SCA2003-43: DETERMINATION OF WETTABILITY OF IRANIAN CARBONATE RESERVOIR ROCKS IN RESTORED-STATE

Mohammad-Reza Esfahani, Iran Research Institute of Petroleum Industry
Ezzat-Allah Kazemzadeh, Iran Research Institute of Petroleum Industry
Seyyed-Majid Hashemi, Iran Research Institute of Petroleum Industry

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ABSTRACT

Many special core analyses, including capillary pressure, relative permeability, and water flooding efficiency are affected by the wettability of the core. In order to achieve reliable results, experiment must be performed on preserved or aged sample. Considering the importance of wettability factor in special core analysis data and their fundamental roles in reservoir studies, the present work was defined to search for wettability characterization of some Iranian carbonate formations. Samples were carefully cleaned and aged at reservoir temperature in order to prepare the restored conditions. Experiments were run at 90 degrees centigrade with crude oil and formation water. In this study a period of 40 days were allowed for aging with crude oil. Petrophysical properties of each sample were measured using both thin section and core flooding techniques.

The lithology of the samples consists of both dolomite and limestone. The qualitative evaluation of carbonate samples using relative permeability curves showed that there was a discrepancy between the result from the role of end-points and those from the role of cross over saturation. The cross over saturations in relative permeability tests at reservoir temperature showed that samples were oil wet.

In this research, the Amott and USBM tests have been run on samples in a combination procedure. The capillary pressure curves obtained by the centrifuge method were used in order to calculate the USBM wettability index. Quantitative results by the Amott method showed that most carbonate samples had intermediate wettability characteristics, but the results from combined Amott-USBM testing indicated that system is mostly oil-wet. Centrifuge experiments also produced capillary pressure curves that were used to evaluate the wettability are according to the method of U.S. Bureau of Mines.

INTRODUCTION

Wettability is a major factor controlling the distribution and flow of fluids in a reservoir. It has a strong influence on capillary pressure and relative permeability, therefore it is of critical importance in a reservoir development and management. To our knowledge, some wettability evaluations have been performed on carbonate rocks and reported in the literature. Treiber et al. [1] evaluated the wettability of 50 reservoir rocks. They showed 8% of carbonate reservoirs were water wet with contact angle between 0.0 and 75 degrees, 8% were of intermediate wettability with contact angle between 75 and 105 and 84% were oil-wet with contact angle between 105 and 180. Chilingar et al. [2] performed contact angle measurement on 161 carbonate samples and concluded that 15% of the rocks were strongly oil-wet, 65% were oil-wet, 12% had intermediate wettability and 8% were water-wet. There are different methods of qualitative and quantitative wettability measurement reported in the literature [3]. Qualitative measurements include imbibition's methods, microscopic visualization of fluids distribution and wettability evaluation using relative permeability curves [4] whereas quantitative methods are contact angle measurements, Amott metho d [3,5], and USBM method [6].

Wettability depends on many variables such as the composition of rock and fluids, temperature, pressure, and core handling procedure [7,8]. The most accurate measurements are made on native and restored-state cores [9]. The wettability of cores can generally be restored by a three-step process. The first step is to clean the core to remove all compounds from the surface. The second step is to flow reservoir fluids into the core. The core is saturated with the formation brine, followed by a crude oil flood to simulate the inflow of oil into the core. In the third step, the core is aged at reservoir temperature for a sufficient time to establish adsorption equilibrium. Certain additives used in drilling and completion fluids may alter the wettability of the core. Cleaning methods must be developed to remove surfactants, allowing restoration of plugs to natural wettability. Some of the ways to clean core samples include Distillation/Extraction (Dean stark and Soxhlet), flow-through core cleaning, centrifuge flushing, gas-driven solvent extraction, and supercritical fluid extraction and critical point drying. To date, only the Distillation/Extraction and flow-through methods have reportedly been used for core cleaning for restoration of wettability, while the other three methods have not been tested to determine their suitability for this purpose [10].

