



**Ministry of Education  
Sebha University  
Faculty of Engineering  
Department of Oil and Gas**

**Project Title:**

**Enhancing Oil Recovery in Sandstone Core Samples  
Using Zeolite, Sodium & Potassium Hydroxide, Slica,  
AL, Addition to Formation Water**

A research project submitted to the department of Oil and Gas in partial  
fulfillment of the degree for B.Sc. in Petroleum Engineering

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## **DEDICATION**

We offer my prayers and thanks to "Almighty Allah" who helped and blessed me with guidance during the courses of my studies.

Thanks goes to my family for their great support and continuous encouragement during the span of my studies.

We would like also to thank the staff members of Sebha University for their efforts and support during my journey at the university.

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## ABSTRACT

This research project explores the effectiveness of adding zeolites to formation water for enhancing oil recovery. The demand for fossil fuels, particularly oil, remains significant for meeting global energy needs. However, primary oil production methods fall short in extracting most of the original oil-in-place (OOIP). Enhanced Oil Recovery (EOR) methods are employed to address this challenge. Among these methods, water flooding plays a vital role in increasing oil production by maintaining reservoir pressure and displacing oil from pore spaces, leading to improved ultimate oil recovery. To enhance the efficiency of water flooding, various chemicals have been tested and proven effective when added to the injected water. These include smart water injection, carbonated water injection, surfactant solutions, polymers, alkalis, mutual solvents, and foam injection. Zeolites, known for their porous structure and ion-exchange properties, have gained prominence in numerous industrial applications, including EOR.

This research investigates the impact of adding zeolites to formation water on oil recovery. The research issue arises from the recognition that formation water alone may not efficiently recover oil due to oil properties and rock characteristics. The aim of this study is to determine whether the inclusion of zeolites in formation water can enhance oil recovery. Experiments are conducted on sandstone core samples obtained from a Libyan field in the Murzuq basin, using oil with a density of g/cc. The findings of this research hold significance for fields subjected to water injection in Libya, as successful implementation of zeolites could substantially increase the country's daily oil production rate.

The experimental results demonstrate that the addition of zeolite, sodium hydroxide, potassium hydroxide, silica, and aluminum significantly improves oil recovery at higher temperatures for sandstone core samples. Furthermore, the inclusion of sodium hydroxide yields superior oil recovery compared to potassium hydroxide. Using 15% of sodium and potassium hydroxides yields better results than other percentages, although the underlying cause is not explored in this study. Additionally, the combination of zeolite, sodium and potassium hydroxides, silica, and aluminum enhances oil recovery, warranting further investigation into the underlying mechanisms.

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## *Chapter One*

# **INTRODUCTION**

## 1.1 Background

Much of the energy needed today comes from fossil fuels, and oil reservoirs are one of its largest sources. However, the production of these resources has many challenges, and it is not possible to produce most of the original oil-in-place (OOIP) with the primary methods. Several recovery methods are used to improve oil recovery. They are called Enhanced Oil Recovery methods (EOR)[1].

Water flooding is an essential technique used in EOR operations to increase the production of oil from reservoirs. Injection of water helps in increasing the reservoir pressure to its initial level and maintain it near that pressure. In addition, water displaces oil from the pore spaces and improves the ultimate oil recovery[2].

The displacement efficiency depends on many factors such as oil viscosity and rock characteristics. Therefore, in order to improve that efficiency, several chemicals were tested and proved its efficiency, added to water during water flooding. For example, smart water injection, carbonated water injection, injection of surfactant solutions, polymers, alkalis and mutual solvents can be considered as various methods of chemical water injection. In addition, sometimes to increase the performance of chemical water, a combination of several types of additives and foam injection is recommended according to the structure of the reservoirs[3].

Zeolites are a group of naturally occurring or synthetic crystalline minerals composed of aluminum, silicon, and oxygen. They have a unique porous structure with regular channels and cavities that can accommodate a variety of cations, such as sodium, potassium, calcium, and magnesium. Due to their high adsorption capacity and ion-exchange properties, zeolites have gained importance in various industrial applications, including enhanced oil recovery (EOR).

In this research, the effect of adding zeolites to formation water in improving oil recovery is investigated.

## 1.2 The Objectives of The Research

**A.** Formation water without additives might not have great efficiency in recovering oil due to oil properties and rock characteristics.

**B.** The current research aims to investigate whether adding Zeolites to formation water would improve oil recovery.



**C.** The experiments in this research will be carried out for sandstone formation using 3 core samples from a Libyan field in Murzuq basin. In addition, an oil of g/cc density will also be used.

**D.** The researcher hypothesizes that adding zeolite to formation water will improve its characteristics and subsequently enhance oil recovery.

**E.** Several fields in Libya are being subjected to water injection, If zeolite has proven its efficiency, and improved oil recovery than the daily production rate of the country will drastically be increased.

## *Chapter Two*

# **LITERATURE REVIEW**

## **2.1 Importance of Water Flooding in EOR Operations:**

Today fossil fuels supply more than 85% of the world's energy. Currently, we are producing roughly 32 billion barrels per year in the world. With global energy demand and consumption forecast to grow rapidly during the next 20 years, a more realistic solution to meet this need lies in sustaining production from existing fields for several reasons [4]:

- Industry cannot guarantee new discoveries.
- New discoveries are most likely to lie in offshore, deep offshore, or difficult to- produce areas.
- Producing unconventional resources would be more expensive than producing from existing fields by enhanced oil recovery (EOR) methods [5].

reduction of production over time, the production process is divided into three parts of primary, secondary Oil reservoirs have different production rates at different stages of exploitation. Following the and tertiary. In the primary stage, production is done by the natural force of the reservoir. The natural force of the reservoir can be supplied by mechanisms such as the solution gas drive, active aquifer and gas cap drive. After natural production with the force caused by the initial pressure of the reservoir, usually water and immiscible gas injection are done according to the economic and technical issues of the reservoir to provide pressure and increase the sweep efficiency. In the third stage, other methods are also used to improve specific parameters affecting oil production. The methods used in tertiary oil production are known as “enhanced oil recovery”. These methods generally include thermal methods and injection of gases such as hydrocarbons, nitrogen, air, carbon dioxide, and factory gases and chemical solutions.

Water flooding is inexpensive and simple to use. Thus, it is dominant among fluid injection methods and is without question responsible for the current high oil producing rate within the America and Canada [6]. Water injection is a technique that uses injection wells and production wells to avail the energy with the water injected for displace oil to the production wells.

pointed out that in determining the suitability of a candidate reservoir for waterflooding, the following reservoir characteristics must be considered:

- **Reservoir geometry**

The areal geometry of the reservoir will influence the location of wells and, if offshore, will influence the location and number of platforms required. The reservoir's geometry will essentially dictate the methods by which a reservoir can be produced through water injection practices. An analysis of reservoir geometry and past reservoir performance is often important when defining the presence and strength of a natural water drive and, thus, when defining the need to supplement the natural injection. If a water-drive reservoir is classified as an active water drive, injection may be unnecessary.

