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Effect of Silica Nanoparticles on Wettability of Oil-Wet Surfaces

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Abstract: *The effect of silica nanoparticles and brine salinity variation on the wettability alteration of oil-wet surfaces was investigated in this paper. The rock and fluids of Kolo oilfield in Nigeria were used as case study and five brine salinities and two nanoparticles' concentrations were investigated. The contact angle and Amott wettability index methods were used to investigate the wettability alteration potential of silica nanoparticles on oil-wet surfaces and intact core plugs, respectively. The results of the contact angle tests showed that the use of 1 g/L and 5 g/L silica nanoparticles reduced the oil-wet surface's angle from 79º to 73º and 71º respectively in aqueous solution. Lower contact angles were however observed with the use of brines of different salinities with the lowest angles (69º and 68º) being obtained with lowest salinity brine (90D). Which shows that the nanoparticles have the tendency to modify oil-wet surfaces towards water-wet condition irrespective of the brine salinities. Also, wettability alteration towards increasing water-wetness was observed with decrease in brine salinities and increase in the concentrations of the nanoparticles. Finally, the results of the Amott wettability index to water show that the application of nanoparticles modifies the intact rock wettability towards water-wet condition and higher oil production was observed with the spontaneous imbibition of nanofluid with 0.25 wettability index relative to that of the formation brine that was 0.1. The sediments of nanoparticles were however observed on top the rock core and at the base of the cell during the spontaneous imbibition process. This work presents experimental study on the wettability alteration potential of the application of silica nanoparticles in Kolo oilfield reservoir rock and fluids. The study presented in this paper is relevant to the design and implementation of nanoparticles enhanced oil recovery process.*

Keywords: *Silica nanoparticles, sandstone, wettability, Kolo oilfield, Amott wettability index and contact angle*.

1. INTRODUCTION

In the context of petroleum engineering, wettability is an important physical property that controls the distribution and motion of immiscible fluids in the reservoir rock pores. Understanding the influence of the surface wettability of rocks can aid in the development of methods to increase oil recovery during displacement processes, as well as improve other oil recovery operations and the geological storage of CO_2 in depleted reservoirs. At the discovery of most reservoirs, they are usually oil-wet or mixed-wet due to long time accumulation of oil after the displacement of the brine [1]. Crude oil can alter the rock surface wettability from water-wetness to oil-wetness or mixed-wetness due to their composition [2]. The degree of the alteration is determined by interactions between the oil components, mineral surface of the rock and brine chemistry [3, 4]. Wettability modification of surfaces has been shown as fundamental factor that influences water flooding behaviour, enhanced oil recovery processes, relative permeability, electrical properties, capillary pressure, and residual oil saturation [5-7].

The use of nanoparticles in oil production systems is an emerging method of which the nanoparticles' ability to modify the surface wettability is one of the proposed mechanisms by which nanoparticles enhance oil recovery [8-10]. This is attributed to their adsorption on the solid surfaces and subsequent formation of a nanostructured surface that promotes water-wetting behaviour [11]. Previous studies have demonstrated this potential in the applications of nanoparticles. For instance, Hendraningrat et al. [12] investigated the wettability alteration potential of lipophobic and hydrophilic polysilicon silica nanoparticles on polished synthetic silica surface and their results showed that the crude oil contact angle reduces and wettability alteration toward increasing water-wetness was observed with an increase in the concentration of nanoparticles. Also, Al-Anssari et al. [13] investigated the effect of the application of silica nanoparticles on calcite surface wettability, and their results showed wettability modification from oil-wet to mixed-wet or water-wet conditions. Other study by Li et al. [14] showed that silica nanoparticles can modify sandstone rock surface from oil-wet to water-wet condition. Furthermore, in the study conducted by Ju and Fan [15], untreated polysilica nanoparticles altered sandstone wettability from oil-wetness to

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water-wetness and improved the effective water permeability. The application of nanoparticles has the following advantages: high surface to volume ratio, good stability, easy modification of size and shape, easy modification of surface chemical properties toward hydrophilic or hydrophobic and environmental friendliness [16]. Thereby making them appealing for oil production enhancement applications.

