over porous rocks under different

Stress and temperature conditions



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#### Abstract

Core flooding tests have a great importance to understand the behavior of reservoir rocks and consequently the results are being used for maximizing of reservoir productivity. Optimizing of the reservoir productivity has paramount importance for the company since it directly related to economic benefit. Laboratory tests are being applied on rock samples which mimicking fluid rock interaction in the reservoir. Initial step is preparation of rock plugs which are usually obtained from core samples or the synthetic rocks. ISRM recommended coring, drying, saturation steps are respectively applied on each sample to determine porosities.

Two main aspects were on the focus within the scope of the work. Firstly, 3 identical carbonate rock samples are used, and they subjected into 4 different temperature and 3 different confinement pressure steps for the assessment of dynamic behavior. Absolute permeabilities are calculated for each of the pressure and temperature regimes and plotted in the same graph to understand effect of temperature and pressure. Moreover, permeability reduction factor (PRF) also calculated and plotted for a better understanding of pressure and temperature effect on the given rock samples.

Second aspect is to determine hydrocarbon in place and recovery factor for other 3 identical samples. Each sample initially fully saturated with brine and then flooded by isooctane (Synthetic oil) and the brine again respectively under same confinement pressure (50 Bar), but different temperatures (20, 40 and 60 °C). With obtained results recovery factors calculated and compared. Final results are discussed and some recommendation were made in the last chapter of the paper.

#### Key words

Core flooding, carbonate rocks, Codaçal limestone, permeability, saturation, overburden stress, reservoir temperature, Enhanced Oil Recovery, Thermal Enhanced Oil Recovery.

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# Nomenclatures

μ	Fluid Viscosity, (Pa.s or cP)
υ	Fluid flow velocity, (m/s)
$\Delta P$	Pressure drop, (Pa, bar or atm)
ρ	Density, [kg/m <sup>3</sup> or g/cm <sup>3</sup> ]
Φ	Porosity
$\Phi_{ m min}$	Minimum Porosity
$\Phi_{\text{max}}$	Maximum Porosity
m	mass (kg or g)
А	Cross section area, $(m^2 \text{ or } cm^2)$
D	Diameter (m or mm)
L	Length, (mm or m)
Κ	Permeability, (md)
$K_{abs}$	Absolute permeability, (mD, Darcy or m <sup>2</sup> )
$\mathbf{K}_{\min}$	Minimum Permeability, (mD, Darcy or m <sup>2</sup> )
K <sub>max</sub>	Maximum Permeability, (m <sup>2</sup> or Darcy m <sup>2</sup> )
$K_{\text{eff}}$	Effective permeability, (mD or Darcy or m <sup>2</sup> )
Ko	Oil permeability, (m <sup>2</sup> D, Darcy or m <sup>2</sup> )
K <sub>rel</sub>	Relative permeability, (mD, Darcy or m <sup>2</sup> )
Р	Pressure, (Pa, bar or atm)
Q	Flow rate (m <sup>3</sup> /d or ml/h)
Т	Temperature, (°C)
V	Volume, (m3 or ml)
$V_b$	Bulk volume, (m3 or ml)
V <sub>p</sub>	Pore volume of the core (m3 or ml)
ml	Milliliter
scc	Standard cubic centimeter
mm	Millimeter
cP	Centipoise (mPa.s)
mD	Millidarcy
P <sub>in</sub>	Inlet pressure (bar)
Pout	Outlet pressure (bar)

So	Oil saturation, fraction
Sor	Residual oil saturation after water flooding, fraction
$\mathbf{S}_{\mathbf{w}}$	Water saturation, fraction
$\mathbf{S}_{wi}$	Initial water saturation, fraction
ASP	Alkaline/surfactant/polymer
AC	Ant Colony
DE	Differential Evolution
EOR	Enhanced oil recovery
GA	Genetic Algorithm
IOR	Improved oil recovery
IFT	Interfacial tension
NA	Neighborhood Algorithm
OHIP	Original hydrocarbon in place
PSO	Particle Swarm Optimization
RCAL	Routine core analysis
RF	Recovery factor [%]
SCAL	Special core analysis

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#### Chapter 1 INTODUCTION

#### **1.1 Motivation**

Although all recent crisis and environmental debates on petroleum industry, we still have a huge dependency on hydrocarbon resources. It is important to keep in mind that petroleum does not only means fuel for our vehicles, but it is also a crucial raw material for petrochemical industry and for energy generation. Only in US annual demand for crude oil for electricity generation is about 1 billion cubic meter per year (U.S. Energy Information Administration, 2011). In comparison with 190-million-ton biomass amount per year it is incomparably more. As consequence at least 50 years period is needed to replace all the products derived from petroleum only in the USA (U.S. Department of Energy, 2005). Which means there will be still a huge demand on the field although rapidly decreasing hydrocarbon reserves. Application of the techniques called thermal enhance oil recovery becomes more feasible with this increasing demand which requires several pilot tests in laboratories such as core flooding. It is also the main motivation for conducing this work.

Core flooding is a technique used to mimicking flooding experiments at the natural environment of the rock samples. By means of these test researchers can determine how fluids or gases moves through the samples in reservoir conditions (Sun et.al., 2015). It is also a common test to determine rock permeability and how will interact a core sample in various fluids.

Due to the high cost of coring from reservoir, core flooding tests are usually performed by an analogues outcrop which has similar petrophysical properties. During the test core sample is placed inside core holder with a requested size and pressurized in a certain temperature in order to simulate different reservoir conditions. Loads in the rock will affect the core's permeability to fluids, so it is important to duplicate them during testing (Soleymanzadeh et.al., 2019). Meanwhile the results show that both stress and temperature are important to both chemical alteration and mechanical deformation (R.C Aadland et.al., 2019). Since every rock is unique effect of temperature and pressure also will be different on each type of rock samples. It means every type of the rock should be investigated individually and this work aims to investigate a specific type of carbonate rock called "codaçal limestone".

#### **1.2 Objectives**

The objectives of the work to observe effects of different confinement pressures and temperature to absolute permeability and recovery factor of core samples. Since the main parameters are permeability and porosity which can be affected, the final recovery also should considerably change with the effect of temperature and pressure.

Absolute permeability alteration is an important parametrical change since it is directly related to ease of flow. That is why investigation of it makes more sense than any other parameter. As scope of this work, it is also main investigated parameter alongside its reduction factor.

The work has two main aspects:

- Effect of temperature and confinement pressure on absolute permeability of investigated sample. This will help to understand pure effects on the sample. 3 samples will be flooded each under 3 different confinement pressure and 4 different temperatures. Final results will be averaged and plotted as different plots.
- Temperature and pressure effect on recovery factor. In this step brine saturated samples will be flooded by isooctane and with brine again to record recoveries and calculate recovery factors in 3 different temperature steps.

## **1.3 Structure of thesis.**

The thesis is divided in following few chapters.

Chapter 1 is the introduction which contains motivation and objective of the work.

Chapter 2 comprehend basics of coring and core analyses. It stars with the general definition of core and then coring techniques, core damages in the site. Finally, chapter ends with general overview of petrophysical parameters of the rocks and methods used to define them on this work.

Chapter 3 describes all the methodologies which were used during the laboratory experiments. It includes sampling of the intended rock, basic measurements such as core diameter and dry weight scaling, saturation procedure, calculation of the effective porosity and core flooding technique.

Chapter 4 contains laboratory experiments which were done on real samples. It shows theoretical background of calculations and the results were obtained from these calculations.

Chapter 5 is the conclusion chapter. All obtained results and recommendations for future works on the laboratory is given.

## CHAPTER 2 Literature review

## 2.1 Core and coring.

There are several methods of coring techniques for core recovery. These are mainly conventional coring systems (full-diameter cores) and wireline retrievable cores. And special coring systems are available for preserving fluids and pressure. In logistically and economically difficult places sidewall coring operations can be also applied (McPhee et.al., 2015).