- **Fluid properties**

The physical properties of the reservoir fluids have pronounced effects on the suitability of a given reservoir for further development by waterflooding. The viscosity of the crude oil is considered the most important fluid property that affects the degree of success of a water flooding project. The oil viscosity has the important effect of determining the mobility ratio that, in turn, controls the sweep efficiency.

- **Reservoir Depth**

Reservoir depth has an important influence on both the technical and economic aspects of a secondary or tertiary recovery project. Maximum injection pressure will increase with depth. The costs of lifting oil from very deep wells will limit the maximum economic water-oil ratios that can be tolerated, thereby reducing the ultimate recovery factor and increasing the total project operating costs. On the other hand, a shallow reservoir imposes a restraint on the injection pressure that can be used, because this must be less than fracture pressure. In waterflood operations, there is a critical pressure (approximately 1 psi/ft of depth) that, if exceeded, permits the injecting water to expand openings along fractures or to create fractures. This results in the channeling of the injected water or the bypassing of large portions of the reservoir matrix. Consequently, an operational pressure gradient of 0.75 psi/ft of depth normally is allowed to provide a sufficient margin of safety to prevent pressure parting.

- **Lithology and Rock Properties**

pointed out that lithology has a profound influence on the efficiency of water-injection in a particular reservoir. Reservoir lithology and rock properties that affect flood ability and success are:

- Porosity.
- Permeability.
- Clay content.
- Net thickness.

In some complex reservoir systems, only a small portion of the total porosity, such as fracture porosity, will have sufficient permeability to be effective in water-injection operations. In these cases, a water-injection program will have only a minor impact on the matrix porosity, which might be crystalline, granular, or vugular in nature.

Although evidence suggests that the clay minerals present in some sands may clog the pores by swelling and deflocculating when waterflooding is used, no exact data are available as to the extent to which this may occur.

Tight (low-permeability) reservoirs or reservoirs with thin net thickness possess water injection problems in terms of the desired water injection rate or pressure.

- **Fluid Saturations**

In determining the suitability of a reservoir for waterflooding, a high oil saturation that provides a sufficient supply of recoverable oil is the primary criterion for successful flooding operations. Note that higher oil saturation at the beginning of flood operations increases the oil mobility that, in turn, gives higher recovery efficiency.

Due to oil and water properties and different rock characteristics several additives are used with the injected water. Additives to injected water are usually engineered and adjusted for better performance and improvement of parameters affecting oil production. This type of water injection is known as chemical water injection[3].

## **2.2 Importance of Adding Chemicals to the Water to Improve Water Flooding Performance:**

Chemical water injection is one of the most common and effective methods of EOR in which by engineering the type and concentration of additives according to the reservoir conditions such as temperature, pressure and salinity, the parameters affecting the reduction of capillary pressure are reached to the desired values.

### **2.2.1 Surfactants:**

A surfactant and co-surfactant are added to an aqueous fluid in order to prepare the surfactant solution and injected into the reservoirs. Surfactant flooding reduces the interfacial tension between the oil and water phases and also alters the wettability of the reservoir rock in order to mobilize the residual oil trapped in the reservoir which improves the oil recovery.

Over the past years, Surfactant floods projects have been applied over a wide range of conditions:

- Reservoir temperatures [70–290] °F.
- Average reservoir permeability from less than 1 millidarcy to thousands millidarcies.
- Oil viscosity from less than 1 cP to thousands of cP for heavier crudes.
- Net pay thickness from a few feet to several hundred feet.
- Remaining oil at start-up [20–90] % of OOIP.

The surfactant selection is a critical stage in designing the surfactant flooding projects, selecting the optimum surfactant would guarantee several advantages such as:

1. Very effective in lab tests [high oil recovery].
2. Surfactant modeling is relatively simple with only a few well-designed experiments needed to provide the most important simulation parameters.
3. Current high-performance surfactants cost less than \$2/lb. of pure surfactant.
4. Recent developments in surfactants solutions for EOR have effectively reduced the required surfactant concentration, lowering the chemical costs required.
5. Recently, new and effective surfactants are derived from plant resources such as sunflower oil, soy and corn oil. It is non-toxic, non-hazardous, and readily biodegradable.

### **2.2.2 Polymers:**

Polymers are high-molecular-weight substances that increase the viscosity of the injected water. The increased viscosity improves the sweep efficiency by reducing water channelling and fingering, allowing for better displacement of oil from the reservoir.

In concept, a water-soluble polymer is used to reduce the mobility ratio of water-oil by increasing the water viscosity which improves the volumetric sweep efficiency. The mechanism of polymer flooding is to increase the water viscosity and also to reduce the permeability of the rock to water, in other words, to reduce the water-oil mobility ratio close to unity or less. Over the past years, polymer floods projects have been applied over a wide range of conditions:

- Reservoir temperatures [46–235] °F.
- Average reservoir permeability [0.6–15,000] mD.
- Oil viscosity [0.01–1494] cP.
- Net pay thickness [4–432] ft.
- Remaining oil at start-up [36–97] % of OOIP.

The advantages of polymer flooding could be summarized as following:

1. Applicable over a wide range of conditions.
2. A reduction in the quantity of water required to reduce the oil saturation to its residual value in the swept portion of the reservoir.
3. An increase in the areal and vertical coverage in the reservoir due to a reduced water flood mobility ratio.
4. Diverting the injected from swept zones.
5. Promising for heavy oil application.
6. Cost-effective.

### **2.2.3 Alkaline:**

Alkaline agents, such as sodium hydroxide (NaOH) or sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), are used to increase the pH of the injected water. Alkaline flooding is particularly effective in reservoirs with high acid consumption, as it helps neutralize acidic components present in the reservoir and reduces the formation of emulsions. Alkaline agents also react with acidic crude oil components, improving their solubility and enhancing oil recovery.

In the Alkaline flooding process, the alkaline agents such as sodium hydroxide solution is injecting into the reservoirs which react with the naturally occurring organic acids in the oil in order to produce surfactants or soaps at the oil-water interface. However, the alkaline agents are less expensive than the surfactant agents, the expected incremental oil recovery by alkaline flooding has not been confirmed by field results and still remains possibility as the process is mainly dependent on the mineral composition of the reservoir rock and its oil.

This EOR method has the same advantages of the surfactant flooding in addition to that its main advantage over the surfactant is the cost of the alkaline agents are cheap compared to the surfactant agents[3].

### **2.3 What is Zeolite and How Does It Improve Water Flooding:**

Zeolites are a group of naturally occurring or synthetic crystalline minerals composed of aluminium, silicon, and oxygen. They have a unique porous structure with regular channels and cavities that can accommodate a variety of cations, such as sodium, potassium, calcium, and magnesium. Due to their high adsorption capacity and ion-exchange properties.

