

**WETTABILITY CHANGES DUE TO THE DEPOSITION  
AND PRESENCE OF ASPHALTENES IN CRUDE OIL.**

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**ABSTRACT**

Wettability is one the most important properties affecting oil recovery efficiency of a reservoir. There is abundant evidence that rock wettability could change due to rock interactions with injected fluids and to the adsorption of the petroleum heavy ends (asphaltene and resin fractions) on the pore walls. In addition to formation damage considerations, the issue of wettability modification is a crucial one for core sampling and core studies. In order to optimize production through core analysis reliability, it is necessary to have a good understanding of the mechanisms controlling these changes. In this work, the effect of oil asphaltene content and eventual precipitation on wettability were investigated.

Fluids and cores of Venezuelan reservoirs with different asphaltene contents and as well as Berea core plugs were used to carry out the study. Wettability was measured using a modified version of the Amott imbibition test. Before performing the tests to determine wettability changes due to the asphaltenes, the wettability of cores "as received" and after cleaning were evaluated. Cleaning procedures reported in the literature were tested on reservoir cores, resulting ineffective, the wettability index was less than the expected value for strongly water-wet conditions, even after a third cleaning. To resolve this, a flow-through core cleaning procedure was developed. Once the reservoir and Berea cores were made partially or strongly water-wet, some of them were saturated with brine, flushed with crude oil and aged at several temperatures for a period ranging from 6 to 38 days and their Amott indices were measured. Others were flushed with pentane, to induce asphaltene precipitation, and then wettability was determined. Cores aged in high asphaltene content crude oil (for 12 a 38 days) and those where asphaltene precipitation was induced, showed similar wettability changes toward the oil-wet conditions. Cores aged in desasphalted oil or low-asphaltene content oil, showed an intermediate-wet state. It was found that core cleaning before aging and the nature of the rock affected the degree of wettability alterations of samples evaluated.

## INTRODUCTION

Recent studies reported in the literature relating to the influence of wettability in oil recovery by water flooding, have shown that recovery is optimum at close to neutral wettability (Amott wettability index ranging from 0,1 to 0,3) (1, 2, 3). Furthermore, others studies (4) have indicated that as many as 50% of all silicate reservoirs and 80% of all carbonate reservoirs, are oil-wet.

These facts show that it is important to know the actual reservoir rock wettability, before the exploitation strategy of an oil field is defined. This property could change due to the rock interaction with injected fluids, as such as drilling oil base muds or solvents (5, 6), and due to the adsorption of the petroleum heavy ends (asphaltene and resins) on the rock surface (7,8,9,10).

This change in rock wettability can be conceived only if the adsorbed water on the largest pores, can be displaced by some surface active compound present in the injected fluids, asphaltenes and resins (11). Specific compound/mineral interactions would control the degree to which such adsorption is irreversible with respect to various organic solvents (12). The factors which control adsorption of asphaltenes and/or resins of petroleum on the mineral surfaces are: chemical and structural nature of the surface mineralogy (13, 14, 15), asphaltene and resin contents of the crude oil (16, 17), physicochemical properties of asphaltenes and resins present, brine PH and composition (18, 19), fluids distribution and saturation (17, 20), pores shape and distribution (21), pressure and temperature (1). Considering the complexity of reservoir rocks and crude oils, each reservoir system is a specific case and its behavior can only be confirmed through experimental work.

In this work, our efforts were geared towards one main purpose: to verify if the adsorption of asphaltenes on mineral surfaces produces changes in the wettability of such a surface using cores and fluids of Venezuela's reservoirs.

## EXPERIMENTAL PROCEDURES

### Materials and Methods

Samples of cores and fluids from Venezuelan reservoirs, were used. Table 1 shows the average properties and compositions of such samples. Crude oils have asphaltene content over 1% (wt). Also, a sample of a desasphalted (n-C7) crude oil F was included in the evaluation. Because of the limited number of available cores, Berea samples of different permeability and porosity were used to accomplish the experiments. Crude oils C, F and M were from similar formations in which the rock was mainly composed of quartz (97%) and a few

percent of carbonates and clays. Crude B come from a non consolidated rock heavy oil reservoir.

