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Laboratory Characterization of crude oil and sandstone reservoir for Chemical Enhanced Oil Recovery

Abstract

Purpose- Because of the increasing global oil demand, efforts have been made to further extract oil using chemical enhanced oil recovery (CEOR) methods. However, unlike water flooding, understanding the physicochemical properties of crude oil and its sandstone reservoir makeup is the first step before embarking to CEOR projects. These properties play major roles in the area of EOR technologies and are important not only for the development of reliable chemical flooding agents, but are key parameters used to evaluate the economic and technical feasibilities of production and refining processes in the oil industries. Consequently, this study is aimed at investigating various important physicochemical properties of crude oil (specific gravity, API, viscosity, pour point, BS & W, wax and SARA components) and sandstone reservoir makeup (porosity, permeability, bulk volume and density, grain volume and density, morphology and mineral composition and distributions) obtained from Malaysian oil field (MOF) for oil recovery prediction and design of promising chemical flooding agents.

Design/methodology/approach- Three reservoir sandstones from different depths (CORE 1; 5601, CORE 2; 6173 and CORE 3; 6182 ft) as well as its crude oil were obtained from the MOF, and various characterization instruments, such as HT-GC and column chromatography for crude's fractions identification, GC-Simdist for boiling point distribution, POROPERM for porosity and permeability, CT-Scan and SEM/EDX for morphology and mineral distribution, wax instrument (wax content), pour point analyser (pour point), visco-rheometre (viscosity) etc, were employed for the characterizations.

Findings- Experimental data gathered from this study shows that the field contains low viscous (0.0018-0.014 Pa.s) sweet and light typed crude because of low sulfur content (0.03 %), API gravity (43.1°), high proportion of volatile components (51.78 %) and insignificant traces of heavy components (0.02 %). Similarly, the rock permeability trend with depth was found in the order of CORE 1 < CORE 2 < CORE 3, and other parameters like pore volume (Vp), bulk volume (Vb), grain volume (Vg) also decreases in general. For grain density, the variation is small and insignificant, but for bulk density, CORE 2 records lower than CORE 3 by more than 1%. In the mineral composition analysis, the CORE 2 contains the highest identified mineral content, with the exception of quarts where it was higher in the CORE 3. Thus, a good flow crude characteristic, permeability trend and the net mineral concentrations identified in this reservoir would not affect the economic viability of the CEOR method and predicts the validation of the MOF as a potential field that could respond to CEOR method successfully.

Originality/value- This paper is first of its kind to combine the two important oil field properties to scientifically predicts the evaluation of an oil field (MOF) as a step forward towards development of a novel chemical flooding agents for application in EOR. Hence, information obtained from this paper would help in the development of reliable chemical flooding agents and designing of EOR methods.

Keywords: Characterization, Enhanced oil recovery, sandstone reservoir, oil field

1. Introduction

Enhanced oil recovery (EOR) strategies promote the extraction of trapped oil after primary and secondary methods have been exhausted (Alvarado and Manrique 2010; Alagorni et al. 2015; Sabet et al., 2016; Hamza et al., 2017). These extraction technologies have become necessary to enable the provision of sufficient oil for increasing domestic and industrial needs (Zaid et al. 2014; Salehi et al. 2014; Patwardhan et al., 2015). Among the EOR processes, chemical EOR (CEOR) has been a subject of renewed interest in the field of hydrocarbon recovery. Reason being that the chemicals used in the