• Full-diameter coring systems are the most widespread techniques of core analysis. The selection priorities of a bit are based on; type of formation, minimum drilling mud-filtrate invasion requirement, optimization of core recovery, rate of penetration and the efficiency of drilling. Table 1 shows relationship between the hole size and retrieved core diameter

Hole Size (in.)	Core Size (in.)
5¾-61/2	29/16
61/2-8	31/2
8–9¼	4
9 <sup>1</sup> /4" - 11	5¼
11-121/2	51/4

Table 1 Typical full-diameter core sizes and hole diameter (McPhee et.al., 2015)

Since the inner barrel of wireline-retrievable core assembly can be moved free from BHA, it offers significant cost and time advantages than conventional coring. Drillers can lift the cored samples without tripping the whole BHA.

• Sidewall cores are very useful in places where the core recovery is lost, or it is not conventional to take a full-size core. It also reduces the cost of coring. Percussion and rotary sidewall coring are two main types of it. Nowadays percussion sidewall coring almost vanished due to the potential damage of core during the shooting. The tool is in on a wireline and it has a series of rotary coring bit for getting samples from the wall of the main hole. They can be differently spaced due to requirements by different clients. The plugs have usually 0.92-inch diameter and 1–1.5-inch length (McPhee et.al., 2015).



Figure 1 Baker Hughes MaxCOR large-diameter rotary sidewall plugs (McPhee et.al., 2015).

The cores will be used have average 100mm length and 30mm diameter limestone (Codaçal limestone) sample available in Geo-lab, CERENA (Instituto Superior Técnico). Codaçal limestone is extensively used for ornamental purposes which is found in the Santo António Plateau of Portugal, but its formation process and history assure for the possible discoveries of hydrocarbon reserves in the future. Figure 2 shows some of the typical core plugs used for this this work



Figure 2 Typical core sample used for this work

#### 2.2 Core analyses.

Harrison argues that it has never been important to take and analyze core samples from complex reservoirs. Analyses just give us insight about lithology and mineralogy; estimating the most essential rock properties; show fluid distribution and flow mechanism inside the pores; and geotechnical parameters for quicker & safer drilling completions. According to Harrison "logs alone are not enough to characterize a reservoir without the information of rock properties so the modelling must depend on a good correlation between logs and cores. So, great uncertainties may occur if we do not have core analyses data (Harrison B., 2009).

For a reliable core analyses a high-quality, undamaged core sample is the first and foremost prerequisite. If the core has been damaged the results will not be reliable even with the best analyses for retrieving petrophysical parameters (McPhee et.al., 2015).

Routine and Special core analyses are two main tests performed on a core sample for obtaining petrophysical parameters.

1) Routine-or basic or conventional core analysis are for measurements of different fluid saturations, obtaining porosity and absolute permeability while using single phase fluids generally at ambient conditions on dry cores. Obtained data are mainly being used for the characterization of reservoir properties and for correlation between logs and cores.

2) Special core analyses-SCAL has several principal analyses tests which usually grouped under the name of SCAL(McPhee et.al., 2015). These include:

- Porosity at stress.
- Resistivity factor of formation.
- Index of resistivity.
- CEC tests (cation exchange capacity).
- Capillary pressure tests (Imbibition and drainage) with low or high pressure Mercury injection, porous plate and centrifuge methods.
- Wettability and contact angle tests (Amott and USBM tests).
- Relative permeability tests among different fluids.

## 2.3 Core damage and core fluid/petrophysical property alteration

While coring and transportation of formation from certain depth to the surface the pore pressure and core temperature are rapidly reduced. These results of them are:

- Original fluid distribution alteration inside the pore space.
- Alteration of sample's texture and mineralogical properties.
- Wettability Alteration of the sample.

These are essential to understand the nature of damages and considering all the retrieved data which must be certainly considered for the further evaluations.

During the core's journey to the surface a water-wet sample can turn into oil wet or less water wet at the surface. Additionally, drop of the pressure under the bubble point may cause liberation of gas from crude oil which is been stored inside the sample. Consequently, relative concentrations of heavier end components and surfactants are increasing, and these may absorb onto the rock surfaces. Solubility of the surfactants in oil also decreases with decreasing temperature and that increases adsorption potential.

If air enters to the core sample during retrieving of it at wellsite or in the laboratory, oxidization can cause an abundance of the asphaltene component and gives an opportunity to increase the oil wettability (McPhee C., 1994). These kinds of wettability alteration can be prevented to design an appropriate anti-oxidization mechanism during handling.

Solution can be the use of linens which physically prevents the sample from chemical and mechanical damages as well as fluid loses.

There are several coring techniques to prevent core damages and fluid/petrophysical alteration during the coring.

- Gel coring systems were developed principally as an alternative to operator intensive wellsite core preservation (Skopec, 1996). Its technical advantage allows encapsulate the core for preventing it from mud invasion and damage after coring. As well as reduce jamming gel also prevents in situ saturation and original fluid content of the rock sample.
- The sponge core system helps to retain water and oil escaping from the sample while operations. For this purpose, polyurethane sponge liners are used in the annular space of coring assembly (Durandeau, 1996).

However, these are out of the objective of this work. In this work only routine core analyses performed, and this general information is given about the techniques of retrieving a core for special core analyses.

#### 2.4 Porosity.

Porosity and permeability are key parameters to describe fluid flow inside reservoir in a laboratory scale. Some recommended laboratory standards must be applied during measurement of these two parameters (Franklin, 1977).

Porosity of a specimen is the measure of the storage capacity for holding fluids inside. It is defined as ratio between void spaces and bulk volume of a rock. This important parameter can be mathematically defined as in below.

$$\emptyset = V_p / V_b \qquad (2.1)$$

During the geological formation process of a rock some void spaces can be isolated from others because of excessive cementation. Thus, many of them are interconnected unlike some of them are totally isolated. Therefore, two distinctive types of porosities can be defined namely

- Absolute porosity
- Effective porosity

Absolute porosity is defined as total void space in rock divided by bulk volume of it. The rock may have considerable void space, but lack of the interconnected voids can lead to nonconductivity. Absolute porosity can be described mathematically as below

The effective ratio in between interconnected pore space and bulk volume of a specimen (Ahmed, T., 2001).

The following ISRM suggested methods should be carefully followed for obtaining porosity in laboratory conditions (Franklin, 1977).

1) Calculation of the bulk volume.

$$V_{bulk} = L^*(\pi D^2)/4$$
 (2.4)

Where

L- is the length of specimen

D – diameter of specimen obtained as an average value.

2) Void volume calculation.

Specimen should be dried inside an oven for at least 24 hours in 105°C temperature and weighted by a highly accurate electronic scale. It should be weighted second time after saturated by fully immersed fluid with a 0.4bar vacuum for 1 hour. Therefore, pore volume ( $V_p$ ) can be defined as

$$V_{p} = \frac{Msat - Mdry}{\rho}$$
(2.5)

Where

$$\begin{split} V_p &- \text{ pore volume } (m^3) \\ M_{sat} &- \text{ mass of dry sample } (kg) \\ M_{dry} &- \text{ mass of dry sample } (kg) \\ \rho &- \text{ density of injected fluid } (kg/m^3). \end{split}$$

3) Calculation of porosity.

 $\Phi = \frac{Pore Volume(Vp)}{Bulk Volume (Vb)} \quad (2.6)$ 

Porosity here is referred as effective porosity and it is the one broadly used in reservoir engineering for the estimation of reserves, recovery factor etc. This important petrophysical parameter because it allows to estimate movable fluids inside interconnected voids.

#### 2.5 Permeability.

Permeability is one of the crucial parameters of the rock which measure ability of fluid flow through the rock. This important parameter controls flow direction and flow rate of fluid inside formation. It was firs time mathematically defined by Henry Darcy in 1856. In fact, equation defined by some measurable parameters and called Darcy's law (Ahmed, T., 2001). This standard mathematical tool can be expressed as below.

$$Q = -\frac{kA(p1-p2)}{\mu L} \qquad (2.7)$$

Were

Q - flow rate  $(m^3/s)$ 

k – permeability (m<sup>2</sup> or Darcy)

p1 and p2 inlet and outlet pressures respectively (Pa)

- $\mu$  viscosity of flowing fluid (Pa\*s or cP)
- L length of sample (m)

A – cross section area of sample  $(m^2)$ 

Darcy's equation is valid under several conditions below.