Wettability alteration has previously been studied with different methods such as contact angle measurements, Amott wettability index, spontaneous imbibition, adhesion etc., of which contact angle method is the most used because of its simplicity and short duration. For example, Treiber et al. [1] used contact angle method to investigate the wettability variation of more than 50 oil producing reservoirs with the aids of quartz and calcite surfaces, synthetic formation brine and dead oil. Khusainova et al. [13] used the adhesion test and contact angle method to study the enzyme wettability modification of calcite using Sea water and crude oil. Mohammadi et al. [17] investigated the effect of γ-Al_2O_3 nanoparticles on calcite surfaces using contact angle method. Also, Ezzati and Khamehchi [18] investigated silica nanoparticles wettability alteration using contact angle method. Although the contact angle method gives an indication of wettability alteration, it is however a fractional representative of the true rock pore topology because it is limited to only a small fraction of the exposed surfaces. The combination of contact method with Amott wettability index method [19] which measures the average wettability of the intact porous cores based on combined spontaneous and forced imbibition process, can improve wettability alteration insight of any process.

The objective of this study is to investigate the effectiveness of silica nanoparticles to modify sandstone rock surface from oil-wet to water-wet using a reservoir in the Kolo Creek oilfield as a case study. Special attention was put on the effect of brine salinity on the effectiveness of nanoparticles wettability alteration potential. The Kolo Creek oilfield is situated in the Niger Delta oil producing zone of Nigeria, and the prospect of commencing enhanced oil recovery operation in the field is being considered but this requires intensive laboratory investigations before field trials. The previous study by Onyekonwu and Ogolo [10] carried out an extensive study on enhanced oil recovery potential of different nanoparticles on sandstone cores from one of the reservoirs in this zone and wettability alteration was one of the mechanisms attributed to the nanoparticles enhanced oil recovery process. They however did not carry out wettability study on the core samples. Also, recent study by Udoh [20] showed that application of silica nanoparticles (1 g/L) in secondary and tertiary injection modes in core plugs from this oilfield can improve oil recovery significantly. The mechanism underlying this positive effect of nanoparticles is not well understood hence, this study aimed at investigating the wettability alteration potential of silica nanoparticles in oil-wet rock sample from the oilfield. The result of this study is relevant to the design and implementation of nanoparticles enhanced oil recovery process.

2. MATERIALS AND METHOD

The materials and method used in this study are presented in this section.

2.1 Samples Preparation

Brine: Table 1 presents the composition and salinities of the brines used in this study. The synthetic brines were prepared based on the formation brine of the reservoir used as a case study with the reagent grade NaCl, $CaCl_2$, $MgCl_2$, KCl and Na_2SO_4 salts in deionized water. The formation brine salinity is 32,000 ppm of which 98.2% is NaCl, 0.6% is $CaCl_2$, 0.8% is $MgCl_2$, 0.2% is KCl and 0.2% is Na_2SO_4. Different ranges of salinity used to investigate salinity effect are 10D, 20D, 50D and 90D brines prepared by dilution of formation brine (FMB) with deionised water based on 10-, 20-, 50- and 90-percentage dilution factors, respectively.

Table 1: Compositional breakdown of brine solutions.

Components	FMB (ppm)	10D (ppm)	20D (ppm)	50D (ppm)	90D (ppm)
NaCl	31424.00	3142.40	1571.20	628.48	349.1556
$CaCl_2$	192.00	19.20	9.60	3.84	2.1333
$MgCl_2$	256.00	25.60	12.80	5.10	2.8444
KCl	32.00	3.20	1.60	0.64	0.3556
Na_2SO_4	32.00	3.20	1.60	0.64	0.3556

Crude oil: The crude oil used in this study is a degassed crude oil from the Kolo Creek oilfield reservoir used for the case study. The properties of the crude oil measured at 25 ^oC are presented in Table 2.

Table 2: Crude oil properties.