CEOR have good working mechanisms in the oil reservoir and have advantage over thermal and instu methods for their ability to reduce the IFT, encouter viscous fingering, promotes sweep efficiency and improved recovery factor. Common examples of these CEOR methods include the injection of surfactant, nanoparticles (NPs) and polymer, and sometimes involves the combination of these chemicals (Legarto et al., 2017). Many laboratory and oilfield scale studies that demonstrate the successful application of CEOR methods have been reported (Hou et al., 2012; Sheng 2013; Kothencz et al., 2017; Towler et al. 2017; Mellat and Jamialahamadi 2017). However, reservoir conditions, crude oil and sandstone mineral make up signnificantly reduce the performance of chemicals used in the CEOR (Gary et al. 2007; Speight 2011; Selley and Sonnenberg 2014; Speigh 2014). These effects could be in the form of precipitation due contact with oil, degradation due to temperature, adsorption due to rock properties, absorption due to mineral content (clay) etc (Soleimani et al. 2014). Similarly, Adepoju et al. (2017) provides a clear understanding of proper CEOR flooding designs by taking cognisance of the chemical and field properties. The authors believed that the field application of the CEOR remains a challenge due to variuos uncertainties like the trapping number, inaccessible pore volume, permeability reduction and reservoir hetrogeineity among others, but emphasized that the reservoir hetrogeinity is the dominant uncertainty and reccommends the reservoir characterization as a key requirement during the evaluation and design of the CEOR. In addition, Abduslam (2012) reported that EOR projects can be better designed through adequate understanding of the petrophysical properties of a reservoir, this could provide proper prediction of a reservoir quality and heterogeneity, and to the assessment of potential reservoir production, and ultimate recovery. As such, careful investigations should be made to fully understand the properties of oil fields under investigation for the purpose of improving recovery and estimation of operational costs of processing facilities (Adetoro et al. 2015). One of the important steps prior to embarking on EOR projects, is to know the relevant physicochemical parameters of crude oil (such as viscosity, density, basic sediment and water, pour point, distillates, saturates, resins, aromatics etc.), and reservoir properties (permeability, porosity, pore volume, grain density, grain volume, mineral distributions etc.) using spectroscopic and analytical procedures. This would provide substantial information that would enable the development of promising flooding chemicals and suitable alternative extraction strategies that could promote the recovery of large trapped oil in the porous media. This paper is aimed at investigating the relevant physicochemical parameters of a Malaysian oil field (MOF) to assess its potential for improving recovery using CEOR methods. The paper serves as a preliminary stage for our ongoing project towards the development of a novel chemical EOR flooding agent for application in MOF.

2. Material and Methods

2.1 Sample collection

From the MOF reservoir, three core samples obtained from different depth were labelled as; CORE 1 (5601 ft.), CORE 2 (6173 ft.) and CORE 3 (6182 ft.). The crude oil sample was collected from the same MOF. The sandstone cores were kept air dried in core laboratory and the crude oil was stored at 20 °C for further analysis. Prior to each analysis, the crude oil was thoroughly shaken for homogeneity.

2.2. Physicochemical analysis of crude oil

2.2.1. Determination of density, specific gravity and American petroleum institute gravity (API)

The density and specific gravity were measured at 15.5, 25, 40, 85 and 90 °C using a bench top digital Anton Paar density meter (DM-40). The temperature was selected to represent reservoir conditions. The meter operates by oscillating U-tube principle and measures $\pm 5 \times 10^{-5} \text{ g cm}^{-3}$ as a precision, as per requirement for ASTM D4052-96 and D5002-99. Built-in Peltier Thermostat helps stabilize the measuring temperature. Approximately, 10 ml of the crude sample was injected into the sample tube and analyzed at the aforementioned temperatures. Density and specific gravity results were then recorded in g cm^{-3} . The same experiments were performed in triplicate to obtain average results in each run for accuracy.

The specific gravity measurement obtained at 15.5 °C (60 °F) was converted to API° using Equation (1);

$$\text{API (60 °F)} = (141.5 / (\text{SG } 60 \text{ °F})) - 131.5 \dots \dots \dots (1)$$

Where; API = American Petroleum Institute
SG = Specific gravity at 60 °F

2.2.2 Viscosity measurement

The dynamic viscosity of the sample was measured according to ASTM D445 using Modular Compact Rheometre (MCR 302). The viscosity was analyzed at temperature between 20-100 °C to ascertain the flow behavior relative to reservoir temperature.

2.2.3 Pour point measurement

Crude oil pour point measurement was done in accordance to the ASTM D97-04 method using cloud and pour point analyzer (Stanhope-SETA-94100-3). For this measurement, the sample was cooled 10 °C above the expected pour point and transferred to cloud and pour point chamber maintained at -7 °C. A measurement was taken at multiple of 3 °C until no movement of fluid was observed within 5 sec by holding the tube in horizontal position. The pour point was reported as a multiple of 3 °C at this observed temperature.

2.2.4 Basic sediment and water (BS & W)

BS & W was determined according to the procedure described in ASTM D4007 manual. The test method describes the laboratory determination of BS & W by a centrifuge machine. Annular grade water saturated toluene was used. Equal volumes of the sample and saturated toluene (50:50) were combined in a 100 ml centrifuge tube with an aliquot amount of demulsifier added to break the emulsion. The centrifuge tube containing the sample mixture was then placed in a water bath at 60 °C for 15 min. It was then centrifuged at 1420 rpm for 15 min and the value of BS & W was recorded in volume %.

