WETTABILITY STUDY OF SAUDI ARABIAN CARBONATE RESERVOIR CORE SAMPLES

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الخلاصـــة :

أُجريت دراسة قابلية الترطب أو التبلل لصخور الكاربونات من مكامن العربية السعودية على أساس ثلاث تقنيات كمية معملية وهي : Amott, USBM وزاوية التلامس ، وتلك الثلاث كانت الأكثر استعمالاً في هذه الدراسة . وتـمَّ معايرة قابلية التبلل باستخدام المحلول الملحي للمكمن وزيت صهريج التخزين المرشح . وتـمَّ بحث عوامل وثيقة الصلة بالموضوع تؤثر على قابلية التبلل مع الاحتفاظ بثبات مركبات زيت الخام . كما تـمَّ اختيار المحاليل الملحية المختلفة التركيز (درجة الملوحة) تحت درجة حرارة وضغط مرتفعين . هذا وقد تم دراسة عوامل اخرى بما فيها درجة الحرارة ، والضغط ، وتعريض العينة الكاربونية المحفوظة والنقية خلال طريقتي : Amott, USBM لاختبار قابلية التبلل وكذلك تم تحضير كاربونات الكالسيوم البلورية الناعمة اثناء قياسات قابلية التبلل بطريقة زاوية التلامس . وتم أيضاً دراسة تأثير تركيز الملوحة ، والضغط ، ودرجة الحرارة ، ونقاوة العينة ، وعرض العينة للهواء على قابلية الابتلال لعينات الكاربونات . وقد ضمنت هذه الدراسة استيعاباً جيداً لقابلية التبلل لصخور الكاربونات والسوائل المستعملة .

من خلال هذه الدراسة باستخدام اختباري Amott, USBM اتضح ان صخور الكاربونات متوسطة التبلل . وباستخدام قياسات زاوية التلامس وُجد أن هذه الصخور معتدلة التبلل .

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ABSTRACT

A wettability study of carbonate rocks from a Saudi Arabian reservoir was conducted based on three widely used quantitative laboratory techniques: USBM, Amott, and contact angle. During the course of this study, wettability was evaluated using reservoir produced brine and filtered stock-tank oil. Several pertinent factors affecting wettability were investigated while maintaining a fixed crude composition. Synthetic brines varying in salinity were tested under elevated temperature and pressure conditions. Other factors studied included pressure, temperature, core exposure to air, and core cleaning.

Both preserved and cleaned carbonate cores were used during the USBM and Amott wettability tests, whereas, smooth calcite crystals were prepared for contact angle wettability measurements. The effects of brine concentration, pressure, temperature, core cleaning, and core exposure on wettability of the carbonate cores were also studied and provided adequate understanding of the wettability of the carbonate rock/fluid system.

USBM and Amott tests indicated that the subject carbonate reservoir rocks were intermediately wet. Contact angle measurements, however, yielded moderate water wetting.

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INTRODUCTION

Wettability of a rock/fluid system can influence fluid distribution in porous media, capillary pressure, relative permeabilities, electrical properties, waterflood performance, and enhanced oil recovery. Several researchers [1–5] have demonstrated the profound influences of wettability on the above reservoir parameters. Recent publications [4, 6–9] on wettability research have focused more attention towards the factors influencing wettability of a rock/fluid system.

Since wettability phenomenon involves a rock and two fluids among which a tendency of preferential wetting exists, factors affecting wettability exhibit rock, fluid, or rock/fluid related properties. Core and fluid preservation and handling procedures also exert significant influence on wettability. Crocker and Marchin [6] reported that alteration in wettability can be attributed to the variations in pore-surface roughness, and mineralogical composition.

Heavy ends of the crude contain polar compounds which adsorb on the rock surface, therefore initiating more potential for oil-wetting. Brine salinity and pH can strongly affect the rock/fluid wettability and promote oil wetting [10]. Trace ions, such as Ca^{+2}, Cu^{+2}, Mg^{+2}, Ni^{+2}, and Fe^{+3} found in brine can shift wettability from water-wet to oil-wet [11].

Elevated temperatures and pressures can induce variations in fluid properties and rock properties [7, 9, 10–12]. Honarpour *et al.* [9] suggested that the changes in rock composition caused by thermal stress not only influence the rock/fluid interaction but also generate variations in fluid flow behavior of the rocks. Moreover, polar compounds that are adsorbed by the rock surface at low temperatures may desorb from the rock surface at high temperatures.

Several researchers [11, 13, 14] have pointed out that cleaned cores could yield relatively more water-wet results when compared with preserved cores. Exposure to air, on the other hand, may induce deposition from the crude thus furnishing more oil-wet cores [15].

In this experimental work, salinity, temperature, pressure, core exposure to air, and core cleaning effects were evaluated among several other factors influencing the wettability of a rock/fluid system. Comparison of USBM and Amott results with contact angle measurements was made in order to determine any anomalies or supplement the findings.

EXPERIMENTAL PROCEDURES

The experimental procedures for each wettability method used in this study are discussed below.

USBM Technique

Preserved core plugs were initially saturated with crude oil in a centrifuge at ambient temperature until irreducible water saturation was obtained. The highest speed used during this oil saturation step was 10 000 rpm. The core plugs were then placed in brine and centrifuged for brine drive at successively increasing speeds until 10 000 rpm. Residual oil saturation was thus achieved upon completion of the brine drive. The volume of the expelled oil corresponding to each incremental speed was recorded.

Oil drive step followed brine drive by displacing brine at successive speeds starting with 500 rpm and ending with 10 000 rpm. The volume of the expelled brine was also recorded at each speed until no more fluid was displaced. Donaldson [16] indicated the requirement of 10 to 30 minutes equilibrium time at each speed of the centrifuge for his cores, however, Omoregie [17] suggested 1000 to 2000 minutes for complete equilibrium. The time required in this study was about 45 minutes. Significant fluid displacement could not be observed when experiment was continued longer. Irreducible brine saturation was obtained when the oil drive was terminated.

