

The Importance of Formation Fluids and Reservoir Rock Mineralogy Characteristics in Chemical Enhanced Oil Recovery (EOR)

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Abstract: Hydrocarbons are produced from the ground to the surface via natural reservoir drive energy. However, substantial amount of hydrocarbons still remain trapped in the reservoir. Enhanced oil recovery (EOR) method has been introduced to recover the remaining trapped oils. EOR is a method to enhance the displacement efficiency of the crude oil. These include reducing the interfacial tension (IFT), increasing the viscosity, and wettability alteration. Presently, the chemical EOR (CEOR) method such as surfactant flooding is utilized due to its efficiency, technical and economic feasibilities. Nonetheless, performance of CEOR is greatly relying upon the characteristics of formation fluids and rock mineralogy. Therefore, uncharacterized these factors prior to CEOR method may result in unsuccessful recovery process. This includes surfactants adsorbed onto the rock surfaces instead of at oil-water interface. Hence, lowering the IFT for mobility control may become impractical as surfactants do not function desirably. Experimental works have been performed to provide comprehensive insight on characteristics of formation fluids in terms of salinity and pH along with rock mineralogy, particularly silica and kaolinite. Effect of zeta potential and surface charge on different rock mineralogy is also discussed to elucidate the behavior of the solid surface when in contact with aqueous solutions.

Index Terms: Keywords: Surfactant Flooding, Formation Fluids, Rock Mineralogy.

I. INTRODUCTION

Inherently, the oil is produced at about 30% to 50% from the total oil-in-place (OIP) through the natural reservoir drive energy and water flooding methods which commonly known as primary and secondary recovery processes, respectively. After the conventional recovery methods have been exhausted, the remaining trapped oil (50% - 70%) from the oil reservoir would be recovered by tertiary recovery method or also called as enhanced oil recovery (EOR) method. The small percentage of crude oils recovery (i.e., 30% - 50%)

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during the primary and secondary recoveries are owing to capillary and mobility issues [1]. At present, the need for EOR method is growing due to the global energy demand and depleting in oil reserves. Hence, sustaining the production from existing fields is a priority due to unguaranteed in new field discoveries as well as cost expensive. Essentially, EOR is a method to enhance the displacement efficiency of the crude oil. Hitherto, myriad of EOR methods have been established which include gas, chemical, and thermal methods. Although, there are ubiquitous EOR techniques developed across the globe, yet the chemical based EOR (CEOR) method in particular surfactant flooding is regard as the auspicious and most promising method because of its exceptional efficiency, technical ability and economic feasibilities along with reasonable capital cost. Ideally, CEOR method increases oil recovery by increasing the effectiveness of injected fluids into the reservoir to displace the oil, although more abstractly, it could signifies a reduction in oil saturation below the residual oil saturation [2]. Depending on the type of chemical EOR process, the injected chemicals such as surfactant will interact and alter the fluid-fluid and/or fluid-rock behavior at the interfaces in the reservoir subsurface environments. These include reducing the interfacial tension (IFT), minimizing the capillary forces, wettability alteration, and emulsion formation [3]–[5]. All these characteristics aforementioned are converged literally to one important factor which is mobility control. Although, scientifically means reducing the residual oil saturation and increase the sweep efficiency.

Nonetheless, the CEOR method of surfactants flooding may not be effective as expected without comprehensive and detailed understanding of the reservoir subsurface environment particularly, formation fluids and rock mineralogy. These two parameters are regard as the major factors in determining the overall performance of surfactant flooding. Uncharacterized formation fluids and reservoir rock mineralogy prior to CEOR scheme (i.e., surfactant flooding) will lead to degradation, precipitation (slug), and inevitable outcome of adsorption of the surfactants onto rock surfaces which ultimately lead to ineffective IFT reduction as depicted in **Fig. 1**. Moreover, the higher degree of adsorption capacity on the rock surfaces will increase the amount of surfactant required for injection purposes during the surfactant flooding process. This is due to pertinent



