Palacký University Olomouc

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Department of Geology

Experimental investigation on the impact of smart water on the wettability alteration of reservoir formations in the Kurdistan Region

Bachelor thesis

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Petroleum Engineering (B0724A330002)

Fulltime study

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Olomouc, May 2023

Experimental investigation on the impact of smart water on the wettability alteration of reservoir formations in the Kurdistan Region

Anotace:

Použití metod zvýšené těžby ropy je nezbytné pro získání většího množství ropy, protože ropa je velkým zdrojem energie, který se v dnešní době nejčastěji využívá. Jedním z možných řešení, jak zvýšit těžbu ropy, je změna smáčivosti hornin ložiska snížením povrchového úhlu kontaktu ropy s pevnou látkou. Hlavním cílem této studie je změnit smáčivost povrchu horniny z povrchu smáčejícího ropu na povrch smáčející vodu snížením kontaktního úhlu pomocí inteligentního vodného roztoku solí KCl a NaCl v různých koncentracích s destilovanou vodou. Použité vzorky hornin pocházely ze čtyř útvarů: Bekhma, Qamchuqa, Pila Spi a Injana. K měření pórovitosti a propustnosti všech jádrových zátek byl použit porozimetr a permeametr. K určení složení vzorků hornin byla použita rentgenová difrakční analýza. Výsledky této studie ukázaly, že přídavek solí KCl a NaCl v různých koncentracích 5000 ppm a 10 000 ppm v různých formacích vedl k různému procentuálnímu snížení povrchového kontaktního úhlu mezi ropou a pevnou látkou. Podle výsledků vedl přídavek KCl v koncentraci 5000 ppm k výraznému snížení kontaktního úhlu o 10,5 % ve formaci Injana. Přídavek solí však neměl žádný významný vliv na kontaktní úhel formace Qamchuqa. Optimálního výsledku bylo dosaženo snížením kontaktního úhlu o přibližně 24 % při použití KCl v koncentraci 5000 ppm v jádrové zátce z formace Bekhma.

Klíčová slova: kontaktní úhel, propustnost, pórovitost, inteligentní voda, změna smáčivosti.

Počet stran: 40

Počet příloh: neni dostupny

Annotation

The application of enhanced oil recovery methods is necessary to produce a larger amount of oil as the oil is a great source of energy that is most commonly used nowadays. One of the potential solutions to increase oil production is the wettability alteration of the reservoir rocks by reducing the oil-solid surface contact angle. The primary objective of this study is to alter the wettability of a rock surface from oil-wet to water-wet by contact angle reduction by using a smart water solution of KCl and NaCl salts in different concentrations with distilled water. The used rock samples were brought from four formations: Bekhma, Qamchuqa, Pila Spi, and Injana. The porosimeter and permeameter were used to measure the porosity and permeability of all core plugs. The X-ray diffraction analysis was used to determine the composition of the rock samples. The results of this study showed that adding KCl and NaCl salts at different concentrations of 5000 ppm and 10 000 ppm in different formations resulted in different reduction percentages of the crude oil-solid surface contact angle. According to the results, the addition of KCl at 5000 ppm resulted in a significant 10.5% reduction in contact angle in the Injana Formation. However, the addition of the salts had no significant influence on the contact angle of the Qamchuqa Formation. The optimum result achieved was a reduction in contact angle by about 24% when KCl at 5000 ppm was employed in a core plug from the Bekhma Formation.

Keywords: Contact angle, Permeability, Porosity, Smart water, Wettability alteration.

Number of pages: 40

Number of annexes: not available

Declaration

I declare that I have prepared the bachelor's thesis myself and that I have stated all the used information resources in the thesis.

In Olomouc, May 4, 2023 Muhammad Kareem Hussein

Acknowledgement

I'd like to thank my supervisor "Dr. Jagar Ali" for providing assistance. The knowledge became essential when developing the thesis. His insightful feedback encouraged me to improve my approach and moved my effort to a higher level. I would like to thank all Palacky University faculty members from the Department of Geology, Petroleum Engineering program, especially Mr. Rebar Mahmmud, and Soran University for assisting me during my studies and research and for their invaluable assistance and support throughout the laboratory work. In addition, I would like to express my great appreciation to my parents, specifically my mother and father, for their sensible advice and unending support and for always being there for me in hard times. Finally, I could not have finished this without the help of my siblings and friends, who provided intellectual talks and pleasurable distractions from my study. Thank you all for inspiring me to do greater.

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List of abbreviations

Abbreviation	Meaning
API	American Petroleum Institute
Ca^{2+}	Calcium
C°	Celsius
CA	Contact angle
cP	Centipoise
DW	Distilled water
EOR	Enhanced oil recovery
KCl	Potassium chloride
KRI	Kurdistan Region of Iraq
LSW	Low-salinity water
Mg^{2+}	Magnesium
mS	milliSiemens
NaCl	Sodium chloride
pH	Potential of hydrogen
PPM	Parts per million
RPM	Revolutions per minute
SW	Smart water
SO_4^{2-}	Sulphate
XRD	X-ray diffraction

Chapter 1: Introduction and Objectives

1.1. Introduction

Enhanced oil recovery (EOR) technology innovation is one of the necessities in the petroleum industry and energy sector. The oil and gas sector needs to concentrate on developing new and emerging practical solutions to improve oil recovery from currently producing reservoirs. One of the possible approaches is to modify the wettability of reservoir rocks in order to isolate more oil from reservoir rocks and increase the recovery factor (Omidi et al., 2020).

There are three main production phases or stages in the production of hydrocarbons over the lifespan of an oil and gas reservoir: primary, secondary, and tertiary. During the primary and secondary phases, reservoir pressure drops substantially. As a result, several strategies have been suggested to maintain pressure and slow down the rate of pressure loss during this stage. On the other hand, water injection is a well-known technique for increasing the recovery of carbonate and sandstone reservoirs worldwide. There has been a huge surge in research efforts to improve oil recovery from hydrocarbon deposits using smart water (SW) injection in recent years. This approach has been linked to positive field and research results in carbonate and sandstone reservoirs. Water injection has been suggested to be the most successful method of improving oil recovery (Rafiei and Khamehchi, 2021). Among the water injection methods, SW injection is considered one of the most cost-effective and efficient techniques for enhanced oil recovery in carbonate and sandstone reservoirs. One of the active methods of SW to rise the production of oil is the alteration of the wettability of the surface of the rock from oil-wet to water-wet conditions (Bahri and Khamehchi, 2021). Similarly, Nowrouzi et al. (2019) reported that SW injection is a control approach, which means the main idea is to control, regulate, and manage the ions in the injection stage to activate the mechanisms of this technique, particularly the wettability modification of formation rock from hydrophobic to hydrophilic states.

As stated in literature, carbonate reservoirs constitute half of the world's oil reserves. Due to difficulties such as poor permeability of the rock, mixed wet to oil-wet conditions, high natural fracture density, and inhomogeneous rock, oil recovery from these reservoirs is challenging (Bahri and Khamehchi, 2021). There are several methods for obtaining oil from these reservoirs. One of the major approaches is to vary the wettability of the water to affect the capillary pressure and relative permeability. The smart water injection is a decent approach for this purpose. Apart from the alteration of wettability, the rate of changing the wettability is an essential parameter in the technique (Rafiei and Khamehchi, 2021).

Reservoir formations, such as Bekhma, Injana, Pila Spi, and Qamchuqa formations, are great formations in the Kurdistan Region of Iraq (KRI) (Omidi et al., 2020). These formations have been selected as investigation cases for this study. The primary objectives of this study are to explore wettability alteration and investigate the steps of wettability alteration by measuring the contact angles of the crude oil on the surfaces of sandstone and carbonate rocks.

1.2. Research objectives

The primary objectives of this study are to study the influence of dissolved ions on wettability alteration and to measure the porosity and permeability of the formation rocks. Crude oil contact angles on the surfaces of sandstone and carbonate rocks retrieved from the Bekhma, Injana, Pila Spi, and Qamchuqa formations are measured for determining wettability.

Chapter 2: Background and Literature Review

2.1. Petroleum reservoir

Oil and gas reservoirs are often situated in sedimentary rocks. Oil and gas, or hydrocarbons in general, can be found in relatively rare circumstances, such as fractured and broken igneous rocks or cracked metamorphic rocks. Both igneous and metamorphic rocks develop under extreme temperature and pressure settings that do not encourage the formation of hydrocarbon reserves (Fanchi, 2002).

Hydrocarbons are not found everywhere on Earth. Oil and natural gas are frequently encountered in petroleum reservoirs. While speaking of reservoir formation and fluids, there are several features and characteristics to consider. These features and characteristics are both of the fluid and the reservoir rock. Lithology, rock permeability, rock porosity, total compressibility, and pay zone thickness are some of these features and characteristics. The above variables and features have an impact on both fluid circulation inside the reservoir and productivity. These features should be studied and investigated extensively as understanding these features and characteristics by the reservoir engineers is crucial for reservoir dynamics simulation and well productivity anticipation. Open-hole logs are usually used to measure the pay zone thickness of a reservoir. In general, a "reservoir" is a pool or deposit of petroleum in porous rock formations buried many feet beneath or subsurface. The term “rock” in a reservoir refers to the natural container that maintains or holds the petroleum distributed out in the pore spaces of the rocks from the position of a reservoir. The term "fluid" refers to anything that flows from a subsurface location to a surface location along a gradient, including gaseous or liquid hydrocarbons (Dandekar, 2013). The hydrocarbon reservoir rock in combination with the reservoir fluids form a system with a specific area and depth which exist at specific pressure and temperature conditions and is explored and exploited economically to produce petroleum.

The most difficult issue for etroleum exploration companies and petroleum producers today and in the future is to substantially increase hydrocarbon recovery from previously identified reservoirs. The compilation of thorough reservoir descriptions, profiles, and characteristics of reservoirs is critical to reaching this aim. A reservoir description provides a complete representation of the three-dimensional (3-D) distribution and continuity of the reservoir and aquifer system's rocks, pores, and fluids, including fluid flow obstacles (Ginsburg et al., 1990). As shown in Figure 1, a petroleum reservoir contains oil, gas, water, and rocks.

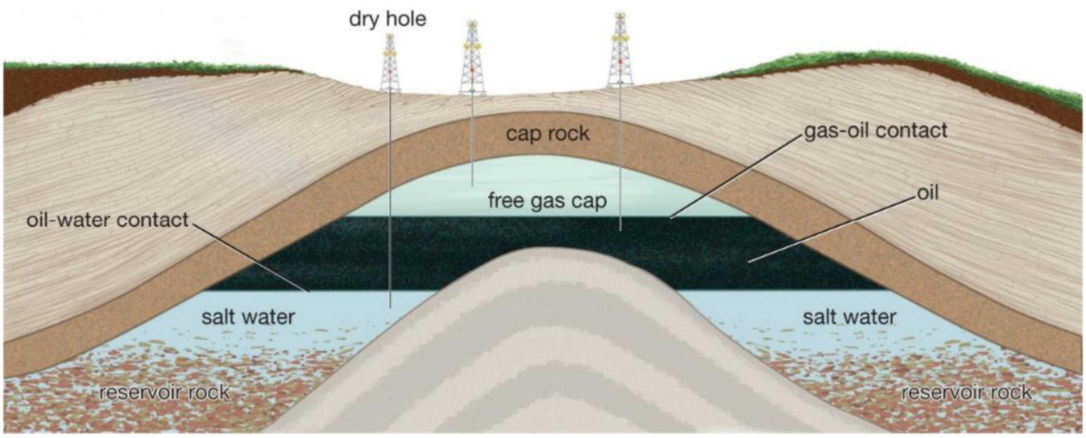


Figure 1 Petroleum reservoir (Druetta et al., 2016).