In this work the wettability has been determined by quantitative and qualitative methods. Crude oil and formation water were used and initial water saturation was established in the cores before the Amott test started. The third quantitative test that is used to measure the wettability is the USBM test developed by Donaldson et al. [6]. The test is relatively rapid. A major advantage over the Amott test is its sensitivity near neutral wettability. The test compares the work necessary for one fluid to displace the other. Because of the favorable free-energy change, the work required for the wetting fluid to displace the non-wetting fluid from the core is less than the work required for the opposite displacement. The required work is proportional to the area under the capillary pressure curve. The USBM method [3,11] then uses the ratio of areas under the two capillary pressure curves to calculate a wettability index, according to the equation

$$W = \log(A_1 / A_2) \qquad (1)$$

where A_1 and A_2 are the areas under the oil and brine drive curves, respectively. In this work, the USBM test was run in combination with the Amott test.

PROCEDURE

The experimental program was performed on eleven carbonate sample rocks from different Iranian oil producing carbonate formations. The reservoirs are denoted as Field M and Field R, and the plugs were taken from various lithological formations in the reservoirs.

First, the thin section of each sample was prepared and analyzed using microscope to study the lithology, mineralogy and pore geometry of samples. Then, the core plugs having the diameter ranged from 2.52 cm to 3.84 cm, and the length ranged from 4.23 cm to 5.27 cm were prepared. The core identifications, depths, intervals and diameters are given in Table 1. The samples were cleaned by Distillation/Extraction method [10]. In this method, the samples are placed in a Soxhlet or Dean-Stark apparatus; the solvent is continuously distilled, condensed, and then distributed over the top of the samples. In the Soxhlet apparatus, the samples soak in the hot solvent, which is periodically siphoned off, then distilled, condensed, and distributed back to the extractors. Toluene was used as a solvent to clean. All cleaned samples were placed in an unhumidified oven at a temperature of 120 degrees centigrade for 48 hours. A helium porosimeter and an air permeameter were used for porosity and absolute permeability measurements respectively. There is good agreement between porosity measurement using core plug and the values evaluated from thin sections. Identical water was used for Field M and R and the composition is shown in Table 2. The result of chemical analyses of a crude oil showed a composition of 30.7% Saturates, 48.2% Aromatics, 19.1% Resins and 2% Asphalthenes.

After initial preparation and evaluation of petrophysical parameters, the following steps were used to restore all samples to reservoir condition:

1- All plugs were placed into the saturator apparatus and vacuumed for 4 hours.
2- All plugs were saturated with formation water.
3- Plug samples were aged for 10 days in order to obtain an ionic equilibrium between rock minerals and formation water.
4- Plugs were then flooded with a minimum of 5 pore volumes formation water at reservoir temperature in order to measure the absolute water permeability.
5- Reservoir oil was injected at reservoir temperature into all plugs until no additional water was produced.
6- All samples were submerged in oil and allowed to age for 40 days at 90 ^{o}C in order to restore the reservoir equilibrium condition [9, 7,12].

After preparation of restored samples, following experiments were performed for determination of wettability. The experiments consisted of two main methods: qualitative and quantitative methods.

Qualitative Experiments

For qualitative experiments, we used relative permeability curves produced for each sample according to Craig [4]; the end-point values of relative permeability curves are measures of wettability. The wetting phase end-point relative permeability will be less than the non-wetting end-point, and the crossover saturation were Krw=Krnw can also serve as a wettability indicator. For example, in water-oil systems if the crossover saturation is more than 0.5 the system is water-wet and is oil-wet if it is less than 0.5. The rules of end-point values and crossover saturation are based on the fact that the non-wetting phase occupies the center of the pores as globules several pore diameter in length while the wetting phase moves through the small pores.