Capillary pressures corresponding to each centrifuge speed was computed for the average fluid saturations. Approximately, 50 psig and 100 psig capillary pressures were determined at 10 000 rpm for oil and brine drives, respectively. Donaldson [16] indicated the necessity of an arbitrary limit of a certain capillary pressure to evaluate wettability from areas by a uniform procedure. His arbitrary limit was 10 psi for the cores and fluids which he used. It was observed that highly porous and permeable Arabian carbonates reached residual oil and irreducible water saturations before reaching 10 psi capillary pressure. Acceptance of a higher capillary pressure limit did not affect wettability indices of permeable samples, but slightly more oil-wet results were obtained for tight samples which do not represent oil producing intervals. Therefore, 10 psi capillary pressure limit was selected in this study. Oil and brine displacement curves were linearly interpolated to obtain 10 and −10 psi pressures [11]. The areas under the two curves were then computed in order to determine the wettability index given by the following equation [18, 19]:

$$WI = \log(A_1/A_2) ,$$

where A_1 and A_2 are the areas under the oil and brine-drive curves, respectively.

The calculated USBM wettability index (WI) was interpreted as follows:

	Water-wet	Neutral	Oil-wet
USBM Index:	near 1	near 0	near −1

Amott Technique

The wettability of core plugs was studied by the Amott Method [20] under ambient conditions. Filtered crude oil, kerosene, and brine were used in conducting the tests. The Amott Tests consisted of four steps: (1) Spontaneous imbibition in brine, (2) Forced displacement by brine, (3) Spontaneous imbibition in kerosene, and (4) Forced displacement by oil [21].

Imbibition was performed in Pyrex fabricated glass cells. Time to reach equilibrium ranged from two to seven days depending on the properties of the tested samples. The minimum recommended time in the literature is 20 hours [9]. Displacement was performed using the centrifuge under ambient conditions. Wettability indices were then calculated for brine and kerosene, using fluid volumes from imbibition and displacements.

The volume of free oil displacement (V_{of}) in Step 1, the total oil displacement (V_{ot}) in Steps 1 and 2, the free water displacement (V_{wf}) in Step 3, and total water displacement (V_{wt}) in Steps 3 and 4 were measured. Displacement indexes by water (δ_w) and by oil (δ_o), respectively, were then calculated for each sample using the following equations:

$$\delta_w = V_{of}/V_{ot}$$

$$\delta_o = V_{wf}/V_{wt}$$

The Amott−Harvey wettability index, I, is then given by:

$$I = \delta_w - \delta_o$$

The results obtained using the Amott method are interpreted as follows:

	Water-wet	Neutral	Oil-wet
Amott−Harvey Index:	+1	0	−1

Contact Angle Technique

Contact angle measurements at elevated test conditions (pressure and temperature) were made using a system that employs high pressure transfer cylinders placed horizontally in an oven on the upstream side of the contact angle apparatus. A control panel provided means for setting the desired test pressures. A bath used for heating the contact angle cell provided a constant temperature setting *via* fluid circulation. A schematic of the system used is shown in Figure 1.

Equilibrated brine and oil were displaced from the transfer cylinders by means of a displacement pump. A dome back pressure regulator, mounted on a cylinder filled with nitrogen was used for setting desired test pressures. A stereomicroscope placed in front of the contact angle apparatus permitted easy viewing of the pendant oil drop, needle tip, and solid surface. A camera attachment on the microscope provided a quick and permanent record of experimental results. Contact angles were measured by a goniometer fitted to the microscope. The applied test procedures are as follows:

1. Preparation for the Test

A polished calcite crystal, 1.4×1.8×0.9 cm in size, was washed in a series of acetone, benzene, absolute ethanol, and boiling distilled water.

2. System Cleaning

The contact angle apparatus was flushed several times with a series of hot distilled water, carbon tetrachloride, hexane, 1.5% hydrochloric acid solution, ethyl alcohol, and Deco 90 (glass cleaning agent) solution [5]. The apparatus was then vacuum cleaned.

3. Charging Fluid to the Cell

The contact angle cell was initially filled under vacuum with oil-equilibrated brine. Then brine was

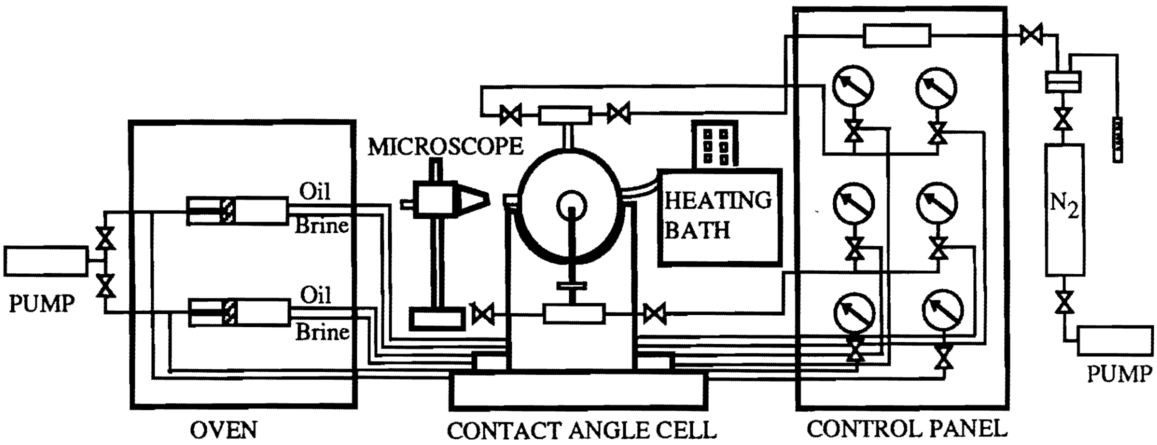

Figure 1. Schematic of Contact Angle Measurement System.

allowed to contact the mineral surface for about four or five days before the crude oil drop was introduced using drop forming tips attached to a metering valve assembly [12, 22].

4. Setting Elevated Test Conditions

The pressure of the brine filling the contact angle cell was initially increased to 1000 psia. Then, the bath circulator was operated to gradually heat the cell to 70°C. As the temperature reached 70°C, the test pressure increased to approximately 2500 psia. Final pressure adjustments were made using the displacement pump and the dome regulator to obtain 2800 psia test pressure. Crude oil was then delivered onto the crystal surface by means of the graduated metering valve.