2.2. Reservoir rock properties

2.2.1. Porosity

A measure of the quantity of void space in a rock is referred to as porosity. This void fraction might exist between particles, as well as within soil or rock holes. Porosity can be expressed as a percentage from 0% to 100% and can be calculated using Equation 1 (Guo, 2019).

$$\phi = \frac{V_{pore}}{V_{bulk}} \quad (1)$$

where;

ϕ: porosity

V_{pore}: pore volume, cm^3

V_{bulk}: bulk volume, cm^3

Porosity in most rocks typically ranges from less than 40%. Permeable rock is a critical component of oil and gas reservoirs. Fluids can be held in porous rocks. Normally, oil and gas are produced from source rocks, moving upwards, and trapped behind sealing layers or impermeable rocks that prevent oil and gas from escaping to the surface (Ganat, 2020). Porosity can also be defined as the reservoir rock's ability to hold fluids. The fluids in the reservoir rocks' pore spaces might be gas, oil, or water. High porosity values indicate that the reservoir's rocks can hold a large percentage of fluids. On the other hand, low porosity values indicate that the reservoir cannot hold a large amount of fluid. Therefore, porosity measurements are commonly utilized to analyze and anticipate the potential amount of hydrocarbon held in a reservoir (Tiab and Donaldson, 2004).

Porosity measurements of retrieved core samples or well logs are utilized to obtain data on the porosity of the reservoir rock. Obtained porosity data from core samples are typically used to evaluate or calibrate porosity data obtained from well logs. Porosity data is used in reservoir characterization for categorizing lithological facies as well as permeability assignment using porosity-permeability transforms. Therefore, porosity data is critical in many reservoir engineering calculations (Wheaton, 2016). Figure 2 shows that when there is material in the rock filling up the gaps and binding the grains together, there is less pore space and this indicates that the porosity is low. Therefore, the greater the space between the grains, the higher the porosity.

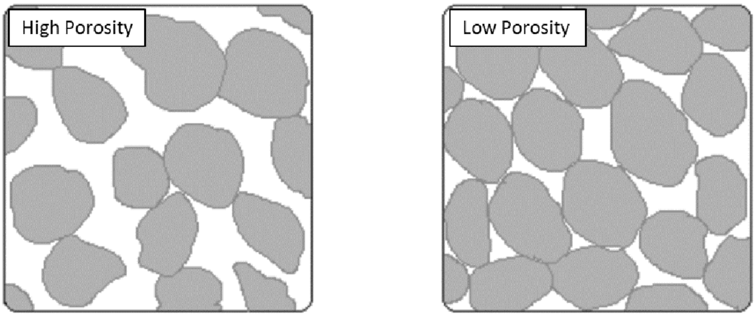


Figure 2 Diagrams demonstrating good porosity and poor porosity (Höök, 2010).

As Tiab and Donaldson (2004) reported, according to engineering classification, there are two classifications of porosity: effective porosity and absolute porosity.

- Effective porosity

The interconnected pores in a rock that allow the flow of fluids constitute effective porosity. Numerically speaking, effective porosity is smaller than absolute porosity, the intercommunicating porosity.

- Absolute porosity

Absolute porosity is defined as the ratio of pore volume to bulk volume. The void ratio is determined by dividing the pore volume by the volume of the solids. Isolated pores are commonly formed owing to the complex internal pore structure in rocks. The isolated pores constitute a component of the total rock porosity. Despite the fact that they make a portion of the total rock porosity, isolated pores are isolated and segregated from the primary body pores and are not associated with the fluid flow mechanism in the reservoir rock.

2.2.2. Permeability

Permeability is one of the important rock properties mentioned above. Permeability is a property of porous medium that describes and reflects the ability of the formation rock to allow the transportation and flow of fluids through it. The rock permeability, k, is an important rock attribute because it controls and regulates the direction and rate of flow of reservoir fluids in the formation rock.

Henry Darcy described this rock characterization quantitatively for the first time in 1856. The equation that he invented which quantifies permeability in terms of measurable quantities is called Darcy's Law. Darcy's fluid flow equation is now the standard mathematical tool for petroleum engineers (Toth and Bobok, 2017).

Fluid can be transmitted through permeable rock. A viable reservoir must be permeable for petroleum to be retrieved. A seal must be mostly impervious to petroleum. The degree to which fluid may be transported is measured by permeability. The darcy (D) is the unit of permeability, while the permeability of many reservoirs is measured in millidarcy (mD). There are three types of permeability: absolute, effective, and relative (Gluyas and Swarbrick, 2004). Figure 3 shows good permeability where the pores are connected (left) and poor permeability where not all the pores are connected (right).

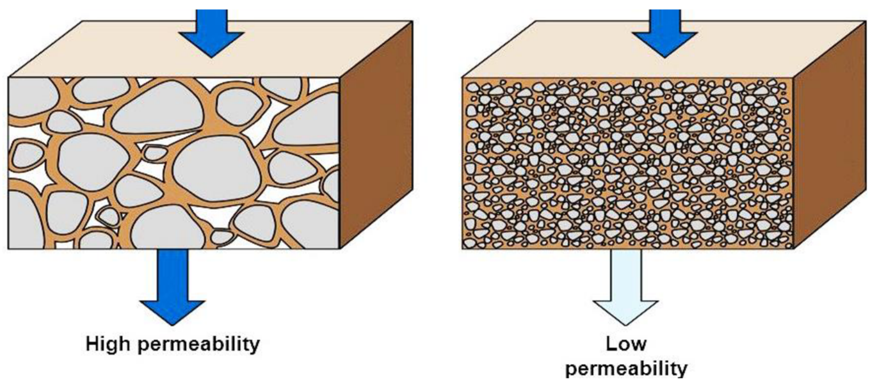


Figure 3 Diagrams illustrating high permeability and low permeability (Malo, 2023).

2.2.3. Fluid saturation

Fluid saturation is defined as the ratio of fluid volume in a particular core sample to the pore volume of the sample. Saturation can also be defined in other words as the fraction of connected pores, or apertures, filled by a specific phase. The pore spaces may contain and hold oil or gas. The saturation of a phase is the percentage of pore volume taken up by that phase. In addition to hydrocarbons, the pores in reservoir rock can hold water (Satter and Iqbal, 2016). Fluid saturation for each phase could be determined using Equations 2-4.

$$S_w = \frac{V_w}{V_p} \quad (2)$$

$$S_o = \frac{V_o}{V_p} \quad (3)$$

$$S_g = \frac{V_g}{V_p} \quad (4)$$

where S_w is water saturation, V_w is water volume, V_p is pore volume, S_o is oil saturation, V_o is oil volume, S_g is gas saturation, and V_g is gas volume.

Owing to the fact that pores can hold oil, gas, and water, only knowing the porous volume is insufficient to determine how much oil and gas are present in the formations. It is required to determine the percentage of the porous volume being occupied by each fluid (Speight, 2017).

The S_w is critical for reservoir analysis, and any erroneous estimate will almost certainly exacerbate the problems (Li et al., 2021). The number of fluids in the pore space in a formation is always greater than one. In light of this, it is noteworthy to state that in an oil reservoir, oil and water occupy the void space; however, in a gas reservoir, the void space is occupied by gas and water. Furthermore, a combination of oil, water, and gas might fill and occupy the pore space of an oil reservoir at some time during its production. The petrophysical characteristic of fluid saturation describes the amount of each fluid type in the pore space (Speight, 2017).

It can be seen in Figure 4(a) that the water is in a physical contact with and covered the rock in the water-wet reservoir. However, Figure 4(b) depicts an oil-wet reservoir, where the oil meets with the rock's surface.

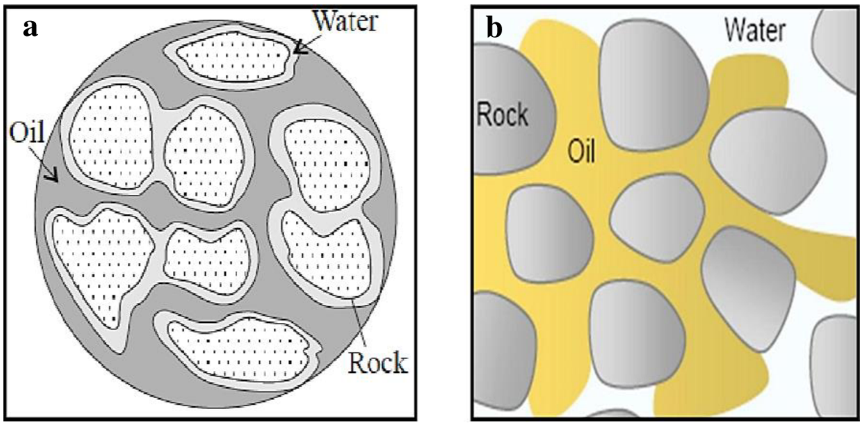


Figure 4 Fluid saturation: a) water-wet reservoir and b) oil-wet reservoir (Sylvester and Bibobra, 2014).

2.2.4. Capillary pressure

Capillary pressures originate and develop in reservoir rock pores (capillaries) where interactions and contacts of pressure between two immiscible fluids occur. One of the phases is recognized simply as the wetting phase, and the other as the non-wetting phase.

Yet, there are certain intermediary instances that could complicate the situation further. The drainage case in which a non-wetting phase substitutes a wetting phase pertains to hydrocarbon rushing into brine-saturated rock. Imbibition data, which is the inverse of drainage data, represents the displacement of a non-wetting phase by a wetting phase. Consequently, drainage data may be employed to estimate non-wetting phase saturation across multiple sites in a reservoir. Imbibition data, on the other hand, can be employed to evaluate the relative contributions of viscous force and capillary force in dynamic systems. (Fernø et al., 2010). Capillary pressure is frequently used in reservoir simulation programs in order to determine the fluids' initial distribution. Another application of capillary pressure is in fractured reservoir flow models where it is used to control the flow of fluids between the fracture and the rock matrix. The capillary pressure notion is frequently employed in flow equations to ease handling phase pressures and the variations in phase pressures (Fanchi, 2006).

2.2.5. Wettability

When two immiscible fluids come into contact with the surface of a solid, one phase tends to stick to the solid surface more than the other. This is owing to the intermolecular tensions and surface energy of immiscible fluids and solids being balanced. Wettability is defined as the measurement of the degree to which two fluids stick to a solid surface. In other words, the degree to which one fluid preferentially spreads to the surface of a porous medium holding two or more immiscible fluids is known as wettability (Schön, 2011).

According to Schön (2011) and Tiab and Donaldson (2012), there are three main types of wettability: water-wet, oil-wet, and intermediate wettability.

i. Water-wet: the water covers the rock or mineral's surface, with the oil and gas filling the largest pores in the center.

ii. Oil-wet: Compared to a water-wet, the relative positions of the oil and water are reversed; the oil covers the rock's surface and the water is in the center of the largest pores.

iii. Intermediate wettability: The term "intermediate wettability" (ii) describes reservoir rocks that encourage water and oil to adhere to the pore surface.

As shown in Figure 5, the contact angle that is less than 90 degrees indicates that the surface is wettable; it is water-wet. However, when the angle is more than 90 degrees, the solid is more difficult to wet; it is oil-wet.

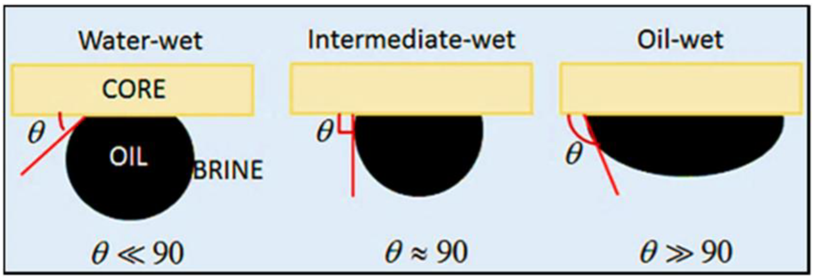


Figure 5 Schematic representation of the theory behind the estimation of the wettability behavior of reservoir rock when two immiscible phases exist (Omidi et al., 2020).