Relative permeability curves were obtained on five core plugs in Field M at reservoir temperature. A hot air bath was used to control the temperature of core holder during the experiment. Five to ten pore volumes of the reservoir oil were injected into the aged samples in order to measure the effective permeability to oil at initial water saturation. The incremental pore volumes of formation water were injected to displace the oil. Water-oil relative permeabilities were calculated using graphical techniques by the unsteady-state Jones and Roszelle method [13].

Quantitative Experiments

In this study, two quantitative methods were used for evaluating the wettability of core plugs, Amott method [3,5,11] and combined Amott/USBM method [3,11,14]. Note that all measurements were done at reservoir temperature (90^{o}C). Amott tests were performed at two conditions. 1-Amott test using the core flooding system. 2-Amott test using centrifuge for forced displacement of fluids. In Amott test using core flooding system the brine was first injected into the oil-saturated samples until the samples reached residual oil saturations (Sor), and then following steps were used to displace water by oil and subsequently displacing oil by water:

1- Spontaneous displacement of water by oil during 40-50 days; the displaced water was recorded as Vwsp.
2- Forced displacement of water by flooding until no more water was displaced; the displaced water was recorded as Vwd.
3- Spontaneous displacement of oil by water during 40-50 days; recording the displaced oil as Vosp.
4- At the final stage, the forced displacement of oil by water using flooding system; the displaced oil was recorded as Vod.

Based on above experiment steps, 3 different indices are defined as:

1- Water wettability index (WWI) as the ratio of Vosp to total displaced oil ……………………………….(Vosp+Vod)
2- Oil wettability index (OWI) as the ratio of Vwsp to total displaced water ……………………………….(Vwsp+Vod)
3- Amott-Harvey index as the difference between WWI and OWI.

In the Amott test, wettability is determined by the Amott-Harvey index ranging from +1 for complete water wetting to –1 for complete oil wetting. Samples with Amott-Harvey index –0.3 to +0.3, have intermediate wettability.

The samples were then cleaned by Dean-stark extraction with toluene, then dried in an unhumidified oven at 120 degrees centigrade for 48 hours, then saturated with formation water and aged for 10 days. The Amott and USBM tests using centrifuge were performed. A step-by-step description of the wettability tests and capillary pressure measurements are presented in the following.

1- The water saturated core plugs were centrifuged in oil at a given rotational speed to obtained initial water saturation.
2- All plugs were submerged in oil and allowed to age for 40 days at 90°C in order to restore the reservoir equilibrium condition.
3- The core plugs were submerged in formation water for 40-50 days, and the produced volume of oil was recorded (Vosp).
4- The cores were centrifuged in water using inverted core holder. Several rotational speeds were run and complete negative imbibition capillary pressure curves were obtained (Vod).
5- The core plugs were submerged in oil for 40-50 days, and the produced volume of water was recorded (Vwsp).
6- Finally the core plugs were centrifuged in oil. The complete secondary drainage capillary pressure curve was obtained, together with the final production of water (Vwd).

The combined Amott/USBM index is the logarithm of the ratio of areas under centrifuge-measured capillary pressure curves in both wetting phase saturation increasing and decreasing directions. It ranged characteristically from –1 (oil-wet) to +1 (water-wet).

RESULTS

The petrophysical properties including the porosity and absolute permeability of all the samples are presented in Table 1. The mineralogical composition and pore geometry of the core plugs from formations in the reservoirs are given in Table 3. All end-points and the crossover saturations from the relative permeability tests are shown in Table 4. The measurement of relative permeabilities was done only on restored core plugs at 90^{o} C.

Tables 5 and 6 presents the main results from the Amott wettability tests. The combined Amott/USBM wettability index was calculated from the secondary drainage and the negative imbibition curves according to equation (1) that is given in Table 6. Figures 1 to 10 show the capillary pressure curves for each sample from combined Amott/USBM tests.