2.2.5 Wax content

Wax content was analyzed using Oxford instrument (Oxford 4000). The working temperature was 40 °C. Approximately, 1.5 ml of the crude oil sample was transferred into the wax tube and heated at 90 °C for 1 hour. At the end of the predetermined time, 0.5 ml chloroform was added, shaken thoroughly and placed in a water bath at -28 °C for 3 hours. The mixture was then analyzed by the instrument and result reported in weight by volume %.

2.2.6 High temperature simulated distillation (HT-SimDist) and high temperature gas chromatography (HT-GC)

Simulated distillation is a gas chromatography technique, which separates crude oil fractions in order of boiling point and mass. ASTM D7169-11 method was used for this separation using GC-7 SimDist and DHA-FE. Prior to the measurement, the sample was diluted with CS₂ and then transferred into vials. The diluted sample was then loaded into the GC-7 magazine for analysis. Another separate sample was also diluted with internal standard (iso-octane) and analyzed using DHA-FE. The results obtained were merged to improve the accuracy of the analysis. The carbon distribution pattern of the crude oil was analyzed using a high temperature gas chromatography (HT-GC) spectrometry.

2.2.7 Saturate, aromatic, resins and asphaltenes (SARA) analysis

SARA were qualitatively and quantitatively determine by refluxing the sample in n-heptane, Dichloromethane and methanol on a heating mantle, respectively. Thereafter, the fractions obtained according to their boiling points were concentrated using a rotary evaporator and dried until constant weights were obtained. These were then separated according to their polarities in a column packed with alumina, at a constant flow rate of 2 ml min⁻¹.

2.2.8 Determination of total sulfur content

Total Sulfur was analyzed using an Automated Sulfur Content Analyzer instrument (iPRO 5000) in accordance to ASTM D5453. The sample and dibutylsulfide were used to prepare a stock solution of 100 ml. Thereafter, a toluene was used to prepared a series of aliquot solutions (0.5-55 ml) from the sample stock solution. The solutions were then loaded into the instrument and the average concentration of sulfur was reported in ppm.

2.3. Sandstone core sample analysis

Three core samples collected from different depths were labeled; CORE 1 (5601 ft), CORE 2 (6173 ft) and CORE 3 (6182 ft), respectively (Figure 1). Direct measurements were carried out to determine the length and diameter using the Digital Vaniar Caliper (CD-6" C), while the weights were measured using a weighing balance. In order to check the physical conditions of the cores, CT-Scan was conducted at 0 and 90 °C using a CT-Scan machine. Porosity and permeability were also determined using POROPERM (CORVAL 30) to measure the cores grain space and connectivity in the cores. Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX) was carried out to investigate the morphology and mineralogical compositions in the cores (Yuan et al., 2015).

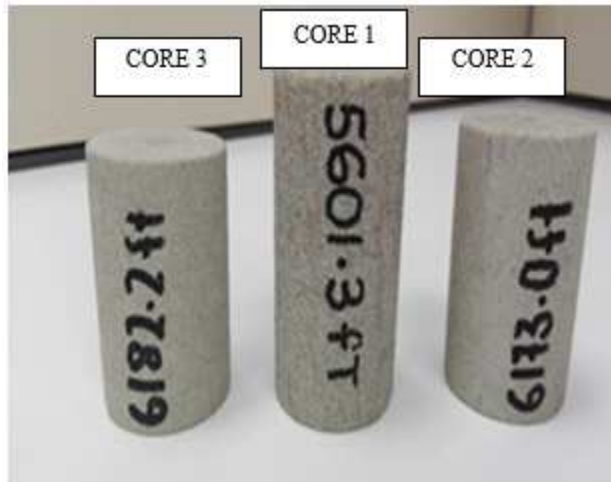


Figure 1 Three different MOF core samples characterized in this study

3. Results and Discussion

Table 1, shows the results of physical and chemical properties of MOF. From the table, it can be seen that the field contains sweet and light typed crude having a total sulfur content of 0.03 % and an API gravity of 43.1°, respectively. Crude oil that contains total sulfur < 0.5 % and API > 10° is regarded as a sweet and light crude oil which can easily be processed into different fractions. Sweet and light crude is not problematic, and has less environmental impact during exploration, processing and transportation (Jadoon et al. 2016). This indicates that, due to the low cost of processing of light crudes, execution of EOR method in MOF would be economically and technically feasible and with less environmental concern.

Both the density and specific gravity were found to decrease with increase in temperature (15-90 °C), which was due to increase in kinetic energy which caused molecules to collide with one another and spread apart, resulting to fewer molecules per given volume. This is typically the characteristic of light crudes, which is also in accordance with the findings of Ekaputra, et al (2014). This means that the injected chemical should be designed to have higher density greater than the crude oil to overcome the effect of density segregation.