Water will fill the smaller holes and wet the majority of the surfaces of the bigger pores in a water-wet brine-oil-rock system. In regions with high oil saturation, the oil floats on a thin layer of water that spreads out throughout the surface. Water will seep into the tiny pores if the surface of the rock is preferentially and the rock is saturated with oil, displacing oil from the core as the rock system is exposed to water. Even if the rock is entirely oil-wet, the core will absorb oil into the microscopic pores, dislodging water when it comes into touch with it. Consequently, if an oil-saturated core takes in water, it is water-wet, and if it aims to absorb oil, it is oil-wet. The spectrum of wettability of a system can range from strongly water-wet to strongly oil-wet depending on the water-oil interactions with the surface of the formation rock (Tiab and Donaldson, 2012).

Dong et al. (2019) provide an insightful example of the effect of wettability on fluid transfer in pore spaces. For instance, in the example shown in Figure 6A, a water coating that develops on the water-wet proppant particles' surface can create an interconnecting water flow channel and reduce water flow resistance. On the other hand, the oil phase can be broken up into droplets and left in the pore mouths intermittently. Hence, water-wet proppants can increase water mobility while decreasing oil movement. Similar to this, when proppants are oil-wet, an interconnecting fluid channel of the oil phase might emerge at the surface of the proppants (Figure 6B). As a result, it promotes oil flow while making it more difficult for water to move through the oil-wet proppants pack.

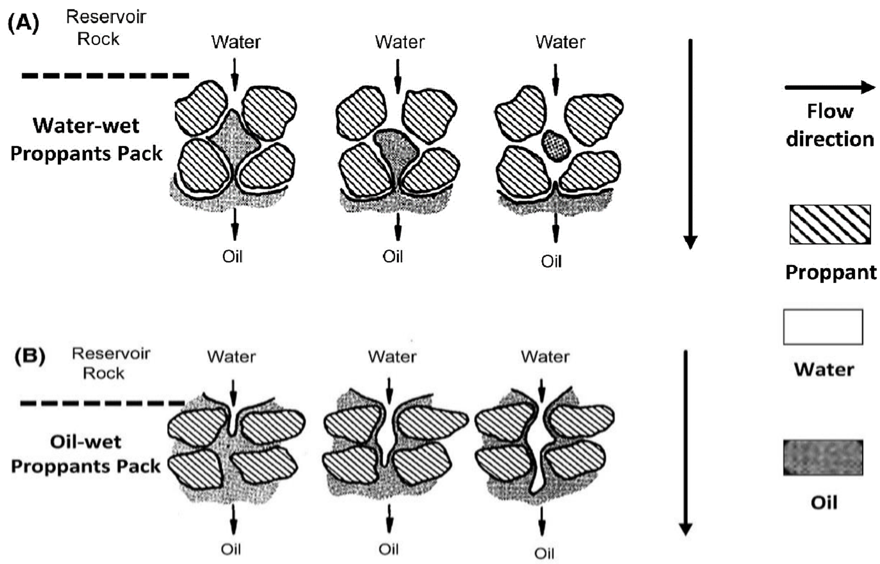


Figure 6 Fluid transfer inside the hydraulic fractures filled with (A) water-wet proppants and (B) oil-wet proppants (Dong et al., 2019).

2.3. Wettability alteration under the influence of smart water

The technique of making reservoir rock more water-wet is referred to as wettability alteration. This is particularly valid for carbonates that are naturally hydrophobic, fractured reservoir rocks, and heavy-oil systems. This change in wettability enhances oil recovery in oil-wet or weakly water-wet reservoirs, boosting total oil recovery. There are several methods to change wettability. Thermal and chemical methods are two main methods that have been used to alter wettability (Yao et al., 2021).

Wettability has progressively been recognized as a phenomenon influenced by numerous minor environmental variables within the pores of rocks. The recognition and study of wettability began in the 1930s. As its implications on production became increasingly apparent, several features of wettability were researched utilizing the beneficial differences in capillary pressure behavior (Fernø et al., 2010; Deng et al., 2019).

Utilizing carbonate rocks and low salinity water (LWS) injection determined that wettability changes occur as a result of rock dissolution. In a study, Derkani et al. (2018) reported that when granite is subjected to high temperatures, it returns to its pre-disturbed form of water of saline formation. When the system is subjected to a low voltage, the bivalent ions (Ca^{2+}, Mg^{2+}) present in the salinity environment tend to re-establish their positions, and the rocks seek to relocate towards the brine equilibrium. Meanwhile, the polar ends of crude oil adhering to the carbonate surface are released, enhancing the crude oil's solubility in the saline around it. As a result, oil recovery improves.

The features of rocks, minerals, pore structure, pore geometry, pore surface chemistry, and pore size, among other things, have been identified as essential factors for recognizing wettability and altering it to improve oil recovery (Donaldson and Alam, 2008). As shown in Figure 7, altering the wettability of the surface of a rock from oil-wet to water-wet results in a reduction in adhesive forces of capillarity and leads to an increase in the reservoir's oil permeability (Gbadamosi et al., 2019).

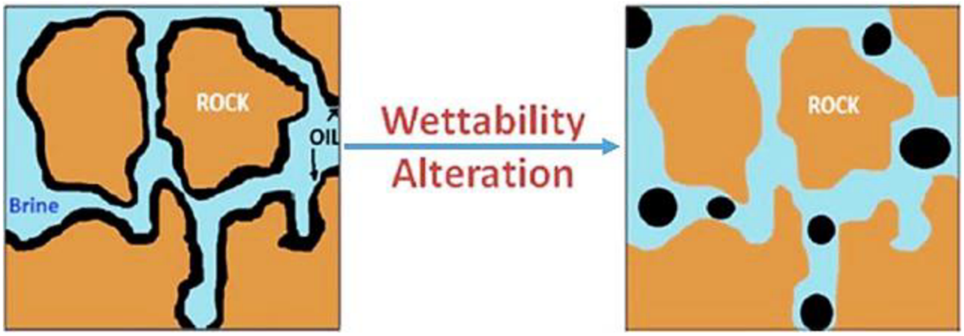


Figure 7 Improved permeability due to wettability alteration of rock from oil-wet to water-wet (Gbadamosi et al., 2019).

2.4. Impact of dissolved ions on wettability alteration

In reservoirs made of carbonate and sandstone, smart water injection is a popular and affordable EOR technique. By modifying water chemistry with salinity variations and composition optimization, we may obtain the ideal smart water composition. In a study by Rafiei and Khamehchi (2021), optimized SW has been proven to have a stronger impact on wettability alteration compared to other compositions. The findings of their study also showed how distributed nanoparticles affected wettability changes in different SW compositions. The goal of injecting SW can be affected by the incompatibility of

SW and formation water. Water containing the least scale due to incompatibility shows the greatest impact on wettability alteration. They also found out that SW composition that was incompatible with the formation water led to a reduction in the wettability alteration.

As a low-cost EOR method, SW application in carbonate reservoirs has recently attracted the attention of researchers. For the first time, the behavior of multiple smart water samples was examined affected the change in wettability (Shirazi et al., 2019). In another study on SW, Manshad et al. (2017) reported that in order to balance the calcium level, calcium carbonate is dissolved in the brines. More calcium ions are released and linked to the carboxyl groups when the rock is dissolved, resulting in the production of more oil. These techniques encourage oil separation from carbonate surfaces and improve oil efficiency. The type of ions used in this technique defines how SW changes wettability. The ions Ca^{2+} and SO_4^{2-} have a greater impact on wettability alteration. When SO_4^{2-} ions lower the number of the positive charges on the rock surface, cations can get closer to the adsorbed carboxylic acids. Mg^{2+}, a bivalent cation, increases the ability of other positive ions to remove carboxylic acids off the surface of rocks when added to smart water.

The mechanism for changing the wettability of SW from carbonate rock that is oil-wet to water-wet is schematically depicted in Figure 8. According to the results of Habibi et al., (2020), bivalent cations and adsorbed acids will come into contact with the rock surface more readily with the addition of SW. As a result, the wettability will alter from hydrophobic to hydrophilic. With the dissolution of carbon dioxide in smart water, solution acidity plays a key role in the breakdown and dissolution of carbonate minerals and acidification of the rock surface.

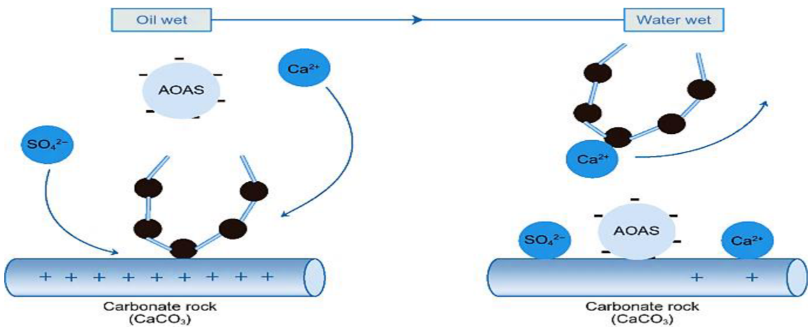


Figure 8 The schematic of the wettability alteration mechanism for smart water with nanofluid (Habibi et al., 2020).

2.5. Stratigraphic Column in Kurdistan Region

The stratigraphic column in the KRI remained unchanged over a lengthy period. However, the stratigraphic column was altered later after a large number of sedimentological and stratigraphical studies of northeastern Iraq had been done. The primary changes involved eliminating all prior unconformities between the subsequent formations: Qamchuqa with Dokan and Kometan, the latter with Dokan and Gulneri, Kometan and Shiranish, Qamchuqa and Bekhma, and Tanjero and Kolosh. The contacts between the aforementioned formations are now conformable, and the new chronostratigraphic column designates their boundary as gradational. Furthermore, the Pila Spi Formation, which is separated from the Walash Naoperdan Series by a paleo-high, is correlated with and indicates the tectonic and geographic location of the series during the Eocene. Instead of using Iraq's prior broad geographic location, the new chronostratigraphic column placed emphasis on the stratigraphic units' locations in relation to the country's recent tectonic zones (the Low and High Folded, Imbricated, and Thrust Zones) (Karim, 2010). Figure 9 shows the recent stratigraphic column of the KRI.

During the Cretaceous, the Arabian Platform's tectonic and depositional history underwent extensive research, particularly the area that is located in the Kurdistan Region, Northeastern Iraq. Karim and Taha (2009) reported that Bellen et al. in 1959, demonstrated several unconformities between the formations using a chronostratigraphy column. On the basis of breaks in the sedimentation and tectonic stages, Buday (1980) has categorized the rocks of the Late Cretaceous into a number of cycles and subcycles.