DISCUSSION

The relative permeability data from five depths interval of Field M were derived at reservoir temperature. The crossover saturations of these samples are less than 0.5. This is considered as an indication of oil wetness. However, the end points of Krw at Sor are less than Kro at Swi, which is not indicative of oil-wet conditions. From this discrepancy, we concluded that these qualitative experiments are not sufficient for wettability characterization in our carbonate rock samples and decided to continue to perform more quantitative experiments. Heaviside et al. [16] found that for rock with intermediate wettability, the correlation between wettability and relative permeability is not very impressive. For the investigated rock-fluid system a normal correlation between end point relative permeabilities and wettability dose not exist. Cuiec [15] reports similar wettability studies based on the Amott procedure where the centrifuging is replaced with forced displacement and measurements of end point relative permeabilities. These relative permeability values can give additional information about the wettability, but it is risky to evaluate the wettability of a reservoir solely from such data [15]. Poston et al. [17] pointed out that in consolidated sands, the changes of wettability were all in a direction suggestive of an increase in water wetness contrary to temperature effects on wettability in carbonate rocks. Weinbrandt et al. [18] studied the effect of temperature on relative and absolute permeability of sandstones and showed that due to increase in temperature, the irreducible water saturation increases. But, the residual oil saturation decreases. And also the relative permeability to water and oil increases.

It is noted that all temperature effects are related to fluid property changes such as interfacial tension and viscosity, but changing temperature creates some thermally-induced mechanical stresses that may be the source of wettability change [18].

By comparison of Amott test results using core flooding system and centrifuge, we can see a good agreement between them. These results show intermediate wettability unless interval D of Field R that shows wettability to water. The experimental results from the combined Amott/USBM wettability test on core plugs from Field M and R indicates that these intervals are oil-wet, unless interval D of Field R that is weakly water-wet. The most results from the Amott tests are different with combined Amott/USBM indices. The combined Amott/USBM method is more informative than the Amott or USBM test alone. The combined method is easily performed at high temperatures and by using an automated centrifuge system; the method is almost as time efficient as the Amott test.

CONCLUSIONS

1- The relative permeability curves cannot be used to characterize the wetting of the carbonate rocks used in this study, because the end point role indicates water wetness while crossover saturation indicate oil wetness.

2- Most of the carbonate samples in Amott test using either the flooding system or centrifuge had intermediate wettability at restored-state.

3- Most of the carbonate samples at restored-state show oil wetness when combined Amott/USBM tests are performed.

4- There is a good agreement between Amott index using the core flooding system and Amott index measured using the centrifuge and both are different with combined Amott/USBM index.

5- At combined Amott/USBM method, the initial water saturation has a lower value than from relative permeability test.

6- At oil wet conditions the secondary drainage capillary pressure curve (oil displacing water) is flat while the forced imbibition curve (water displacing oil) is quite steep. This indicates that oil enters almost spontaneously while a high water pressure is needed to displace oil.

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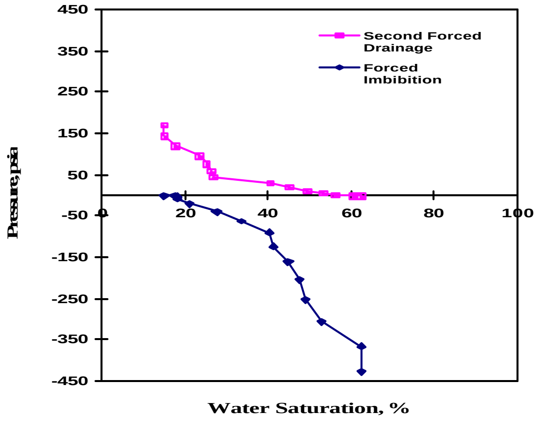