Pour point and wax content were found to be 15 °C and 8.51 %, respectively. Which are within the accepted range for crude oil to have smooth flow in the formation. Higher pour point and wax

deposition affect the flow of crude oil in the reservoirs which result to poor production rate. In crude oil, a lower pour point is generally associated with a low paraffin content (basically straight chain molecules which can precipitate at lower temperature). Therefore, It has become necessary during the design of CEOR technologies to examine these parameters in oil fields to evaluate the production rate and profitability (Kelechukwu 2011).

BS & W was also reported as 0 % (below detection limit). Low BS & W reduces emulsion formation which improves the mobility of the crude. Similarly, Low BS & W indicates the purity of crude and make it potential pricing benchmark for many countries (Valadkhani et al. 2013).

SARA analysis from Table 1 revealed that the crude contains high proportions of volatile compounds (51.78 %) as the main hydrocarbon fractions followed by saturates (33.33 % wt) with little traces of asphaltenes (0.02 % wt) and inorganic salts (0.01 % wt). This information is crucial for identifying crudes with asphaltenes dropout problems which can cause pore clogging. All these data conform to reported literature (Mcclafin and Whitfill 1984; Kelechukwu 2011).

Viscosity is a key parameter used to determine the flow properties of crude oil in the reservoir under certain conditions of temperature and pressure (Naiya 2015). Figure 2, shows the effect of temperature on the viscosity of crude oil. From the graph, it can be observed that, viscosity of MOF crude at varied temperatures was found to be decreasing with an increase in temperature. This indicates that it is apparent for MOF crude to have a smooth flow characteristics in the underground formation.

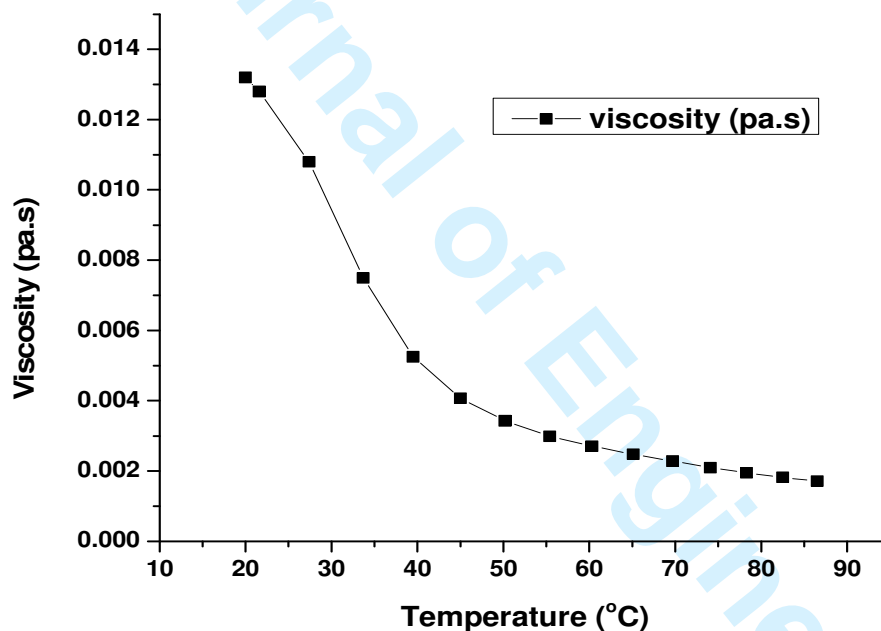


Figure 2 Effect of temperature on the viscosity of MOF crude oil

The result of the carbon distribution pattern of MOF crude is shown in Figure 3 and Table 2, it can be seen from the results that the crude has compounds from C_4 to C_{42} carbon distributions with heavy component ($C > 22$) found in trace amounts. The presence of trace amounts of heavy components or bulky molecules is a credit to this crude due to the anticipated clogging of pores by heavy components. EOR extraction of crude oil containing light components is more economical than the crude with heavy molecules. This crude would have greater extraction advantages using CEOR methods which can be economically processed.