Cores of reservoir F were unpreserved and cut with an oil base drilling mud, however, previous wettability measurements carried out on preserved ones F indicated an oil-wetting behavior (Amott Index ranging form -0.443 to -0.859). Cores of reservoir M were also unpreserved. Berea plugs were cored under fresh water and were used without further treatment. All samples were approximately 2.36 in. (6 cm) in length and 1.5 in. (3.81 cm) in diameter.

In order to evaluate changes, wettability was measured by a modified Amott test on samples "as received", after cleaning, after aging with crude oil and/or after asphaltene deposition induced by pentane. All measurements performed on samples "as received" and "after aging" were duplicated.

### Measurement of Wettability

The wettability index of each core sample was determined using a modified Amott method (4, 20). Measurements were performed at 22°C. A refined oil (Soltrol 170, 1,5 cp) was used as the oil phase. Because brine pH can have an important effect on wetting behavior of crude oil, the water phase used was syntetic formation brine corresponding to the reservoir evaluated. In the case of Berea samples the brine used was syntetic formation brine corresponding to the crude oil present in the sample before carrying out the Amott test. For the forced displacement following spontaneous imbibition, a constant rate of 0.55 cc/min, was used. This rate was low enough such as to avoid core damage, in particular to Berea samples. Test time in the present study was about two weeks.

From these tests, the wettability index (WI) is obtained as the difference, between the wettability index to water ( $I_w$ ) and the wettability index to oil ( $I_o$ ). The wettability index (WI) can have values ranging from -1 for a strongly oleophilic (hydrophobic) rock, to +1 for a strongly hydrophilic rock. Cut-off values have been choosen to separate various wettability classes (20). The wettability scale adopted is shown up in Figure 1.

Analyses of data from duplicated samples indicated that values of wettability index were reproducibile within +/- 0.05.

### Core Cleaning Procedures

The main objetive of core cleaning is to remove all organic compounds without altering the basic pore structure of the rock (5). In the litterature, a wide range of combinations and/or sequences of solvents have been proposed, but

procedures are far from standardized (20,22,23). In this study we employed standard extraction procedures (Soxhlet) using two different set of solvents, resulting ineffective to make samples water-wet. Therefore, additional flow-through cleaning methods with a sequence of solvents were evaluated.

The method was a trial-and-error process. Wettability was measured on the core "as received", then remeasured after each cleaning attempt to examine progress.

#### Induced Deposition Procedure

To examine the effect of asphaltene precipitation on wettability an induced deposition procedure was performed, using dry Berea plugs. They were first saturated with crude oils M and F, aged for two days at room conditions and then flooded with 1 PV of n-pentane. After this procedure, Amott wettability indices were determined for each sample.

#### Aging Process

In order to determine the effect of core exposure to crude oils, with different contents of asphaltene, reservoir cores after cleaning and Berea plugs, were flooded with syntetic formation brine, followed by the crude oil until irreducible water saturation ( $S_{wi}$ ). The samples were then aged in crude oil contained in a covered beaker or in a pressure cell, for a time period of 6 to 38 days, at temperatures ranging from 22°C at 150°C and at pressures from 14.7 psi to 500 psi. Once the aging period was finished, cores were returned to room temperature and their wettability index determined.

Furthermore, to evaluate the effect of core cleaning on the degree of wettability alteration, some samples whose wettability after cleaning was not stongly water-wet were used.

### RESULTS AND DISCUSSION

Wettability indices were determined on 42 core samples: 13 of reservoir F, 8 of reservoir M and 21 Berea cores. The total number of tests was 103, wich included 35 test carried out to select the best cleaning method. Overall results show that several factors control the trend in wettability change under the conditions tested.