5. Contact Angle Measurement

The oil drop surrounded by the aqueous phase was allowed to age on the crystal surface. In the meantime, advancing and receding contact angles were measured by expanding or contracting a drop between crystal surface and syringe tip [23]. It was observed that advancing and receding contact angles were very close to each other. The true contact angle was obtained when the crude oil reached equilibrium with the crystal.

The following relationship between wettability and contact angle suggested by Anderson [11] was considered in this report to interpret wettability from contact angle measurements:

Wettability	Minimum angle (degree)	Maximum angle (degree)
Water-wet	0	60 – 75
Neutrally-wet	60 – 75	105 – 120
Oil-wet	105 – 120	180

RESULTS AND DISCUSSION

Cleaned Core Measurement

USBM Tests of Cleaned Cores

Six cleaned samples were tested for wettability using the USBM method. Two consecutive measurement were conducted at 70°C. In the first, the subject cores were saturated with 20 000 ppm brine, centrifuged, flushed with filtered crude oil to their irreducible brine saturation, and then tested. At the end of this run, the plugs were cleaned with toluene followed by alcohol, and then oven-dried overnight.

In the second run, the cleaned and dried plugs were initially saturated with synthetic brine (200 000 ppm). The same testing procedures were then used to measure the wettability of the samples.

The USBM results of cleaned cores are given in Table 1. These results show that the subject core plugs were neutrally wet. Only two plugs showed weak water preferential wettability when tested with crude oil and 200 000 ppm brine at 70°C.

Amott Tests of Cleaned Cores

The Amott method was performed on another group of six cleaned plugs. The tests were conducted

Table 1. USBM Wettability Results of Nonpreserved Samples.

Sample No.	Depth (ft)	Porosity (%)	Permeability (md)	20 000 ppm Brine Oil Drive A1 (cm^2)	20 000 ppm Brine Water Drive-A2 (cm^2)	20 000 ppm Brine Wettability Index (WI)	200 000 ppm Brine Oil Drive A1 (cm^2)	200 000 ppm Brine Water Drive-A2 (cm^2)	200 000 ppm Brine Wettability Index (WI)
2	6509.3	29.1	56.6	8.54	6.13	0.14	6.59	6.47	0.008
22	6543.4	26.4	367.9	2.34	1.95	0.08	2.54	3.52	−0.14
31	6545.3	26.5	82.0	4.60	4.41	0.02	3.63	5.06	−0.14
40	6547.8	26.7	557.8	2.72	3.22	−0.08	2.93	3.54	−0.08
44	6548.8	26.0	62.0	5.21	3.03	0.24	4.49	5.90	−0.12
62	6590.9	25.9	291.4	3.86	3.76	0.01	2.54	2.84	−0.05
Averages	26.7	236.3			0.07				−0.09

with filtered crude oil and synthetic brine at ambient conditions. Although the minimum recommended time is 20 hours, the free displacement experiments in both oil and brine was continued for over one week, but free displacement was not obtained. This indicates neutral wettability of the rock under the testing conditions.

Preserved Core Measurements

USBM Tests of Preserved Cores

A total of 54 USBM wettability measurements were performed on 40 preserved core plugs that measure 1 inch in diameter and 1 inch in length. Core plugs were selected for wettability testing based on the lithological variations. All USBM measurements were performed using filtered crude oil. The measurement results are given in Table 2. The test conditions employed were as follows:

1. Tests at 70°C using 20 000 ppm brine and preserved samples,
2. Tests at 25°C using 20 000 ppm brine and preserved samples,
3. Tests at 70°C using 200 000 ppm brine and preserved samples,
4. Tests at 25°C using 200 000 ppm brine and preserved samples,
5. Tests at 25°C using formation brine and preserved samples,
6. Tests at 70°C using formation brine and preserved samples, and
7. Tests at 70°C using 20 000 ppm brine and samples exposed to the atmosphere (Core plugs were exposed to air at room temperature for approximately 80 days prior to testing).

Physical properties of test fluids are given in Table 3 and chemical analysis of formation brine is presented in Table 4.

Several runs were performed at ambient temperature and at an elevated temperature of 70°C. As is shown in Table 2, the USBM wettability index indicated at intermediate wettability for the overall conditions.

Amott Tests of Preserved Cores

The Amott test results of 18 preserved plugs for various testing fluids are presented in Table 5. In all tests, filtered crude oil and synthetic brine of 20 000 ppm salinity were used under ambient conditions. Kerosene was selected during the free displacement stage of some experiments. Core samples were selected to represent various lithologies.

1. Tests of preserved samples: Two sets of six preserved samples were tested using 20 000 ppm brines and stock tank crude oil under ambient conditions. Testing conditions were the same for both sets.
2. Tests using kerosene for free brine displacement: Experiments on a set of six plugs which were previously used in Item 1 was designated to study the effect of kerosene on the wettability. The free brine displacement stage of the Amott test in the third Set was conducted using filtered crude oil instead of kerosene.
3. Tests of samples exposed to the atmosphere: A set of six plugs were tested after being exposed to air at room temperature for approximately two months. Testing fluids used were filtered oil, kerosene, and brine.
4. Tests of cleaned samples: A set of six samples, which was previously used for preserved Amott testing, was retested after cleaning in Dean Stark and Soxhlet extractors in sequence.

Table 2. USBM Wettability Testing of Preserved Samples.