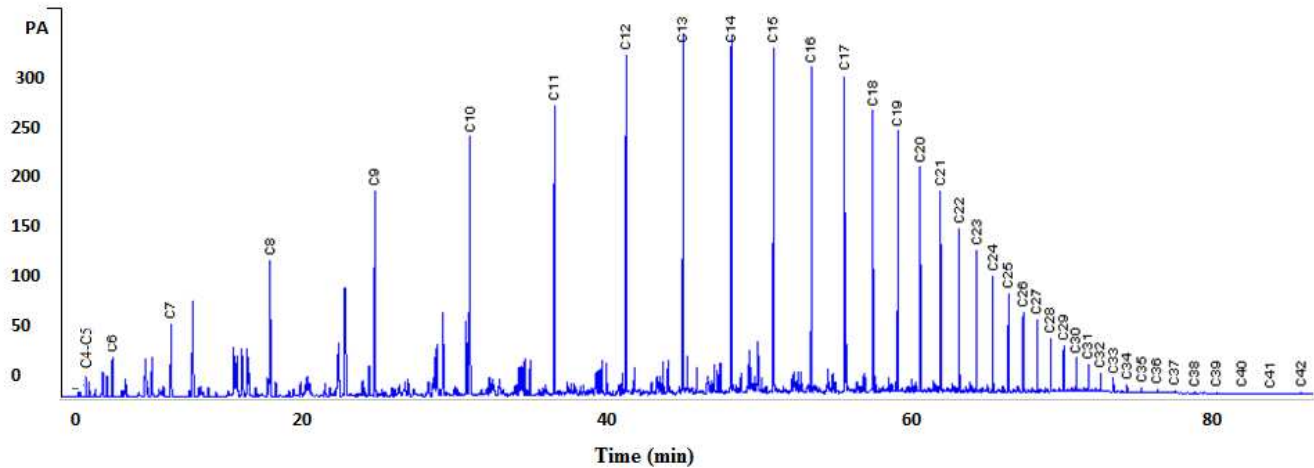


Figure 3 Carbon distribution of MOF crude oil using HTGC

Table 1 Physical and chemical properties of MOF crude

Test Parametre	Method	Unit	Result
<u>Density at Temperature (°C)</u>			
15.5	ASTM D4052-96	g/cm ³	0.8064
25		g/cm ³	0.7992
40		g/cm ³	0.7886
85		g/cm ³	0.7562
90		g/cm ³	0.7527
<u>Specific gravity at Temperature (°C)</u>			
15.5		g/cm ³	0.8101
25		g/cm ³	0.8016
40		g/cm ³	0.7948
85		g/cm ³	0.7807
90		g/cm ³	0.7798
API		°	43.100
Pour point	ASTM D97-04	°C	15.000
Wax content		%	8.5100
Total Sulfur	ASTM D5453	%	0.0300
Basic sediment and water (BS & W)	ASTM D4007	Vol %	0.0000
<u>SARA</u>			
Volatiles	Column chromatography	mass%	51.780
Inorganics		mass %	0.0100
Saturate		Mass %	33.333
Asphaltene		mass %	0.0200
Resins		mass %	12.800
Aromatics		mass %	3.8700
Total recovery		mass %	101.80

HT-SimDist experiment was conducted to determine the boiling point distribution of the crude's components and it describes the volatility behavior of hydrocarbon mixture . Figure 4, shows the plot of boiling point and mass recovery. From the graph, it can be seen that the crude components started to recover at initial boiling point (IBP) of 60.5 °C and up to 99 % mass at 506.0°C. This indicates that, the crude contains light to medium fractions (Table 2).

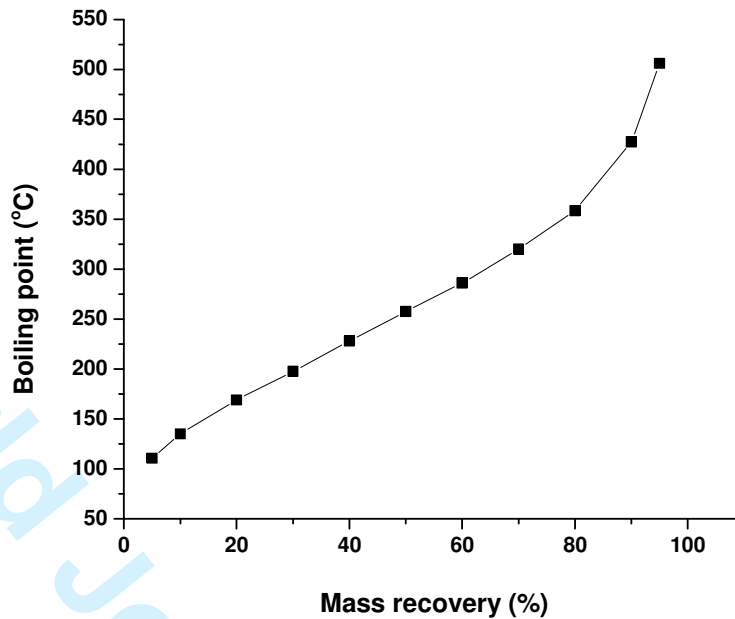


Figure 4 Boiling point curve distribution of MOF crude oil

These properties further supported the extractability properties of this crude. These light fractions can dilute the higher fractions, as a result improved mobility of the crude during CEOR could be achieved. The shape of the curve obtained is in agreement with the work of Behrenbruch and Dedigama (2007).