## Cleaning Treatment Screening

As indicated before, the standard extraction procedure (Soxhlet) was evaluated using two different sets of solvents: toluene - methanol and dichloroethane - methanol. The Amott indices for samples "as received" and after cleaning by these process, are shown in Table 2. The cores "as received" were neutral-wet to slightly oil-wet. Soxhlet extraction with both sets of solvents tested left the cores intermediate-wet, except for sample F-13-6, which never was strongly water-wet, even after undergoing two cleaning processes.

A summary of the flow-through methods used for core cleaning is given in Table 3. The Amott Indices for some plugs cleaned with these treatments are shown in Table 4 and Figure 2. Different series of solvents, flood temperatures (22°C, 50°C, 65°C) and the use of oven drying as the final step were tested. Samples used were "as received" or "after aging" (see sample Berea-6B in Table 4 and Figure 2). In general, methods where the samples were dried in the unhumidified oven at 230°F (110°C) for 8 to 12 hours, were ineffective. Therefore this step was replaced by formation brine flood at the cleaning process temperature.

Solvent named M.L. is a mixture often used to remove oil base mud contaminants in well bores, which contains xylene, gasoil, a mutual solvent and a surfactant compound. This mixture was used to remove possible contaminants in reservoir core samples F but it was ineffective in making cores strongly water-wet, despite of the change in the final step mentioned above. The cleaning treatment using only dichloroethane (4) was one the most inefficient since some samples cleaned with this method become even more oil-wet than "as received" (see Figure 2).

Finally the sequence dichloroethane-methanol, followed by formation water at 65°C (5) was selected as the best cleaning procedure since samples cleaned with this method did not show any spontaneous oil imbibition and resulted strongly water-wet, even after only one cleaning cycle (see sample M11-3).

### Effect of asphaltene precipitation.

The wettability indices measured in Berea samples after inducing asphaltene deposition are given in Table 5. The wettability of dry, untreated Berea samples was strongly water-wet ( $WI > 0.94$ ) with  $I_w$  and  $I_o$  values similar to those reported by Wolcott et al. (16). Wettability alteration was observed in all samples examined. Berea samples became only intermediate-wet or neutral-wet, when they were previously saturated with oil M which had a low asphaltene content, while samples saturated with oil F, became oil-wet due to the greater asphaltene content present in oil F.

In order, to determine if the degree of wettability alteration could be increased by adsorption of more asphaltenes precipitated, the samples saturated before with oil M (Berea 1B and 2B) were again employed after measuring their wettability indices. They were saturated with oil F, aged for 2 or 10 days, flooded once again with 1 PV of n-pentane and then their Amott indices were measured. The values of these indices are given in Table 5. The smaller change in wettability showed after two deposition cycles indicates that for these intermediate-wet surfaces, no further adsorption or wettability alteration can occur by asphaltene precipitation. This result can be extrapolated to surfaces oil-wet.

#### Effect of presence of asphaltenes in crude oil.

A summary of measurements of wettability index carried out on samples "before aging" and "after aging" is given in Table 6 and Figures 3 and 4. This results showed that there was wettability changes in all cases studied and that the direction of these changes was always toward the oil-wet state. The degree of wettability alteration was found strongly to depend on the core cleaning, the nature of the rock, the asphaltene content in oil and the physicochemical properties of asphaltenes. There were few obvious trends in this data, and it was difficult to correlate the wettability alteration with the aging conditions.

The greatest wettability change was found in the system oil C - sample Berea 4A wich was aged for 12 days at 22° C and 14.7 psi and the least one was found in the system oil M - sample M11-1, aged for 12 days, 55° C and 14,7 psi. In the latter the wettability of sample M11-1 before aging was not strongly water-wet because to an inefficient cleaning (treatment 6). Otherwise the oil M produced a great wettability alteration in sample M11-3 wich was strongly water-wet before aging. The same behavior was observed in systems oil F - sample F13-4 and - sample F13-5 where the degree of change obtained was less than the one obtained in the system oil F - sample F13-1A, in spite of the samples F13-4 and F13-5 were exposed to aging conditions quite extreme (150° C, 500 psi, 38 days).

