

Wettability Alteration of Gas Condensate Reservoir Rocks to Gas Wetness by Sol-Gel Process Using Fluoroalkylsilane

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ABSTRACT: Natural gas is becoming a very important energy source in recent years. A sharp reduction has been observed in gas deliverability in many low permeability gas reservoirs due to bottom-hole pressure drops below the dew point pressure. It is an established fact that altering the wettability of reservoir rocks from liquid wetness to gas wetness could improve the gas deliverability. In this study, a new polymeric surfactant coating for altering the wettability of Sarkhun reservoir rocks was prepared by using a sol-gel process. Perfluorodecylsilane (PFDS) and triethoxysilane (TEOS) were used to obtain an inorganic-organic network via hydrolysis and polycondensation reactions. The effect of this method was examined by various characterization tests such as FTIR, SEM, and EDX, static contact angle and imbibition tests for wettability alteration. The experimental results revealed that, the prepared polymeric network including the terminal fluorine groups were effective and provided sufficient repelling characteristics towards oil and water. Static contact angles were raised from 30 to 130° for water, and from <5 to 60° for condensate after sol-gel treatment. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 128: 4077–4085, 2013

KEYWORDS: fluoropolymers; surface modification; surfactants

Received 25 February 2012; accepted 20 September 2012; published online 17 October 2012

DOI: 10.1002/app.38632

INTRODUCTION

Production of natural gas around the world is kept increasing in the last few decades. However, many problems are often encountered during the gas production in low permeability gas condensate reservoirs. In such reservoirs, when flowing bottom-hole pressure falls below the dew point pressure of the fluid, liquid saturation drops out from gas phase. This condensate accumulation develops “condensate banking” or “condensate blockage” in near wellbore region and results in significant loss of both gas and condensate productivity (as shown in Figure 1).

Today, several methods are proposed to remove drop out liquids and recover well production. Hydraulic fracturing, gas recycling and solvent injection such as methanol and propanol have been developed to confront this problem, though they are not a permanent solution due to their temporary restoration effect of well productivity.¹ A recent successful method has focused on altering the wettability of rocks near the wellbore region from strongly water/oil wet to gas wet. This method can provide a long-term solution to treat damage caused by liquid accumulation. Wettability alterations using fluoro-components have been studied extensively in the past two decades.^{2–7}

Li and Firoozabadi⁴ altered the wettability of rocks to preferential gas wetting by using fluoro-polymers consisting of FC754

(cationic surfactant, soluble in water) and FC722 (fluoro-polymer, insoluble in water). The results showed that in glass capillary tube, θ altered from 50 to 90° and from 0 to 60° by using FC754 for water-air and decane-air systems, respectively. Before treatment, the percentage of imbibition was 60% for water, while after chemical treatment it decreased to zero. For FC722, θ altered from 50 to 120° and 0 to 60° for water-air and decane-air systems, respectively and there was no imbibition after treatment.

Kumar et al.⁵ presented a successful treatment using fluoro-surfactant in methanol and water solution at reservoir conditions to alter the wettability of sandstone and limestone cores. The relative permeability for both gas and condensate for sandstone cores was improved by a factor of about two for temperatures over the range of 145 to 275°F.

Liu et al.⁶ performed a laboratory study to demonstrate improved gas deliverability by altering the wettability of sandstone core with proper chemicals from strongly water-wet to neutral-wet. Capillary rise, contact angle, imbibitions, and core flood tests were conducted to provide a cost-effective method in reservoirs where water blockage occurs.