Table 2 Carbon number distribution of Malaysian crude with respect to % mass recovered

S/N	Carbon Number	Mass %	S/N	Carbon Number	Mass %
1	iC4	0.02	22	C23	1.61
2	nC4	0.02	23	C24	1.28
3	iC5	0.12	24	C25	1.15
4	nC5	0.08	25	C26	0.89
5	C6	0.81	26	C27	0.88
6	C7	2.76	27	C28	0.76
7	C8	7.30	28	C29	0.75
8	C9	8.68	29	C30	0.62
9	C10	9.30	30	C31	0.60
10	C11	8.05	31	C32	0.51
11	C12	7.61	32	C33	0.45
12	C13	7.04	33	C34	0.33
13	C14	6.76	34	C35	0.28
14	C15	7.18	35	C36	0.18
15	C16	5.31	36	C37	0.11
16	C17	4.46	37	C38	0.03
17	C18	4.01	38	C39	0.02
18	C19	3.24	39	C40	0.01
19	C20	2.64	40	C41	0.01
20	C21	2.32	41	C42	<0.01
21	C22	1.84			

The result of the CT-Scan analysis obtained is shown in Figure 5. From the Figure it can be observed that CT-Scan experiment revealed the core's physical conditions, no defects were observed. This is necessary to ensure accurate porosity and permeability data. Appearance of white traces is the indication of mineral distributions in the cores.