Oil Properties	Quantity
Density at 25 ^oC (g/cc)	0.9067
API at 25 ^oC (◦)	24.5673
Viscosity at 25 ^oC (cp)	15.2206

Nanoparticles: The silica nanoparticles used in this study are 99.5% non-porous silica (SiO_2) with no surface treatment. The SiO_2 was supplied by Skyspring Nanomaterials, Inc., Houston, USA in powdered form. The aqueous stock solution of

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nanoparticles was prepared with deionised water under continuous mixing until all the particles were completely dispersed in the solution. The properties and description of the nanoparticles are presented in Table 3, while the TEM image is presented in Figure 1 (Skyspring Nanomaterials Inc. website).

Table 3: Properties and description of SiO_2 nanoparticles

Properties	Quantity
Bulk density (g/cm^3)	0.01-0.10
Surface area (m^2/g)	160.00
Size (nm)	20.00
Morphology	Spherical
Colour	White

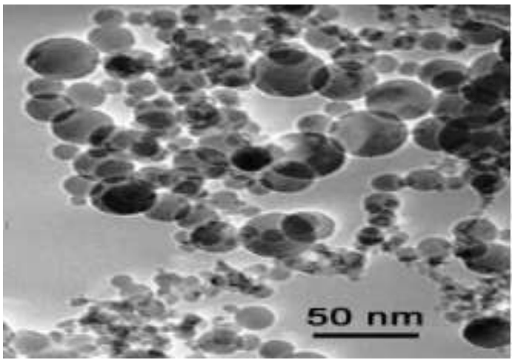

Figure 1: Image of the SiO_2 nanoparticles.

Solid surface and rock samples: For the contact angle experiments, the smooth microscopic glass slides (28 by 48) from Menzel-Glaser, Germany were used as the representative of rock surface. While intact sandstone core plugs were used for the Amott wettability index experiments. The core samples were obtained from the Kolo oilfield reservoir that is used as a case study in this work. X-ray diffraction (XRD) analysis of the rock sample shows that it has different compositions, of which Quartz (silica) is the largest component with a total concentration of 64.94%. The compositions of the sandstone rock are similar to that of the glass with both having silica as the dominate composition, although the glass has a higher composition (72.2%) than sandstone rock with a total composition of 64.94%. The compositional breakdown of the microscopic glass and the sandstone rock are presented in Tables 3 and 4, respectively.

Table 3: Compositional breakdown of the microscopic glass.

Microscopic glass	
Composition	**Quantity (%)**
SiO_2	72.20
Na_2O	14.30
K_2O	1.20
CaO	6.40
MgO	4.30
Al_2O_3	1.20
Fe_2O_3	0.03
SO_3	0.30

Table 4: Compositional breakdown of the Kolo sandstone rock.

Sandstone Rock	
Composition	**Quantity (%)**
Davyne: ($Na_4K_2Ca_2Si_6Al_6O_{24}(SO_4)Cl_2$) K_2O (6.78%), Na_2O (10.12%), CaO (12.57%), Al_2O_3 (29.15%), SiO_2 (32.14%), SO_3 (4.43%), Cl (6.77%).	2.30
Quartz: (SiO_2)	57.20
Garnet: ($A_3B_2Si_3O_{12}$) A: (Fe^{2+}, Ca^{2+}, Mg^{2+}, Mn^{2+}); B: (Fe^{3+}, Al^{3+}, Cr^{3+}), Si_3O_{12}.	12.40
Celestine: ($SrSO_4$) SrO (56.41%), SO_3 (43.59%)	10.60
Allophane: ($Ca_3Al_2(SiO_4)_3$) CaO (37.35%), Al_2O_3(22.64%), SiO_2 (40.02%).	17.50

2.2 Experimental Methods

Contact angle: To investigate the effect of brine salinities and nanoparticles concentration on the wettability of oil-wet surfaces, a series of sessile drop measurements were performed using the Ossila contact angle goniometer (L2004A1). Each fluid droplet was 5 µl with each measurement repeated three times and the average values presented in the results section. Prior to measurement, the glass slides were cleaned thoroughly with ethanol and deionized water, dried, and then aged in crude oil for a minimum of 6 weeks at 100 °C to ensure the surfaces were thoroughly oil-wet as required for rock surface wettability alteration [21]. The experiments were carried out in two phases with salinity effect been investigated in the first phase and silica nanoparticles effect been investigated in the second phase. For the salinity effect, five brines (FMB, 10D, 20D, 50D and 90D) were used, while for the nanoparticles, two concentrations (1 g/L and 5 g/L) were used. All tests were carried out at 25 ^oC.