Mohanty et al.⁷ presented a chemical treatment by using 10 surfactants to change the wettability of carbonate and sandstone

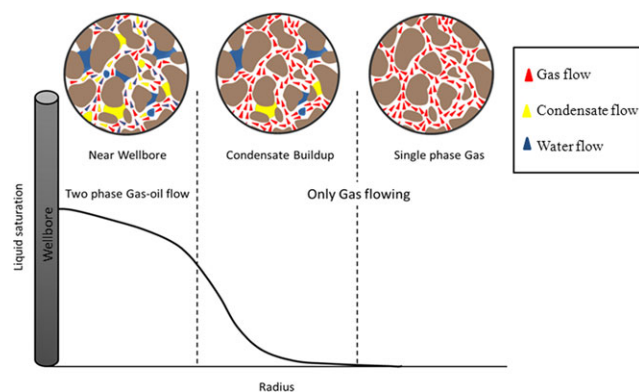


Figure 1. Condensate and water build-up in gas condensate reservoirs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

rocks from water wet to intermediate wet conditions. He recognized that among fluorosilanes, with higher number of fluoro-groups, samples tend to become less water wet, and 1 day of aging period with 1 wt % concentration is sufficient for altering wettability.

Since increasing gas permeability of these reservoirs is primarily associated with the alteration of wettability, the major goal of this study was to change the wettability of limestone rocks in such a manner that would show remarkable characteristics of hydrophobic/oleophobic properties. The samples were collected from Sarkhun reservoir, one of the gas condensate reservoirs located in Hormozgan, Iran. Sarkhun has low permeability and it is predicted that it may undergo condensate blockage in near future. To make the limestone cores hydrophobic/oleophobic, i.e., amphiphobic, we have used a type of monomeric surfactant and conducted the sol-gel process to prepare fluorinated polymeric network that behaves as a repellent towards water and oil.

Up to now, various studies have been performed to modify the surface properties by treating the inorganic materials with various coupling agents such as alkylsilanes and fluoroalkylsilanes.^{8–12} Their current applications are useful for windshields of automobiles, dust-free and self-cleaning surface for solar cells, building walls, photovoltaic, exterior architectural glasses, and the reduction of frictional drag on ship hulls.^{13–15} Among these approaches, the sol-gel process is found to be an effective and simple technique for making coating films onto substrates. To the best of our knowledge, there is no study yet reported on sol-gel process to prepare fluorinated coating for wettability alteration of the gas reservoir rocks.

In this project, limestone surface modification was achieved by using triethoxy-1H,1H,2H,2H-perfluorodecylsilane (Figure 2) and triethoxysilane (TEOS). The alkoxy silane monomers underwent polymerization and converted into a cross-linked polymer onto the surface. The presence of reactive –OH groups on the mineral grains of the stone could lead to a molecular attachment of the polymer chains to the mineral grains. The fluorine groups with the tendency to migrate towards the air/film interface, reduced the surface energy while TEOS increased the density of sites of –OH groups.

We primarily collected full descriptions of rocks, fluids, chemicals used for rock modification and treatment procedure. To demonstrate the performance of the sol-gel process, then we performed characterization tests such as Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and electron dispersive analysis of X-ray (EDX). Static contact angle measurements and imbibition tests were run to confirm degree of wettability alteration. We also examined the adhesion of applied sol-gel coating onto the limestone. Finally, we showed the results before and after core treatment stages with and without sol-gel modification.

EXPERIMENTAL

Rock

In this study, the rock samples were selected from Sarkhun reservoir. The porosity was around 25% measured by Helium porosimeter (Vinci, France), using Boyle's-Mariotte's law. The cores had a gas permeability of 3–3.5 mDarcy, measured by N₂ at a flow rate of 100 mL/min using core flood test. Darcy's law was applied to calculate permeability as follows:

$$k = \frac{q\mu L}{A\Delta P} \quad (1)$$

where q is the core flow rate, μ is the flowing fluid viscosity, L is the length of the core, A is the cross-sectional area of the core, and ΔP is the steady state pressure drop across the core.⁵ The chemical analysis of the rock samples was performed by XRF analysis.

Fluids

In this study, air was the gas phase in contact angle and imbibition measurements. The aqueous phase was synthetic brine of 2.0 wt % NaCl prepared in distilled water with 1.01 specific gravity and 1cp viscosity at 20°C. We also used normal decane (nC₁₀), with 0.73 specific gravity and 0.92 cp viscosity at 20°C, as condensate (DAE JUNG, Korea). Distilled water was prepared in-situ in PVT laboratory, Shiraz University, Iran. The samples were dried with N₂ gas (purity 99.99%) which was purchased from Abughaddare Gas Co., Shiraz, Iran.