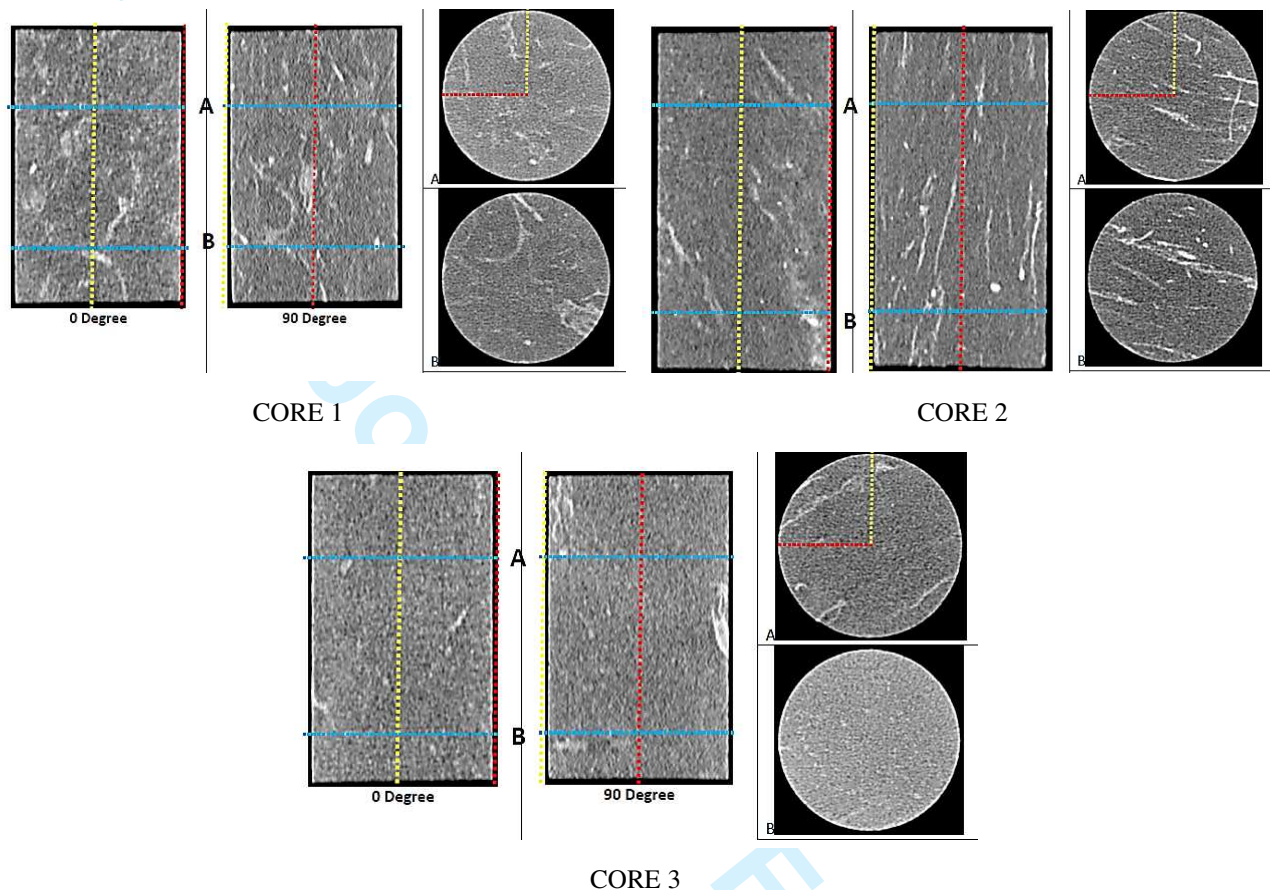


Figure 5 CT-Scan of three MOF reservoir stones

The results of the physical analysis conducted are presented in Table 3. From the same table, it is clearly seen that the permeability were noticed to decrease with increasing depth, this is due to effective stress and compaction of solid matrix as a result of increased in reservoir temperature and pressure with depth (Ramm and Bjorlykke 1994). Similarly, decrease in the bulk (V_b) and pore volume (V_p) with the depth, also influences the decrease of the rock permeability, other controlling permeability factors in support of this study have been explained in detail in the work of Ballas et al (2015). The variation of porosity is mostly governed by mud content and cementation. Other parameters like pore volume (V_p), bulk volume (V_b), grain volume (V_g) also decreases in general. When referring to bulk density and grain density, a slight abnormality was observed as depth increases. For grain density the variation is small and insignificant, but for bulk density, CORE 2 records lower than CORE 3 by more than 1%. This may be attributed to the composition of the minerals present at these depths. Based on the above observation, it can be assumed that the injected chemical fluids would have the possibilities to recover the residual oil in the formation, since flow connectivity (permeability) was observed to increase toward the surface (decrease in depth).

Table 3 Physical properties of three sampled reservoir cores

S/N	Sample code	Depth (ft)	Weight (g)	Length (mm)	Diameter (mm)	\emptyset (%)	V_p (cc)	V_b (cc)	V_g (cc)	ρ_g (g/c)	ρ_b (g/c)	K (mD)
1	CORE 1	5601	153.9	72.4	37.4	26.6	21.1	79.5	58.4	2.64	1.94	1066
2	CORE 2	6173	138.0	61.1	37.5	23.1	15.6	67.6	51.9	2.65	2.04	198.6
3	CORE 3	6182	124.6	56.3	37.6	24.3	15.2	62.6	47.34	2.63	1.99	61.72

To investigate the authigenic and other mineralogical compositions in the cores, SEM/EDX was conducted as seen in Figure 6. From the figure, portion of the rocks were analysed and the result presented in the Table 4. From the table, it was found out that the CORE 2 contains the highest identified mineral content, with the exception of quartz where it was higher in CORE 3. That could further explain the low porosity observed in CORE 2, because, mineral deposition affects net porosity and this is in accordance with the work reported in (Ramm and Bjorlykke 1994).