Amott wettability index: Two intact core samples of the Kolo oilfield reservoir were used for the Amott wettability index test. The objective of this test is to investigate the wettability alteration potential of silica nanoparticles relative to the formation brine in the rock cores. This is relevant to water flooding processes and the application of nanoparticles enhanced

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oil recovery. Before the commencement of the test, the core samples were first cleaned with methanol and toluene in a Soxhlet extractor for 24 h. The cores were then dried in the oven for 48 h at 100 ºC. After cooling, the cores were saturated with formation brine under vacuum for 24 h, and then further saturated under pressure with injection of two pore volume of formation brine to ensure all the pores were saturated with the brine. Finally, the cores were flooded with crude oil down to the irreducible water saturation point and then aged in crude oil for six weeks at 100 ºC. for wettability alteration. After ageing, each core sample was then subjected to spontaneous imbibition in the Amott cells containing the relevant aqueous solution for eight weeks and the volume of produced oil was recorded until no oil production was observed. The forced imbibition process was carried out on the cores with injection of brine identical to the one used in the preceding stage at 1 mL/min flow rate until no further oil production was observed. The volume of oil produced from spontaneous and forced imbibition were used to calculate the Amott wettability index of water using Equation 1 [21].

$$I_w = \frac{\Delta S_{ws}}{\Delta S_{ws} + \Delta S_{wf}}, \qquad (1)$$

where ΔS_{ws} is the change in water saturation during spontaneous imbibition and ΔS_{wf} is the change in water saturation during forced imbibition.

3. RESULTS AND DISCUSSION

Figure 2 shows the results of the contact angle measurements of the oil-wet glass surfaces in which the effect of varied nanoparticles concentrations on wettability alteration under varied saline conditions was considered. Generally, reduction in contact angle was observed with increase in the concentration of silica nanoparticles, which signifies a modification in surface wettability from oil-wetness toward water-wetness. The lowest wettability alteration (i.e., highest contact angles) was observed with the use of deionised water without any electrolytes, while the highest alteration (i.e., lowest contact angles) was observed with the low salinity brine. The significant effect of solution composition and salinity effect is shown in Figure 3, where increase in contact angle values was observed with increase in brine salinity with or without nanoparticles. From the results, it is obvious that the wettability alteration potential of silica nanoparticles reduces with increase in brine salinity irrespective of the concentration be used. Also from this result, it is evident that higher concentration (5 g/L) of silica nanoparticles does not significantly alter the surface wettability relative to lower concentration (1 g/L). This shows that the effectiveness of this silica nanoparticles is better enhanced in the presence of small concentration of salt relative to deionised water and high salinity brines.