Chemicals

The chemicals used included a perfluorinated surfactant, triethoxy-1H,1H,2H,2H-perfluorodecylsilane of 97% purity (Alpha Aerosol, Germany), abbreviated as PFDS in this article and ethanol 99.8 wt % (Merck, Germany) as solvent. We also used tetraethoxysilane (TEOS, 98%) [Si(OC₂H₅)₄] from Merck,

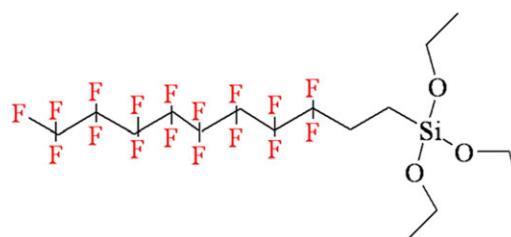


Figure 2. Molecular structure of triethoxy-1H,1H,2H,2H-perfluorodecylsilane. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Germany and HCl 37 wt % (Merck, Germany) for sol–gel process. All the chemicals were used as received.

EXPERIMENTAL PROCEDURE

Preparation of Solutions and Treated Cores

To study the effect of sol–gel method to produce polymeric surfactant as compared with monomeric one, two samples were made. We first prepared monomeric surfactant solution, designated as sample A, by dissolving PFDS, water and ethanol having a composition of 94 wt % ethanol, 4 wt % water, and 2 wt % PFDS, at room temperature. The second coating solution (polymeric surfactant coating) was prepared by mixing PFDS, TEOS before dilution with ethanol at room temperature. Distilled water and HCl were subsequently added drop-wise to the solution described above under stirring for 2 h in a capped glass bottle. The mass ratio of the mixture was 1:0.25:10.625:0.5:0.125, TEOS/PFDS/ETOH/Water/HCl. Compositions of water and PFDS were the same as the first solution i.e., 4 and 2 wt %, respectively. This procedure led to formation of a transparent sol–gel solution (solution for sample B). The cores were cleaned and dried by N₂ gas until a constant weight was reached. Then the clean cores were aged in both solutions (3 × pore volume) for 48 h. The aged cores were subjected to heat treatment at 80°C for 1 h to remove the extra liquid chemicals completely. Note that, this temperature of drying step is within the range of the gas condensate reservoirs temperature.

FTIR Spectra

Fourier transform infrared measurements were performed by KBr method using a Perkin Elmer FTIR spectrophotometer (spectrum RX1). The spectra were recorded between 600 cm⁻¹ and 4000 cm⁻¹ to investigate the chemical bonding between the groups present in the sol–gel system.

SEM-EDX

Scanning electron microscopy (SEM) was employed on sol–gel treated and untreated samples at room temperature to assess morphology information (Lica Cambridge LTD, England). The samples were connected to an SEM stub and were covered by conductive coating. Energy dispersive X-ray (EDX) was also conducted for the identification of sol–gel coated samples, by penetrating into the cores using model ZEISS, Germany.

Contact Angle

The static contact angle (θ) for the liquid-gas-rock system was measured by placing a droplet of brine and n-decane onto the surface of air-saturated samples before and after the treatments (DSA100, Germany). To evaluate the homogeneity of samples, each test was carried out on at least three spots of samples with droplets ~ 5 μ L. The contact angle data were average values of three different droplet spots. The image of the droplets was captured at equilibrium state at room temperature.

Spontaneous Imbibition

Spontaneous imbibition is a phenomenon that exists in gas condensate reservoirs. We conducted this experiment by immersing the air-saturated core samples into liquid phase (brine 2.0 wt % and n-decane) before and after chemical treatments to evaluate the effectiveness of wettability alteration. The increase in weight

Table I. Chemical Composition of the Sample

Formula	(wt %)
SiO ₂	1.15
Al ₂ O ₃	0.45
Fe ₂ O ₃	0.14
CaO	29.1
MgO	20.9
IL	47.7
Total	99.44

with time was recorded regularly until no significant change was detected.