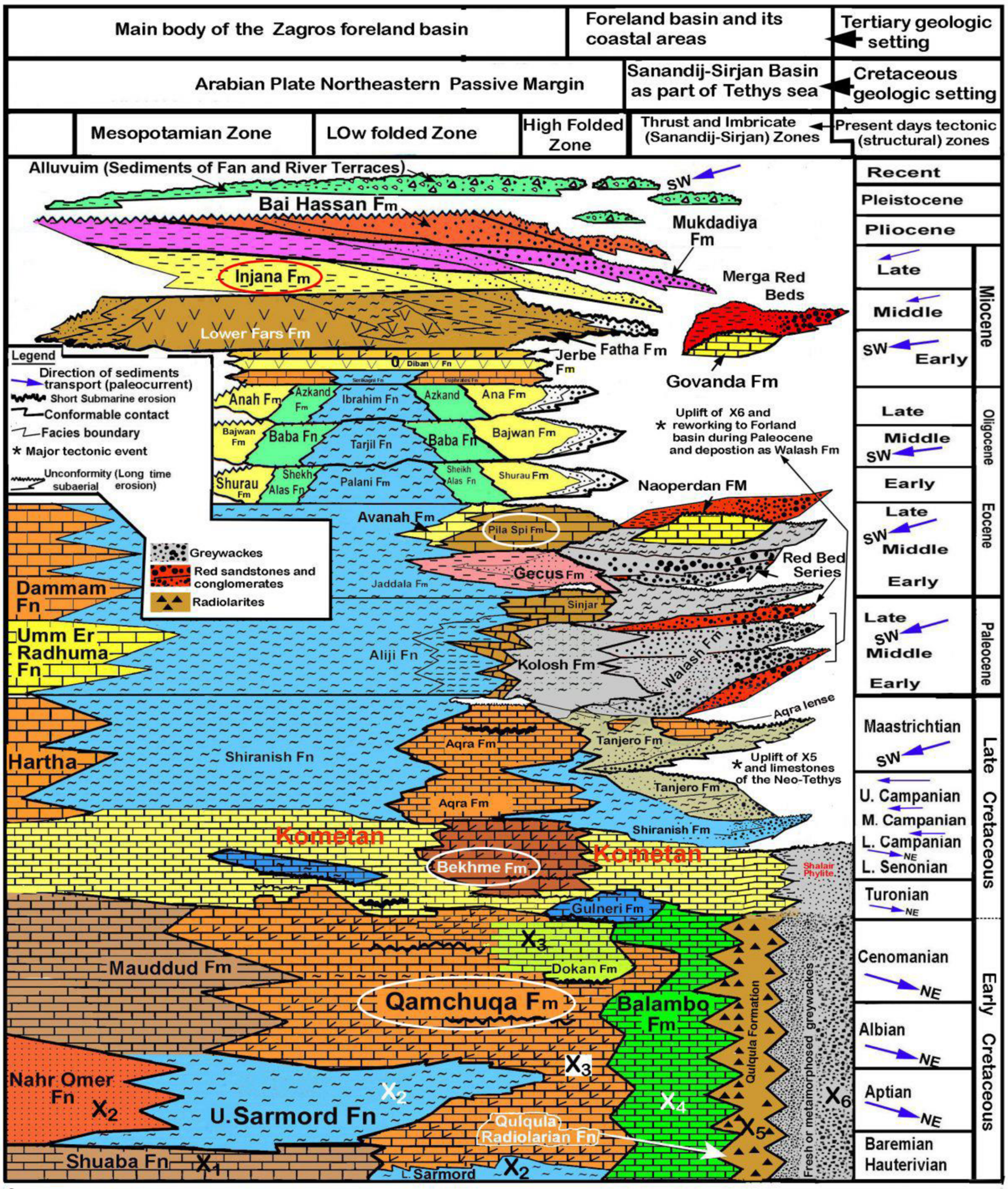


Figure 9 Stratigraphic column of Kurdistan Region (Karim, 2021).

i. Bekhma Formation

The Bekhma Formation was formed during the Late Campanian-Maastrichtian cycle. The cycle begins with a broad violation, practically encompassing the whole country, that happened following the conclusion of the upheaval induced by Cretaceous orogeneses in the middle Cretaceous. The cycle is complete. This was followed by another ascent and regression induced by the paroxysmal Laramide Orogeny stages along the Cretaceous-

Tertiary boundary. The Bekhma Formation (Figure 10) is one of seven other formations: Hartha, Aqra, Tayarat, Tanjero, Hadina, Shiranish, and Digma (Al-Mutwali et al., 2008).

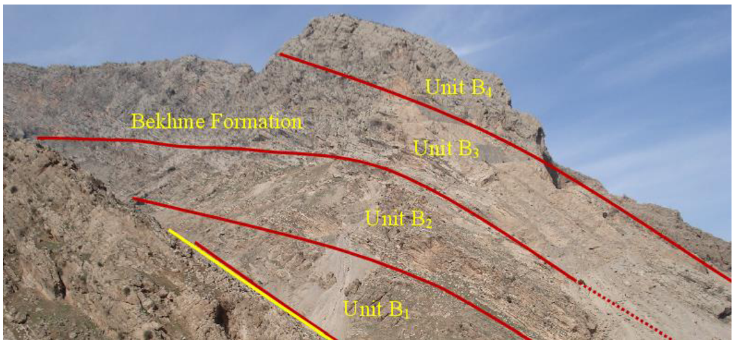


Figure 10 Bekhma Formation and lower contact of the Bekhma Formation (Ali, 2010).\

ii. Qamchuqa Formation

Both the outcrop in north-northeast Iraq and the foothill zone in southern Iraq are extensively covered by the shallow-water carbonate known as the Qamchuqa Formation. The Qamchuqa Formation is widely and clearly exposed in northeast Iraq's High Folded Zone. In the lower south of the High Folded Zone, Qamchuqa has thick reservoir units and is strongly dolomitized. The Early Cretaceous Neo-Tethys Ocean shelf limit is where Qamchuqa was deposited. It merges south- and southeastward to the Arabian shallow marine shelf and deep basin accumulations further north (Ghafur et al., 2020). Figure 11 shows the Qamchuqa Formation in the KRI.

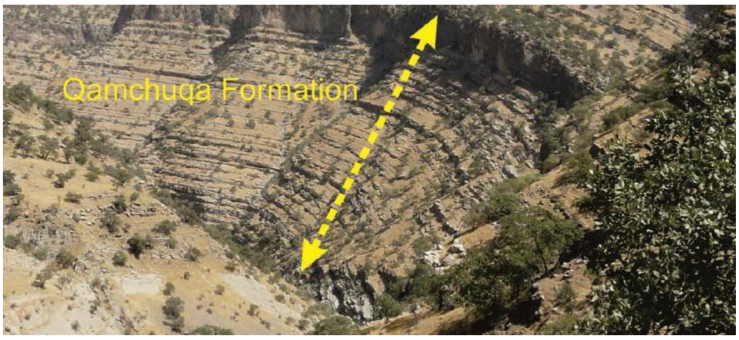


Figure 11 The Qamchuqa Formation that forms high cliffs (more than 1000 m thick) in the Barzan Area close to Dore Village (Ahmed et al., 2015).

iii. Pila Spi Formation

The Pila Spi Formation is reported to be made of dolomite, dolomitic limestone, and scarce marl and limestone. As a result of the high dolomite component, the bulk rocks of Pila Spi cannot be used for cement production (Sissakian et al., 2019). Figure 12 depicts the Pila Spi Formation in Duhok City.

Figure 12 Two symmetrical oval valleys in the Pila Spi Formation in the vicinity of Sharya village on Zawa Mountain, south of Duhok (Karim and Hama, 2019).

iv. Injana Formation

Injana Formation (Upper Miocene) in Zawita, Amadia, and Zakho localities. The Injana Formation sandstone is divided into two types: litharenite and feldspathic litharenite. Because the Injana Formation's rock fragments are mostly sedimentary, the sandstones present are classified as sedarenite and, more specifically, chertarenite due to the predominance of chert rock fragments (Tamar-Agha and Salman, 2018). Figure 13 shows Injana Formation in the Kurdistan region.

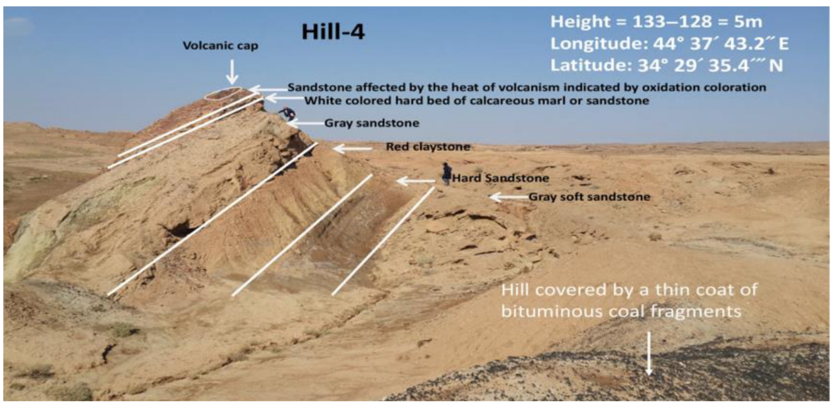


Figure 13 Field view of the Injana Formation in the Kurdistan region (Kettanah and Abdulrahman, 2022).

Chapter 3: Materials and Methodology

3.1. Materials

i. Reagents

Purified chemical regents, including methanol, ethanol, toluene, and salts, sodium chloride (NaCl), and potassium chloride (KCl), were purchased from Merck Company. In addition, crude oil was collected from Tawkee Oilfield having a density of 0.87 g/cm^3 (32.6° API) and viscosity of 13.32 cP. Tawkee Oilfield is located in north of Duhok City by 50 km in the KRI. The crude oil's density and viscosity were measured using the Anton-Paar density meter and Brookfield viscometer, as shown in Figure 14. The density, API gravity, and viscosity values of the crude oil are shown in Table 1.

Table 1 Properties of the crude oil.

Properties	**Value**
Density (g/cm^3)	0.877
API (°)	32.6
Viscosity (cP)	13.32

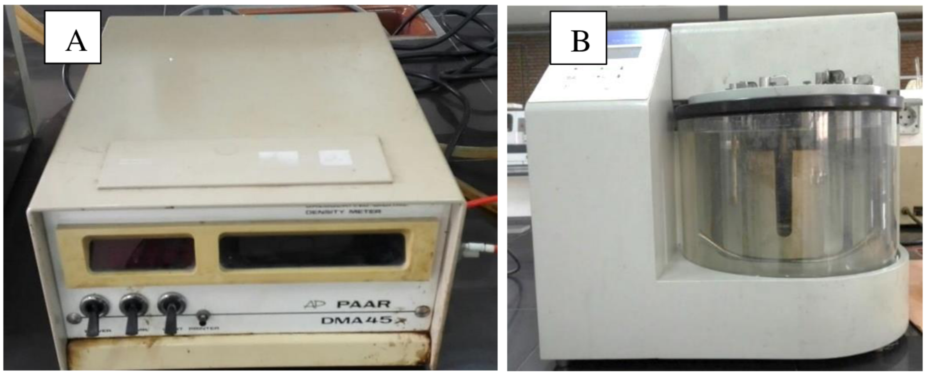


Figure 14 (A) Anton PAAR DMA45 density meter, (B) Vst 2400 kinematic viscometer.

ii. Rock samples

The study areas included four different locations which are Bekhma, Hujran, Spilk, and Amedi as shown in Figure 15 and 16. The outcrops are spread across large areas in the KRI. Outcrop cores were taken horizontally in these study areas and from the formations located there, including the Bekhma Formation in Bekhma District-

Erbil with coordinate of (36°40'12"N 44°13'57"E), the Qamchuqa Formation in north Hujran, Shaqlawa District-Erbil (36°24'43.7"N 44°15'20.0"E), the Pila Spi Formation in Spilk District-Erbil with coordinate of (36°36'49.6"N 44°18'15.2"E), and the Injana Formation in Amedi District-Duhok (37°03'20"N 43°31'55"E). The mineralogy of the collected rock samples was studied using X-ray diffraction (XRD) analysis.

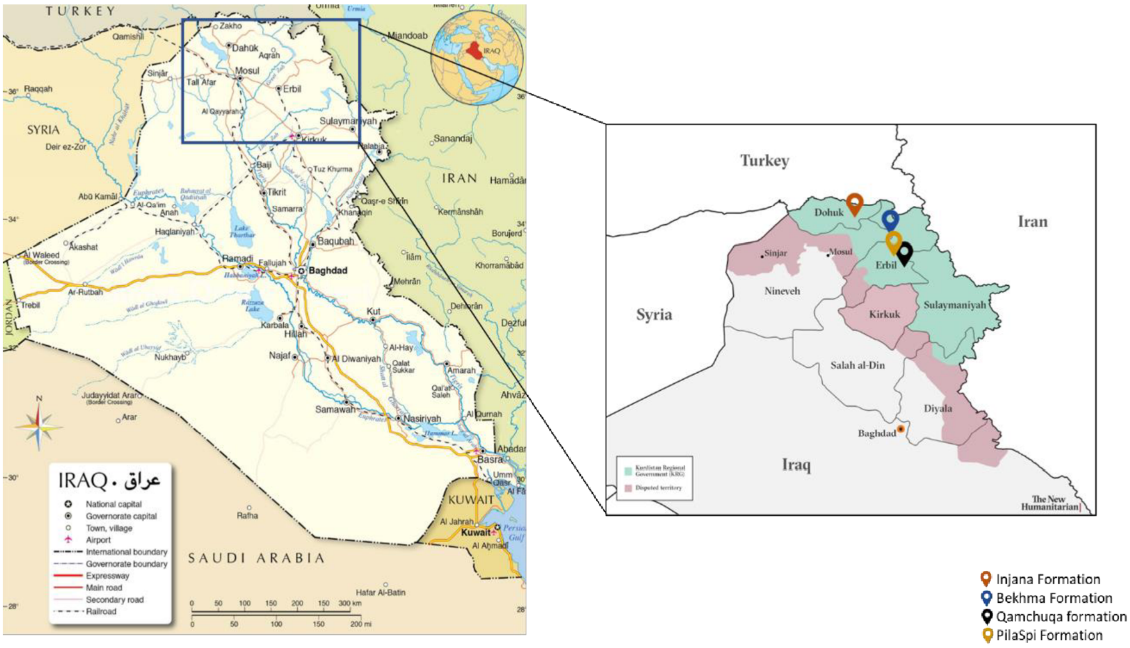


Figure 15 The map of the formations under study in the Kurdistan Region - Iraq.