- Fully saturated core with a single-phase fluid
- No chemical reaction between fluid and rock
- Steady state, laminal flow regime for flowing fluid.

The unite of permeability is  $m^2$  in SI units, but it is convenient to use Darcy (1D) or even milli Darcy (1mD) since permeability usually has very small values.  $1mD=10^{-15}m^2$  (Graue., 2006).

For our case on cylindrical specimen Darcy's law can be described as in Figure 3 and expressed as in equation 2.8.



Figure 3 schematic description of laboratory test on core sample

$$\mathbf{K} = \frac{Q\mu L}{A(P1 - P2)} \qquad (2.8)$$

When a porous media is fully saturated with one fluid (it is usually air or brine) permeability to this fluid is called as absolute permeability. When a porous media has more than one fluid it has some resistance for other fluid and this measure of the resistance is called effective permeability (Colin McPhee., 2015).

#### 2.6 Core flooding procedure in laboratory.

Core flooding tests or routine core analyses should be done by following steps (Avasare., 2016)

- 1. Core sample should be cleaned by Ethanol
- 2. After cleaning it should be dried inside an oven under 105°C for at least 24 hours. Another way (such as flushing by N<sub>2</sub>) of drying are also available depend on facilities of laboratory.
- 3. Saturation of the samples with desired fluid. Usually formation water (brine) used for saturation and absolute or effective permeabilities can be achieved due to injection of second fluid. If same fluid (brine) injected again absolute permeability can be calculated by the help of equation 2.8.
- 4. Then crude oil flooding process for achieving the full saturation and followed by aging for 14 days in the reservoir temperature.
- 5. 2 to 4 weeks of oil flooding to ensure initial water saturation  $(S_{wi})$ .

Figure 3 schematically describe core flooding apparatus. The high precision chromatography pump is designed to inject fluids with accuracy of 0.01ml/h up to 10ml/h flow rate. Backup pressure can be obtained by help of a manual hydraulic pump. Inlet and outlet gauges allow to take pressure measurements. Flow rate can be detected by help of a computer or manually (Figure 4).



Figure 4 Simplified core flooding procedure

## CHAPTER 3 Methodology

## 3.1 Methodology.

The chapter focus on summary of all the procedures applied through the entire thesis. Figure below is a brief roadmap of the work.



Figure 5 Roadmap of the thesis

## 3.2 Geological context & sampling.

Cores are drilled from the limestone quarry (Codaçal limestone) sample in the picture below by a core-driller in GeoLab of IST. The samples used for this experiment are obtained from the Lusitanian Basin, located in western Iberian basin (Figure 6). It covers about 20000 km<sup>2</sup> area under west-central part of Portuguese mainland and some adjacent continental shelf.



Figure 6 Study area of outcrop (Wikipedia.org)

Lusitanian basin is one of the families of Atlantic margin-rift basin, which formed by result of Atlantic Ocean opening in Mesozoic era (Rasmussen et.al., 1998). However, the outcrop in use belongs to the middle Jurassic.

Although all Jurassic basin rocks are usually within hydrocarbon generation windows, this is not case for entire Lusitanian basin. That is explained by its highly heterogeneity. However around 100 exploration wells are drilled in the basin and 80 % of the wells from Triassic,

Jurassic and Cretaceous strata produced oil with a 27% recovery on the surface. While there are these reserves, Portugal imports 100% of its fossil fuels. Pre-salt and subsalt are two main types of petroleum system in the basin (Uphoff., 2002).

Sampling is done according to the suggested methods by International Society of Rock Mechanics (J. Franklin., 1997). Cores were cut in cylindrical form with the tops cut in planes parallel to each other and perpendicular to the core axis (Figure 7). Possible discontinuities were avoided, to guarantee the absence of any irregularities, areas of weakness or heterogeneities present in the core, however, is expected some geological variability that is reflected in the properties that will be studied it the cores obtained are around 38 mm of diameter and length between 84 and 100 mm, ensuring a ratio between length and diameter between 2 and 3 as established by ISRM suggested methods. On the other hand, the overall core dimensions must be at least 10 times larger than the size of the largest grain of the rock and the surfaces must be flat and smooth to comply with ISRM methods (Pinto., 2020).



Figure 7 core plug matrix (a), the core drill apparatus (b)

## **3.3 Measurements and preparation of specimens.**

All the measurements in the lab should be followed International Society for Rock Mechanics (ISRM). There are several simple measurements we must define as part of routine core analysis such as diameter and weight measurements.

A digital caliper (Figure 8) must be used for measuring length and diameter of the core.



Figure 8 Digital caliper

Each measurement is repeated for 5 times to minimizing errors for geometrical parameters (Appendix A, Table A-1). Average length, diameter, and cross section area is calculated for each of the 6 sample (AL 1, AL 2, AL 3, AL 4, AL 5, AL 6) and summarized in the table below.

Core sample	Average diameter (mm)	Average Length (mm)	Section area (mm²)
AL1	37.818	100.166	1123.277
AL2	37.752	100.156	1119.360
AL3	37.758	100.036	1119.716
AL4	37.838	99.992	1124.466
AL5	37.898	100.240	1128.035
AL6	37.830	100.044	1123.990

Table 2 Geometrical measurements of core samples.

The cores must be placed inside an oven for drying in 105°C temperature for at least 17 hours. This step helps to get rid of all environmental changes of the sample during handling. Finally, samples should be weighted immediately after taking from the oven with a sensitive electronic scale (Fgure 9).



**Figure 9 Digital scale** 

## **3.3 Fluid preparation and specimen saturation.**

Two types of fluid are needed for our experiment. The first is brine with a 35g/L (35000 ppm) NaCl concentration. Brine is easy to prepare. 35 g of NaCl (salt) is added to 1L of distilled water in a room temperature (20°C) and with a 1.05 cP estimated viscosity (Kestin, 1981).

The second fluid is isooctane. It is used as a substitute for oil (synthetic oil). It is also known as 2,2,4-Trimethylpentane and formulated as  $(CH_3)_3CCH_2CH(CH_3)_2$ . Isooctane is isomer of octane  $(C_8H_{18})$  and one of the important components of gasoline. It is frequently used for increasing knock resistance of petroleum. Isooctane is flammable and colorless. It can affect people in case of a direct inhalation. These are some properties of isooctane below.

Molar mass:  $114.232 \text{ g} \cdot \text{mol}^{-1}$ Density: 693 kg/m<sup>3</sup>

Viscosity: 0.51cP

Vapor pressure: 5.5kPa at 21°C.

Due to its high flammability and evaporation, it should be kept inside a dark place and special care needed to measure it at outlet. Usually, injected fluid is collected in a scaled cylinder to measure the rate (Figure 10).



Figure 10 Scaled cylinder

Although brine can be directly collected inside an open cylinder glass should be sealed for the case of isooctane. Especially in case of small flowrates such as 2ml/hour it becomes more crucial.

Core saturation process is also done by ISRM suggested methods in lab conditions. Vacuum equipment used for saturation purposes is described in figure below.



Figure 11 Vacuum device for core saturation

The process of saturation as in below step-by-step.

- Core plugs should be placed inside a container filled by 2/3 level of saturation fluid (brine). The pump turned on for making 0.4 bar pressure (vacuum) and keep the sample for an hour inside the vacuum. That allows the fluid to fill pore spaces.
- 2) After an hour of saturation, samples should be fully emerged inside brine and put inside the vacuum for another 1 hour.
- 3) Then the pump must be turned off and suction valve should be closed for isolating samples another 18 hours. This is the time needed to make sample fully saturated.
- 4) Finally, samples can be weighted on a digital scale and weight difference between dry and wet conditions must be reported.

## **3.4 Calculation of effective porosity.**

After applying all the ISRM suggested methods, we can finally calculate the porosity of the samples. Since the porosity is ratio between pore spaces and total volume (equation 2.6), those two parameters are inputs. Bulk volume can be simply calculated by multiplying length of sample to cross section area. Calculation of pore space is based on calculating volume of brine inside these pores. The weight difference between dry and fully saturated samples represents amount of brine inside interconnected pores and that leads us to find void volume by dividing this weight to brine density.