Adhesion of the Applied Sol–Gel Coating onto the Limestone Rock

The main objective in this part was to examine the covalent molecular bond formation with limestone surface using FTIR analysis of ethanol which has been subjected to sol–gel coating in different steps. Initially, the FTIR spectra were carried out for pure ethanol and the transparent sol solution before applying to the surface. To investigate the adhesion strength of sol–gel coating on the limestone surface after completion of the process, the treated cores were initially washed before and after drying step with ethanol (3 × pore volume) for probable extraction of sol compound. The extraction was performed at room temperature for 30 min. Subsequently, the IR spectra were recorded on liquid extractions of treated samples.

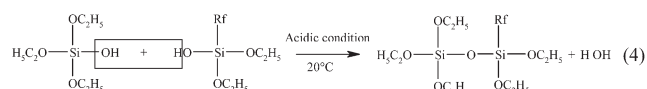
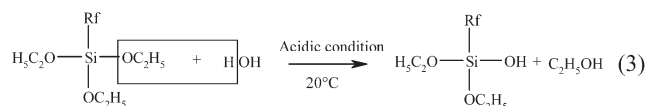
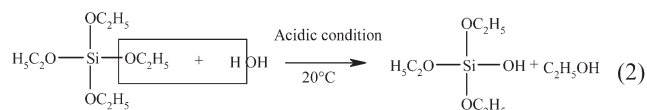
RESULTS AND DISCUSSION

Chemical Composition of the Studied Samples

Table I shows the chemical composition of the limestone samples selected in this study. The XRF result indicates that the samples consist of significant amount of calcite.

Sol–Gel Reactions and FTIR Analysis

Sol–gel process consists of two main steps: hydrolysis and polycondensation reactions. In the first step, the reactions occur by oxygen attack of water on alkoxy group of alkoxy silanes. As we can see in eqs. (2) and (3), TEOS and PFDS react with water and produce hydrolyzed TEOS, PFDS, and an alcohol as a by-product at acidic condition. In this step, HCl was used as a catalyst for rapid hydrolysis reactions (R_f = CF₃(CF₂)₇CH₂CH₂).



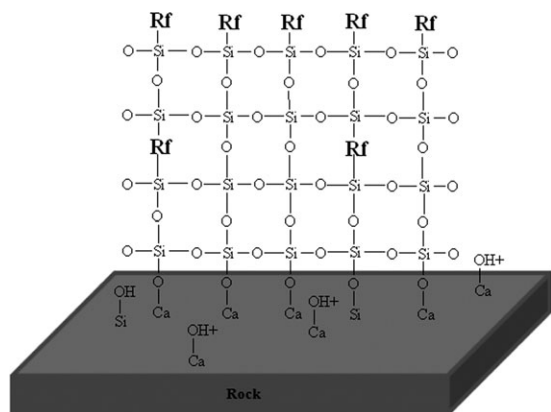


Figure 3. A schematic model of modified limestone rock by PFDS and TEOS coating.

In the next step, as we can see in equation (4) two silanol molecules, produced from hydrolyzed reactions, undergo polycondensation and form dimers, trimers, tetramers, and finally a polymeric network. It was in this step that the mixture was applied onto the samples. On the surface of samples, as indicated by the compositional test, there exist CaOH^+ and small quantity of SiOH species, which can react chemically with silane groups and attach to mineral grains. When ethanol evaporates, the polymer remains on the stone. This cross-link structure contains —Si—O—Si— called siloxane and fluorine groups that provide sufficient oil and water repellency. The schematic formation of PFDS/TEOS sol-gel system on Sarkhun rock is shown in Figure 3. In this process TEOS increases the density of the reaction sites of the hydroxyl groups with the surface and make the film more durable. This leads to greater resistance towards water and condensate formation.