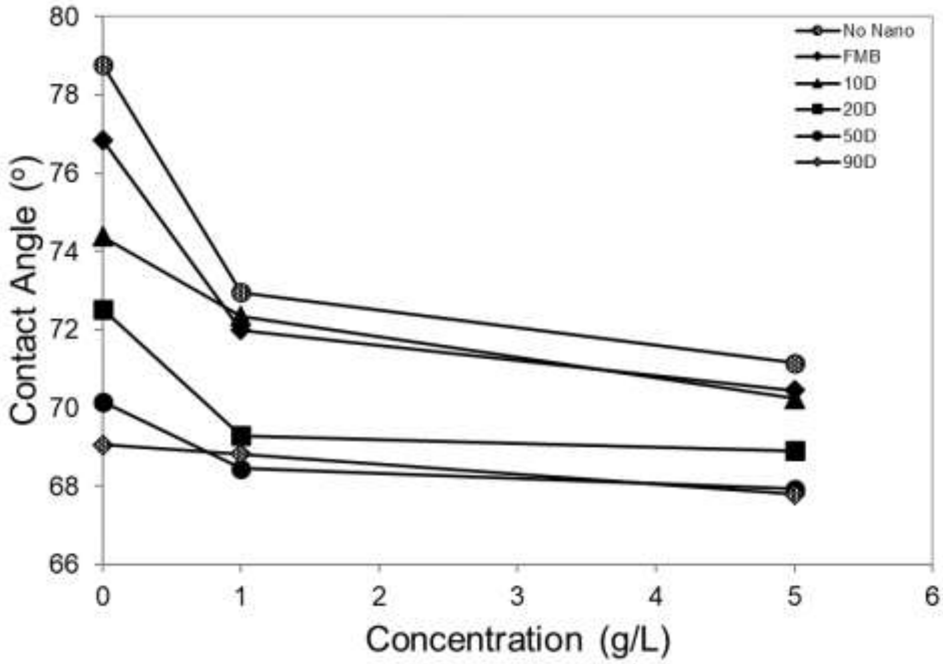

Figure 2: Contact angle measurements of the effect of varied concentration of silica nanoparticles in different solutions.

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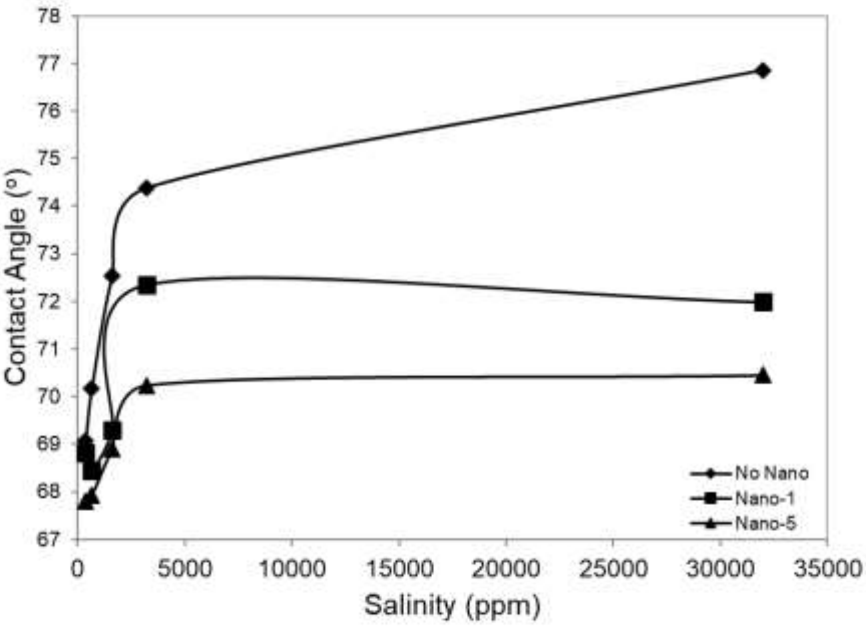

Figure 3: Contact angle measurements of the effect of varied brine salinities.

Following the oil-wet surface wettability alteration investigation using the contact angle method, the Amott wettability index test was conducted on two oil-wet core plugs. The first core imbibes the formation brine (FMB) while the second core imbibes the 1 g/L silica nanoparticles solution. This lower concentration was used based on economic factor since not much wettability modification was achieved with the use of higher concentration. Figure 4 shows the images of the spontaneous imbibition process that lasted for eight weeks. At the end of the imbibition process (Figure 4b), higher oil production was achieved with the solution of nanoparticles relative to that of the formation bine. Worthy of note is the sedimentation of the nanoparticles at the base of the imbibition cell and on top of the core plug that was observed during the process (Figure 4c). This shows that most of the dispersed nanoparticles in the solution at the commencement of the spontaneous imbibition process (Figure 4a) did not imbibe the core plug during the imbibition process as evident by the deposition of these particles