Fig. 1- Capillary pressure curves of combined Amott/USBM test of plug no. 11

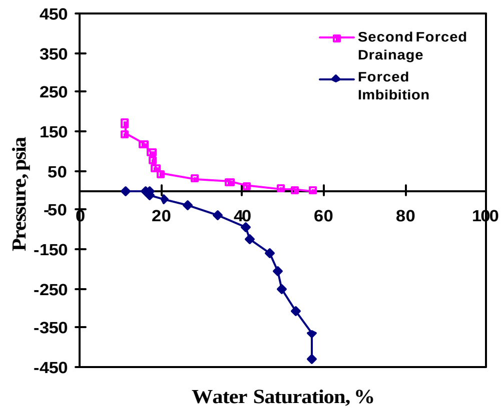

Fig. 2- Capillary pressure curves of combined Amott/USBM test of plug no. 12

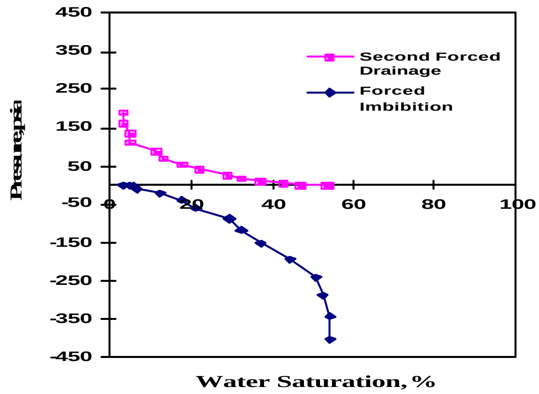

Fig. 3- Capillary pressure curves of combined Amott/USBM test of plug no. 25

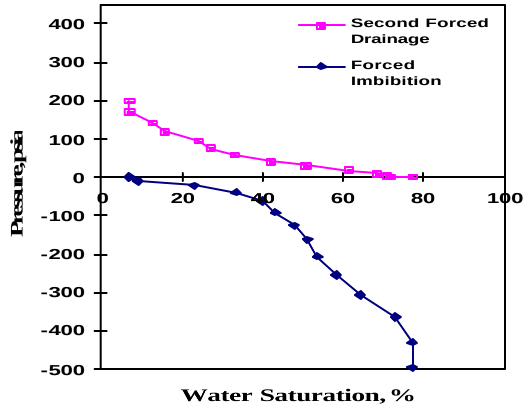

Fig. 4- Capillary pressure curves of combined Amott/USBM test of plug no. 40

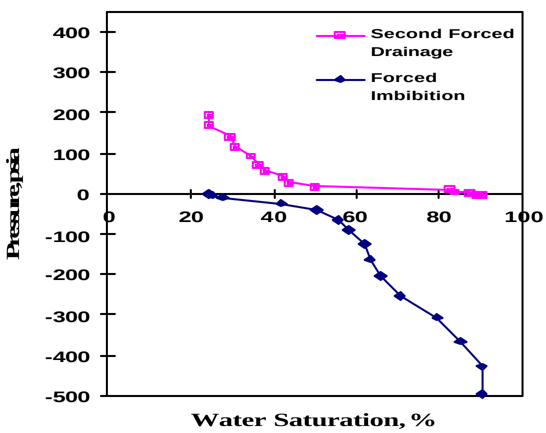

Fig. 5- Capillary pressure curves of combined Amott/USBM test of plug no. 41

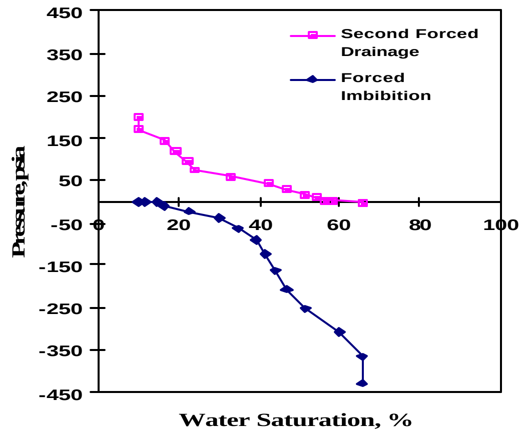

Fig. 6- Capillary pressure curves of combined Amott/USBM test of plug no. 42

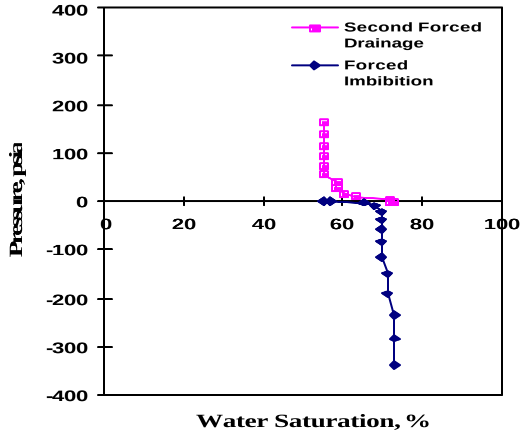

Fig. 7- Capillary pressure curves of combined Amott/USBM test of plug no. 5