Figure 4 shows FTIR spectra of the gel powders derived from coating solution, with 0.2 mass ratios of PFOS/TEOS. The

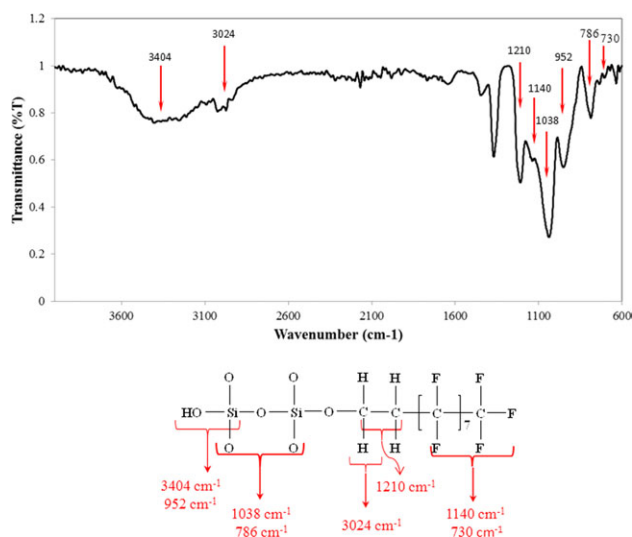


Figure 4. FTIR spectra on the gel powders derived from coating solution with schematic model of the formation of fluorosilane and its wavenumbers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.wileyonlinelibrary.com).]

absorption band at 786 cm^{-1} is related to Si—O—Si symmetric stretching and strong absorption peak at 1038 cm^{-1} is related to an asymmetric stretching. Other bands at 952 cm^{-1} , which may be related to Si—OH , and hydrogen bonded Si—OH at 3404 cm^{-1} are also observed. The bond around 3024 cm^{-1} is due to C—H band of ethoxy group ($\text{—OC}_2\text{H}_5$) and the peak at 1210 cm^{-1} is associated with C—C band. In the spectra of the coating film, there is a small peak related to C—F stretching of —CF_3 and —CF_2 around 1140 cm^{-1} and 730 cm^{-1} , which confirm the hydrophobic and oleophobic characteristics of the coating films. All these results indicate that sol-gel reactions were successfully accomplished.

SEM-EDX

The linkage created between limestone and products was studied both at surface and in depth by SEM-EDX analysis at room temperature. SEM images ($1000\times$) for treated and untreated top surface of samples are shown in Figure 5(a,b). Compared with the blank image, we can obviously see the surface uniform coverage with surfactant after modification. SEM images of the cross-section of the samples were also obtained at 1000 magnification for investigating the penetration depth of sol-gel (Figure 6). The images show coating onto the mineral, which reveal the presence of sol-gel bonding to hydroxyl group of the rocks. This indicates that the sol compound has good ability to