#### **2.3.1 What is Zeolite:**

Zeolites can be defined in two ways. They are hydrated tectoalumino silicates with the general formula[7]:  $M_X M_y N_z [T_m T_n \dots O_{2(m+n)} (OH)_{2z}] (OH)_{br} (aq)_p \cdot qQ$

where M, M' are exchangeable and non-exchangeable cations, respectively; N are non-metallic cations (generally removable on heating); (aq) represents chemically bonded water (or other strongly held ligands of T-atoms); Q are sorbate molecules; T, T' are Si and Al, but also Be, B, Ga, Ge and P, among others. This formula is particularly useful when describing natural zeolites[8], but also those synthesized from natural or waste materials, due to their complex chemical composition.

On the other hand, zeolites can be more graphically, as shown in Figure 1, defined as crystalline inorganic polymers consisting of  $[SiO_4]$  and  $[AlO_4]$  tetrahedra, having the structure filled ions and water molecules, having great freedom of movement.



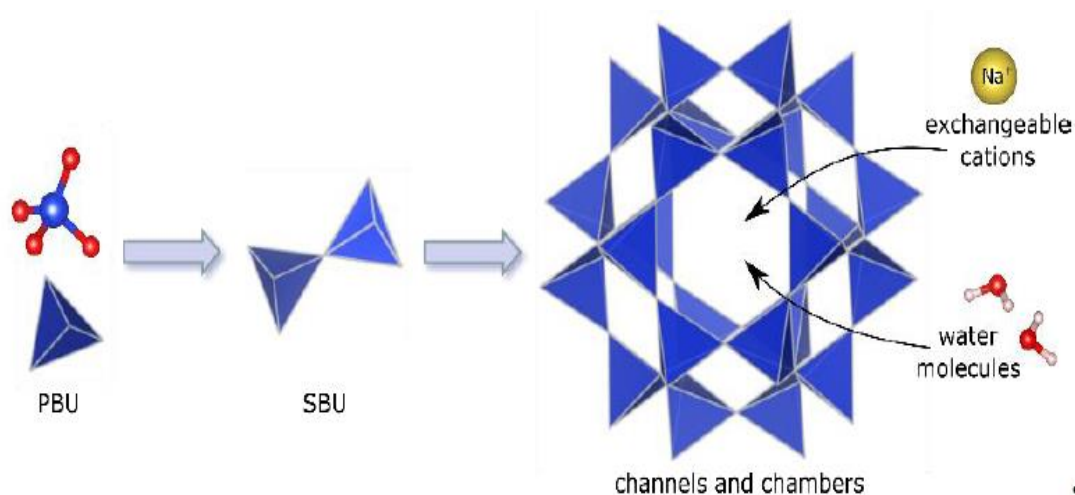


Figure 2. 1 Scheme of Zeolite Structure[8]

The specific structure of the zeolites gives them a number of unique properties. The most important regarding potential uses include [9]:

- Low density and large volume of free space.
- The existence of channels and chambers of strictly defined dimensions (shape-selectivity).
- High degree of hydration and the presence of so-called "zeolite water".
- High degree of crystallinity.
- Possibility of sorption of molecules and ions.
- Ion exchange capacity.
- Catalytic properties.

### 2.3.2 Natural Zeolites:

Natural zeolites are hydrothermal and of mainly volcanic origin. They can occur both in crystallized forms found in igneous and metamorphic rocks, as well as in grains of smaller diameters accumulated in sedimentary rocks [10]. Ocean bottom sediments are relatively huge and rich in zeolites, but these deposits are so far inaccessible to humans. However, these minerals may also constitute important components of tuffs or clay. Such surface retention of zeolite sediments, and therefore relatively simple mining using the opencast method, creates perfect conditions for their wider use[11].

It should be mentioned here that the zeolites naturally occurring in nature, possessing operational significance, are: analcime, chabazite, clinoptilolite, erionite, mordenite and phillipsite.

### 2.3.3 Synthetic Zeolites:

Zeolites have been recognized as minerals of natural origin, but currently more than one hundred Types different of zeolite structures are known which can be obtained synthetically [12]. Under natural conditions zeolites were formed as a result of the reaction of volcanic ash with the waters of the basic lakes. This process lasted several thousand years. In laboratory conditions, an attempt can be made to imitate hydrothermal processes using elevated temperature or pressure and using natural raw materials and/or synthetic silicates. The synthesis reaction requires appropriate equipment, clean substrates and energy. As a result, the price of the product may be much higher than the price of natural zeolite. Therefore, research often focuses on the search for cheaper and available substrates for the production of zeolites, while striving to reduce the cost of the reaction itself. The current trends in research on the synthesis of zeolites are shaped by environmental aspects, which implies the use of natural or waste raw materials for this purpose[13]. The most common are Synthetic Zeolites A, X, Y and ZSM-5.

#### 2.3.3.1 Synthesis Methods

The oldest of the works on the synthesis of aluminosilicates under hydrothermal conditions dates back to the 1950s of the last century. They show that, by heating the aluminosilicate raw materials in the presence of alkaline solutions within a few hours or days, depending on the type of raw materials and process conditions (temperature, pressure), a final product can be obtained.

Today, many different methods for the synthesis of zeolites are known. The most important of them should be mentioned:

- Hydrothermal synthesis (at normal or elevated pressure).
- Molten salt method.
- Fusion method.
- Alkali activation.
- Microwave-assisted synthesis.
- Synthesis by dialysis.

The first method is relatively commonly used. This process generally reflects the natural conditions in which rocks containing zeolite minerals were formed. Hydrothermal (80–350 °C) synthesis of zeolites requires the supply of components that are a source of Si and Al, followed by treatment with an alkaline solution (pH > 8.5). The reactions, during which processes such as dissolution, condensation, gelatinization and crystallization take place are carried out in

autoclaves, are often at elevated pressure. The appropriate control of process parameters favors the formation of desired products.

The estimated cost of zeolite material obtained by the above methods is between natural and synthetic zeolite. However, taking into account the fact that fees for the storage and utilization of waste will probably increase, the implementation of one of these technologies may prove to be the most cost-effective solution[13].

#### **2.3.3.2 Benefits of Synthetic Zeolite**

Numerous scientific studies confirm the benefits of synthetic zeolites compared to natural ones. The efficiency of natural zeolites in the removal of radioactive waste from the environment (Cs and Sr radionuclides) was found to be lower than that of synthetic zeolites. Synthetic zeolites also show much higher adsorption capacity for heavy metal ions (eg.  $\text{Cd}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cr}^{3+}$ ) than the natural zeolites.