Sample No.	Depth (ft)	Lithology	Porosity (%)	Permeability (md)	Testing Brine	Sample Type	Wettability 70°C	Wettability 20°C
2A	6519.5	Ool Biocl Grst	25	92.0	20K	P	0.30	–
2B	6519.5	Ool Biocl Grst	26	201.0	FB	P	0.30	0.28
2C	6519.5	Ool Biocl Grst	26	150.0	200K	P	0.21	na
8A	6521.5	Biocl Pkst	26	4.0	20K	P	0.08	0.13
8B	6521.5	Biocl Pkst	24	5.0	20K	P	–	−0.06
10A	6522.5	Ool Biocl Grst	27	35.0	20K	P	na	0.24
10B	6522.5	Ool Biocl Grst	25	53.0	20K	E	0.12	–
10C	6522.5	Ool Biocl Grst	26	10.0	200K	P	0.04	0.03
12A	6523.5	Biocl Grst	28	458.0	20K	P	0.16	–
12B	6523.5	Biocl Grst	27	504.0	FB	P	0.27	0.06
12C	6523.5	Biocl Grst	26	529.0	200K	P	0.04	−0.07
16A	6525.2	Peld Biocl Pkst	29	36.2	20K	P	0.29	–
16C	6525.2	Peld Biocl Pkst	28	24.0	20K	E	−0.14	–
20A	6527.0	Peld Biocl Pkst	25	17.6	20K	P	−0.07	–
23A	6528.3	Peld Biocl Pkst	28	17.6	20K	P	0.04	–
27A	6529.8	Peld Biocl Grst	25	84.9	20K	E	−0.34	–
31A	6539.2	Dol Biocl Wkst	23	5.5	20K	P	0.23	0.11
31B	6539.2	Dol Biocl Wkst	21	4.0	20K	P	–	0.15
31C	6539.2	Dol Biocl Wkst	22	5.0	20K	E	0.21	–
32A	6539.6	Dol Biocl Wkst	15	2.5	20K	P	0.37	–
32C	6539.6	Dol Biocl Wkst	19	3.0	20K	E	0.02	–
38A	6541.9	Dol Intcls Biocl Pkst	21	4.9	20K	P	0.24	–
38B	6541.9	Dol Intcls Biocl Pkst	18	4.0	FB	P	0.00	0.35
38C	6541.9	Dol Intcls Biocl Pkst	22	5.0	200K	P	na	0.13
41A	6543.4	Biocl Intcls Grst	16	4.7	20K	E	0.05	–
44A	6544.5	Biocl Intcls Grst	21	244.3	20K	P	0.13	–
51A	6547.3	Biocl Intcls Grst	25	883.9	20K	P	0.11	–
54A	6549.2	Biocl Intcls Grst	26	125.0	20K	P	0.00	−0.12
54B	6549.2	Biocl Intcls Grst	26	140.0	20K	E	−0.23	–
54C	6549.2	Biocl Intcls Grst	25	159.0	200K	P	0.16	0.06
56A	6619.3	Dol Biocl Pkst	20	19.0	20K	P	–	0.29
56B	6619.3	Dol Biocl Pkst	23	55.0	200K	P	0.28	−0.07
56C	6619.3	Dol Biocl Pkst	19	28.0	200K	P	0.22	0.10
59A	6620.2	Dol Biocl Wkst	19	4.5	20K	P	0.36	–
59B	6620.2	Dol Biocl Wkst	19	12.0	20K	E	−0.16	–
59C	6620.2	Dol Biocl Wkst	20	13.0	20K	E	−0.30	–
65A	6622.4	Dol Biocl Wkst	23	32.0	20K	P	0.05	–
68A	6623.4	Biocl Wkst	24	21.0	20K	P	−0.03	–
72C	6625.5	Dol Intcls Biocl Wkst	13	1.7	20K	E	0.00	–
105A	6746.3	Biocl Intcls Pkst	12	13.0	20K	P	0.07	–

20K: 20 000 ppm brine
200K: 200 000 ppm brine
FB: Formation brine
P: Preserved
E: Exposed to atmosphere

Table 3. Physical Properties of Testing Fluids.

Fluids	25°C			70°C		
	Density (g/cc)	Viscosity (cps)	API	Density (g/cc)	Viscosity (cps)	API
Arab-D crude oil	0.8553	26.80	33	0.8409	7.3	30
20K ppm brine	1.0140	1.05	–	1.0110	0.58	–
200K ppm brine	1.1456	1.48	–	1.1206	0.95	–
Formation brine	1.1520	–	–	1.1230	–	–

Table 4. Chemical Analysis of Formation Brine Used in Some Wettability Tests.

Parameter	Concentration (mg/I)
Sodium	58 297
Calcium	19 840
Magnesium	2439
Sulfate	355
Chloride	131 705
Bicarbonate	2226
Boron	66.7
Barium	12.7
Copper	0.005
Potassium	2700
Lithium	31
Manganese	0.08
Lead	0.005
Rubidium	2.68
Silicon	23
Strontium	1000
Zinc	0.05
pH	6.3
Specific Gravity	1.164

Source: Saudi Aramco

Contact Angle Measurements

Contact angles were measured using filtered dead crude and various brines under different test conditions (Table 6). Contact angle variations with aging are plotted in Figure 2. Test results are discussed below:

1. Tests under ambient conditions using 20 000 ppm brine: Two tests (Tests 1 and 2 in Table 6) yielded equilibrium contact angles of 61° and 60°, respectively, which implies neutral wettability for the fluids tested at ambient conditions.
2. Tests under elevated pressure and temperature using 20 000 ppm brine: Test 3 was run to investigate the wettability of 20 000 ppm brine and dead oil at 70°C and 2800 psia. The equilibrium contact angle under these conditions was found to be 32°.
3. Tests under ambient conditions using 200 000 ppm brine: In Test 4, equilibrium contact angles of 42° for advancing and 40° for receding were measured.
4. Tests under ambient pressure and 70°C using 200 000 ppm brine: In Test 5, two different oil drops were aged on the calcite surface and angles of 29° and 28° were measured under these conditions.
5. Tests under ambient conditions using formation brine: In Test 6, both receding and advancing angles were measured and an equilibrium angle of 40° was obtained for both.
6. Tests under elevated pressure and temperature using formation brine: Formation brine and filtered crude oil were tested at 70°C and 2800 psia during Test 7. The equilibrium contact angle measured was 27°.

Tests using 20 000 ppm brine (Tests 1 and 3) indicated a shift in wettability from neutral-wet (61°) to moderately water-wet (32°) as the test temperature was increased from ambient to 70°C. Similarly, in 200 000 ppm tests (Tests 4 and 5) increasing temperature from ambient to 70°C lowered the contact angle from 42° to 28°, thus resulting in a higher degree of water-wetting.

Considering ambient conditions only (Tests 1 and 4), the contact angle decreased from 61° to 42° as the salinity was increased from 20 000 ppm to 200 000 ppm. Yet under elevated temperature conditions (Tests 3 and 5), same salinity change did not demonstrate any significant variation in the contact angle (32° *versus* 28°). Formation brine (213 000 ppm) which contains several salts (Table 4) also showed a similar

Table 5. Amott Wettability Testing Results of Preserved Samples.