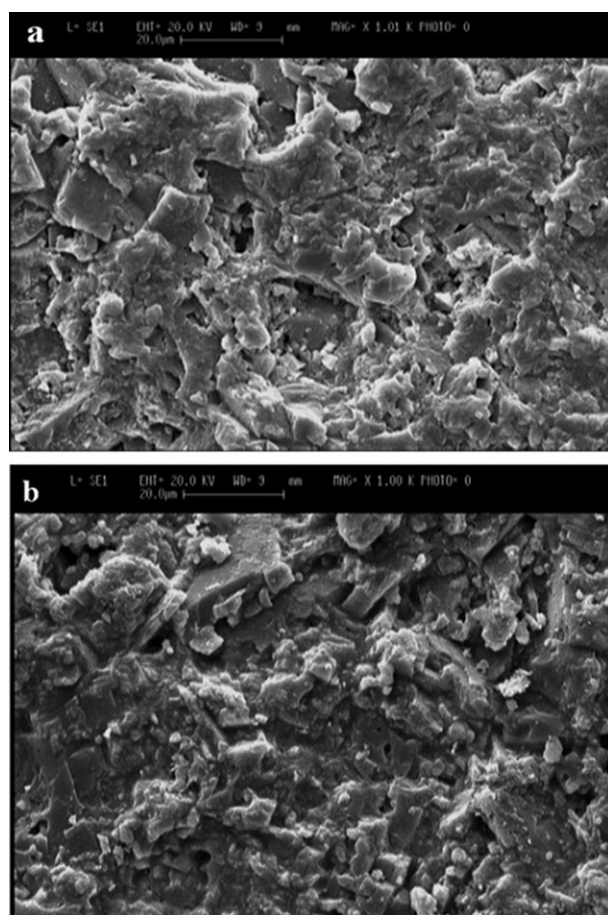


Figure 5. Top surface SEM images ($1000\times$) of limestone (a) before and (b) after treatment.

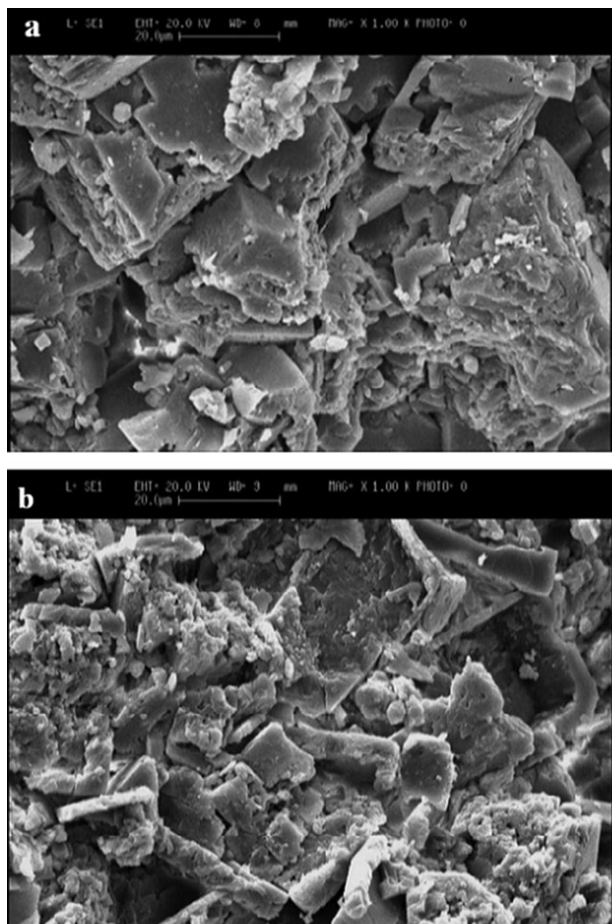


Figure 6. Cross section SEM images (1000 \times) of limestone (a) before and (b) after treatment.

Table II. Atomic Composition of Coating Films Measured by EDX

Atom	wt %
F	19.39
Si	3.11
Mg	2.66
Ca	0.86
C	29.28
O	44.71
Total	100

penetrate deeply in the porous media because of its small amount of alkoxy silane and low molecular weight before drying step.

EDX result illustrates the distribution of elements inside the treated rock. As shown in Figure 7 we performed the EDX spectra for various elements such as Si, F, Ca, O, Mg, and C. In this section we have also compared the mass composition measured by EDX (Table II) with the mass composition of the calculated from initial precursor values to demonstrate the successful coating on limestone. For this calculation, we assumed that PFAS and TEOS completely react and form $R_f-Si-O_3-Si-R_f$ and silicone oxide after polycondensation towards hydrolysis, respectively.¹⁵ Based on this assumption, it is found that on the one hand, the percentage of fluorine content in the solution B was ~ 9 wt % and it increased to 19.39 wt % (measured by EDX) after treating on limestone, and on the other hand the silicone content of initial mass composition decreased from 14 to 3 wt %. These values show that fluorine groups originating from perfluorodecylsilane are accumulated at top layer coating and are able to coat $-O-Si-O-$ groups, originating from TEOS. The