Sample	Dry	Saturated weight	Weight difference	Bulk Volume	Pore	Effective
	Weight(g)	(g)	(kg)	(m3)	volume(m <sup>3</sup> )	porosity
AL1	258.588	273.130	0.01454	0.000113	1.41902E-05	0.126119
AL2	260.570	274.340	0.01377	0.000112	1.34369E-05	0.119854
AL3	259.720	273.960	0.01424	0.000112	1.38955E-05	0.124054
AL4	258.280	272.800	0.01452	0.000112	1.41688E-05	0.126014
AL5	262.470	275.990	0.01352	0.000113	1.31929E-05	0.116675
AL6	261.470	274.900	0.01343	0.000112	1.31051E-05	0.116543

Table 3 Effective porosities of samples.

Density of saline water at room temperature (20°C) been taken as 1024.79 kg/m3 according to calculations by Millero et.al (Millero., 1980).

## 3.5 Core flooding equipment.

Experiment conducted in a simple way due to the limitation of lab conditions. Figure 12 illustrates simple core flooding system with an injection fluid, injection pump, core holder, manual hydraulic pump, and container in a line.



Figure 12 Core flooding system.

Injection pump, core holder and manual hydraulic pump are the main core flooding equipment. Injection pump is an electronic high precision pump P-500 and has a flow rate range of 1-499 ml/hour. However, it also has 4MPa (40 bar) pressure limit due to the material of cylinders (Figure 13).



Figure 13 High precision pump (left) and manual hydraulic pump (right)

Manual pump is for supplying confinement pressure inside the core holder. It can supply pressure up to 700 bars. Reservoir conditions can be mimicked inside the core holder by means of both pressure and temperature. For heating up the core holder and consequently core inside it we need to apply temperature by an electric core mantle which is shown in figure 14.



Figure 14 Electric heater and electronic thermo controller.

Thermic controller is designed such as way to keep the temperature constant for a long time. Its sensor can measure the temperature continuously and keeps it around given range. It covers the core holder. Due to the conductivity of metal and oil inside it takes around an hour (conduced study) for the temperature to reach to the specimen. Therefore, this should be considered before starting the injection.

The core holder we use is a "Hassler" core holder and it has a rubber cylinder inside to seal the core from pressurizing oil. It can be simple depicted as in figure 15 below.



Figure 15 Hassler core holder and its structure

## CHAPTER 4 laboratory experiments.

#### 4.1 Core flooding with brine.

The core sample 100 percent saturated with brine. Absolute and relative permeabilities can be obtained by flooding with different fluids. Flooding with brine will give absolute permeability, while flooding by isooctane gives relative permeability to isooctane. Before flooding with any fluid, we need to consider some constrains of laboratory equipment.

The main constrain is the limitation of maximum allowable pressure of the injection pump. Since this limit is 40 bars, we should not exceed this value. This value is related to many things including injection flow rate, flooding fluid's viscosity, temperature, and confinement pressure. Confinement pressure is another parameter to be defined due to the equipment limitations. The objective of this work is not defining this laboratory specific values, it aims to study effect of these values on core and fluid properties in allowable extend. Therefore, predefined value ranges for injection rate and confinement pressure will be used in this work. Instead, effect of temperature, confinement pressure and salinity will be studied.

All the samples are saturated and flooded with brine (35g/l of salinity) for defining absolute permeability. Allowable confinement pressures and flow rate (due to the limitation of the equipment) can be taken from previous work (Mu., 2018). Due to the high viscosity of the brine, injections rate and confinement pressures should be small. For our experiment 50, 100 and 150 bar confinement pressure and 5ml/h injection rate were applied.

## 4.1.1 AL4.

12 tests are performed on sample AL4 under three (100, 150 and 200 bar) constant confinement pressure and four (20, 40, 60, 80°C) different temperatures for each of the constant confinement pressure steps. The inlet pressure and flowrate should be recorded frequently until it becomes stable for application of the Darcy's law. Darcy's law can basically be applied as in below (Equation 2.8)

$$\mathbf{K} = \frac{Q \ \mu \ L}{A \ \Delta P}$$

Where Q - is injection flow rate (5ml/h),

 $\mu$  - dynamic viscosity of brine (function of temperature and salinity)

L – length of the sample,

A - cross section of the sample,

 $\Delta P$  – pressure drop on sample ( $\Delta P = P_{inlet} - P_{outlet}$ )

Since a constant injection flow rate (5ml/h) applied the main changing parameters are  $\mu$  dynamic viscosity of the brine and  $\Delta P = (P_{inlet} - P_{atm})$  the pressure difference. Dynamic viscosity of brine is a function of temperature and salinity. The values calculated by the help of a special calculator (<u>www.ifh.uni-karlsruhe.de</u>) based on empirical values obtained by El-Dessouky and Ettouney (El-Dessouky, Ettoney., 2002).

Additionally, Permeability Reduction factor (PRF) will be calculated for each of temperature and pressure regimes (Appendix B, C, D). PRF is a factor indicates absolute permeability reduction with respect initial absolute permeability at 20°C temperature and 100 bars. It can be calculated as in below

PRF=1-
$$\frac{Kn}{Ki} = (1 - \frac{Kn}{Ki}) * 100\%$$
 (2.9)

Where  $K_n$  – Absolute permeability of the sample at any temperature step

K i – Initial absolute permeability at 20°C and 100 bars confinement pressure.

Table 4 below summarizes results of the test on AL4.

Temperature (°C)		20		40		60		80
Pressure(bar)	K <sub>abs</sub> (mD)	PRF (%)	K <sub>abs</sub>	PRF (%)	K <sub>abs</sub>	PRF (%)	K <sub>abs</sub>	PRF (%)
			(mD)		(mD)		(mD)	
100	0.22	0.00	0.22	1.01	0.18	18.95	0.16	27.77
150	0.19	14.31	0.18	20.86	0.16	29.11	0.16	30.11
200	0.19	14.31	0.18	20.86	0.16	29.11	0.16	30.11

Table 4 Absolute permeability and PRF for AL4

The table represents absolute permeabilities and permeability reduction factors under different confinement pressures and temperatures for sample AL4. Figure 16 depicts results of absolute permeability relationship with temperature and confinement pressure.



Figure 16 K abs, vs Confinement pressure under different constant temperatures for AL4

Also, permeability reduction factors can be plotted as function of temperature and pressure. Figure 17, a) shows PRF versus temperature at constant pressure and figure 17, b) PRF versus confinement pressure under constant temperature.



Figure 17 a) PRF vs Temperature (constant pressures) b) PRF vs Pressure (constant temperatures).

## <u>4.1.2 AL5.</u>

Same procedures are also applied on AL5 (12 flooding tests under different confinement pressure and temperature regimes) to investigate effect of confinement pressure and temperature on the given sample. The main differences are on intrinsic parameters of samples such as average length, cross section, and porosity of the specimen, which were measured and calculated in the table1 and table 2. Consequently, also  $\Delta P$  will be different than obtained values for AL 4. 5ml/h injection rate was applied again, and brine viscosities calculated by the viscosity calculator as it is functions of temperature and salinity (35g/l). Table 4 below shows results of absolute permeabilities and PRF (%).

Temperature (°C)	20		40		60		80	
Pressure	K abs	PRF (%)	K abs	PRF (%)	K abs	PRF (%)	K <sub>abs</sub> (mD)	PRF (%)
(bar)	(mD)		(mD)		(mD)			
100	0.13	0.00	0.11	17.57	0.11	21.25	0.10	27.84
150	0.12	9.10	0.11	17.57	0.11	21.25	0.10	27.84
200	0.11	16.69	0.11	17.57	0.11	21.25	0.10	27.84

Table 5 Absolute permeability and PRF for AL5

Absolute permeability values can be graphically represented as function of confinement pressures as in figure 18 below.



Figure 18 K abs, vs Confinement pressure under different temperatures for AL5

Permeability Reduction Factor is also function of both confinement pressure and temperatures. PRF values here are also calculated with respect to initial PRF values at 20°C and 100 bars confinement pressure. Table 19 a) and b) shows graphical representation of PRF values versus temperature and confinement pressures.