Another advantage of synthetic zeolites over natural ones is the significantly larger pore size. This allows for the sorption of larger molecules, which extends the range of potential applications. For example, it was found that synthetic zeolites have two-times higher oil sorption capacities than the natural clinoptilolite, so synthetic zeolites are a promising alternative for natural mineral sorbents for land-based petroleum spills cleanup. Furthermore, zeolites with smaller pore sizes, used as catalysts, suffer from pore blockage and ultimately poisoning and deactivation, while zeolites with large interconnected channels remain stable much longer in reactions.

During the synthesis of zeolites, the Al content can be adjusted. Zeolites with low Si/Al ratio are much more polar and thus exhibit stronger sorption capacities. Zeolites with high silicon content are also characterized by greater power of active centers, which promotes them for catalytic applications [48]. On the other hand, high silica zeolites have a more homogeneous surface characteristic and exhibit hydrophobic properties. They have been used in reactions in which resulting water poisons the acid sites of the catalysts.

It should be noted that the synthetic zeolites are not without disadvantages. The worrisome problem is that synthetic zeolites are mostly in the form of finely grained crystalline and highly dispersive powder (single crystals have a size to a few microns), which certainly limits their use. For natural zeolites, after mining the deposit, isolation processes, such as crushing and pellet formation, are usually used to obtain zeolites in usable form. In contrast, the synthetic ones can be used in the form of hard, wear-resistant granules. There are many reports related to receiving zeolite granulates, but for the most part they are not yet used in practical applications[14].

### 2.3.4 Zeolite Applications

Natural zeolites have a high selectivity for heavy metal ions and ammonium ions[9]. Thus, zeolites have found important uses in environmental protection and agriculture. Wastewater treatment from heavy metal ions or radioactive isotopes can take place in sorption columns filled with zeolite. Ammonium ions contained in municipal, industrial and agricultural wastes can also be removed in a similar way[15]. In agriculture, zeolites can be used as carriers of agrochemical compounds, in the treatment of soil and fish ponds and as a feed additive[16]. Attempts to modify the structure to give them catalytic or antibacterial properties are also made[17]. After all, they are also widely used in many households as pet litter.

On the other hand, natural zeolites have limited applications in industry because, as already mentioned, their properties are strictly dependent on their crystal structure. The main disadvantage is that the channel diameters are too small (in the case of clinoptilolite, which is the most common in nature, it is 0.30–4 nm [12], which do not allow for the adsorption of larger gas molecules and organic compounds. In addition, zeolite deposits are a non-renewable resource. The need for the synthesis of molecular sieves and adsorbents with very specific parameters means that numerous attempts were made to obtain zeolites in the laboratory.

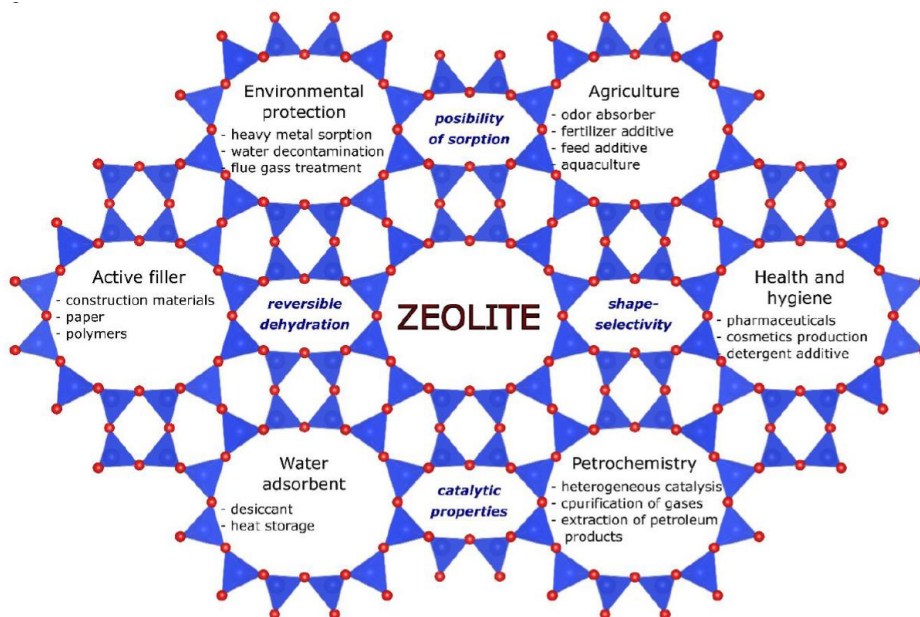


Figure 2. 2 Zeolite applications[9].

## *Chapter Three*

# **RESEARCH METHODOLOGY**

### 3.1 Experimental Materials

The materials used in this research project experiment includes:

1. **Core samples:** Three sandstone core samples, **Figure 3.1**, from a Libyan field in Murzuq basin was used.



*Figure 3. 1 Sandstone core sample*

2. **Reservoir oil sample:** A 40.1 API and 0.8243 gm/cc oil sample was used for the experiment. **Figure 3.2** shows the oil sample used for this research project.



*Figure 3. 2 Oil Sample*

3. **Reservoir water sample:** Use one sample of reservoir water with for our study. **Figure 3.3** shows the reservoir water used for this research project while **Table 3.1** shows the properties of the reservoir water.



*Figure 3. 3 Reservoir water sample*

*Table 3. 1 Properties of reservoir water sample*

<b>Conductivity Electrine CE (mc/cm)</b>	13440
<b>Salinity (ppt)</b>	6.2
<b>PH</b>	8.23
<b>TDS (mg / l)</b>	8810

4. **ZSM-5 manufactured zeolite:**

**Figure 3.4** shows the ZSM-5 manufactured zeolite.



*Figure 3. 4 Zeolite*

## 5. Silica:

Silica is a granular, vitreous, porous substance and is a form of silicon dioxide created synthetically from sodium silicate. Silica gel is hard and harsh. It is more solid than common household silica gel and its gelatin or agar versions. It is a natural mineral that is purified, processed, and converted into any form other than granules or beads. As a result of its dryness, it has an average pore size of 2.4 nanometers and has a strong affinity for water molecules.