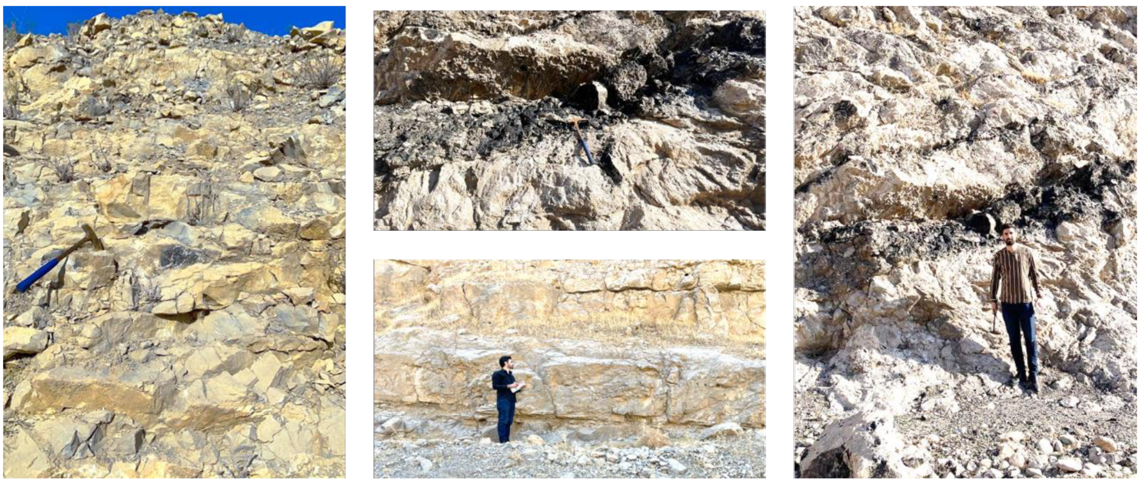


Figure 16 Taking cores from outcrops.

3.2. Core plug preparation

The core plug samples were prepared by using a core plugger. The core plugger drills the plugs to different sizes. The core plugs were then trimmed by the core saw trimmer for obtaining good shapes. A ruler was employed to measure the dimensions of the core plugs to ensure the correct dimensions have been achieved. The Soxhlet extractor, which is specially designed to clean core plugs from filthy residuals, was then used to clean the core plugs. Following that, toluene was used in the Soxhlet extractor to clean the core samples. The core samples were fully cleaned after the clean samples were dried for approximately two days. Figure 17 shows the steps of core plug preparation in the laboratory.

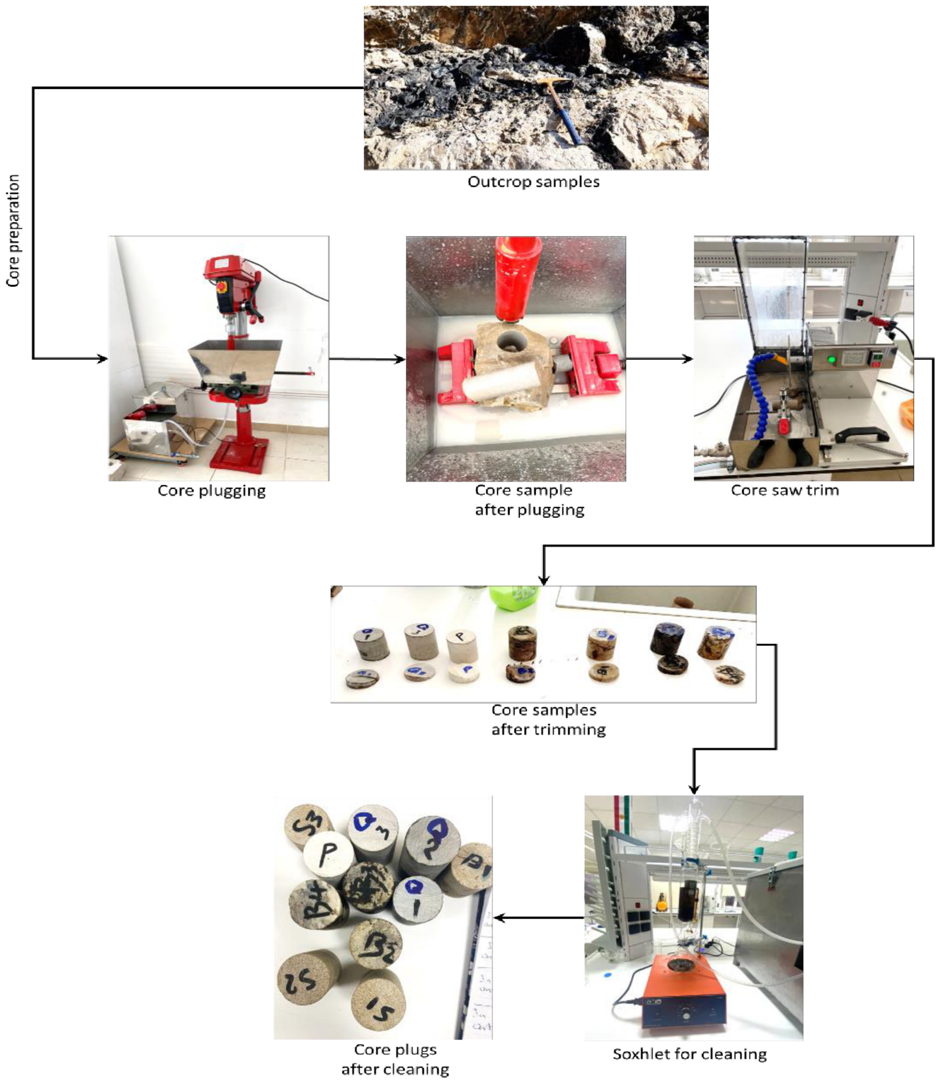


Figure 17 Diagram of preparation of core plugs in the laboratory.

3.3. Porosity measurement

The porosity of core samples is assessed and measured using a porosimeter. The methods to perform this either involves drawing fluid from the rock or injecting a fluid into the rock's pore spaces. The samples are placed in the sample cell (cylinder) of the helium porosimeter, which houses the core, and pressure is applied to the samples' voids by the air compressor. The starting pressure, P1, and the end pressure, P2, are measured and recorded. Using this pressure, the pore volume is calculated. Porosity is then calculated using P1 and P2. The dry and clean core plug samples were tested by the porosimeter in the laboratory, and the outcomes were recorded. Figure 18 depicts an air compressor and a helium porosimeter machine in use in the laboratory.

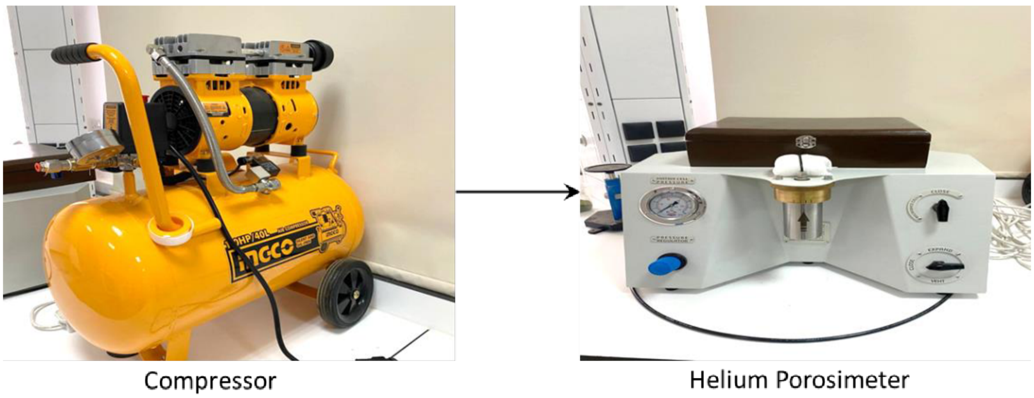


Figure 18 Air compressor and helium porosimeter.

3.4. Permeability measurement

The permeameter was utilized to measure the permeability of all core plugs (Figure 19). Permeameter is a device that is designed to measure permeability by Darcy law, and it consists of two valves, an inlet and outlet pressure valves, a pressure regulator, and a core holder. This instrument will measure the flow rate, and this flow rate will be used in the Darcy equation to calculate permeability in mD.

Figure 19 Gas permeameter machine.

3.5. Preparation of thin sections

Thin sections were made after using a trim saw for small distinct units, then a grinding and polishing machine was utilized by hand to reduce friction in the thin sections. After that, the thin sections were well-prepared. Figure 20 depicts the procedure of preparing thin sections.

Figure 20 Preparing of thin sections.

3.6.Preparation and characterization of smart water solutions

For the preparation of smart water solutions, distilled water (DW) was used for concentrations of 5000 ppm and 10 000 ppm of KCl and NaCl salts. The salts were mixed with 100 mL distilled water. The properties of the solutions are shown in Table 2. After that, a heater was used for preparing the smart water solutions at 800 RPM for about 30 minutes at a temperature of 30℃. Then the solutions were placed in the homogenizer machine, and they needed to stay in it for about an hour for better stabilization of salts as it helped in dissolving the powders in the water better. Figure 21 shows the heater and the homogenizer machine used for the smart water preparation.

Table 2 Properties of smart water solution.

Dissolved Ions	**Concentration [ppm]**	**PH**	**Conductivity [mS]**
NaCl-1	5000	6.36	30.6
NaCl-2	10 000	6.69	52.1
KCl-1	5000	6.58	31.6
KCl-2	10 000	6.54	58.7

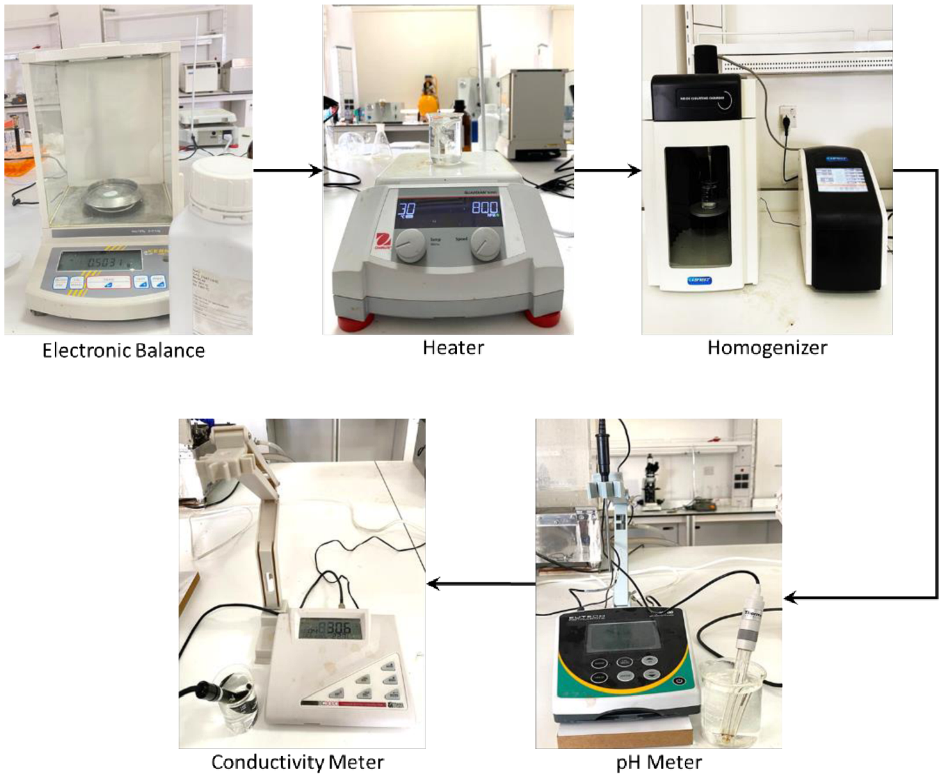


Figure 21 Heater and homogenizer for preparing smart water solutions.