Sample No.	Depth (ft)	Lithology	Porosity (%)	Permeability (md)	Sample type	Medium for free brine displacement	Displacement: Under 20K ppm brine: Free crude oil (cc)	Displacement: Under 20K ppm brine: Crude oil by centrifuge (cc)	Displacement: Under oil or kerosene: Free brine (cc)	Displacement: Under crude oil: Brine by centrifuge (cc)	Wettability index: δ_w	Wettability index: δ_o	Wettability index: I
8C	6521.5	Biocl Pkst	24	4.7	E	K	0.00	0.70	0.10	0.70	0.00	0.13	−0.13
16B	6525.2	Peld Biocl Pkst	28	35.7	P	K	0.00	1.30	0.30	1.50	0.00	0.17	−0.17
16B	6525.2	Peld Biocl Pkst	28	35.7	P	Oil	0.00	1.40	0.00	1.50	0.00	0.00	0.00
16B	6525.2	Peld Biocl Pkst	28	35.7	C	Oil	0.00	0.70	0.00	1.30	0.00	0.00	0.00
20B	6527.0	Peld Biocl Pkst	30	15.9	P	K	0.00	2.00	0.00	1.50	0.00	0.00	0.00
20C	6527.0	Peld Biocl Pkst	29	23.2	E	K	0.00	1.40	0.15	1.20	0.00	0.11	−0.11
23B	6528.3	Peld Biocl Pkst	29	21.8	P	K	0.05	2.00	0.25	1.60	0.02	0.14	−0.12
23B	6528.3	Peld Biocl Pkst	29	21.8	C	K	0.10	1.00	0.10	1.30	0.09	0.07	0.02
23C	6528.3	Peld Biocl Pkst	29	27.1	E	K	0.00	1.50	0.15	1.30	0.00	0.10	−0.10
32B	6539.6	Dol Biocl Wkst	19	2.0	P	K	0.00	1.40	0.15	0.60	0.00	0.20	−0.20
32B	6539.6	Dol Biocl Wkst	19	2.0	C	K	0.00	0.50	0.00	0.40	0.00	0.00	0.00
41B	6543.4	Biocl Intcls Grst	20	21.6	P	K	0.00	0.80	0.10	0.80	0.00	0.11	−0.11
41B	6543.4	Biocl Intcls Grst	20	21.6	P	Oil	0.00	0.70	0.00	1.00	0.00	0.00	0.00
41B	6543.4	Biocl Intcls Grst	20	21.6	C	Oil	0.05	0.60	0.00	0.50	0.08	0.00	0.08
44B	6544.5	Biocl Intcls Grst	27	387.6	P	K	0.15	1.70	0.25	1.50	0.08	0.14	−0.06
44C	6544.5	Biocl Intcls Grst	22	670.6	E	K	0.24	1.20	0.15	1.60	0.17	0.09	0.08
51B	6547.3	Biocl Intcls Grst	28	2045.0	P	K	0.35	1.80	0.00	1.70	0.10	0.21	−0.11
51B	6547.3	Biocl Intcls Grst	28	2045.0	P	Oil	0.20	2.00	0.45	1.70	0.16	0.00	0.16
51B	6547.3	Biocl Intcls Grst	28	2045.0	C	Oil	0.20	1.80	0.20	1.30	0.10	0.13	−0.03
51C	6547.3	Biocl Intcls Grst	26	1855.4	E	K	0.10	1.50	0.30	1.60	0.06	0.14	−0.08
61A	6621.3	Biocl Pkst	20	3.9	P	K	0.15	1.80	0.35	0.60	0.16	0.37	−0.21
61A	6621.3	Biocl Pkst	20	3.9	P	Oil	0.10	0.80	0.00	0.60	0.11	0.00	0.11
61B	6621.3	Biocl Pkst	20	2.7	P	K	0.00	0.80	0.40	0.30	0.00	0.57	−0.57
61B	6621.3	Biocl Pkst	20	2.7	C	K	0.00	0.80	0.00	0.00	0.00	0.00	0.00
65B	6622.4	Dol Biocl Wkst	24	49.2	P	K	0.50	0.80	0.15	1.40	0.39	0.10	0.29
65B	6622.4	Dol Biocl Wkst	24	49.2	P	Oil	0.40	0.80	0.00	1.40	0.33	0.00	0.33
68C	6623.4	Biocl Wkst	21	6.0	E	K	0.15	0.55	0.20	0.70	0.21	0.22	−0.01
84C	6631.3	Sil Dol	6	0.2	P	K	0.00	0.10	0.00	0.00	0.00	0.00	0.00
84C	6631.3	Sil Dol	6	0.2	P	Oil	0.00	0.10	0.00	0.00	0.00	0.00	0.00
97B	6739.9	Biocl Intcls Pkst	–	12.0	P	K	0.00	0.30	0.00	0.00	0.00	0.00	0.00

C: Cleaned sample E: Exposed to air I: Amott–Harvey index K: Kerosene P: Preserved sample δ_o: Displacement by oil δ_w: Displacement by water