Figure 19 a) PRF vs Temperature (constant pressures) b) PRF vs Pressure (constant temperatures).

#### <u>4.1.3 AL6.</u>

AL6 is the last sample subjected to these procedures and table5 summarizes obtained results below.

Temperature (C)		20		40		60		80
	K abs		K abs		K abs		K abs	
Pressure (bar)	(mD)	PRF (%)						
100	0.13	0.00	0.11	17.57	0.11	17.82	0.11	19.80
150	0.13	0.00	0.11	17.57	0.11	17.82	0.11	19.80
200	0.12	9.10	0.11	17.57	0.11	21.25	0.10	24.03

Table 6 Absolute permeability and PRF for AL6



Figure 20 illustrates relationship between absolute permeability and confinement pressure on constant temperatures.

Figure 20 K abs, vs Confinement pressure under different temperatures for AL6

Finally, PRF values as a function of both temperature (Figure 21 a) and pressure (Figure 21 b).



Figure 21 a) PRF vs Temperature (constant pressures) b) PRF vs Pressure (constant temperatures).

#### Averaged results.

For having a better result, it is decided to plot averaged values of permeability and permeability reduction factors. Since all the samples were obtained from the same outcrop all of them have identical petrophysical characteristic and averaging the results are allowable for the shake of simplicity and conclusion. Table 7 represents averaged values of K <sub>abs</sub> and PRF values.

Temperature (C°)	20		40		60		80	
Pressure	K abs		K abs		K abs		K abs	
(Bar)	(mD)	PRF (%)						
100	0.16	0.00	0.15	12.05	0.13	19.34	0.12	25.14
150	0.15	7.80	0.13	18.67	0.13	22.73	0.12	25.92
200	0.14	13.37	0.13	18.67	0.12	23.87	0.12	27.33

Table 7 Averaged absolute permeability and PRF for AL4, AL5 and AL6

Figure 22, 23 and 24 below also represent averaged K <sub>abs</sub> and PRF values for AL4, AL5 and AL6 (which is calculated as sum of values divided by 3) versus confinement pressure and temperature for different temperature steps.



Figure 22 Averaged K abs, vs Confinement pressure under different temperatures.



Figure 23 Averaged PRF vs Temperature (constant pressures)



Figure 24 Averaged PRF vs Pressure (constant temperatures).

#### 4.2 Core flooding by isooctane. Recovery factor calculation.

#### <u>4.2.1 AL 1.</u>

In this step we will investigate effect of temperature on first 3 samples (AL1, AL2 and AL3). Absolute and relative permeabilities will be calculated as well as isooctane recovery. All the samples are saturated with brine and brine, isooctane, brine injection will be carried on respectively to define absolute/relative permeabilities and recovery factor.

Sample AL1 is heated up to 40°C and brine started to be injected after an hour in order ensure the temperature conduction inside the core holder. 50 bar confinement pressure supplied by a manual hydraulic pump. According to the applicability of Darcy's law the flow rate and differential pressure should be constant in order to apply Darcy's law. For this reason, the experiment should be carried out for enough time to obtain a constant inlet pressure since we have outlet pressure of an atmospheric pressure. Plotting of pressure and measured flow rate versus time, we can obviously determine that stabilization as in Figure 25.



Figure 25 Pressure and flowrate stabilization time of AL1.

As we observed pressure starts to stabilize after 50 minutes and then Darcy's law (2.8) can be applied for determining absolute permeability as in below (Appendix E, Table 1).

$$K = \frac{Q \ \mu L}{A \ \Delta P} = \frac{5 \times 0.708 \times 0.100166 \times 10^{-9}}{60 \times 60 \times 1123.277 \times 448675 \times 10^{-6}} \ m^2 = 1.9543 \times 10^{-16} \ m^2 = 0.1980 \ mD$$

Where Q = 5ml/h is flow rate,

 $\mu = 0.708 \times 10^{-3} \text{kg/m} \text{ s brine viscosity from table (El-Dessouky., 2002).}$ 

L= length of sample,

A= Cross section

 $\Delta P$ = Differential pressure between inlet and outlet of specimen.

In second step we need to inject different fluid to define relative permeability (Appendix E, Table 2). For mimicking hydrocarbon migration, synthetic oil (Isooctane) is injected. The test is carried out with same confinement pressure (50 bar) and temperature (40°C).



Figure 26 Pressure and production profile of AL1

Due to the low viscosity of isooctane it takes some hours to get brine production. After 2 hours isooctane starts to displace brine from outlet of the core holder. As it can be seen from figure 14 the pressure became stabile after 320 minutes during isooctane production. Darcy's law (2.8) can be applied here also to determine relative permeability to isooctane as in following.

$$K_{rel} = \frac{Q \ \mu L}{A \ \Delta P} = \frac{5 \times 0.51 \times 0.100166 \times 10^{-9}}{60 \times 60 \times 1123.277 \times 798675 \times 10^{-6}} \ m^2 = 7.90863 \times 10^{-17} m^2 = 0.0801 \ mD$$

Where Q = 5 ml/h is flow rate,

 $\mu = 0.51 \times 10^{-3} \text{kg/m} \times \text{s}$  isooctane viscosity.

3<sup>rd</sup> step of the test aims to determine oil recovery factor of the sample (Appendix E, Table 3). Since the sample (AL1) is fully saturated with brine initially and flooded by isooctane in the 2<sup>nd</sup> step, we must flood again with brine as mimicking of enhance oil recovery by water injection. Recovery factor is the ratio in between recovered oil amount and total oil in place. Recovered oil amount can be easily determined by measuring it by a metric cylinder glass placed at outlet but measuring amount of isooctane initially in place requires to determine irreducible (connate) water saturation inside porous space. Total Isooctane amount is basically pore space minus connate water saturation. Therefore, recovery factor can be determined as in below.

$$RF = \frac{Produced \, Isooctane}{Total \, Isooctane} = \frac{V_{recovered \, isooctane}}{V_p - V_{wi}} = \frac{V_{recovered \, isooctane}}{V_p - (V_p - V_{recovered \, water})} = \frac{V_{recovered \, isooctane}}{V_{recovered \, water}} \quad (3)$$

Where V recovered isooctane -is the amount of isooctane recovered at 3<sup>rd</sup> step,

V  $_{p}$  – porous space, which is calculated as  $\frac{wet weight - dry weight}{brine density}$  previously,

V wi-irreducible water amount,

 $V_{recovered water}$  – is the amount of water recovered during  $2^{nd}$  step as the result of isooctane injection. Recovered water amount should be less than initial water amount ( $V_p$ ) due to the irreducible water. Since isooctane displaces water in  $2^{nd}$  step, the amount of oil in place should be same with recovered water in  $2^{nd}$  step. Finally, recovery factor is

$$RF = \frac{V_{recovered isooctane}}{V_{recovered water}} = \frac{4.2 \times 10^{-6}}{8.2 \times 10^{-6}} \times 100\% = 51.21\%$$

51% of recovery factor calculated for first confinement (50°C, 50bar confinement pressure and 35g/l salinity).

## <u>4.2.2 AL 2.</u>

Same procedures applied for the next specimen (AL2) but with a different temperature (60°C). Again, 50bar of confinement pressure and 35g/l of saline water is used during the experiment (Appendix F, Table 1).

First flooding step is started after applying 60°C outside the holder for at least 1 hour to guarantee temperature conduction to the sample. Brine is injected to fully saturated sample to define absolute permeability by using Darcy's law.

Pressure and flowrate versus time graph shows the stabilization time when Darcy's law can be applied (Figure 27)



#### Figure 27 Pressure stabilization time AL2

If we apply Darcy's law (2.8) again,

$$\mathbf{K} = \frac{Q \ \mu \ L}{A \ \Delta P} = \frac{5*0.507*0.100156*10^{-9}}{60*60*1119.36*598675*10^{-6}} \ m^2 = 1.05865*10^{-16} = 0.1066 \ \text{mD}$$

2<sup>nd</sup> step is injecting isooctane at 50 bar and 60°C. This will help to determine relative permeability to isooctane and comparison will be made to see how the temperature effects it later(Appendix F, Table 2). Pressure profile is plotted in figure 28.