3.7. Aging core samples for wettability alteration

The prepared thin sections were aged in crude oil for about 10 days for wettability alteration, and later they were put in ethanol for them to be cleaned. They were placed in an oven for about five hours to be dried out. Figure 22 shows the aging process of the thin sections, and that they are ready to be placed in smart water solutions.

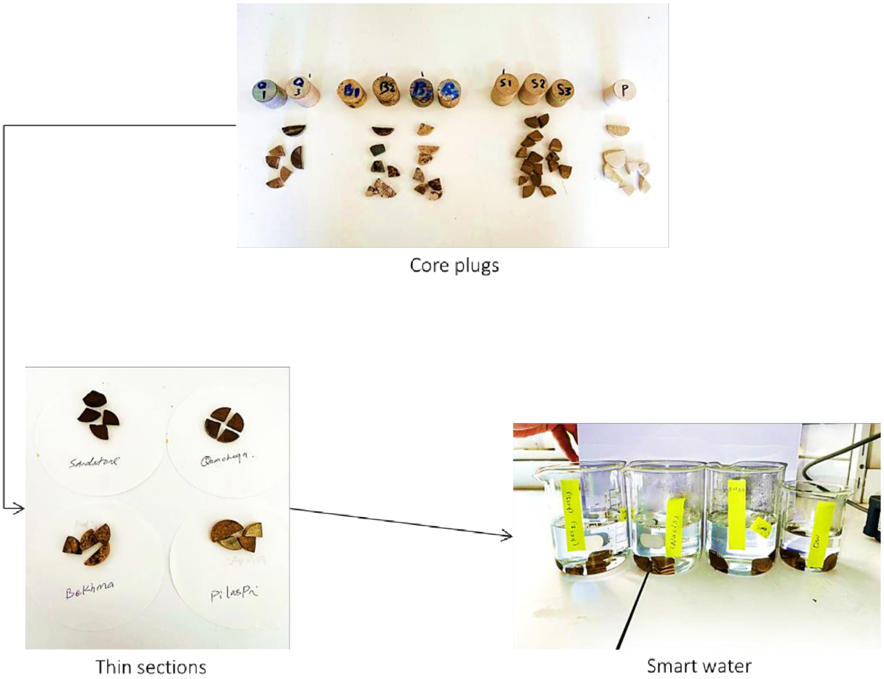


Figure 22 Schematic diagram for thin sections preparation for being placed in SW solutions.

3.8.Contact angle measurement

The contact angle measurement setup is used to quantify a solid using liquid. If the contact angle between the surface of the solid and the produced tangent line is less than 90°, the surface is hydrophilic; if it is greater than 90°, the surface is hydrophobic.

The contact angle measurement system (Figure 23) was built at Soran University. The system consisted of a video camera, a syringe, a computer, a light source, and a variable sample stage. The procedure to measure the contact angle was as follows:

- flattened the stage for the droplet to be stable during deposition,
- dropped the liquid (crude oil) on the stage,
- brightened the droplet from behind with the light,
- recorded the image using the camera,
- analyzed the image using computer software, and
- determined the contact angle.

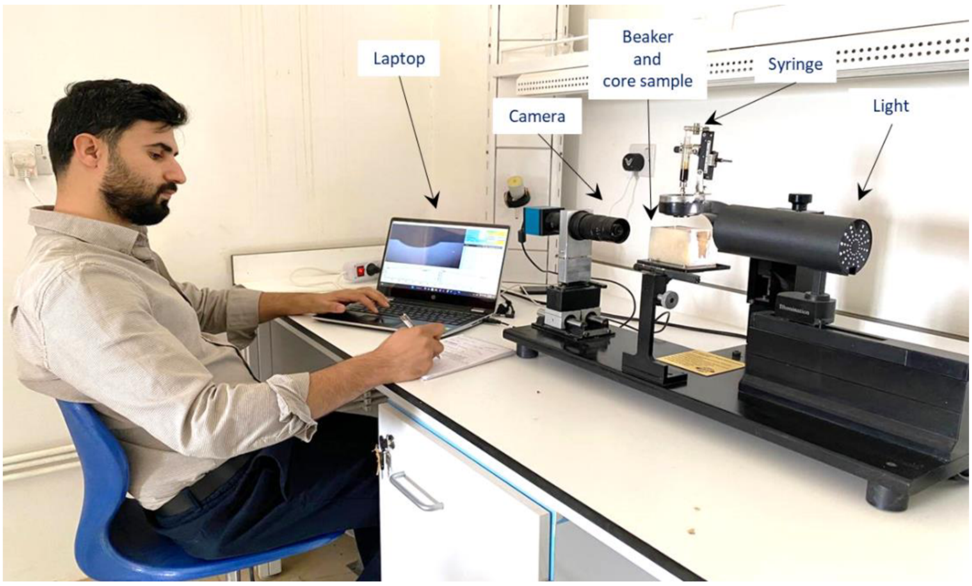


Figure 23 Contact angle measurement setup.

Chapter 4: Results and Discussions

4.1. Compositions of rock samples

X-ray diffraction (XRD) is a non-destructive technique that is used to examine and characterize the position of atoms in a sample, their arrangement in each unit cell as well as the spacing between the atomic planes. In this study, the XRD technique was used to determine the composition of rock samples from different formations and to identify the major mineral of the rock samples. Based on the XRD analysis in Figure 27, the Bekhma Formation contains both dolomite and calcite minerals; however, its vast composition is dolomite as there are more peaks of dolomite. The major mineral of the Qamchuqa Formation is calcite. The main minerals constituting the Pila Spi Formation are ankerite and sinnerite. The four minerals constituting the Injana Formation are quartz, calcite, fresnoite, and dolomite.

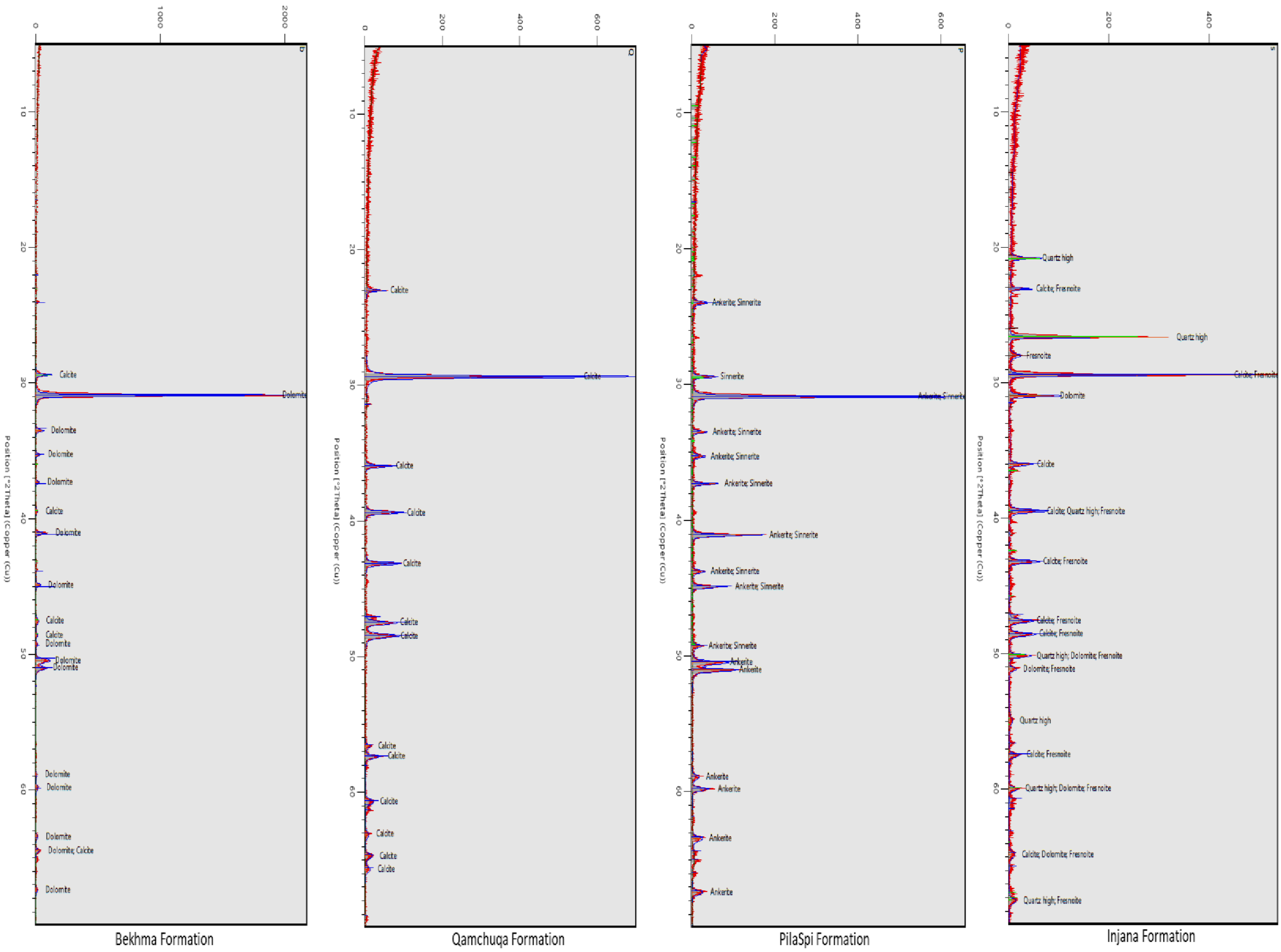


Figure 24 XRD graph.

4.2. Porosity

The porosity values of the retrieved plugs from the studied formations are tabulated in Table 3. The porosimeter was used for measuring the porosity of the plugs. The results show that Plug#10 from the Injana Formation showed the highest percentage of porosity, which is 14.03%. However, Plug#5 from the Qamchuqa Formation has the lowest percentage of porosity (6.92%).

Table 3 Porosity's result of the cores of the study area.

Core sample	Formation	Diameter (cm)	Length (cm)	Porosity (%)
Plug#1	Bekhma Formation	3.6	5	7.97
Plug#2		3.73	5.43	7.14
Plug#3		3.73	5.16	10.78
Plug#4		3.74	5.35	6.95
Plug#5	Qamchuqa Formation	3.71	4.26	6.92
Plug#6		3.71	5.38	7.81
Plug#7	Pila Spi Formation	3.71	5.32	10.53
Plug#8	Injana Formation	3.6	5.38	13.17
Plug#9		3.6	5.2	11.51
Plug#10		3.7	5.38	14.03

4.3. Permeability

For the purpose of calculating the permeability of the core plugs, the pressure difference along the core plugs was measured. The permeameter was used to measure the permeability of the core plugs. The results are shown in Table 4. Plug#2, which belongs to the Bekhma Formation, possessed the maximum permeability of 0.90 mD among all the others. However, the permeability results for Plugs#5 and #6 from Qamchuqa Formation, also Plug#7 from the and Pila Spi Formation, respectively, are all 0 mD.

Table 4 Permeability's result of the cores of the study area.