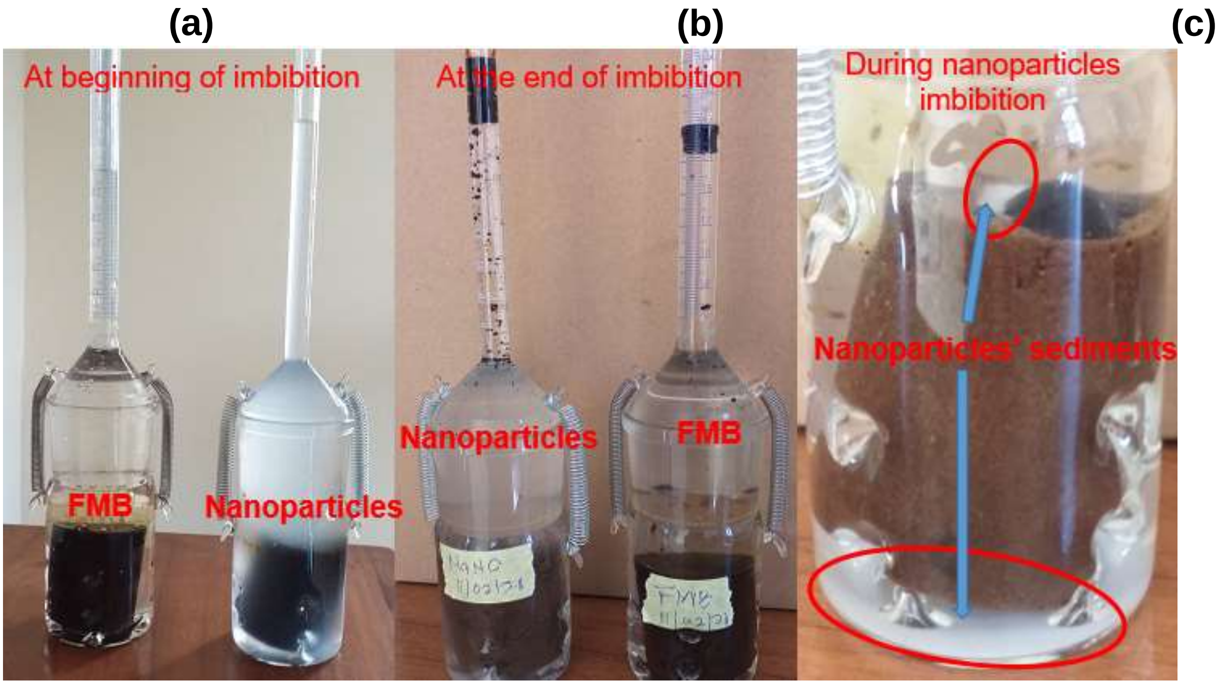

Figure 4: Spontaneous imbibition of formation brine (FMB) and silica nanoparticles (Nano) in oil-wet core plugs. (a) image of the oil-wet cores in imbibition solutions at the commencement of the imbibition process, (b) oil production at the end of eight weeks imbibition process and (c) nanoparticles sedimentation observed at the base of the cell and on the core during nanoparticles imbibition process.

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in cell. The observed higher oil production from the process however suggests that some of these nanoparticles imbibed the core and enhanced oil production. Furthermore, lower oil production was made during the force imbibition of the solution of nanoparticles relative to that of the FMB. This further confirms higher spontaneous imbibition of the nanofluid in oil-wet core.

The results of the Amott wettability index to water calculated using Equation 1 are presented in Figure 5. Higher wettability index was observed with nanoparticles imbibition process which means the presence of nanoparticles in the brine makes the rock imbibed more water and expelled more oil, thereby signifying its wettability modification capacity. Since higher Amott wettability index to water indicates increase in water-wetness, the results of this test showed that the application of silica nanoparticles in oil-wet core plug imbibition process has the potential to modify the wettability towards water-wetness. The results of this imbibition process agree with the results of the contact angle measurements in which the application of nanoparticles modified oil-wet surfaces towards water-wetness. This wettability alteration process has the tendency to improve oil production as evident in higher oil expulsion from the rock pore during nanoparticles imbibition process. The result of this study is consistent with the previous studies [13, 22] that observed reduction in contact angle with decrease in brine salinity and increase in contact angle with increase salinity. The wettability alteration potential of nanoparticles is associated with their interaction with brine in the system that will result in formation of amorphous silica nanoparticles structure at the rock-oil-brine interface and hence, alteration of the rock surface wettability [23, 24]. Furthermore, the ions of the nanoparticles interact with the ions of the salts in brines thereby modifying the electric double layer at the interface [23]. The theoretical equation for the calculation of the thickness of diffuse electrical layer is given by Equation 2 where I is the ionic strength of the salt solutions [25]. From this equation, it is obvious that brines with higher ionic strength will be characterised by reduced electric layer thickness and at very low salt concentrations the nanoparticles dispersion becomes less stable. This explains while the lowest wettability alteration was observed nanoparticles-deionised water relative to nanoparticles-brines system. The results of this study are relevant to enhanced oil recovery application of silica nanoparticles in Kolo oilfield reservoir. This study has showed that the application of nanoparticles in the reservoir rock has the potential to modify the rock surface wettability from oil-wet toward water-wetness. This is consistent with previous studies [9-11] that attributed the increased oil recovery from the application of nanoparticles to wettability alteration.