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surfactant loss and marginal alteration in desired IFT reduction and thereby elevates the overall operational cost. On the contrary, understanding the adsorption behavior of surfactants will facilitate the selection process of appropriate surfactants and its concentration range for the given reservoir conditions. Gogoi, (2009) in her study on adsorption of surfactant on reservoir rock also reported that, rock mineralogy has not been studied comprehensively upon its critical influence on surfactant adsorption behavior. This signifies that, the determination and characterizations of formation fluids and reservoir rock mineralogy ahead of surfactant flooding operation are highly important. Surfactant/chemical flooding has been used extensively as a chemical based EOR to manipulate the phase behavior of the reservoir fluids and alter the capillary forces trapping the oil in the porous media during the enhanced oil recovery [7]. Nevertheless, the desirable reduction of IFT in the reservoir is always the primary objective in the surfactant flooding, however, the element of reservoir conditions such as high temperature, pH and salinity have to be taken into consideration for a successful enhanced recovery process.

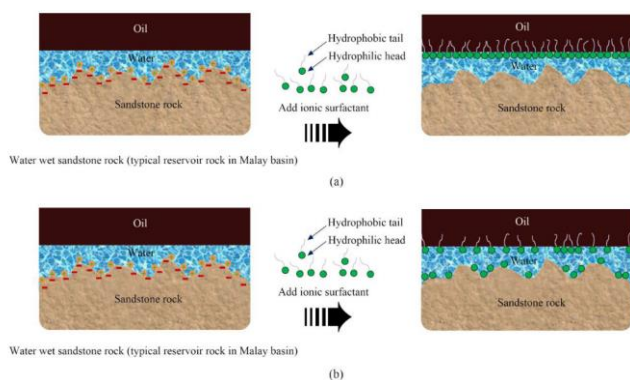


Fig. 1. Schematic representation of the adsorption of surfactant during the EOR surfactant flooding process (a) ideal condition of surfactant flooding in the reservoir rock (b) non-ideal condition in the EOR process with the presence of surfactant molecules on the solid surface in the reservoir rock.

Therefore, this paper aims to investigate the reservoir conditions such as formation fluids (i.e., formation water salinity) and rock mineralogy (i.e., silica and kaolinite) characteristics prior to surfactant flooding process. Various experimental techniques are employed to explicate the influence of these characteristics having on enhanced recovery process particularly, surfactant flooding. In this study, the surfactant adsorptions behaviors including surface potentials are also discussed. This is to elucidate the significant effect of zeta potential from various rock mineralogy such as silica and kaolinite in subsurface environments towards IFT reduction in CEOR process. Besides that, effect of surface charge from different rock mineralogy is also important in the surfactant adsorption process. This is due to the fact that surfaces of minerals show complex behaviour when in contact with aqueous solution. The development of the surface charge is primarily based on rock composition and mineralogy, and the surfactant is chosen by these parameters [8]. For these reasons, surface

charge is an inevitable factor to be considered in this study. Several protocols are implemented in laboratory practices to characterize the formation fluids and rock mineralogy. All experiments are conducted at Universiti Teknologi Malaysia (UTM) and the materials utilized in this study are typical rock samples and formations fluids from Malay basin.

II. LITERATURE REVIEW

In a recent study by Saha et al., (2017), they explained that rock mineralogy (i.e., illite, montmorillonite, and kaolinite) and its types have a significant effect on the surfactant adsorption characteristics. Succinctly, reservoir rock and its mineralogy has to be taken into consideration to ensure the success of the surfactant flooding, or the entire EOR process will come at cost expansive due to inefficient oil recovery as surfactants do not function desirably as predicted. Another important criterion to take into account in the surfactant (chemical) flooding is the surface charge. The principles of surface charges have been discussed and widely reported in the literature see review article by [9],[10]. Hence, the discussion on theory in this paper has been kept to a minimum. In general, most of the reservoir rocks are charged, either negatively or positively, depending on the reservoir parameters, such as pH and salinity [11]–[13]. Apart from that, rock composition along with its mineralogy are also the key factor in determination of surface charge and in most cases, the surfactant is selected by considering these parameters [8], [14], [15]. The original surface charge can be altered by the presence pH and salinity, however, the mechanism of modification of the surface charge by these effects still remains a major debate.