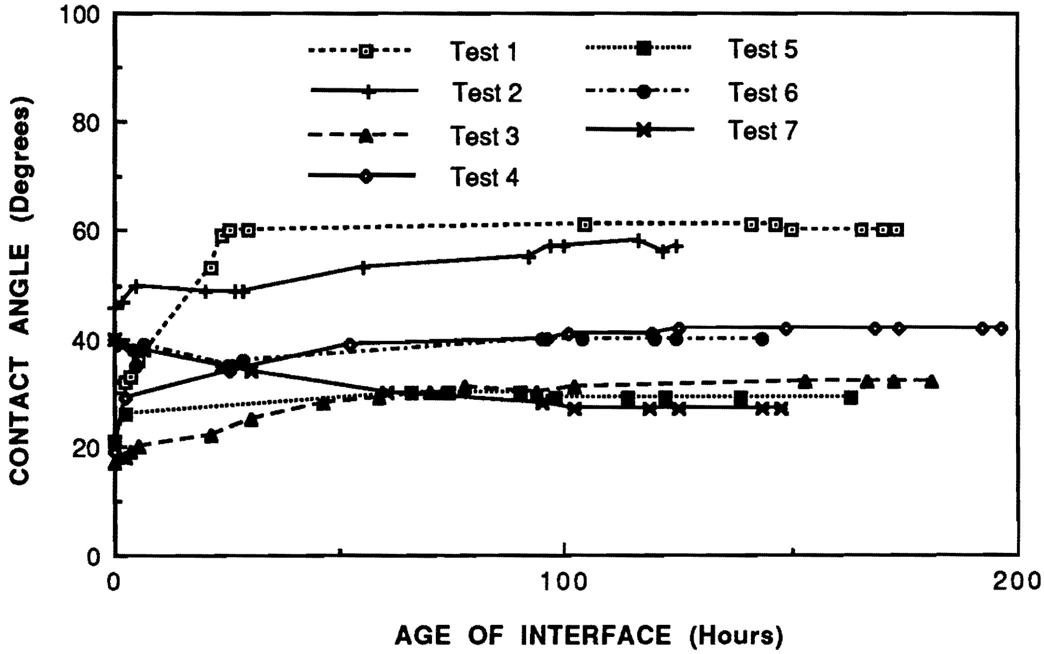

Figure 2. Contact Angle Measurements Performed on a Calcite Surface.

Table 6. Contact Angle Measurements Using Filtered Arab-D Oil on Calcite Surface.

Test No.	Test time (hrs)	Temperature (°C)	Pressure (psia)	Brine type	Contact Angle (degrees)	Remarks
1	174	21.5	20	20K	61	Neutral-wet
2	125	21.5	20	20K	60	Neutral-wet
3	181	70.0	2800	20K	32	Water-wet
4	606	21.0	20	200K	42	Water-wet
5	163	70.0	20	200K	28	Water-wet
6	143	22.2	20	FB	40	Water-wet
7	148	70.0	2800	FB	27	Water-wet

20K: 20 000 ppm brine
200K: 200 000 ppm brine
FB: Formation brine

trend as 200 000 ppm synthetic brine under the same test conditions. At ambient conditions, 200 000 ppm synthetic brine yielded 42° and formation brine 40° contact angles, respectively. Similarly, at 70°C, 200 000 ppm brine and formation brine test results were 28° and 27°, respectively.

Comparison of Test 5 with Tests 3 and 7 clearly indicated that pressure did not affect the wettability. Contact angles for Tests 5, 3, and 7 were 28°, 32°, and 27°, respectively. The temperature was maintained at 70°C while the pressure was varied from 20 psia in Test 5 to 2800 psia in Tests 3 and 7. Although different brines were used in these three tests, it was already determined that salinity effect was almost negligible, under elevated temperature (70°C). Therefore, these test results were utilized to interpret the pressure effect on the contact angle.

Drop sizes were varied in most of the tests. During each test, more than one drop of various sizes were monitored for their contact angles. This, in turn, indicated that the contact angle is not a function of drop size.

Both receding and advancing angles were recorded during some of the tests. Results of these tests showed that receding and advancing contact angles were very similar. This implies that the calcite crystals used were clean, homogeneous, and smooth [23].

All tests indicated that the contact angle reached equilibrium at around 100 hours of aging of interface, and did not change with further aging. This result was also verified by Test 4 which continued over 600 hours.

ASSESSMENT OF VARIOUS EFFECTS

Temperature Effect

Several USBM runs were successfully performed to investigate the temperature effect on rock wettability. Table 7 presents the data related to the temperature effect. A total of 22 core plugs from three different lithological groups [24] were tested: five samples from oolitic bioclastic grainstone, seven plugs from bioclastic grainstone, and the remaining ten samples from dolomitic bioclastic packstone and wackestone. USBM runs were conducted at ambient conditions and at an elevated temperature of 70°C. Measured wettability indices at both temperatures were averaged for each group.

Figure 3 shows the plot of the USBM wettability index *versus* testing temperature for the three different rock types. The average wettability index ranged from −0.02 to 0.18 at ambient temperature and from 0.12 to 0.21 at 70°C indicating slightly more water-wetting at a higher temperature, but still in the intermediate range. This observation is in conformance with Donaldson and Siddiqui's [25] results.

Brine Salinity Effect

In order to examine the brine salinity effect on the wettability of carbonate cores, three brines, each with a different salinity were selected. In the course of this investigation, two synthetic brines with salinities of 20 000 ppm and 200 000 ppm and a produced formation brine were used. Tested core samples were grouped according to the lithological description into two main groups: (1) grainstone and (2) dolomitic packstone/wackestone.

Experimental data and results used in studying the salinity effect are given in Table 7. An average wettability index was calculated for each rock type. Figure 4 is the graphical presentation of the test data. As shown in the plot, wettability index slightly decreased with increasing salinity for synthetic (NaCl) brines. When 200 000 ppm synthetic brine results were compared with formation brine (213 000 ppm) results, a slight increase in wettability was observed. Yet, all these variations were within the intermediate wettability range.

Table 7. The Wettability Averages of Some Lithology Groups.

Lithology group	Temperature effect		Brine effect			Exposing to air effect		Cleaning effect	
	25°C	70°C	20K	200K	FB	Preserved	Exposed	Preserved	Clean
1. Grainstone	–	–	0.14	0.10	0.29	–	–	–	–
2. Oolite bioclast grainstone	0.18	0.21	–	–	–	–	–	–	–
3. Bioclast grainstone	−0.02	0.12	–	–	–	0.18	−0.12	0.00	0.00
4. Intraclast bioclast grainstone	–	–	–	–	–	0.08	−0.09	−0.06	0.03
5. Dolomitic bioclast packstone & wackestone	0.12	0.17	0.18	0.11	–	0.21	0.07	−0.16	0.01
6. Bioclast wackestone	–	–	–	–	–	0.17	−0.17	–	–