$$k^{-1} = \frac{1}{3.288\sqrt{I}}\ (nm). \qquad (2)$$

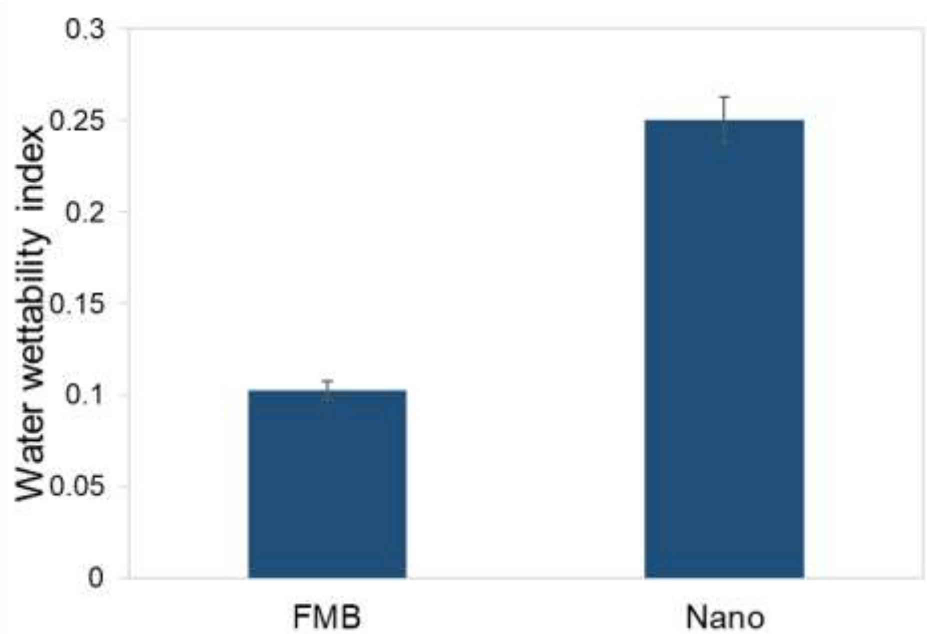

Figure 5: Amott water wettability index using formation brine (FMB) and 1 g/L nanoparticles solution.

4. CONCLUSION

The wettability alteration potential of the application of nanoparticles in oil-wet surfaces and core plugs was investigated in this study. The results of the study showed that oil-wet surfaces wettability can be modified toward increased water-wetness with reduction in brine salinity with or without addition of nanoparticles as evident by reduction in contact angles values from 79^{o} to 71^{o}. Addition of nanoparticles to brines of varied salinities also reduces oil-wet surfaces contact angles from 79^{o} to 68^{o} and modifies the surface wettability towards water-wetness. Increase in the concentration of nanoparticles result in increase in oil-wet surface wettability alteration toward water-wetness. Application of nanoparticles in oil-wet core

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spontaneous imbibition results in higher oil production relative to the formation brine imbibition. Finally, the application of nanoparticles resulted in higher Amott wettability index to water of 0.25 relative to 0.1 of formation brine thereby demonstrating wettability alteration potential toward water-wetness. Hence, application of silica nanoparticles in Kolo oilfield reservoir rock has the potential to modify the rock surfaces toward water-wetness and increase oil production.

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