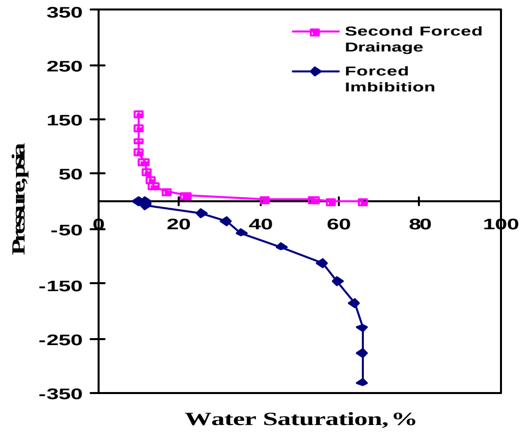

Fig. 8- Capillary pressure curves of combined Amott/USBM test of plug no. 13

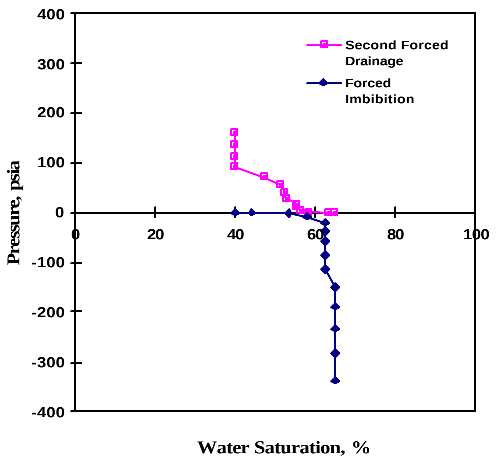

Fig. 9- Capillary pressure curves of combined Amott/USBM test of plug no. 6

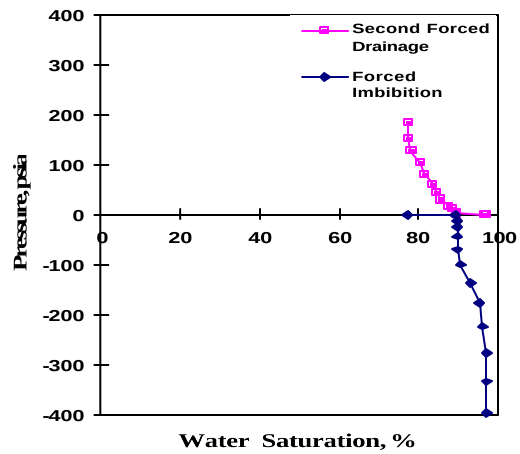

Fig. 10- Capillary pressure curves of combined Amott/USBM test of plug no. 19

Table 1- Core plug data

Field	Core ID	Depth Interval	Length (cm)	Diameter (cm)	Porosity (%)	Absolute Perm. (md)
M	11	1	5.21	3.82	16.0	2.7
	12	2	5.19	3.83	14.8	2.9
	25	3	4.85	3.84	13.5	2.3
	40	4	5.23	3.84	11.2	2.2
	41	5	5.19	3.83	14.4	3.2
	42	6	5.23	3.84	10.0	1.8
R	5	A	4.34	3.84	6.5	0.2
	6	B	4.23	2.52	12.3	1.6
	13	C	4.32	2.56	9.4	0.3
	19	D	5.27	3.78	11.7	1.0
	27	E	4.24	2.56	3.6	0.3