FB: Formation brine

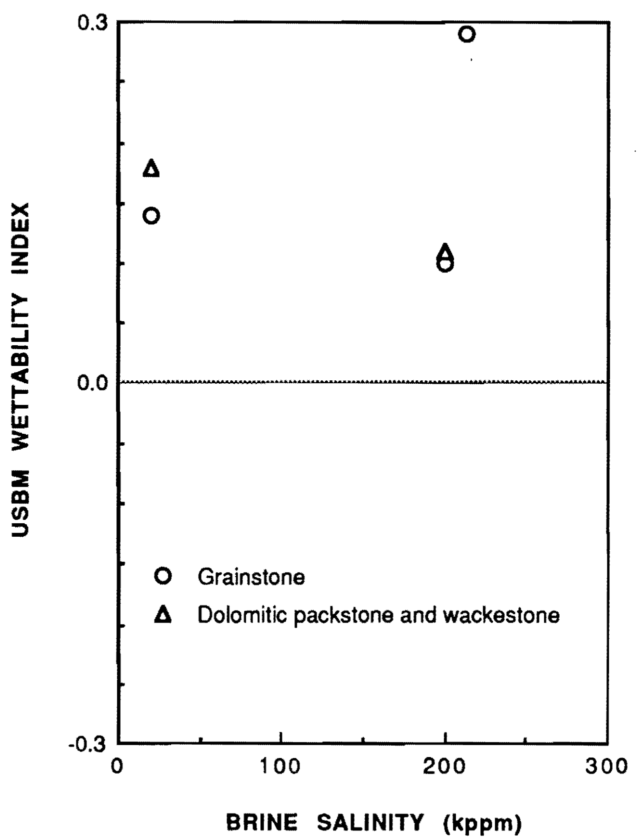

Figure 3. Temperature Effect on Wettability.

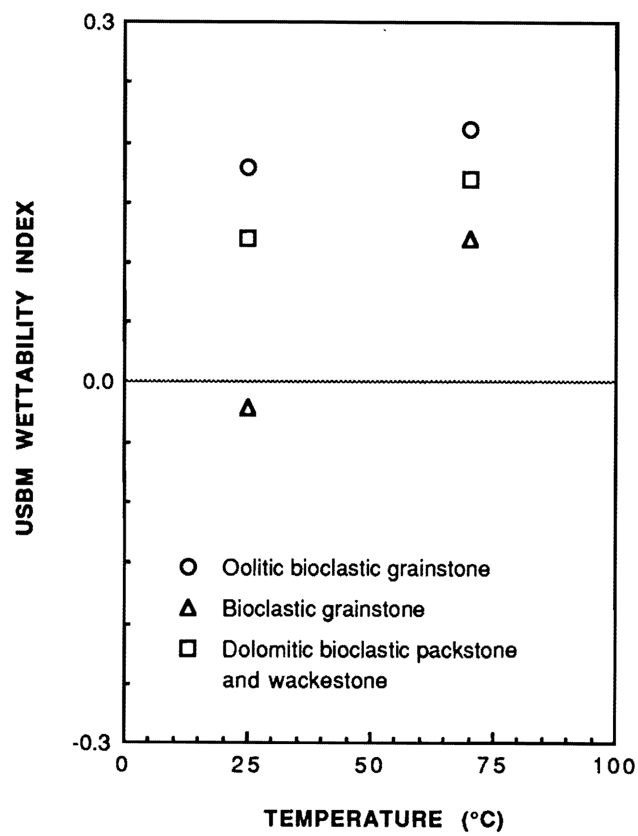

Figure 4. Brine Salinity Effect on Wettability.

Core Exposure Effect

Several core plugs were selected and exposed to air for approximately 80 days at ambient conditions to examine the importance of exposure effect. Based on lithological rock description, tested cores were divided into four groups. Table 7 includes experimental results for these four groups. The comparison was made between the test results of preserved cores and exposed cores of the same lithological type. Measured USBM wettability indices fell into the intermediate wettability zone for all plugs. Figure 5 gives the plot of USBM wettability index against time of exposure to air in days. As shown in this graph, core samples become less water-wet with increased exposure time for all lithological groups studied. Hence, exposing the core samples to air could significantly affect the original wettability.

Core Cleaning Effect

Selected preserved cores were tested according to the Amott method, then upon completion of testing, cores were cleaned with toluene, alcohol, and oven dried under vacuum. These dried and cleaned cores were retested for wettability.

The tested samples represent three different lithological rock types. These groups are bioclastic grainstone, intraclastic bioclastic grainstone, and dolomitic bioclastic packstone/wackestone. Figure 6 is the plot of the Amott Wettability Index for preserved and cleaned state cores of the three lithological formations. As shown, the average wettability index for the first group remains unchanged after cleaning with toluene. The average wettability indices for the other two groups are more water-wet for cleaned cores than they are at preserved conditions. The wettability indices ranged from −0.16 to 0.0 for preserved cores and from 0.0 to 0.03 for cleaned cores. This indicates that cleaning the cores with toluene has minimum effect on wettability of subject cores.

Measurement Method Effect

The USBM test results of preserved cores yielded wettability indices ranging from 0.07 to 0.36. Results

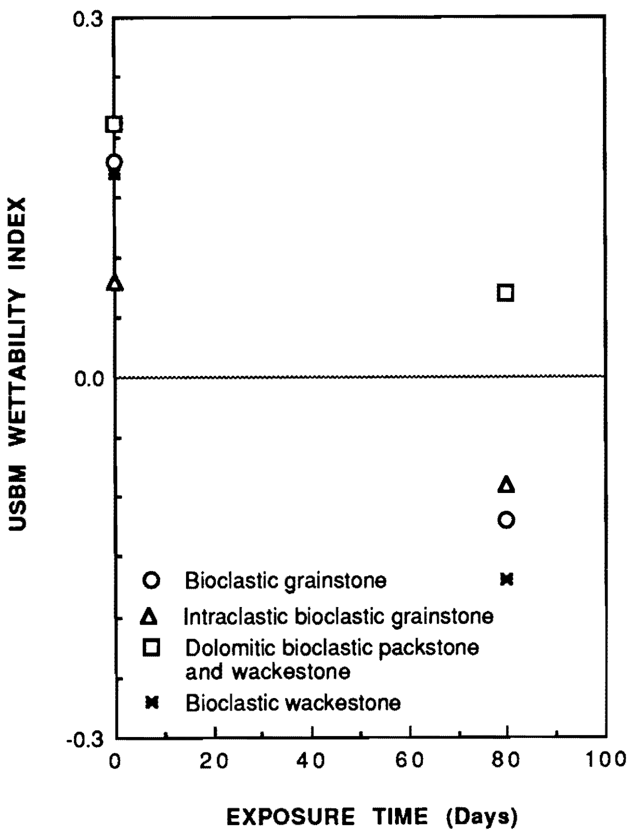

Figure 5. Core Exposure to Air Effect on Wettability.