Core sample	Formation	Diameter (cm)	Length (cm)	Permeability (k) (mD)
Plug#1	Bekhma Formation	3.6	5	0.27
Plug#2		3.73	5.43	0.90
Plug#3		3.73	5.16	0.69
Plug#4		3.74	5.35	0.89
Plug#5	Qamchuqa Formation	3.71	4.26	0
Plug#6		3.71	5.38	0
Plug#7	Pila Spi Formation	3.71	5.32	0
Plug#8	Injana Formation	3.6	5.38	0.86
Plug#9		3.6	5.2	0.73
Plug#10		3.7	5.38	0.51

4.4. Wettability

4.4.1. Distilled water

In proportion to the application of smart water and the nature of the rock, as well as the covering tendency of the fluids for the solid surface, the contact angle will be changed, either increased or decreased. In this study, both indications have been observed. Some of the types of formations have been impacted by these synthetic brines, and the SW led to contact angle reduction, which is one of the main targets of enhancing oil recovery.

In order for the remaining oil to be produced, the angle formed by the adsorbed fluid and the rock surface should be decreased. Contact angles for Bekhma, Qamchuqa, Pila Spi, and Injana were measured in different concentrations (5000 ppm and 10 000 ppm). The impact of the contact angle changed by using the application of SW. The application of SW reduced the contact angle and led to improved production of oil.

Figure 28 and Table 5 summarize the contact angles for each of the formations showing the impact of smart water employment by using only distilled water. The measured average contact angles are 45.90°, 46.07°, 49.43°, and 61.71° for Bekhma, Injana, Pila Spi, and Qamchuqa, respectively. The results revealed that among the four formations, the Bekhma Formation has the least contact angle when only DW is applied.

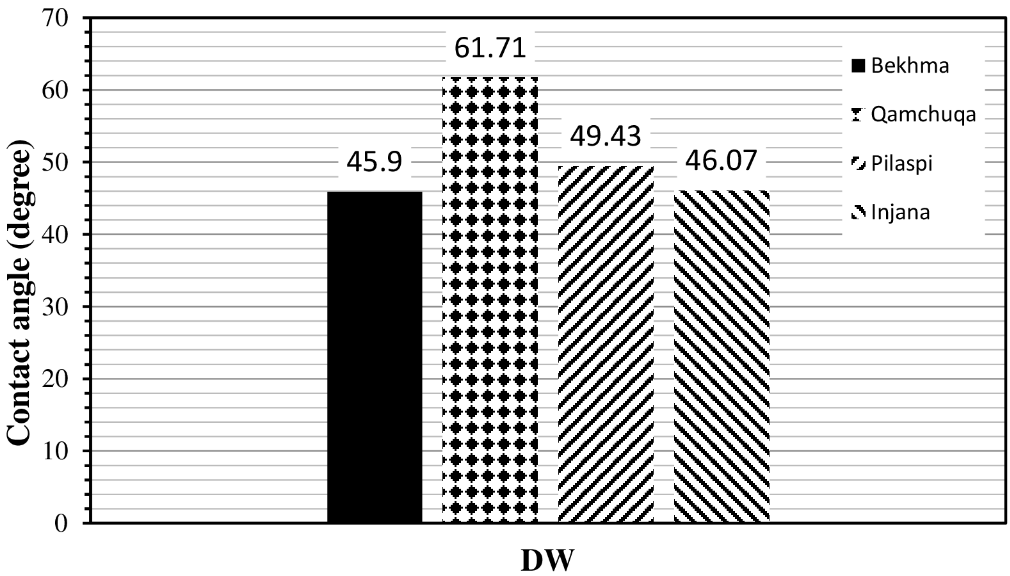
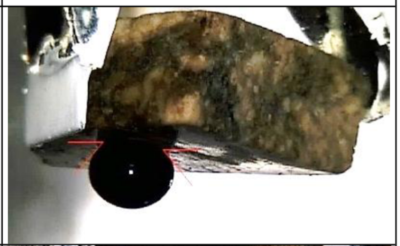
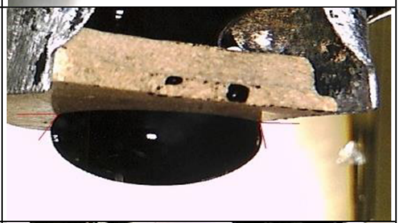
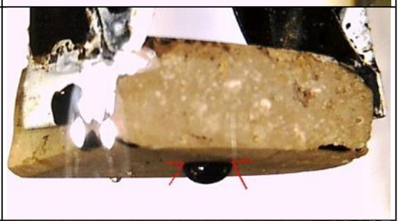
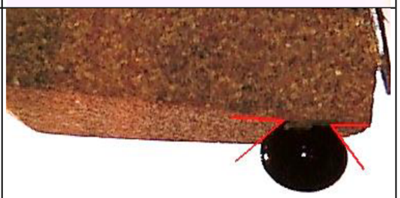


Figure 25 Using DW for the cores of the four formations.

Table 5 The impact of varying SW on the contact angle of formations by using distilled water with shot pictures using CA measurement equipment.

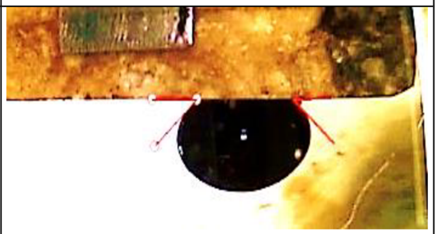
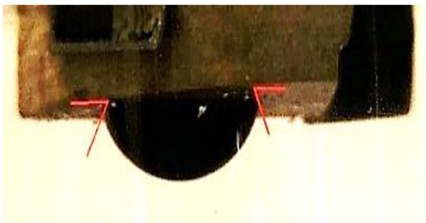
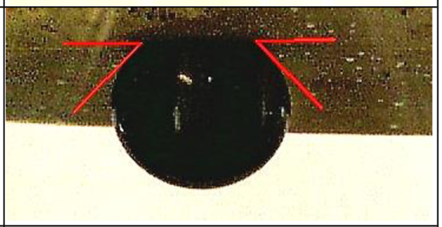
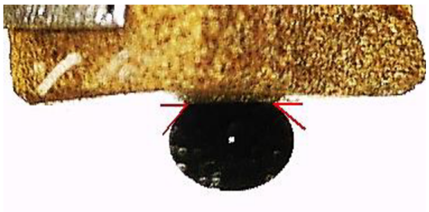
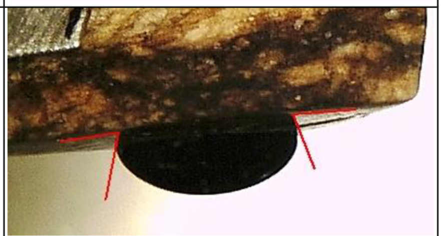
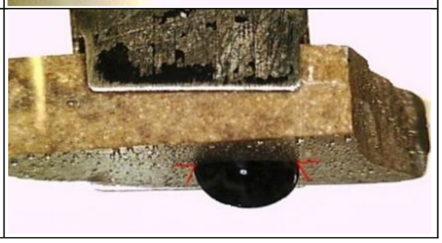
Distilled water (DW)				
Name of formation	**Left angle (degree)**	**Right angle (degree)**	**Average angle (degree)**	**Image**
Bekhma	44.69	47.10	45.90	
Qamchuqa	60.12	63.31	61.71	
Pila Spi	50.21	48.64	49.43	
Injana	49.41	42.72	46.07	

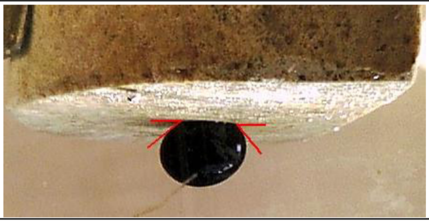
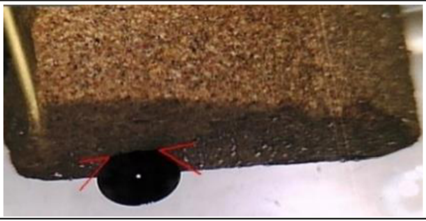
4.4.2. Effect of KCl

The impact of smart water by using KCl in two different concentrations of 5000 ppm and 10 000 ppm. As shown in Table 6, for a concentration of 5000 ppm, the measured average contact angles for the Bekhma, Qamchuqa, Pila Spi, and Injana formations are 37.01°, 60.59°, 45.02°, and 41.24°, respectively. This revealed that when a concentration of 5000 ppm of KCl is used, the Bekhma Formation was affected the most.

Additionally, when KCl is added in a concentration of 10 000 ppm, the contact angles were found to be 73.07°, 65.55°, 41.70°, and 41.68° for Bekhma, Qamchuqa, Pila Spi, and Injana, respectively. The results showed that for this concentration, it is the Injana Formation that is affected the most. Figure 29 illustrates the measured average contact angles of each of the formations when using concentrations of 5000 ppm and 10 000 ppm of KCl.

Table 6 The impact of varying SW on the contact angles of the formations by using KCl with shot pictures using CA measurement equipment.

KCl					
Name of formation	**Concentration**	**Left angle**	**Right angle**	**Average angle**	**Image**
Bekhma	5000	42.16	31.85	37.01	
Qamchuqa	5000	57.80	63.38	60.59	
Pila Spi	5000	44.57	45.48	45.02	
Injana	5000	36.10	46.37	41.24	
Bekhma	10 000	75.51	70.82	73.07	
Qamchuqa	10 000	69.56	61.55	65.55	

Pila Spi	10 000	36.18	47.26	41.70	
Injana	10 000	41.10	42.25	41.68	

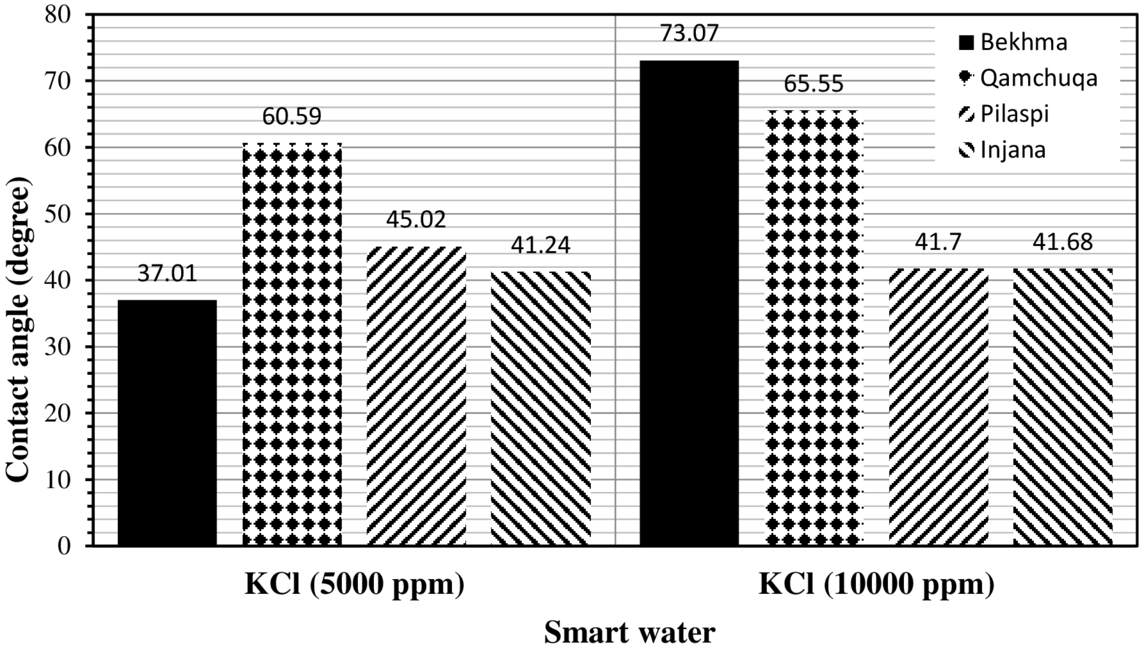


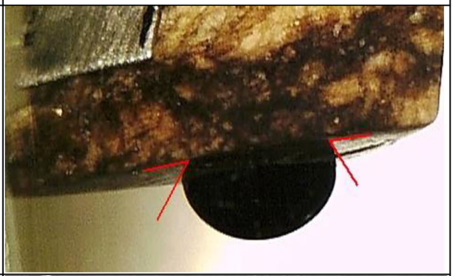
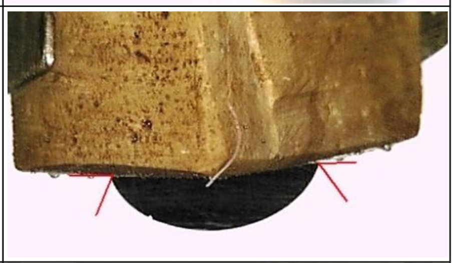
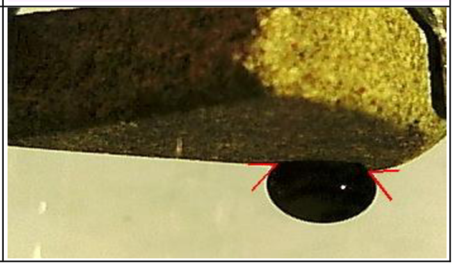
Figure 26 Using KCl in concentrations of 5000 ppm and 10 000 ppm for all cores.