According to Hiorth et al., (2010), the process of sweeping the oil components depends upon the rock minerals and the surface charge of the pore surface. In this regard, the oil trapped in the reservoir rock or porous carrier beds that comprised of sandstone (i.e., rich in silica) and shale or presence of inorganic minerals in petroleum reservoir such as clay (kaolinite, montmorillonite, chlorite, illite and quartz) normally carry a net negative or positive surface charge. In the case of negatively charged of subsurface environment (i.e., reservoir rock), the surfactant used for EOR process might not thoroughly success in sweeping all the trapped oils from the reservoir rock. This is essentially due to the opposite charges between the injected fluids from the EOR process and the surface of the rock which creates an electrostatic attraction at the interface between the two different elements as shown in Fig. 1(b). This will induce some of the surfactant molecules to attract at the solid surface and eventually increase the surfactant concentration at the rock interface. In this respect, the EOR method of surfactant flooding has become inefficient and may results in higher operation cost due to unnecessary increase in total volume of surfactant employed owing to pertinent surfactant loss and marginal changes in desired IFT reduction. As elucidated by Saha et al., (2017), the adsorption capacity of surfactant is highly dependent upon the mineral content of rock. This is due to the fact that, between the rock minerals and fluid interface

there is a different in charge species, rock surface area, salinity, pH, and temperature which contribute critically to the surfactant adsorption behavior [16],[17]. Through their work, Saha et al., (2017) reported that, after screening process of the desired surfactant in regard to IFT analysis and nature charge on reservoir rock surface, the results showed that adsorption capacity of different types of surfactant depends greatly on the rock mineralogy. Hence, thorough pre-understanding of these factors particularly the charge elements of the different rock minerals particularly, prior to the actual CEOR implementation will greatly assist in deciding the appropriate surfactants and its concentration range.

The main idea of injecting surfactant in the EOR process is to lower the interfacial tension (IFT) between oil and water in order to provide an efficient oil recovery process. As far as the reservoir rock samples of Malay Basin (Peninsular of Malaysia) are concerned (where most of the rocks are found in the form of sandstone) the adsorption of surfactant onto the rock surface is more likely to occur due to the strong water-wet character of sandstone as depicted in Fig. 2. Since water is a polar molecule and reservoir rock consists of charges, hence water-wet condition at the rock surface resulting from the mutual intermolecular interactions between the fluids and the solid may cause the surfactant to be attracted towards the water-rock surface instead of oil-water surface which inhibits the desired IFT reduction for better sweep of oil recovery. According to Farajzadeh et al., (2017), the magnitude of the interfacial tension reduction by surfactants strongly depends on the ionic strength as well as the hardness (concentration of divalent cations) of the aqueous phase. A more detailed understanding of the nature of the interactions between the rock and the fluids residing in the pore can be found in the book by [18]. Another important criterion to be considered in ensuring efficient EOR process of surfactant is to avoid the aggregate structures of surfactant molecules such as lamellar liquid crystals (slug of surfactant molecules) which may prevent further reduction of IFT. The system should be that of mixing between the fluids and the surfactants, or a surfactant slug will occur. Dispersion of surfactant at the oil-water interface would form a desired condition in the porous media. A small change or reduction in surfactant concentration at the interface will significantly affect the interfacial tension. In this regards, some of the surfactant molecules may have concentrated on the solid surface of porous media (reservoir rock) by virtue of surface charge effect. Other application of surface charge can also be found in the nanoparticle technology as a potential solution in EOR schemes. The application of nanoparticles in EOR is discussed in detail in a review article by [19].