Figure 28 Pressure and production profile of AL2

From the profile pressure starts stabilizing after 180 minutes and Darcy's law (2.8) can be applied here again to determine relative permeability to isooctane as in below.

$$K_{rel} = \frac{Q \ \mu L}{A \ \Delta P} = \frac{5*0.51*0.100156*10^{-9}}{60*60*1119.36*898675*10^{-6}} \ m^2 = 7.05249*10^{-17} = 0.0715 \ mD$$

The last  $(3^{rd})$  step is to define the recovery factor of the isooctane production (Appendix F, Table 3). As in AL1 we also need to flood sample with brine again and record amount of recovered isooctane at outlet. Simplified equation for recovery factor (3) has already been defined for the lab experiment as in below

$$RF = \frac{V_{recovered isooctane}}{V_{recovered water}} = \frac{3*10^{-6}}{8.3*10^{-6}} = 0.3614 = 36.14\%$$

## <u>4.2.3 AL 3.</u>

80°C temperature is applied on the  $3^{rd}$  sample (AL 3). The sample is flooded by brine in the  $1^{st}$  step to define absolute permeability (Appendix G, Table 1). Pressure and flowrate stabilization time on a given flowrate can be plotted as below.



Figure 29 Pressure stabilization time AL3

Darcy's law (2.8) applied as

$$K = \frac{Q \ \mu L}{A \ \Delta P} = \frac{5*0.387*0.100036*10^{-9}}{60*60*1119.716*298675*10^{-6}} \ m^2 = 1.607*10^{-16} = 0.1629 \ mD$$

 $2^{nd}$  step on specimen AL3 is for defining relative permeability to isooctane by injecting isooctane to the sample at 80 °C (Appendix G, Table 2). Production and pressure profile for the test can be plotted as in below



Figure 30 Pressure and production profile of AL3

So, Darcy's law (2.8) can still be applied here after having the stabilization time as following

$$K_{rel} = \frac{Q \ \mu L}{A \ \Delta P} = \frac{5*0.51*0.100036*10^{-9}}{60*60*1119.716*598675*10^{-6}} = 1.05705*10^{-16} = 0.10710 \ mD$$

Although we tried to define recovery factor in the 3<sup>rd</sup> step of the test, we could not due to some unexpected property changes of isooctane with effect of the temperature (Appendix G, Table 3). Therefore, temperature effects isooctane physical properties in such a way that we could not distinguish border with brine inside scaled discharge glass in outlet. The phenomenon can be explained by highly volatility of Isooctane. Relatively high vapor pressure (0.568 bar at 80° C) and being close to boiling point (99°C) makes it to evaporate at the outlet (Williamham et. al., 1945).

#### **Chapter 5 Conclusion and recommendations.**

## 5.1 Conclusions.

As the main scope of this work effect of the temperature and confinement pressure investigated on a specific type of carbonate rock. That Investigated sample is "codaçal limestone" which extracted from Lusitanian Basin, located in western Iberian. It covers about 20000 km<sup>2</sup> area under west-central part of Portuguese mainland and some adjacent continental shelf. 6 samples (AL1, AL2, AL3, AL4, AL5, AL6) were cored from the same outcrop with identical geometrical and petrophysical parameters. Having this identical parameter is the main advantage of summarizing results for the rock itself.

All the samples dried and saturated with ISRM suggested methods. With basic calculation porosities are calculated and obtained results confirmed to be consistent with previous works on same type of rock. Since porosity is one of the important petrophysical parameters, determination of porosity was attentively done with maximum avoidance of measurement errors. For this reason, several geometrical measurements done, and averaged numbers were used to minimize them errors.

All rock samples subjected to different experimental procedures. First 3 samples AL4, AL5 and AL6 (nomenclatures are trivial) is used for defining of pressure and temperature effects on absolute permeability. 12 injection tests were repeated on each of the sample under 3 different confinement pressure (100, 150 and 200 bar) and 4 different temperature (20, 40, 60 and 80 Celsius) for each of the pressure steps. For securing temperature conduction from the heater (mantle powered by electricity) to the sample inside of core holder, pre-calculated conduction time (1 hour) applied before each temperature step. Due to the pressure limitation of the injection pump also pre-defined range of injection rate is used (5ml/h). During the experiments repetitive measurements were taken with the help of a stopwatch which makes a distinctive sound in each adjusted time step and it allows observant to measure injection pressure and flow rate versus time. Procedures were repeated till constant flowrate and injection pressure values obtained for at least 3 iteration. This is one of the requirements by Darcy's law which was used to determine absolute permeability values for each experimental step.

It is defined that absolute permeability is the main parameter which represents ease of fluid flow inside the porous medium and therefore temperature and pressure effect on absolute permeability change is crucial. After all the absolute permeability calculation the results needed to be plotted for having a clear vision on temperature and pressure effect on them. Also, permeability reduction factor (PRF) was introduced which represents the percentage reduction of permeability with respect to initial permeability values at 20 °C and 100 bar confinement pressure.

Three dependency were plotted for each specimen: absolute permeability versus pressure under different temperatures, PRF versus temperature under different confinement pressures and PRF versus confinement pressure under different temperatures. Since all the specimens are from same rock sample, the obtained results are expectedly identical. Averaged results showing a nice tendency which are consistent with previous research on carbonate rocks.

Averaged permeability versus pressure plot shows a tendency of decreasing permeability with increasing confinement pressure and this rate is higher in lower temperature steps (Figure 21). It can be better expressed in permeability reduction factor (PRF) versus pressure plot (Figure 23). Increasing of confinement pressure led decreasing of permeability and consequently increasing of PRF values. It is also found that these rates higher for a lower temperature step. For instance, PRF increases about 13% (from 100 bar confinement pressure to 200 bars) for 20°C constant temperature, meanwhile these values are as 8%, 5% and 2% for 40, 60 and 80°C, respectively. That means effect of the temperature on absolute permeability is more significant than effect of the confinement pressure. The main reason why rate of permeability reduction is lower in higher temperature. According to the Darcy's equation absolute permeability is directly proportional to fluids viscosity. Brine viscosity versus temperature

itself is a nonlinear function where viscosity decreasing rate is more significant for higher temperature steps. Also, the reason why increasing pressure causes reduction on permeability values might be explained with stress-strain relationship of the sample. More the confinement pressure means more packing of the grains (more deformation and more tortuosity) and lowering the value of porosity (S. K. Sanyal, 1974). This explains obtained results well enough.

Figure 22 represents plotted date of PRF versus temperature at constant confinement pressure values. It can be noticed the permeability reduction rate decreases with increasing temperature at considered pressure. It is important to observe the trends of permeability reductions for higher pressures (for our case for 150 and 200 bar). Two curves are very identical and even converge in one point. It means effect of confinement pressures on absolute permeability is relatively limited. Obvious if the experiments were repeated in higher values of pressures the lines would completely converge. Benzagouta and Amro performed experiments on higher values of effective pressures and the results confirm it (Mohammed Benzagouta, 2009). Moreover, reduction of absolute permeability is not only effect of confinement pressure it can be related to deformation mechanism of grains which constitute the rock. Sayers and Kachanov argues that reduction in pore space and consequently absolute permeabilities are related to pore space orientation order (C. M. Sayers., 1995).

Experiments with other 3 samples (AL1, AL 2 and AL3) focused on temperature effect on absolute permeability and recovery factors. Each of the samples subjected to same-50 bars confinement pressure but different temperatures (40, 60 and 80°C) and flooded by brine, isooctane, and brine respectively. In firs flooding process by brine absolute permeabilities were calculated for all the samples. Which shows an unpredicted tendency of absolute permeability. Expected tendency should be an increasing on absolute permeability with increasing temperature, but AL3 shows a higher absolute permeability than AL2 (Table 7).