**Figure 3.5** shows the silica used for the research project.

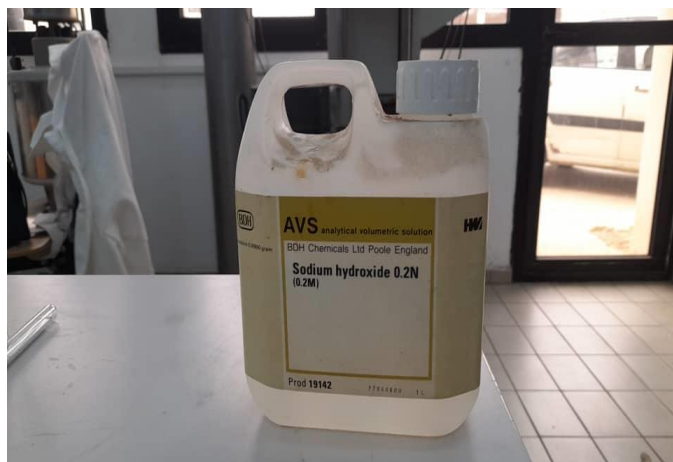


*Figure 3. 5 Silica*



## 6. Sodium Hydroxide:

Sodium hydroxide is sometimes called caustic soda or lye. It is a common ingredient in cleaners and soaps. sodium hydroxide is a white, odorless solid. Liquid sodium hydroxide is colorless and has no odor. It can react violently with strong acids and with water. Sodium hydroxide is corrosive. NaOH can react with moisture from the air and may generate heat as it dissolves. This heat can be enough to cause a fire if it is near flammable materials. **Figure 3.6** shows the sodium hydroxide used for the research project.



*Figure 3. 6 Sodium Hydroxide*

## 7. Potassium Hydroxide:

Potassium hydroxide solution appears as a clear aqueous solution. Metal and tissue corrosion. Not combustible. Used in the chemical industry, petroleum refining and vehicle cleaning. **Figure 3.7** shows the potassium hydroxide used for the research project.



*Figure 3. 7 Potassium hydroxide*

### 3.2 Experiment Devices:

1. **Na, K ratio meter:** to measure the sodium and potassium content in the formation water.



*Figure 3. 8*

#### 2. **PH and Electrical Conductivity (EC) Meter:**

A pH meter is an analytical instrument that measures the activity or basicity of a solution, a key parameter for many applications in various industries.

Conductivity electrodes, available in two- to four-cell models, are used with conductivity meters to measure a solution's ability to convey an electrical flow. They feature built-in temperature compensation, as well as high, standard and low conductivity measuring ranges. A variety of probe materials are available including epoxy/graphite, glass/platinum, and stainless steel. **Figure 3.9** shows the PH and EC meter.



*Figure 3. 9 EC meter*

3. **Filter Paper:** Semi-permeable paper used to separate the fine solids in a liquid during a filtration process. **Figure 3.10** shows the filter papers used for the research project.



*Figure 3. 10 Filter Papers*

4. **Glass Cups:** Glass cups were used in reservoir water treatment process in the experiments. **Figure 3.11** shows the cups used for the research project.



*Figure 3. 11 Glass Cups*

5. **Sample Preservation Tube:** Figure 3.12 shows the tubes were used to hold water samples for analysis after treatment.



*Figure 3. 12 Sample preservation tube*

6. **Vacuum pump:** Figure 3.13 shows vacuum pump used to deflating air from the vacuum chamber.



*Figure 3. 13 Vacuum Pump*

7. **Vacuum Chamber:** Air deflating was carried out in the pellicle vacuum chamber shown in **Figure 3.14**. The vacuum chamber was also used for saturating sandstone sample with reservoir water, then for saturating the same sample with reservoir oil to measure the oil recovery.



*Figure 3. 14 Vacuum Chamber*

8. **Vernier Caliper:** Vernier caliper, shown in **Figure 3.15**, was used to accurately take the dimensions of the rock samples during the experiment in order to calculate the core bulk volume.



*Figure 3. 15 Vernier Caliper*

9. **Drying oven:** The drying oven, shown in **Figure 3.16**, was used for drying the core samples during the experiment.



*Figure 3. 16 Drying oven*

### 3.3 Experiments Procedures:

#### 3.3.1 Formation Water Preparation:

The treatment process was carried out for water with a TDS of 8810. Using the active ZSM-5 zeolite catalyst, potassium, and sodium hydroxides the TDS was decreased through the following detailed procedures:

**Step 1:** The first step is to filtrate the formation associated water from sand and oil residuals using paper filters.



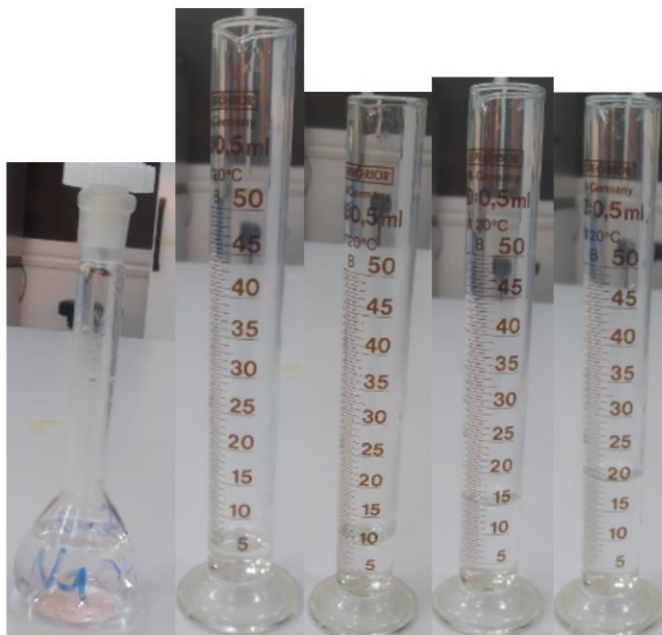
*Figure 3. 17 Associated formation water filtration*

**Step 2:** Zeolite was activated before it is being added to filtered water. The activation is carried out by raising the zeolite temperature to 750 °C for two hours.



*Figure 3. 18 Zeolite activation at high temperature*

**Step 3:** Sodium hydroxide with a concentration of 0.2 M was added to a 100 ml of the filtered associated water at different rates of 5%, 10%, 15% and 20%.



*Figure 3. 19 Adding sodium hydroxide to the filtered associated water*

**Step 4:** 1 gm of zeolite was added to the water mixture.



*Figure 3. 20 Zeolite*

**Step 5:** The mixture was put in a water bath at 50 °C with a mixer at a speed of 500 rpm for half an hour.





*Figure 3. 21 Mixing device*

**Step 6:** The mixture was left in a Polypropylene vial for 24 hours at room temperature as per **Figure 3.22.**



*Figure 3. 22 Polypropylene vial*

**Step 7:** After 24 hours the water was filtered from the zeolite.



*Figure 3. 23 Filtrating water from silica and zeolite*

**Step 8:** Filtered water was analyzed.

**Step 9:** The process was repeated again with replacing the sodium hydroxide with potassium hydroxide with a concentration of 0.1M with the same rate and procedure.

**Step 10:** Another two scenarios were investigated:

**Scenario 1 (Cat1):** Using 15% sodium hydroxide sample, a 1:1 ratio of pure silica and aluminum was prepared and added to water in order to have a better efficiency in terms of total dissolved solids.

During preparation they were activated at a temperature of 450 °C for a period of two hours. The mixture was then put in a water bath with stirring for half an hour and being left in poly poplin bottle.