III. MATERIALS AND METHODS

A. Materials

The materials used in this study are silica and kaolinite which considered as dominant minerals presence in the Malay basin formation. The materials are sourced from Sigma-Aldrich and subjected to further verification via detailed experiment to identify the purity and the chemicals content. The synthetic formation water is prepared using

sodium chloride (NaCl). In this case, NaCl is selected due to its abundancy in the typical formation water as shown in **Table I**, particularly, for Malay basin. Concentration for NaCl in this study is set at 0.0001M until 5.0000M with an increment by a factor of 10. The range of salinity employed in this study is covered from the lowest to the highest salinities presence in the reservoir [20]. The ionic strength is calculated from the equation, $I = 0.5 \sum C_i Z_i^2$ where, C and Z represent the NaCl concentration and the charges of the aqueous solution, respectively. The pH of the aqueous solution is adjusted using 0.01M sodium hydroxide (NaOH) and 0.01M hydrochloric acid (HCl).

Table I: Types of ions traced in the formation water

| Ions | Cl ⁻ | Ca ²⁺ | Mg ²⁺ | Na ⁺ | K ⁺ | Fe ²⁺ | SO ₄ ²⁻ | Oth ers | Total salinity |
|-------|-----------------|------------------|------------------|-----------------|----------------|------------------|-------------------------------|------------|-------------------|
| Unit | 119 | 77 | 161 | 68 | 7 | 3 | 74 | 2080 | 21252 |
| (ppm) | 00 | | | 80 | 7 | | | | |

B. Methods

1) Sample Physical Characterizations

The samples are characterized using specific surface area adsorption method and X-ray fluorescence (XRF). XRF analysis for chemical composition is obtained by Bruker S4 PIONEER X-ray fluorescence Spectrometer at 4 kW power. Specific surface area adsorption of samples are measured using Micromeritics ASAP 2020 by physisorption of N₂ using Brunauer–Emmet–Teller (BET) method. The N₂ adsorption isotherms is measured at temperature degree of -196 °C. The samples are degassed at 160 °C for 4 hours before the analysis. This is to ensure the impurities and mist are completely removed from the surface of the solid samples. Scanning electron microscopy (SEM) and electron dispersive X-ray (EDX) analysis are recorded by using LEO S430 scanning electron microscope coupled with energy dispersive X-ray analyzer model Oxford LINK ISIS for surface morphology and elemental analysis. Samples are prepared by dispersing dry powder on double sided conductive adhesive tape. Samples are coated with carbon by arc discharge method prior to SEM- EDX analysis.

2) Zeta Potential Characterizations

Zeta potential measurement employed in this study is operated using an automated electrophoresis instrument (Litesizer 500) from Anton Paar. The instrument is equipped with electrophoretic light scattering (ELS) process which enables a direct measurement of particle size with less than 10 microns ($d < 10\mu\text{m}$) as well as zeta potential (ζ) of the suspended particles in the solution. In the sample preparation procedures for zeta potential measurement, a 100mg (0.1g) of sample is prepared and transferred into a 100mL of an aqueous solutions. Magnetic stir is applied in order to keep the samples well dispersed in the solution. The number of runs, attenuation filter and focus position is automatically optimized by the instrument. The pH of the suspension is measured using Mettler Toledo Five Easy and adjusted using NaOH and NaCl from pH of 2 to 12. Apart from pH alteration, the ionic strength of the solution is also adjusted by

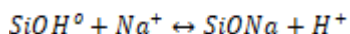
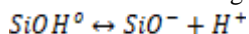


adding NaCl, consecutively. The relative accuracy of the measurement is about ± 0.01 pH unit at room temperature. The zeta potential measurement is performed using the Smoluchowsky approximation in automation mode by the Kalliope software.