Samples	K abs (mD)	K rel (mD)	RF (%)	Porosities
AL1	0.198025	0.080134	51.21951	0.126119
Al2	0.106637	0.071459	36.14458	0.119854
A13	0.162909	0.107105	-	0.124054

Table 8 Results of last 3 samples

This can be explained as the effect of porosity differences. Although the samples have identical petrophysical parameters, they are still slightly difference and it can affect other related parameters (K <sub>abs</sub> and K <sub>rel</sub>) as well. Most obvious of these differences are porosities and geometrical parameters of the samples (especially the length since it is in Darcy's equation). Obviously, AL 3 has a higher porosity value than AL2. Even permeability is not a direct function of porosity in our calculation method (Darcy's law), its effect on absolute and relative permeabilities are considerable and more than effect of temperature (Benzagouta, 2009).

In the second step the samples were flooded by isooctane and it was found that the results are strongly affected by porosity more than temperature. But also effect of temperature has a certain effect on relative permeability especially when comparison made between AL 3 and AL1. Although AL3 has slightly less porosity than AL1, AL3 ends up with more relative permeability. That might be due to the phase change of isooctane with effect of high temperature (80°C). It is already investigated that 80°C is considerably close to isooctane's boiling point and it is highly evaporative due to its high vapor pressure (Williamham et.al., 1945).

In the third step samples were flooded by brine again to calculate recovery factor of isooctane. Basic formula was composed for the calculations. AL1 and AL2 showed expectedly good results due to the lower operation temperatures while AL3 was out of track to be detected. It became impossible to distinguish isooctane on the outlet of flooding apparatus due to the temperature effect. Again, this might be the result of high vapor pressure of isooctane and being close to its evaporation temperature which makes it highly evaporative in outlet. Generally, it was concluded that experiment with AL3 is not reliable (except absolute permeability determination at first step).

## 5.2 Recommendations.

The main recommendations are related to improvement of lab equipment. Although there were all the necessary facilities in CERENA lab of IST, some of them need to be renovated for having a more precise results of measurements. Especially pressure gauges and flowmeter needed to be replaced with electronic ones since these two are most direct sources of our data sources. Observant can make some measurements errors while recording from the mechanical gauges and graded tube for flowrate. Replacement graded tube by an Arduino based flowmeter was already performed some years ago in the lab, but due to some financial reasons and pandemic the project was uncompleted. The main recommendation is having this measurement all automated and a software base.

Another recommendation is towards application of an outlet pressure and temperature to the outlet. During our experiment outlet pressure and temperature was always atmospheric pressure and room temperature, which can result by evaporation of some injected fluids and led to incorrect measurements. Also, availability of volume measurements of injected fluid would be better although injection pump has a flowmeter. This especially may result in a better estimation of recovery factor.

The last recommendation is the possible application of Scanned Electron Microscopy (SEM) which allows to detect textural distribution, pore space orientations, fractures, veins, vugs and most importantly assessing petrographic and textural changes on samples after each flow. However, SEM might be crucial for relatively higher confinement pressures when the sample expected to be in non-elastic stress state. For our case maximum applied confinement pressure is 200 bar and this type of carbonate rock supposed still to be in elastic state.

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# Appendixes

# Appendix A – Geometrical and petrophysical measurements

Table-1 Average length and diameter calculation

	Length	Average	Diameter	Average
AL1	100.03	100.166	37.78	37.818
	100.35		37.76	
	100.11		37.92	
	100.08		37.79	
	100.26		37.84	
AL2	100.7	100.156	37.76	37.752
	100.17		37.77	
	99.98		37.75	
	100		37.76	
	99.93		37.72	
AL3	100.03	100.036	37.75	37.758
	100.11		37.79	
	100.01		37.75	
	100.05		37.79	
	99.98		37.71	
AL4	99.85	99.992	37.81	37.838
	100.11		37.98	
	100.12		37.8	
	99.82		37.79	
	100.06		37.81	
AL5	100.25	100.24	37.85	37.898
	100.3		37.87	
	100.26		37.9	
	100.2		37.87	
	100.19		38	
AL6	100.26	100.044	37.8	37.83
	99.91		37.82	
	100		37.79	
	100.02		37.9	
	100.03		37.84	

# Appendix B – Historical data for AL4.

Temp.	20	C	40 C		60	С	80 (	C
Time	Press.	Water	Press.	Water	Press.	water	pressure	water
(min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)
0	0	1	0	1	0	0	0	1
10	4	1	5	1.3	4	1.2	4	1.4
20	7	1	5	2	4.5	2	4	2.4
30	7	1	5	3	4.5	2.8	4	3.2
40	7	1	5	3.8	4.5	3.2	4	4
50	7	1.4			4.5	4.2		
60	7	2.4						
70	7	3.2						
80	7	4						

Table 1 AL 4 - 100 bar pressure and flow rate stabilization

Table 2 AL 4 – 150 bar pressure and flow rate stabilization

Temp.	20 (	C	40	С	60 (	C	80 C	
time(	pressure	water	pressure	water	pressure	water	pressure	water
min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)
0	0	1	0	1	0	1	2	5
10	0	1	6	1.2	5	1.4	4.1	5.5
20	0	1	6	2	5	2.2	4.1	6.5
30	1	1	6	2.8			4.1	7.5
40	1	1	6	3.2				
50	3	1						
60	4	1						
70	5	1						
80	5	1						
90	6	1						
100	7	1						
110	8	1						
120	8	1						
130	8	1						
140	8	1.6						

Table 3 AL 4 - 200 bar pressure and flow rate stabilization

Temp.	20 C		40 C		60 C		80 C	
time( min)	pressure (bar)	water (ml)	pressure (bar)	water (ml)	pressure (bar)	water (ml)	pressure (bar)	water (ml)
0	1	1	0	0	2	5	2	5
10	7	1.2	5	1.2	5	5	4.1	5.5
20	8	2	6	2	5	5.5	4.1	6.5
30	8	3.6	6	2.8	5	6	4.1	7
40	8	4.4	6	3.6	5	6.5	4.1	

# Appendix C Historical data for AL5

Temp	20 C		40 C		60	0	80	80 C	
time(	pressure	water	pressure	water	pressure	water	pressure	water	
min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	
0	2	5	9	5.5	0	5	0	5	
10	7	5	9	6	7	5.5	6	5.5	
20	11	5	9	6.5	7	6.5	6	6.5	
30	11	5	9	7	7	7			
40	11	5	9	7.5					
50	11	5.5							
60	11	5.5							

Table 1 AL 5 – 100 bar pressure and flow rate stabilization

Table 2 AL 5 – 150 bar pressure and flow rate stabilization

Temp.	20 C		40 C		60	60 C		80 C	
time(	Pressure	water	pressure	water	pressure	water	pressure	water	
min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	
0	0	1	0	1	0	1	0	1	
10	5	1	8	1.2	7	1.2	6	1.4	
20	10	1	9	2	7	2	6	2.2	
30	11	1	9	2.8	7	3	6	3.2	
40	11	1	9	3.6					
50	10	1.2							
60	12	1.6							
70	12	2.2							
80	12	3							

Table 3 AL 5-200 bar pressure and flow rate stabilization

Temp.	20 C		40 C		60 C		80 C	
time(	pressure (bar)	water	pressure (bar)	water (ml)	pressure (bar)	water	pressure (bar)	water (ml)
0	2	5	1	5	2	5	2	5
10	12	5.5	7	5.5	7	6	6	5.5
20	13	6.5	9	6.5	7	6.5	6	6
30	13	7	9	7	7	7.5		

# Appendix D Historical data for AL6

Temp.	20 C		40 C		60	60 C		80 C	
time(	pressure	water	pressure	water	pressure	water	pressure	water	
min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	
0	4	1	0	1	2	1	1	1	
10	11	1.1	8	1.4	6.75	1.4	5.5	1.4	
20	11	1.6	9	2.2	6.75	2	5.5	2.2	
30	11	1.8	9	3	6.75	2.4			
40	11	2.6	9	4.8					
50	11	3.6							

Table 1 AL 6-100 bar pressure and flow rate stabilization

Table 2 AL 6 – 150 bar pressure and flow rate stabilization

Temp.	20 C		40 C		60	60 C		80 C	
time(	pressure	water	pressure	water	pressure	water	pressure	water	
min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	
0	2	1	7	1.2	2	1	1	1	
10	11	1.6	9	1.8	4	1.5	5	1.8	
20	11	2.4	9	2.6	6.5	2.3	5	2.6	
30	11	3.2			6.5	3.2			
40	11	4							
50	11	5							