**Scenario 2 (Cat2):** The same scenario was repeated but the 15% sodium hydroxide sample was replaced by the 15% potassium hydroxide sample.

The results of both scenarios were reported. All reported results shall be discussed in the next chapter.

### **3.3.2 Core Samples Preparation and Preliminary Measurements:**

**Step 1: Core Samples Cleaning:**

Three core samples were used in previous experiments and might be saturated with oil. Therefore, they were cleaned with toluene by Soxhlet device.



*Figure 3. 24 Soxhlet device used in core cleaning*

### **Step 2: Core Sample Drying:**

After cleaning, core samples were put in the oven at temperature from 100 to 120 °C to allow toluene to evaporate from pores. After 24 hours course were taken out from the oven and their weight was measured and then returned to the oven. The process was repeated until weight become constant.

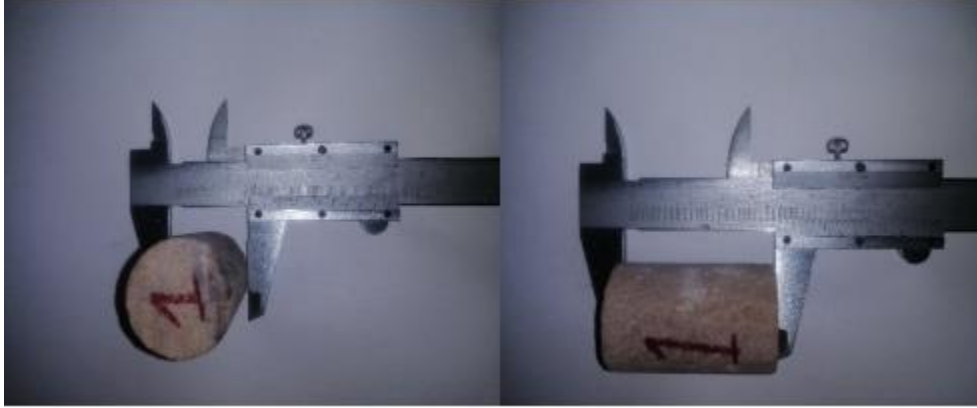


*Figure 3. 25 Cores Sample in Oven*

### Step 3: Core samples bulk volume measurement:

In order to measure the bulk volume, the following steps were carried out:

1. The length and radius were measured using vernier caliber.



*Figure 3. 26 Core samples dimensions measurement using vernier caliber*

2. Bulk volume for the three core samples was calculated using the following equation:

$$V_B = ((\pi / 4) * D^2) * L$$

Where:

$V_B$ : Bulk volume (cc).

$D$ : Diameter of core (cm).

$L$ : length of core (cm).

### Step 4: Core samples saturation in formation water:

The following steps were carried out to saturate the samples with formation water, as per

#### **Figure 3.27:**

1. Core sample and reservoir water were put into vacuum chamber.
2. Vacuum chamber was closed and coupled with the vacuum pump using rubber pipe.
3. The pump vacuum was run to allow air to move out from vacuum chamber and observing out the air moving from core sample pores.
4. The system was left to run for a long time to ensure of saturation the core sample by reservoir water.



*Figure 3. 27 Core samples reservoir water saturation process*

5. The wet weight of core samples was measured and reported.

**Step 5: Core samples drying form saturation water:**

1. The cores were then put into the oven at a temperature from 100 to 120 °C, as per **Figure 3.28**, to allow reservoir water and brine to evaporate from pores.
2. The dry weight was then measured to be used in the original oil in place calculations.



*Figure 3. 28 Core samples drying inside the oven*

**Step 6: Core samples saturation in formation oil:**

The following steps were carried out to saturate the samples with oil, as per **Figure 3.29**:

1. Core sample and reservoir water were put into vacuum chamber.
2. The vacuum chamber was closed and coupled with the vacuum pump using rubber pipe.
3. The pump vacuum was run to allow air to move out from vacuum chamber and observing out the air moving from core sample pores.
4. The system was left to run for a long time to ensure of saturation the core sample by oil.



*Figure 3. 29 Core samples oil saturation process*

#### **Step 7: Oil density measurement:**

Oil density was measured through the following procedure:

1. The weight of a pycnometer with known volume of 70 cc was measured when it is empty.
2. The weight was measured again when it was then filled with oil.
3. The density of oil was the calculated based on the difference in weights and the pycnometer volume using the following equations:

$$W_o = W_{pyc} - W_{pyco}$$

**Where:**

$W_o$ : Weight of Oil (g).

$W_{pyc}$ : Weight of pycnometer without oil (g).

$W_{PYCO}$ : Weight of pycnometer with oil (g).

$$\rho_o = W_o / V_o$$



**Where:**

$\rho_o$ : Density of the oil (g/cc).  $W_o$ : Weight of the oil (g).  $V_o$ : Volume of the oil (cc).



*Figure 3.30 Weighting the pycnometer with oil*

**Step 7: Samples OOIP measurements:**

1. Once saturated with oil, the three samples were taken out and placed on filter papers until the oil is removed from outside the samples.



*Figure 3.31 Core samples on filter paper*

2. The oil saturated core samples weight was measured to be used for OOIP measurements.



*Figure 3.32 Oil saturated core samples is being measured*

3. The OOIP was calculated and reported using the following equation of for three core samples.

$$N = W_{cs} - W_{cd} / \rho_o$$

**Where:**

**N:** Original oil in place (cc).

**Wcs:** Weight of oil saturated core sample (g).

**Wcd:** Weight of dry core sample (g).

**$\rho_o$ :** Density of oil (g/cc)

### 3.3.3 Wettability Measurement of Filtered Waters:

Wettability was evaluated using contact angle measurement through the following steps:

**Step 1:** Core samples were saturated with oil.

**Step 2:** Saturated core samples were submerged into three different solutions, filtered formation water, Cat1 and Cat2. Samples are shown in Figure

**Step 3:** Using a high-resolution camera, drop shots were taken for the three cases.

**Step 4:** Using image software and the drop shot the contact angle was measured for the three cases.

**Step 5:** Results of all cases were reported and will be shown in the next chapter.



*Figure 3.33 Wettability Measurements*



### 3.3.4 Spontaneous Imbibition Test:

1. Oil saturated core samples were placed inside the test tube.
2. The test tubes were filled with original reservoir water, Cat1 and Cat2 treated water until the core samples were completely submerged.
3. Test tubes orifices were closed using aluminum foil and paper tape in order to prevent oil evaporation.
4. The volumes of produced oil were measured through several steps at different temperatures starting from room temperature, 30 °C, 40 °C, 50 °C, 60 °C and 70 °C. At each step the samples were left for two days.
5. Oil recovery factor was calculated using the following equation:

$$Rf = NP/N * 100$$

Where:

**Rf:** Oil Recovery Factor (fra).

**NP:** Oil Production (cc).

**N:** Original Oil In Place (cc)

6. All recovery factor results were reported and shall be presented in the next chapter.

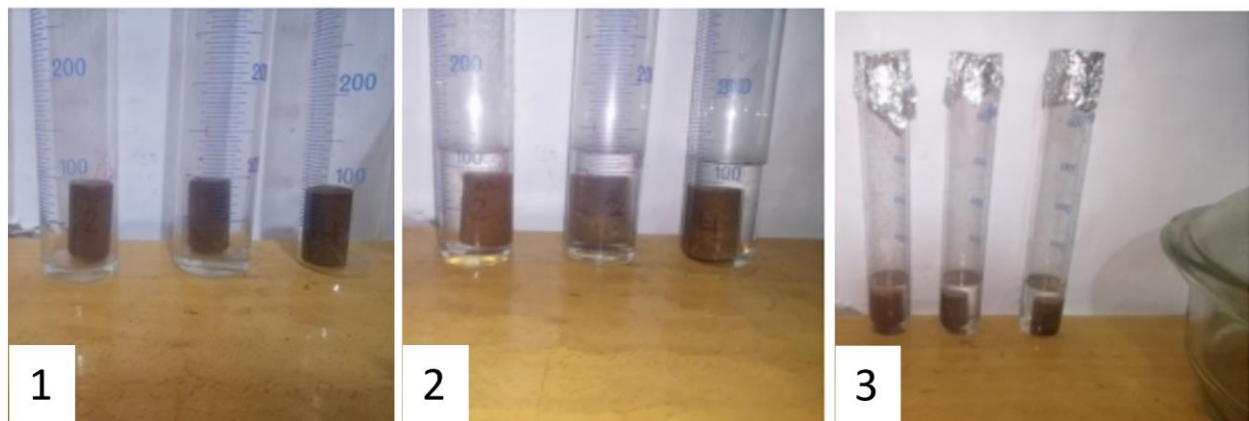


Figure 3. 34 Spontaneous imbibition test steps

## *Chapter Four*

# **DISCUSSION OF RESULTS**

#### 4.1 Formation Water Preparation Results:

The results of all of the experiments will be presented and discussed in this chapter.

Starting from Table 4.1 which shows the results of the original formation water sample after filtration. Tables 4.2 to 4.5 show the formation water with the addition of sodium hydroxide with a percentage of 5%, 10%, 15% and 20%.

Form those tables, the higher the percentage of sodium hydroxide the lower the percentage of TDS. are showing the water analysis for different water samples.

*Table 4. 1 The Parameters of Original sample*

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.23	-	ASTM D-1293
2	Electrical Conductivity , EC	13440	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	1500	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	750	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	750	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	300	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	182	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2700	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	155	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	8810	mg/L	Measured

*Table 4. 2 The Parameters of sample with NaOH (5%) (N1)*

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.21	-	ASTM D-1293
2	Electrical Conductivity , EC	12750	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	860	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	280	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	580	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	112	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	141	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2100	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	140	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	8450	mg/L	Measured

*Table 4. 3 The Parameters of sample with NaOH (10%) (N2)*

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.29	-	ASTM D-1293
2	Electrical Conductivity , EC	12120	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	310	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	120	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	190	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	48	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	46	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2275	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	120	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	7730	mg/L	Measured

*Table 4. 4 The Parameters of sample with NaOH (15%) (N3)*

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.29	-	ASTM D-1293
2	Electrical Conductivity , EC	12120	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	310	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	120	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	190	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	48	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	46	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2275	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	120	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	7730	mg/L	Measured

Table 4. 5 The Parameters of sample with NaOH (20%) (N4)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.75	-	ASTM D-1293
2	Electrical Conductivity , EC	12940	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	Nil	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	Nil	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	Nil	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	Nil	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	Nil	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	1975	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	705	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	6670	mg/L	Measured

Tables 4.6 to 4.9 show the formation water with the addition of potassium hydroxide with a percentage of 5%, 10%, 15% and 20%.

Form those tables, the higher the percentage of potassium hydroxide the lower the percentage of TDS. are showing the water analysis for different water samples.

Table 4. 6 The Parameters of sample with KOH (5%) (K1)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.41	-	ASTM D-1293
2	Electrical Conductivity , EC	13900	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	1250	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	600	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	650	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	240	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	158	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2500	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	320	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	6710	mg/L	Measured

Table 4. 7 The Parameters of sample with KOH (10%) (K2)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.23	-	ASTM D-1293
2	Electrical Conductivity , EC	13120	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	1000	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	5000	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	5000	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	200	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	122	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2400	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	380	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	6700	mg/L	Measured

Table 4. 8 The Parameters of sample with KOH (15%) (K3)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.51	-	ASTM D-1293
2	Electrical Conductivity , EC	12570	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	720	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	240	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	280	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	96	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	117	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2275	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	540	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	5870	mg/L	Measured

Table 4. 9 The Parameters of sample with KOH (20%) (K4)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.40	-	ASTM D-1293
2	Electrical Conductivity , EC	12650	μS/cm @ 25°C	AWWA 2510
3	Total Hardness	540	mg/L	ASTM D1126
4	Calcium Hardness as CaCO <sub>3</sub>	300	mg/L	ASTM D1126
5	Magnesium Hardness as CaCO <sub>3</sub>	240	mg/L	ASTM D1126
6	Calcium (Cs <sup>-1</sup> )	88	mg/L	ASTM D511
7	Magnesium ( Mg <sup>+2</sup> )	78	mg/L	ASTM D511
8	Sodium (Na <sup>+</sup> )	2050	mg/L	ASTM D-2791
9	Potassium (K <sup>+</sup> )	676	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	6030	mg/L	Measured

Table 4.10 shows the formation water with the addition of 15% sodium hydroxide, silica and aluminium. The sample was labelled as Cat1 and was later used for imbibition process.

Table 4.11 shows the formation water with the addition of 15% potassium hydroxide, silica and aluminium. The sample was labelled as Cat2 and was later used for imbibition process.

Table 4. 10 The Parameters of sample with NaOH + Silica+ Aluminum (Cat1)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	7.28	-	ASTM D-1293
2	Electrical Conductivity , EC	11350	μS/cm @ 25°C	AWWA 2510
8	Sodium (Na <sup>+</sup> )	2700	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	5430	mg/L	Measured

Table 4. 11 The Parameters of sample with KOH + Silica+ Aluminum (Cat2)

	PARAMETER	TEST RESULT	UNITS	METHOD
1	pH -Value @ 25 °C	8.27	-	ASTM D-1293
2	Electrical Conductivity , EC	12510	μS/cm @ 25°C	AWWA 2510
8	Potassium (K <sup>+</sup> )	500	mg/L	ASTM D-2791
10	Total Dissolved Solids (TDS)	6760	mg/L	Measured

## 4.2 Experiments Procedures Results:

Table 4.12 and Table 4.13 show the measurement results of core samples dimensions and bulk volume. Table 4.14 shows the volume of produced fluids in mL for the different samples at temperatures from 30 °C to 70 °C.