IV. RESULTS AND DISCUSSIONS

Details of minerals characterizations are conducted to evaluate their nature of charges, minerals content, surface properties, pore size and shape. **Table II** indicates the chemicals contents present in silica and kaolinite. From the table, SiO₂ is found to be the dominant composition in both silica and kaolinite, followed by Al₂O₃. The results reflect that, both silica and kaolinite are anionic by nature. This is further supported by EDX analysis as shown in **Fig. 2**, wherein oxygen and silica are the two major elements (peaks) discovered along with other traces of elements such as Aluminium (Al), Magnesium (Mg) and Ferum (Fe).

Reservoir rock is primarily consists of sandstones, and silica is known as the main mineral. Silica consists of two primary structures at the surface namely, siloxane group (Si₂O) and silanol group (SiOH). Jaafar, (2009) reported that, the reactions of silanol group can be described as follows:



From the reactions written above, H⁺ ion is released into the solution due to deprotonation process [21]–[23]. Consequently, the mineral surfaces of the reservoir rock (sandstone) become negatively charged. However, the reaction for siloxane group can be neglected due to chemically inert characteristic and considerably low of protonation process. On the other hand, for clay minerals such as kaolinite, the solid particles always display negative charges in the solution across all pH values above the isoelectric point [24]. Isomorphous substitution plays an important role in determining the charge of the clay particle due to ions exchanged. This phenomenon occurs during the formation of clay crystals which are different in structure compared to silica [23]. Kaolinite is non-expanding clay structure with one octahedral sheet in a 1:1 layer structure.

Table II: Chemical composition of silica and kaolinite from XRF analysis.

| Chemical composition | Silica | | Kaolinite | |
|--------------------------------|---------------------|----------|---------------------|----------|
| | Concentration (ppm) | Mass (%) | Concentration (ppm) | Mass (%) |
| SiO ₂ | 997000 | 99.75 | 586000 | 58.65 |
| Al ₂ O ₃ | 2470 | 0.25 | 372000 | 37.23 |
| Fe ₂ O ₃ | 30.6 | 0.00 | 5500 | 0.55 |
| MnO | 17.6 | 0.00 | 134 | 0.01 |
| CaO | - | - | 1110 | 0.11 |
| K ₂ O | - | - | 24500 | 2.45 |
| P ₂ O ₅ | - | - | 3740 | 0.37 |
| TiO ₂ | - | - | 6240 | 0.62 |

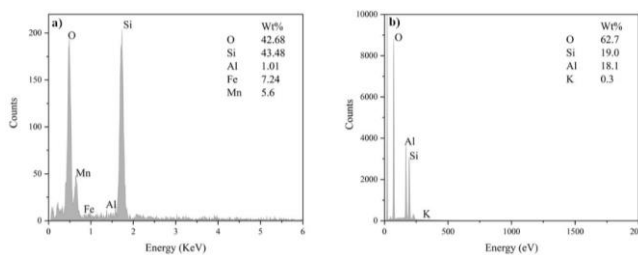


Fig. 2: Characterization of reservoir minerals by EDX (a) silica and (b) kaolinite.

Table III shows the surface area, pore volume and pore diameter of the silica and kaolinite. According to BET analysis in **Table III**, the surface area of silica is observed to be higher than kaolinite. In this respect, silica is expected to have greater tendency of adsorption (surfactants) due to large charge distribution on the solid surface. In spite of that, the pore structures, sizes, and surface area of silica as well as kaolinite are depicted in **Fig. 3**. It clearly shows that, the average particle size of silica is in several magnitude smaller than kaolinite (silica $\approx 0.10 - 0.20 \mu m <$ kaolinite $\approx 7.00 - 8.00 \mu m$). Having said that, silica ultimately contains greater pore volume and average pore diameter compared to kaolinite. In addition, the surface chemistry of kaolinite resides in its bilayer structure which composed of one sheet of silica and a sheet of alumina. It exists as a microscopic plate-like layers of silica tetrahedral and alumina octahedral sheet and these can be seen distinctly in **Fig. 3(b)**.