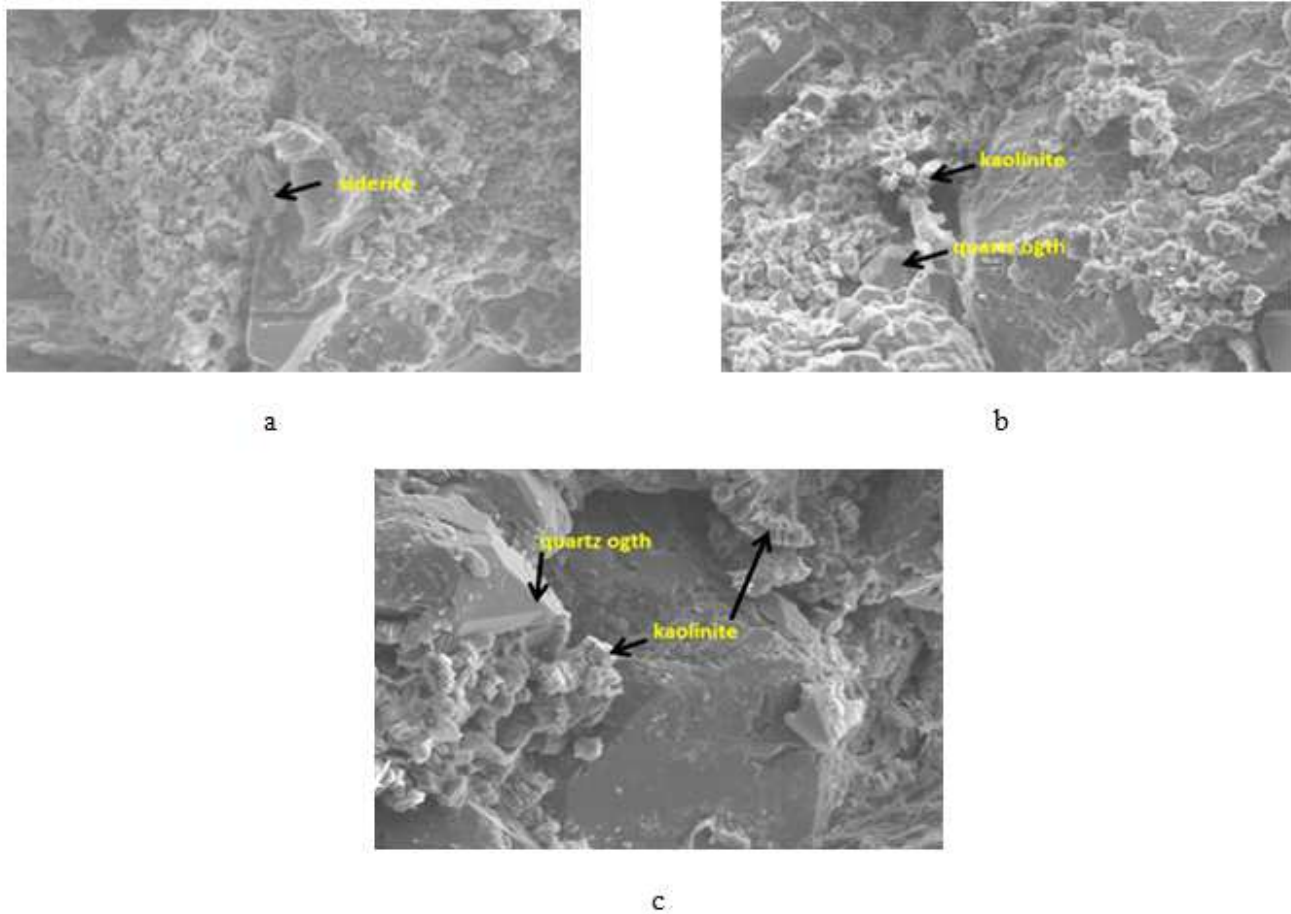
**Figure 6** SEM/EDX of MOF reservoir stones; a: CORE 1, b: CORE 2 and c: CORE 3

Table 4 Mineralogical compositions of cores

Rock comp.	CORE 1 (wt %)	CORE 2 (wt %)	CORE 3 (wt %)
Quartz	71.2	61.9	74.2
Plagioclase	7.30	9.10	6.10
K-feldspar	2.60	4.30	0.80
Calcite	3.30	3.60	3.30
Dolomite	4.10	4.50	4.20
Siderite	5.50	6.20	5.20
Pyrite	0.00	0.00	0.00
Total clay	6.00	10.4	6.20

Further analysis (clay composition) in three cores was carried out. The results are presented in Table 5. From the same table the distribution of clay composition was found to be unrelated to depth. This can be attributed to the deposition mechanism of the sandstone. The concentrations of minerals identified in this reservoir were low which would not affect the economic viability of CEOR method, reason being that, high mineral depositions, particularly clay, which has a high degree of reactivity could absorb and reduce the amount of injected chemical intended to push the oil to the surface, making EOR uneconomical (Michaelides et al. 2012; Adepoju et al., 2017).

Table 5 Clay composition in three cores

Clay Comp.	CORE 1 (wt %)	CORE 2 (wt %)	CORE 3 (wt %)
Kaolinite	28.0	25.5	26.5
Chlorite	13.8	14.3	14.5
Illite	26.2	23.3	18.6
Mixed layer I/S	30.6	33.9	39.5
Smectite	1.4	3.00	0.90

4. Conclusion

The physicochemical properties of crude oil and reservoir core samples investigated and discussed in this paper are essential and are the key indicators governing the prediction of the successful EOR operations. Based on the preliminary present findings, the MOF field could respond to CEOR methods. This research could help in the development and design of suitable chemical flooding agents with good properties relative to this field and other reservoirs.

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