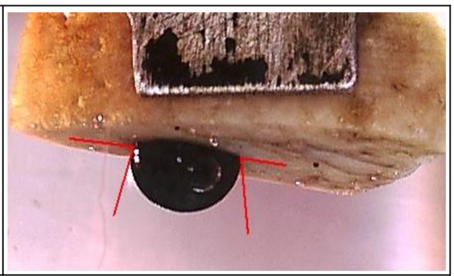
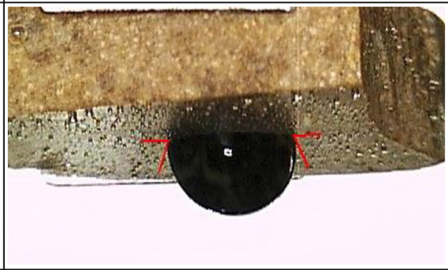
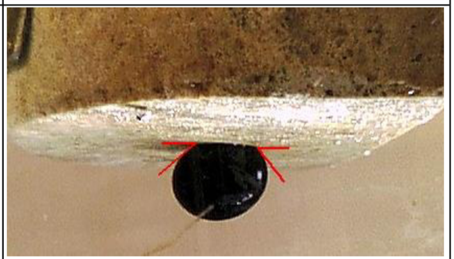
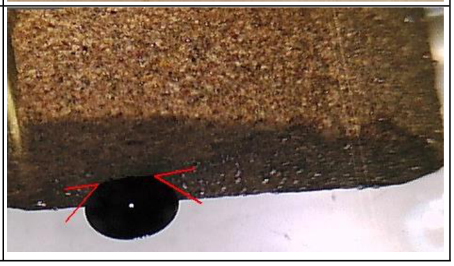
4.4.3. Effect of NaCl

The effect of SW on wettability alteration using NaCl in concentrations of 5000 ppm and 10 000 ppm is presented in numerical values in Table 7. When a concentration of 5000 ppm of NaCl was used, the average contact angles for Bekhma, Qamchuqa, Pila Spi, and Injana were 52.01°, 69.51°, 62.69°, and 55.14°, respectively. However, when a concentration of 10 000 ppm of NaCl was used, the average contact angles were 76.25°, 65.55°, 46.72°, and 41.68° for Bekhma, Qamchuqa, Pila Spi, and Injana, respectively.

The results showed that a concentration of 5000 ppm of NaCl is most effective in the Bekhma Formation while the Injana Formation is the most affected formation when a concentration of 10 000 ppm of NaCl was used. Figure 30 depicts the average contact angles of each of the formations using the two different concentrations of NaCl.

Table 7 The impact of varying SW on the contact angles of the formations by using NaCl with shot pictures using CA measurement equipment.

NaCl					
Name of formation	**Concentration**	**Left angle**	**Right angle**	**Average angle**	**Image**
Bekhma	5000	47.03	56.99	52.01	
Qamchuqa	5000	74.05	64.97	69.51	
Pila Spi	5000	69.35	56.02	62.69	
Injana	5000	52.54	57.75	55.14	

Bekhma	10 000	75.35	77.16	76.25	
Qamchuqa	10 000	69.56	61.55	65.55	
Pila Spi	10 000	44.18	49.26	46.72	
Injana	10 000	41.10	42.25	41.68	

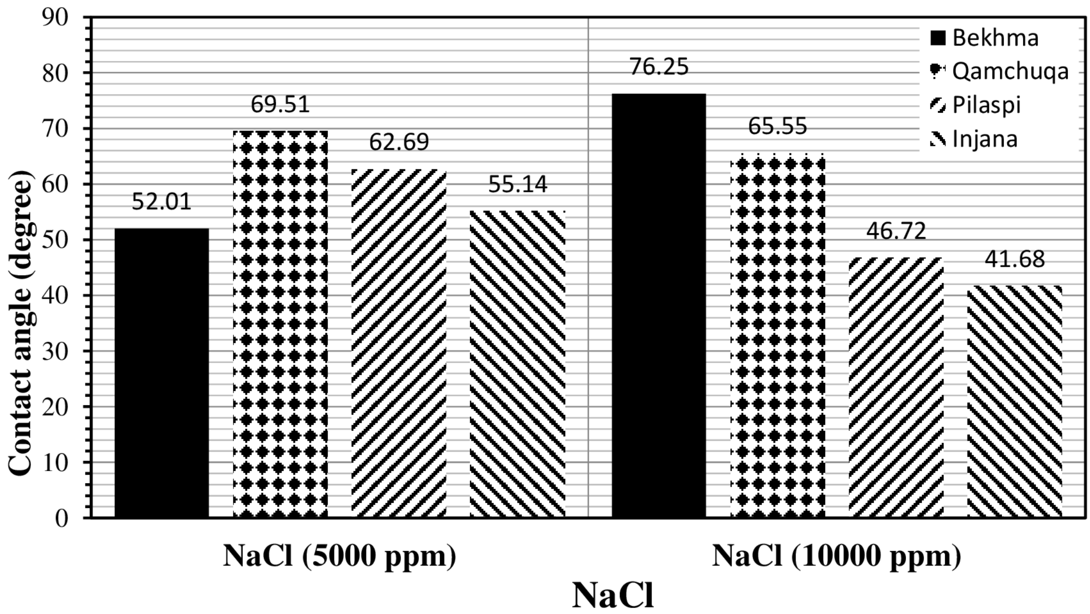


Figure 27 Using NaCl in concentrations of 5000 ppm and 10 000 ppm for all cores.

4.5. Summary

Figure 31 summarizes the effect of the smart water solutions at different concentrations on the rock samples from the studied formations.

- The initial contact angle of the Bekhma Formation was 45.9° when DW was used and it reduced to only 37.01°, a reduction of around 24%, when a concentration of 5000 ppm KCl was added (Figure 31A).
- The addition of KCl in a concentration of 10 000 ppm caused a reduction in the contact angle of the Injana Formation from 46.07° to 41.68° (Figure 31B). This means the contact angle was reduced by 10.5%.
- The employment of NaCl in a concentration of 5000 ppm reduced the contact angle of the Pila Spi Formation by 8.9%. That is, the contact angle was reduced from 49.43° to 45.02° (Figure 31C).
- The contact angle from the core of the Injana Formation reduced from 46.07° to 41.68° (Figure 31D) when NaCl was added in a concentration of 10 000 ppm. This accounts for a 9.5% reduction in contact angle.
- Among all the studied core plugs from the four different formations, the biggest reduction in contact angle (around 24%) was recorded for the Bekhma Formation when a concentration of 5000 ppm KCl was used.

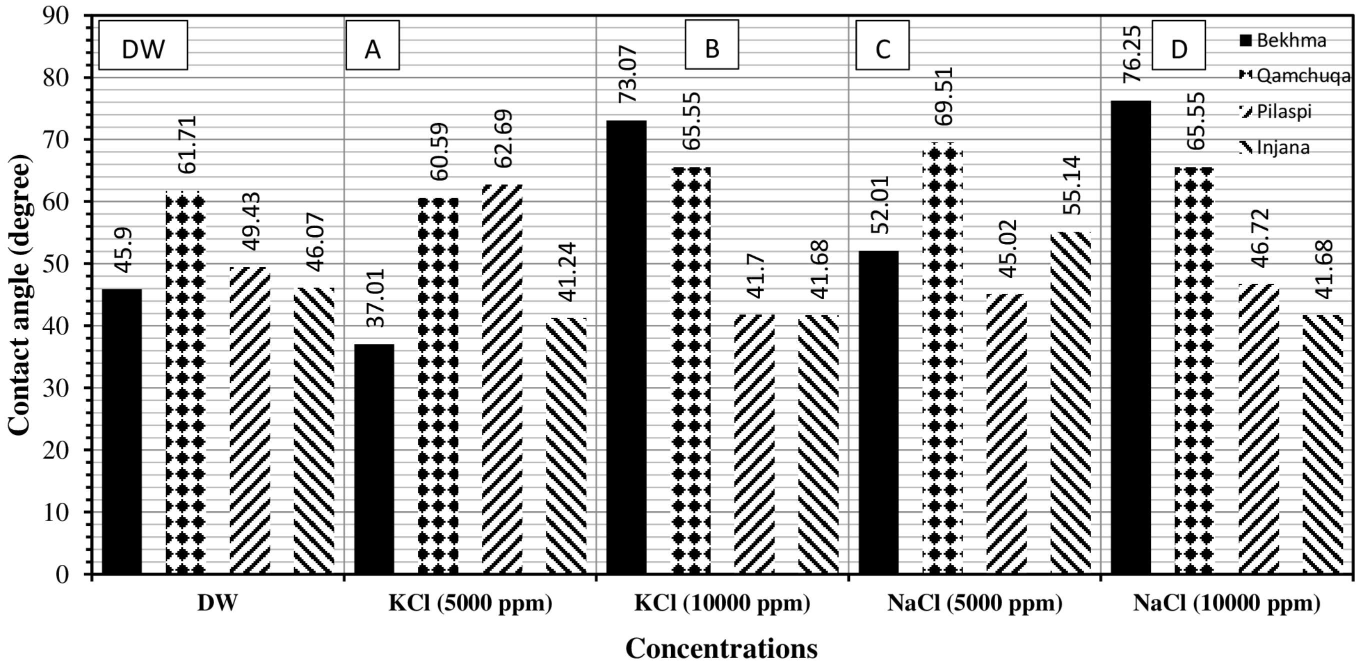


Figure 28 The summary of the impact of SW on contact angle reduction for all cores.

Chapter 5: Conclusions and Recommendations

5.1. Conclusions

This study mainly investigated the impact of smart water on wettability alteration by utilizing two different types of dissolved salts (NaCl and KCl) at different concentrations (5000 ppm and 10 000 ppm) in order to achieve wettability alteration through contact angle reduction. Depending on the kind of mechanism, the wettability could be altered by using smart water which reduces the contact angle and can assist in increased oil recovery. The results obtained from this study can be concluded as follows:

- The addition of KCl in a concentration of 5000 ppm has the greatest effect on the Bekhma Formation as it reduced the contact angle by about 24%.
- Reducing the contact angle of the Bekhma Formation by employing KCl at 10 000 ppm and NaCl at 5000 ppm and 10 000 ppm was unsuccessful because it had no significant effect on reducing the contact angle.
- The Qamchuqa Formation had an initial (before using smart water) high contact angle. The addition of the salts did not impose a significant effect on its contact angle. As a result, it can be concluded that the employment of KCl and NaCl at concentrations of 5000 and 10 000 ppm is ineffective in the Qamchuqa Formation.
- The employment of KCl at 5000 ppm led to a significant contact angle reduction of 10.5% in the Injana Formation.

5.2.Recommendations

In this bachelor project work, the contact angle was determined for various types of formations by the application of the prepared smart water and utilizing laboratory equipment.

The author of this study would like to suggest some recommendations for further work and investigation.

- The author recommends that when taking pictures of the droplets on a solid surface, attention should be paid as a level surface is required for a precise angle measurement.
- Sufficient time should be provided for the cores to be immersed in smart water.

- After immersing the cores, they must be preserved and kept away from high temperatures because the temperature has an effect on them.
- The author also suggests that other solutions and salts with varying concentrations be employed to achieve the best feasible contact angle reduction.

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