Table III: BET surface analysis.

| Reservoir mineral | Surface area m ² /g | Pore volume cm ³ /g | Average pore diameter nm |
|-------------------|--------------------------------|--------------------------------|--------------------------|
| Silica | 172.3602 | 0.4379 | 10.1623 |
| Kaolinite | 7.1299 | 0.0306 | 17.1693 |

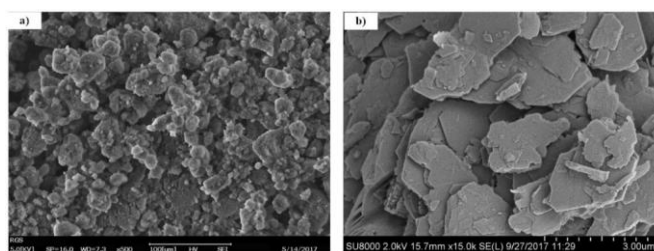


Fig. 3: Surface morphology by the SEM image (a) silica and (b) kaolinite

On the other hand, **Fig. 4** illustrates the N₂ adsorption and desorption isotherms with hysteresis loops at value of approximately 0.6 and 0.45 for p/p₀ for the silica and kaolinite, respectively. Based on international union of pure and applied chemistry (IUPAC) standard, the isotherms indicate a type II (H3) classification with presence of some mesoporous property due to the existence of hysteresis significant gradient uptake as well as abundance of macro pores. The results reflect the significant of adsorption capacity of the solid surface. In general, adsorption capacity is



found to be dependent upon the porous structure of the adsorbent (i.e., surface area, pore volume, and average size). Hence, high adsorption will less likely to occur at infinitesimal pore size [25].

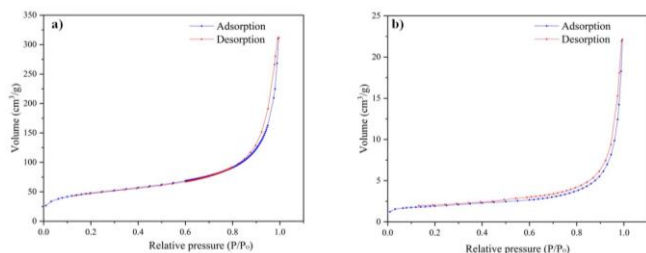


Fig. 4: BET adsorption and desorption curve for N₂ (a) silica and (b) kaolinite.

Fig. 5(a) presents the behavior of zeta potential at different ionic strengths of an aqueous NaCl solution for silica and kaolinite along with the decay of Debye length. The results implicitly describe the increment of zeta potential with decreasing NaCl concentration. In this case, the increase of zeta potential is due to the expansion of the electrochemical double layers or rather briefly, increase of Debye length. The results further indicate that at high salinity of NaCl concentration, the electrical double layer is compressed as ionic strength increases and affects the electrical potential near the charged surface to fall rapidly with distance. Therefore, the results demonstrate that, the reservoir rock particularly sandstone (silica) and clay (kaolinite) are not suitable at high salinity environment as attraction force override the electrostatic repulsion and promote higher chances of agglomeration of particles or rather abstractly, collapse of electrical double layer at the solid surface.