The effect of the nature of the rock can be observed in systems where reservoir samples and Berea cores were exposed to oils F and M (see Figure 3). For similar aging conditions (55° C, 14,7 psi, 6 to 12 days), oil F produced a greater wettability alteration in cleaned reservoir samples F than in Berea samples: samples F became oil-wet, Berea became only intermediate -wet. Similarly, reservoir samples M exposed to crude oil M showed a higher degree of wettability change than the one showed in systems oil M - Berea samples whose wettability index values after aging were never negative. It is quite possible that

this difference was originated by the water content and distribution established prior to aging these samples which depend mainly on the hydrophilic surface sites present in the rock, the distribution of this property throughout the pore space (11), the pore shape and distribution. These results indicate that the nature of rock is an important key variable in the aging procedure if one wants to obtain a more realistic and reproducible wetting state, relevant to those occurring in reservoirs.

Tests carried out in Berea samples with different oils confirm that the asphaltenes present in these crude oils caused a large change in the wettability of the core. Although precipitation is often invoked as the principal mechanism of wettability alteration by asphaltenes, no precipitate was visible in these experiments, indicating that adsorption alone could alter wetting state. Systems oil F - Berea 6B and oil F desasphalted -Berea 3B under the more extreme conditions (150°C, 500 psi, 38 days) became strongly oil-wet and intermediate-wet respectively. It could be possible that this desasphalted oil F still had some traces of asphaltene, negligible with respect to the accuracy of measurement. Wolcott et al. (16) found that a minute quantity of organic matter influenced wettability.

According to some authors it can not be assumed that the degree of wettability alteration is proportional to asphaltene content (2), however when the results obtained in Berea systems with oils B, C and F (Berea samples 13A, 14A, 11A, 12A, 2C) are compared with the ones obtained with oil M (Berea samples 1A, 9B), under comparative aging conditions, it is observed that the crude with the lower asphaltene content caused the least change: oil M left the samples after aging from slightly-water-wet to intermediate-wet, while oils B, and C, which contained higher asphaltene concentration, left the samples from slightly to strongly-oil-wet.

We assumed that the differences obtained in systems with oils F and C, in spite of having similar asphaltene content, and the comparable behavior observed in systems with oils B and C, although having different asphaltene content, were due to the individual physicochemical properties of asphaltenes of each oil. Similar behavior was previously obtained by Donaldson (24).

## CONCLUSIONS

- The Soxhlet extraction method with toluene-methanol or dichloroethane-methanol did not achieve a water wet-condition for cores that initially were not water-wet.
- The drying-oven of samples after cleaning did not improve the effectiveness of cleaning treatments evaluated.
- The best treatment to make samples strongly water-wet was a flow- through method using the sequence dichloromethane-methanol-formation brine at 65° C. After using this method none of the cores had spontaneous oil imbibition.
- Asphaltene precipitation can change the wettability of Berea cores from strongly water-wet to neutral-wet or to oil-wet. This change depends on asphaltene content in the oil, the higher content the greater degree of wettability alteration.
- In intermediate-wet surfaces, no further adsorption or wettability alteration can occur by asphaltene precipitation. This result can be extrapolated to oil-wet surfaces.
- The presence of asphaltenes in Venezuelan crude oils altered the wetting state of all reservoir cores and Berea samples evaluated. The direction of these changes was always toward the oil-wet state. Under similar aging conditions and for a particular type of rock, the degree of wettability alteration depend on the amount and the physicochemical properties of asphaltenes present in the crude oil.
- Core cleaning before aging and the nature of the rock affect the degree of wettability alteration after aging.