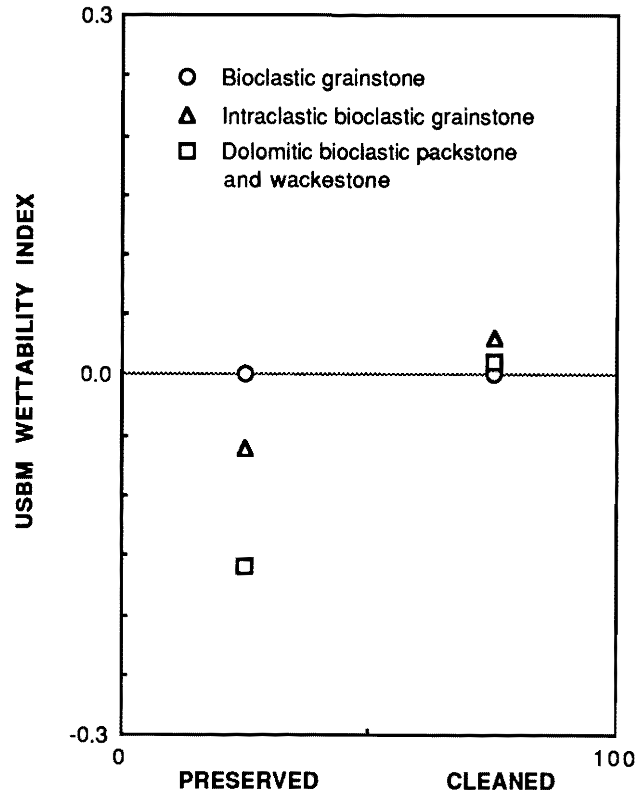

Figure 6. Core Cleaning Effect on Wettability.

obtained from Amott tests were consistent with USBM results. The wettability indices ranged from −0.21 to 0.33 on the Amott–Harvey scale. Amott and USBM testing of the carbonate rock samples at various conditions, showed that the subject cores are intermediately wet. However, contact angle measurements using filtered crude oil and formation brine on calcite surface yielded moderate water-wet condition. A difference between contact angle wettability and Amott and USBM wettabilities was observed. Wettability results of the USBM, Amott, and contact angle methods are shown in Figure 7.

CONCLUSIONS

Preserved and nonpreserved core samples were tested for wettability evaluation under various laboratory conditions. A total of 60 USBM runs and 24 Amott tests were performed for wettability determination. Finally, the contact angle test was carried out for testing wettability of fluids on a calcite surface. Both ambient and elevated temperatures and pressures were considered for most of the laboratory measurements. Based on the present investigation, the conclusions can be summarized as follows:

1. Amott and USBM testing of the carbonate rock samples at various conditions, showed that the subject cores are intermediately wet. However, contact angle measurements using filtered crude oil and formation brine on calcite surface yielded moderate water-wet condition.
2. The USBM test results of preserved cores yielded wettability indices ranging from 0.07 to 0.36.
3. Results from USBM runs conducted to investigate the effect of temperature demonstrated that, core samples become more water-wet with increasing temperature.

   As temperature was increased from ambient to 70°C, the contact angles decreased. Moreover, regardless of type of fluids used, more water-wet condition was observed with increasing temperature.
4. The USBM tests on preserved cores using formation brine yielded slightly more water-wet results

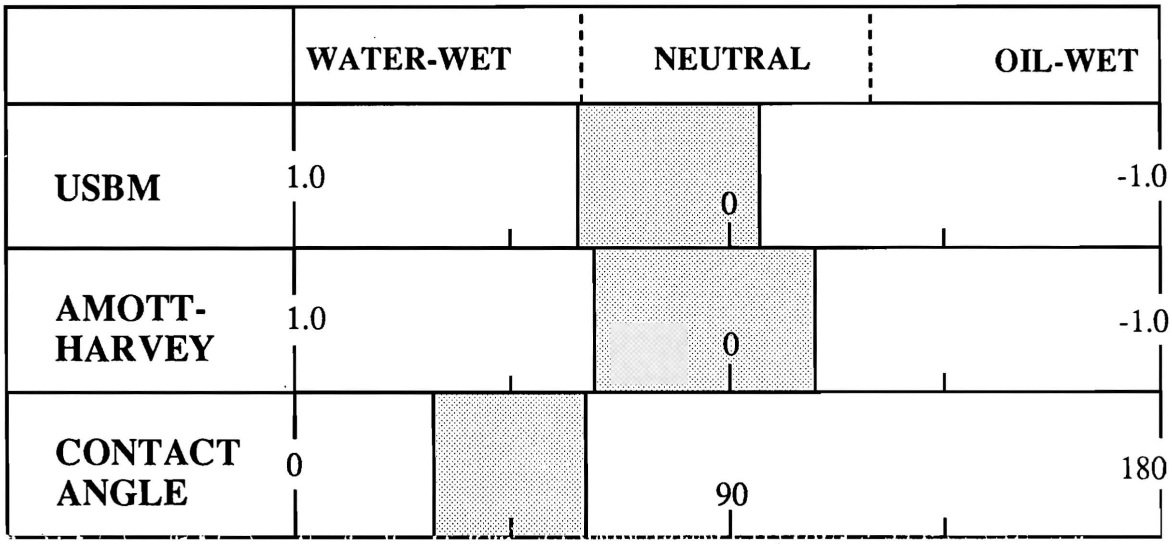

Figure 7. Comparison of Various Wettability Measurement Methods.

than tests using synthetic brines. However, contact angle results were compatible for both brines.

It was concluded that increasing brine salinity decreased the contact angles at ambient temperature, although this effect was not observed at elevated temperature (70°C).

5. Exposure to air and partial drying indicated that samples become more oil-wet as compared to preserved samples.
6. Results obtained from Amott tests were consistent with USBM results. The wettability indices ranged from −0.21 to 0.33 on the Amott−Harvey scale.
7. Wettability results using the Amott method showed that cleaned cores were more water-wet than preserved cores.
8. It was observed from all contact angle tests that the equilibrium age of interface was around 100 hours.
9. It was determined that pressure has no influence on the contact angle measurements.

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