The following equation (1) is used to calculate the Debye length:

$$\text{Debye length} = \sqrt{\frac{\epsilon_0 \epsilon_r k_B T}{2 N_A e^2 I}} \quad (1)$$

Whereby, I can be written as follows:

$$I = \frac{1}{2} \sum_{i=1}^n C_i Z_i^2 \quad \text{where, } i = 1, 2, 3, \dots, n \quad (2)$$

In the equations (1) and (2) above, ϵ_0 is permittivity of free space, ϵ_r is the relative permittivity of liquid, k_B is Boltzmann constant, T is temperature in Kelvin, e is elementary charge in Coulombs, N_A is the Avogadro constant, I is the ionic strength, C is the ion concentration, and Z is the ion charges. From the results obtain in **Fig. 5(a)**, the fundamental concept from equation (1) is proven through the experimental works. From equation (1), the Debye length decreases with an increasing ionic strength. This indicates that, the experimental results obtained are matching with the theoretical model.

Fig. 5(b) compares the zeta potential of silica and kaolinite at various pH. It is important to note that, pH is the most important parameter of the liquid phase that affects the zeta

potential. Therefore, it is essential to study the behavior of zeta potential at different pH particularly, at reservoir environment wherein, the pH is normally observed in the range of pH 5 to pH 8. In addition, isoelectric point (IEP) is another significant parameter that strongly indicates the chemistry of functional group which are present on the solid surface. IEP is related to either acidic or basic strength. IEP at 0 represents the equilibrium of electrokinetic charge density between negatively and positively charged surface groups which ideally, give zero net charge. However, in this study, IEP is found to be appeared at pH value of 3 for silica and is not observed for kaolinite. This emphasizes that, kaolinite is predominantly negatively charged which is analogous to the results discussed in **Fig. 2**. In this regard, the findings explicitly describe that, cationic surfactants may not suitable for the surfactant flooding (CEOR) within the scope of study. High adsorption capacity may be expected on the solid surfaces owing to attraction of opposite charges. It is important to note that, a decrease in zeta potential at high pH demonstrates an increase in hydrophilicity (water affinity) compared to hydrophobicity (water repellent). Hence, pre-selection of surfactant types is critical in ensuring the successful of CEOR employed by minimizing the potential of surfactant adsorption. In general, it is obvious that from both results in **Fig. 5(a) and (b)**, the addition of salt (NaCl) and modification of pH can alter the charging behavior of the solid surface. Particular interest should be focused on pH 5 to 8 wherein, typical reservoir pH value is commonly found. Zeta potential for kaolinite ($\zeta_k \geq -25mV$) and silica ($\zeta_s \geq -8mV$) between pH 5 to pH 8 is a strong indication of surface functionality which provides information of liquid-on-solid surface adsorption process and electrostatic interactions with the surrounding liquids. In summary, both parameters (salt and pH) are the crucial criteria to be thoroughly investigated prior to implementation of CEOR.

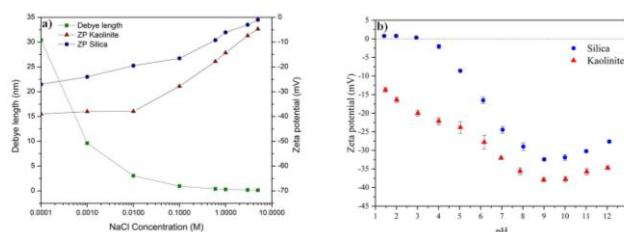


Fig. 5: (a) Dependence of zeta potential for silica and kaolinite on the ionic strength of an aqueous NaCl solution together with the decay of Debye length. (b) pH dependences of the zeta potential for silica and kaolinite.

V. CONCLUSION

This study emphasizes the important of pre-characterizing of the formation fluids and reservoir rocks prior to CEOR implementation. The results demonstrate that both silica and kaolinite are negatively charged by nature as proven from mineralogy characterization and experimental work. However, IEP is not observed in kaolinite while, IEP of silica is found at pH = 3. This confirms that, below the pH of 3, silica becomes positively charged with weak zeta



potential. In addition, electrical double layer is compressed as a result of high salinity environment (strong ionic strength) and conversely. Ultimately, the knowledge and findings will assist in deciding the appropriate selection of surfactant used and ways in minimizing the adsorption of surfactants.

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