Table 3 AL 6 – 200 bar pressure and flow rate stabilization

Temp.	20	С	40 C		60	С	80	80 C	
time(	pressure	water	pressure	water	pressure	water	pressure	water	
min)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	(bar)	(ml)	
0	0	1	0	1	0	1	0	1	
10	5	1	7	1.2	7	1.4	5.75	1.3	
20	9	1	9	1.8	7	2.1	5.75	2.2	
30	12	1	9	2.2	7	3	5.75	3	
40	12	1	9	3					
50	12	1.4							
60	12	2.2							

# Appendix E Historical data for AL1

Time (min)	Pressure (bar)	Water (ml)	Rate(ml/10min)	Rate (ml/h)
0	0	1	0	0
10	0	1	0	0
20	2	1	0	0
30	4	1	0	0
40	5	1	0	0
50	5.5	1	0	0
60	5.5	1	0	0
70	5.5	1	0	0
80	5.5	1	0	0
90	5.5	1.6	0.6	3.6
100	5.5	2	0.4	2.4
110	5.5	2.6	0.6	3.6
120	5.5	3.4	0.8	4.8
130	5.5	4.2	0.8	4.8
140	5.5	5	0.8	4.8

Table 1 AL1 – Flooding with brine for absolute permeability determinati	on.
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Table 2 AL1 – Flooding with isooctane for relative permeability determination.

time(min)	press(bar)	water (ml)	total	isooctane	water rate (m3/s)	isooctane rate
0	0	0			0	0
10	0	0			0	0
20	0	0			0	0
30	0	0			0	0
40	0	0			0	0
50	0	0			0	0
60	0	0			0	0
70	0	0			0	0
80	0	0			0	0
90	0	0			0	0
100	0	0			0	0
110	0	0			0	0
120	0	0			0	0
130	0	0			0	0
140	1	0			0	0
150	2	0			0	0
160	2	0			0	0
170	5	0			0	0
180	7	0			0	0
190	8	0			0	0
200	8	0.5			8.33E-10	0
210	9	1.2			1.17E-09	0
220	6	1.6			6.67E-10	0
230	9	1.6			0	0
240	10	2.4			1.33E-09	0
250	10	3.2			1.33E-09	0
260	10	4.2			1.67E-09	0
270	10	6			3E-09	0

280	10	7.5			2.5E-09	0
290	10	8.2			1.17E-09	0
300	10		2.4	1.4	0	2.33E-09
310	10		3.4	2.4	0	1.67E-09
320	9		4.4	3.4	0	1.67E-09
330	5		5	4	0	1E-09
340	9		5.6	4.6	0	1E-09
350	9		6.5	5.5	0	1.5E-09
360	9		7.4	6.4	0	1.5E-09
370	9		8.4	7.4	0	1.67E-09
380	9		9.2	8.2	0	1.33E-09

Table 3 AL1 - Flooding with brine for recovery factor determination.

time(min)	press(bar)	isooctane	total	water(ml)	brine
0	0	0	1	1	0
10	1	0	1	1	0
20	5	0	1	1	0
30	9	0	1	1	0
40	12	0	1	1	0
50	16	0	1	1	0
60	21	0.8	1.8	1	0
70	22	1.6	2.6	1	0
80	22	2.4	3.4	1	0
90	22	3	4	1	0
100	22	3.8	4.8	1	0
110	22	4	5.2	1.2	0.2
120	20	4.1	5.9	1.8	0.8
130	22	3.9	6.7	2.8	1.8
140	22	4.2	7.6	3.4	2.4
150	22	4.2	8.4	4.2	3.2
160	22	4.2	9.2	5	4

# Appendix F Historical data for AL 2

Time (mins)	Pressure (bar)	Water (ml)	Rate (ml/h)
0	0	1	0
10	1	1	0
20	3	1	0
30	7	1	0
40	7	1	0
50	7	1	0
60	6	1.2	1.2
70	7	2.2	6
80	7	3	4.8
90	7	3.8	4.8
100	7	4.7	5.4
110	7	5.5	4.8
120	7	6.2	4.2
130	7	7	4.8

Table 1 AL2 - Flooding with brine for absolute permeability determination.

Table 2 AL2 – Flooding with isooctane for relative permeability determination.

Time(min)	Pressure (bar)	Water (ml)	Total	Isooctane	Brine rate	Isooctane rate
0	1	0	1		0	0
10	2	0	1		0	0
20	4	0.2	1.2		3.33E-10	0
30	7	0.2	1.2		0	0
40	8	0.4	1.4		3.33E-10	0
50	10	1.1	2.1		1.17E-09	0
60	11	1.8	2.8		1.17E-09	0
70	11	2.6	3.6		1.33E-09	0
80	12	3	4		6.67E-10	0
90	12	3.7	4.7		1.17E-09	0
100	12	4.5	5.5		1.33E-09	0
110	13	5.3	6.3		1.33E-09	0
120	12	6.2	7.2		1.5E-09	0
130	12	7.6	8.6		2.33E-09	5E-10
140	12	8.3	9.6	0.3	1.17E-09	1.17E-09
150	12	8.3	2	1	2.96E-24	1.33E-09
160	11	8.3	2.8	1.8	0	6.67E-10
170	11		3.2	2.2	0	1E-09
180	10		3.8	2.8	0	1.67E-09
190	10		4.8	3.8	0	6.67E-10
200	10		5.4	4.2	0	1E-09
210	10		6	4.8	0	

Time(mins)	Press(bar)	Isooctane	Total	Water(ml)	Brine
0	1	0	1	1	0
10	17	0.2	1.2	1	0
20	21	0.7	1.7	1	0
30	24	1.2	2.2	1	0
40	28	1.4	2.4	1	0
50	28	1.8	2.8	1	0
60	28	2.4	3.4	1	0
70	28	2.8	3.8	1	0
80	28	2.8	3.8	1	0
90	28	3	4.4	1.4	0.4
100	28	3	5	2	1
110	26	3	6	3	2
120	26	3	6.6	3.6	2.6

Table 3 AL2 – Flooding with brine for recovery factor determination.

# Appendix G Historical data for AL 3

time (mins)	Pressure (bar)	water (ml)	rate (ml/h)
0	0	1	0
10	3	1	0
20	4	1	0
30	4	1	0
40	4	1.5	3
50	4	2.3	4.8
60	4	3.2	5.4
70	4	4	4.8
80	4	4.9	5.4
90	4	5.8	5.4
100	4	6.6	4.8
110	4	7.4	4.8

Table 1 AL3 – Flooding with brine for absolute permeability determination.

Table 2 AL3 – Flooding with isooctane	for relative permeability determination.
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time(min)	press(bar)	isooctane	brine	total	brine rate	isooctane rate
0	0	0	0	1	0	0
10	2	0	0	1	0	0
20	6	0	0.6	1.6	1E-09	0
30	7	0	1.4	2.4	1.33E-09	0
40	7	0	2.2	3.2	1.33E-09	0
50	7	0	3	4	1.33E-09	0
60	8	0	3.8	4.8	1.33E-09	0
70	8	0	4.5	5.5	1.17E-09	0
80	8	0	6.2	7.2	2.83E-09	0
90	8	0	7.6	8.6	2.33E-09	0
100	8	0.7	8	9.7	6.67E-10	1.17E-09
110	8	1.4	8		0	1.17E-09
120	7	2.2	8		0	1.33E-09
130	7	2.8	8		0	1E-09
140	7	3.6	8		0	1.33E-09
150	7	4.2	8		0	1E-09

Table 3 AL3 – Flooding with brine for recovery factor determination.

time(min)	press(bar)	isooctane	total	water(ml)	brine
0	0		1	1	-
10	0			1	-
20	2			1	-
30	5			1	-
40	8			1	-
50	12			1	-
60	14			1	-

70	14	0.1	1.1		-
80	14	0.4	1.4		-
90	14	0.4	2.2	1.8	-
100	14	0.4	3	2.6	-
110	14	0.4	4	3.6	-
120	14	0.4	4.5		-
130	14	0.4	5.4		-
140	14		6.2		-
150	14		7		-
160	14		8		-