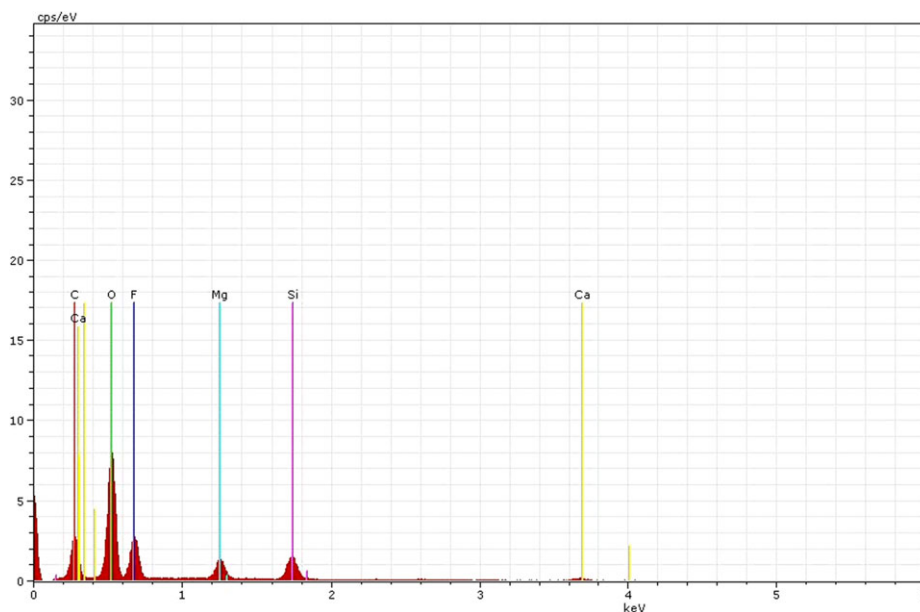


Figure 7. EDX analysis on the cross section of limestone after sol-gel treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

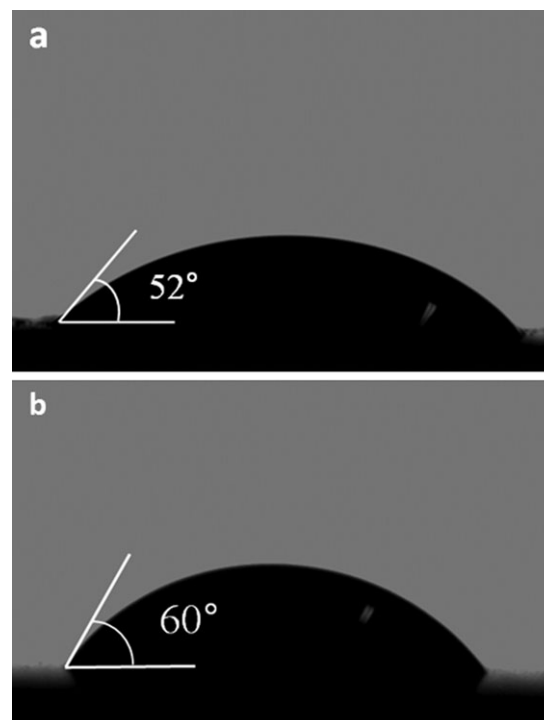
Table III. Contact Angle Values for Samples A and B Before and After the Treatment