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TABLE 1. PROPERTIES OF FLUIDS AND CORES SAMPLES

## OIL CHARACTERIZATION

OIL	ASPHALTENES n-C <sub>7</sub> (% WT)	RESINS (% WT)	°API
M	1.1	N.A	34.5
F	4.2	14.5	23.5
C	4.6	13.5	23.5
B	17.0	18.9	10.2
F(*)	0.0	N.A	28.0

(\*) DESASPHALTED

N.A NOT AVAILABLE

## WATER CHARACTERIZATION

FORMATION WATER	TDS	pH
F	16698	9.42
C & M	14157	9.10
B	2581	8.71

## AVERAGE PROPERTIES OF ROCK SAMPLES

SAMPLE	POROSITY, $\emptyset$ (%)	PERMEABILITY, K <sub>G</sub> (mD)
M	9.3	100.4
F	13.4	109.5
BEREA A	17.2	150.0
BEREA B	18.8	390.0
BEREA C	21.6	2000.0

TABLE 2. EVALUATION OF EXTRACTION TREATMENTS  
AMOTT INDEX

SOLVENTS	SAMPLE	W.I AS RECEIVED	W.I AFTER CLEANING
TOLUENE	F13-4	-0.110	+0.050
METHANOL	F13-5	-0.080	+0.070
	F13-6	-0.140	+0.360
DICHLOROETHANE	F13-1	-0.090	-0.120
METHANOL	F13-3	-0.250	+0.030
	F13-6	+0.360*	+0.420

(\*) : SECOND CLEANING

TABLE 3. CLEANING TREATMENTS

TREATMENT	SOLVENTS	TEMPERATURE	DRYING
1	M.L	22	YES
2	M.L-METHANOL FORMATION WATER	22	NO
3	M.L FORMATION WATER	50	NO
4	DICHLOROETHANE	50	YES
5	DICHLOROETHANE- METHANOL-FORMATION WATER	65	NO
6	TOLUENE-METHANOL FORMATION WATER	65	NO

TABLE 4. WETTABILITY INDEX FOR FLOW-THROUGH  
CLEANING METHODS

SAMPLE	AS RECEIVED	W.I. AFTER CLEANING TREATMENT					
		1	2	3	4	5	6
F13 1A	-0.115	+0.190(1)			-0.447(2)	+0.615(3)	
F13 2A	-0.200		+0.028(3)	-0.326(2)	-0.120(1)	+0.769(4)	
F13 3	-0.120	+0.290(1)			-0.329(2)		
F15 1	-0.926					+0.876(2)	+0.692(1)
BEREA 6B	-0.639				+0.167(1)	+0.912(3)	+0.509(2)
M11 3	-0.008						+0.781

(n) ORDER OF CLEANING TREATMENT

TABLE 5. EVALUATION OF WETTABILITY CHANGES BY  
ASPHALTENE DEPOSITION (n-C<sub>5</sub>)  
AMOTT INDEX

SAMPLE	OIL	AGING TIME (DAYS)	W.I	W.I
			(BEFORE)	(AFTER)
BEREA 1B	M	2	+0.953	+0.168
BEREA 2B	M	2	+0.963	-0.051
BEREA 1B	F	2	+0.168	-0.075*
BEREA 2B	F	10	-0.051	-0.043*
BEREA 8C	F	2	+0.948	-0.494
BEREA 9C	F	2	+0.967	-0.463

( \* ) : AFTER TWO DEPOSITION CYCLES

TABLE 6. EVALUATION OF AGING CONDITIONS  
AMOTT INDEX

OIL	SAMPLE	TEMPERATURE °C	PRESSURE PSI	AGING TIME (DAYS)	W.I BEFORE	W.I AFTER
B	BEREA 13A	55	14.7	12	+0.939	-0.387
B	BEREA 14A	55	14.7	38	+0.945	-0.552
F	F13 1A	55	14.7	12	+0.615	-0.901
F	F13 2A	55	14.7	6	+0.769	-0.348
F	BEREA 10A	55	14.7	6	+0.953	-0.115
F	BEREA 11A	55	14.7	12	+0.965	-0.189
F	F13 4	150	500	38	+0.050	-0.576
F	F13 5	150	500	38	+0.070	-0.493
F	BEREA 6B	150	500	38	+0.943	-0.639
F*	BEREA 3B	150	500	38	+0.934	+0.142
C	BEREA 4A	22	14.7	12	+0.925	-0.746
C	BEREA 5A	22	14.7	38	+0.930	-0.572
C	BEREA 12A	55	14.7	12	+0.939	-0.160
C	BEREA 2C	55	14.7	12	+0.953	-0.304
M	BEREA 1A	22	14.7	38	+0.993	+0.049
M	BEREA 2A	22	14.7	12	+0.900	+0.292
M	BEREA 8B	22	14.7	12	+0.937	+0.115
M	BEREA 9B	22	14.7	38	+0.991	+0.277
M	M11 1	55	14.7	12	+0.393	-0.091
M	M11 3	55	14.7	12	+0.781	-0.755