Table 2-Water composition

Ion	g/l
Na^+	80.6
Ca^{2+}	23.3
Mg^{2+}	3.7
K^+	1.6
Sr^{2+}	1.1
Cu^{2+}	0.006
Ba^{2+}	0.330
Cl^-	149
SO_4^{2-}	0.22

Table 3- Mineralogical composition, and pore geometry of samples

Field	ID	Formation	Lithology, %			Porosity						
			Dolomite%	Limestone%	Anhydrite%	Intera-particle	Inter-particle	Intercrystaline	Vuggy	Moldic	Fracture	Total porosity%
M	11	Asmari	80	10	10	-	-	-	4	6	1	11
	12		90	-	10	-	-	3	-	11	-	14
	25		85	5	10	-	-	4	-	5	-	9
	40		90	-	10	-	-	2	2	7	-	11
	41		98	2	-	-	3	1	12	-	-	16
	42		99	1	-	2	-	2	1	10	-	15
R	5	Sarvak	-	100	-	2	-	1	3	1	1	8
	6		-	100	-	-	-	1	8	1	2	12
	13		-	100	-	1	-	-	9	1	1	12
	19		-	100	-	3	-	-	2	2	3	10
	27		-	100	-	2	-	-	-	-	-	2

Table 4- End point values and crossover saturations

Field	Core ID	Swi (%)	Kro(Swi)	Sor (%)	Krw(Sor)	Sw @Krw=Kro (%)
M	11	22.6	0.65	27.4	0.095	41
	12	20.4	0.94	28.2	0.106	46
	25	14.2	0.93	29.9	0.205	33
	40	17.3	0.54	30.5	0.148	42
	42	18.2	0.56	43.4	0.085	35

Table 5- Results of Amott wettability test on restored core plugs using coreflooding

Field	Core ID	Pore Volume (cm^3)	Amott wettability Index		
			Iw	Io	I
M	11	8.76	0.085	0.050	0.035
	12	7.80	0.168	0.004	0.164
	25	6.22	0.042	0.024	0.018
	40	5.69	0.006	0.003	0.003
	41	7.72	0.003	0.025	-0.022
	42	5.40	0.050	0.004	0.046
R	5	2.96	0.125	0.080	0.045
	6	2.50	0.040	0.250	-0.210
	13	2.16	0.225	0.030	0.195
	19	6.56	0.510	0.020	0.490

Table 6- Results of wettability tests on restored core plugs using centrifuge

Field	Core ID	Swi (%)	Sor (%)	Amott wettability Index			Combined Amott/USBM Index
				Iw	Io	I	
M	11	15.07	37.56	0.053	0.036	0.017	-0.545
	12	11.02	42.69	0.116	0.003	0.113	-0.339
	25	3.54	45.66	0.033	0.019	0.014	-0.452
	40	7.00	22.53	0.005	0.002	0.003	-0.395
	41	24.22	9.72	0.002	0.020	-0.018	-0.572
	42	10.00	34.26	0.030	0.003	0.027	-0.360
R	5	55.07	27.03	0.094	0.057	0.037	-0.310
	6	10.00	34.40	0.029	0.144	-0.115	-0.852
	13	39.81	35.19	0.167	0.074	0.093	0.374
	19	77.13	3.05	0.615	0.008	0.607	-0.098
	27	-	-	0.400	0.140	0.260	-