Table 4. 12 Core sample dimensions

Sample	D (cm)	L (cm)
1	2.5	3.2
3	2.5	3.3
4	2.5	4.3

Table 4. 13 Core samples bulk volume

Sample	Bulk Volume ( $\frac{\pi}{4} \cdot D^2 \cdot L$ )	Unit
1	15.70	cc
2	16.19	cc
3	22.08	cc

Table 4. 14 Produced volumes 1from core samples in (mL) at different temperatures

Temperature (°C)			20	30	40	50	60	70
1	Cat 2	KOH	1	1	1.5	1.5	2	2
2	Cat 1	NaOH	0.5	1	1	1.5	2	2.5
3	Formation Water	F.W	1	1	1	1.5	1.5	1.5

Table 4. 15 Measurements of OOIP in the different core samples using different formation waters

Original Oil in Place (STB)			
1	Cat 2	KOH	$\frac{38.45 - 34.20}{0.84} = 5.05$
2	Cat 1	NaOH	$\frac{52.05 - 48.25}{0.84} = 4.52$
3	Formation Water	F.W	$\frac{42.30 - 38.09}{0.84} = 5.01$



Table 4.16 shows the calculated recovery factor from each sample at different temperatures.

*Table 4. 16 Recovery factor for the 3 core samples using different formation water composition*

Temperature (°C)	Sample	RF
<b>30</b>	Cat 2 - KOH	$\frac{1}{5.05} \times 100 = 19.80$
	Cat 1 - NaOH	$\frac{1}{4.52} \times 100 = 22.12$
	Formation Water	$\frac{1}{5.01} \times 100 = 19.96$
<b>40</b>	Cat 2 - KOH	$\frac{1.5}{5.05} \times 100 = 29.70$
	Cat 1 - NaOH	$\frac{1}{4.52} \times 100 = 22.12$
	Formation Water	$\frac{1}{5.01} \times 100 = 19.96$
<b>50</b>	Cat 2 - KOH	$\frac{1.5}{5.05} \times 100 = 29.70$
	Cat 1 - NaOH	$\frac{1.5}{4.52} \times 100 = 33.18$
	Formation Water	$\frac{1.5}{5.01} \times 100 = 29.94$
<b>60</b>	Cat 2 - KOH	$\frac{2}{5.05} \times 100 = 39.60$
	Cat 1 - NaOH	$\frac{2}{4.52} \times 100 = 44.24$
	Formation Water	$\frac{1.5}{5.01} \times 100 = 29.94$
<b>70</b>	Cat 2 - KOH	$\frac{2}{5.05} \times 100 = 39.60$
	Cat 1 - NaOH	$\frac{2.5}{4.52} \times 100 = 55.30$
	Formation Water	$\frac{1.5}{5.01} \times 100 = 29.94$

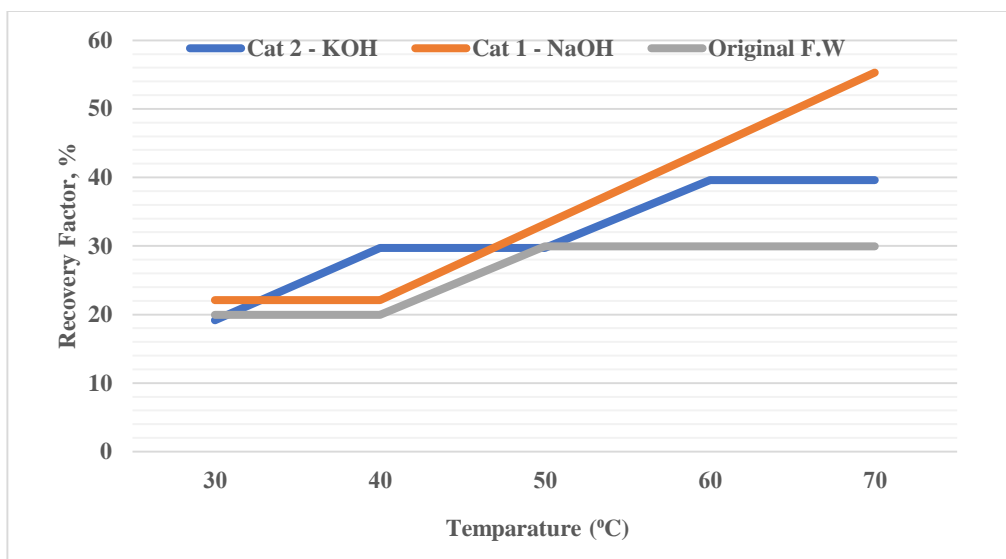


Figure 4. 1 Comparison between recovery factors using different compositions of formation water

Figure 4.1 shows the calculated recovery factor from each sample at different temperatures.

From the presented results chart, it is very clear that the core samples saturated with Cat 1 formation water exhibit a notably higher oil recovery factor at elevated temperatures compared to Cat 2 formation water and the original formation water.

However, at lower temperatures, Cat 2 formation water was performing better in the oil recovery compared to Cat 1 and original formation water. Additionally, the recovery factor for Cat 1 is still going higher, while Cat 2 and original formation water were stable in recovery.

## *Chapter Five*

# **CONCLUSIONS AND RECOMMENDATIONS**

## 5.1 Conclusions

In conclusion, the experimental findings presented in this research project shed light on the significant influence of adding chemicals to formation water on oil recovery. These are some of the conclusions:

- 1- For a sandstone core samples, adding Zeolite, sodium hydroxide, potassium hydroxide, silica and aluminum significantly improves oil recovery at higher temperatures.
- 2- The addition of sodium hydroxide had better oil recovery than potassium hydroxide.
- 3- Using 15% of sodium and potassium hydroxides gave better results than other percentages of 5, 10 and 20%.
- 4- Adding silica and aluminum to the combination of Zeolite, sodium and potassium hydroxides gave better results in terms of oil recovery.

## **5.2 Recommendations**

Based on this research project, these are some recommendations:

- 1- This research project was carried out for sandstone core samples. It is recommended that the experiments be performed for limestone core samples.
- 2- It is recommended to test more percentages and concentrations of sodium and potassium hydroxide and investigate the impact of those percentages and concentrations on the oil recovery.
- 3- It is recommended to test other ratios and quantities of silica and aluminium and investigate their impact on the oil recovery.
- 4- It is recommended to investigate the impact of oil properties on the oil recovery using the different chemicals. To do so, it is recommended to redo the experiments using different oil samples with different properties.
- 5- It is recommended to investigate the impact of original water formation properties on the oil recovery using the different chemicals. To do so, it is recommended to redo the experiments using different formation waters with different properties.

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