(\* ) CRUDE OIL DESASPHALTED

FIG. 2 EVALUATION OF THE CORE CLEANING AMOTT INDEX

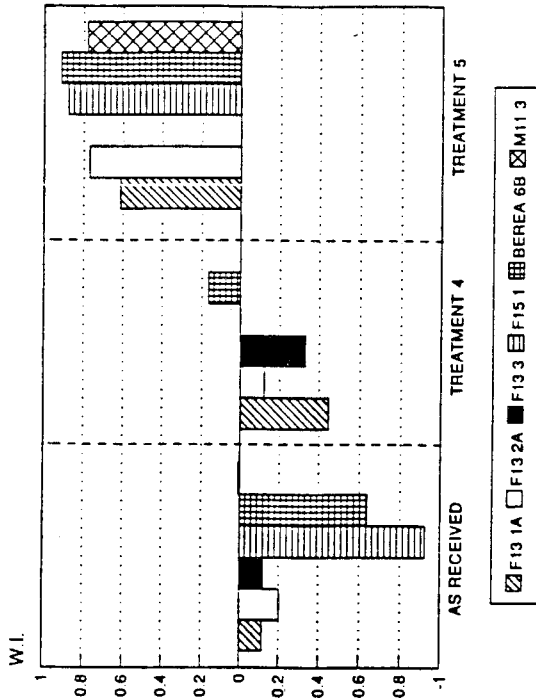


FIG. 4 EVALUATION OF THE EFFECT OF AGING IN CRUDE OIL AMOTT INDEX (38 DAYS)

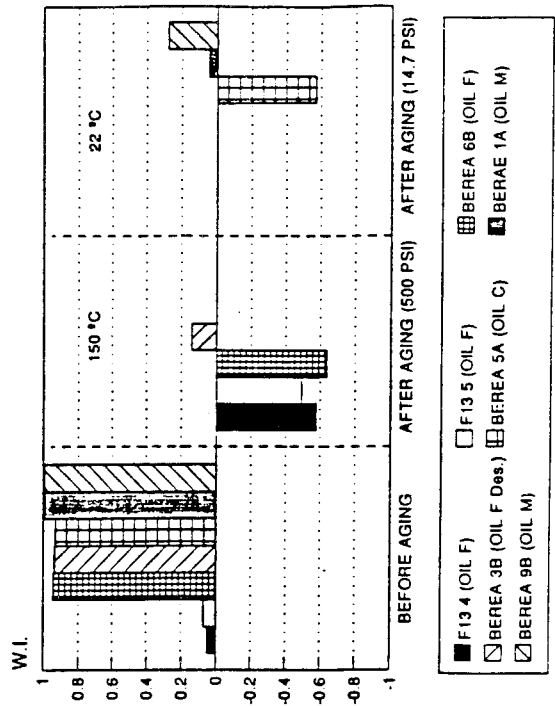


FIG. 1 WETTABILITY SCALE ADOPTED (AFTER REF. 20)

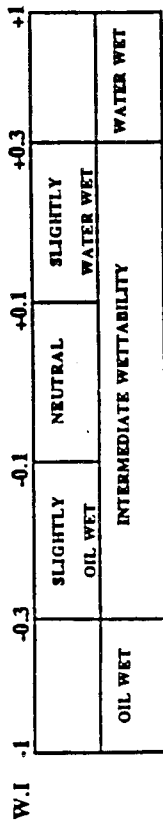


FIG. 3 EVALUATION OF THE EFFECT OF AGING IN CRUDE OIL AMOTT INDEX (12 DAYS, 14.7 PSI)