	Brine	N-decane
Blank	30°	<5°
A	105°	52°
B	130°	60°

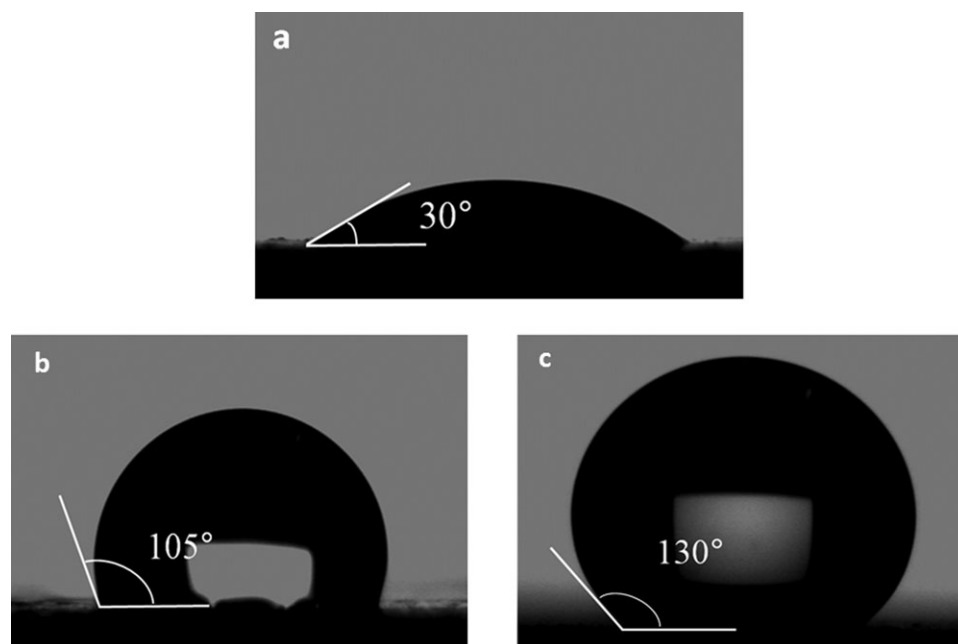
EDX spectra with the SEM micrographs for treated samples indicate that the sol-gel has covered on and inside the samples successfully. These results, which are indications of hydrophobic and oleophobic properties of limestone, are further testified by the two other tests of contact angle and imbibition measurements in the following sections.

Contact Angle

To demonstrate effective wettability alteration, we measured the contact angle for both water (2 wt % brine) and condensate (n-decane). These values are listed in Table III. Figure 8 shows contact angles of brine for samples A and B derived from PFDS and PFDS+TEOS, respectively. Fluorinated coating films with extremely large contact angles for both samples are compared to the blank in this figure. For sol-gel treatment the brine contact angle increases to about 130°, attributed to strong gas wetness, which shows higher hydrophobicity compared to sample A. The results of wettability alteration for condensate are depicted in Figure 9. Before the treatment because of high oleophilicity of limestone, n-decane droplet has penetrated into the core instantly as soon as it is applied onto the core surface; an indication of almost zero contact angle. As it is well known, when the wettability is altered to intermediate gas wet the condensate contact angle should be increased. In this study, we improved it

**Figure 9.** N-decane contact angle measurements after treatment for samples (a) A and (b) B.

from 0° to 52° and 60° for samples A and B, respectively. The results show that we can alter the wettability of rock from strong oil wet to intermediate oil wet. Higher contact angle for sol-gel modifier reflects that TEOS existence in coating provides greater adhesion and higher strength of fluorosilane onto limestone.

**Figure 8.** Water contact angle measurements before (a) and after treatment for samples (b) A and (c) B.

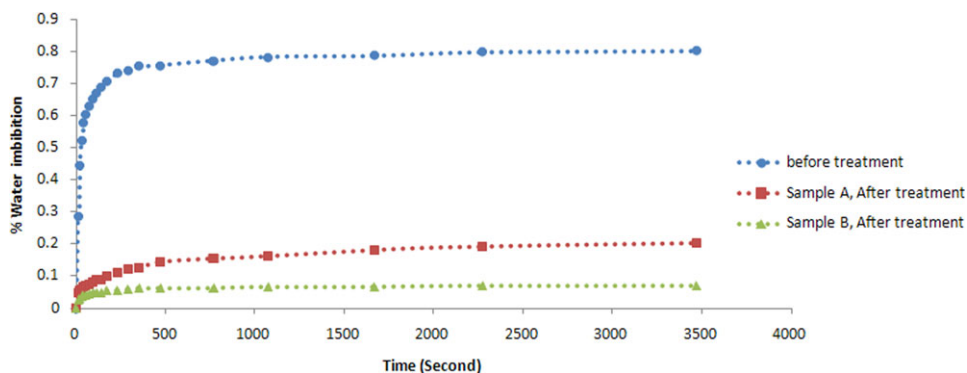


Figure 10. Brine imbibition on samples A and B before and after treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

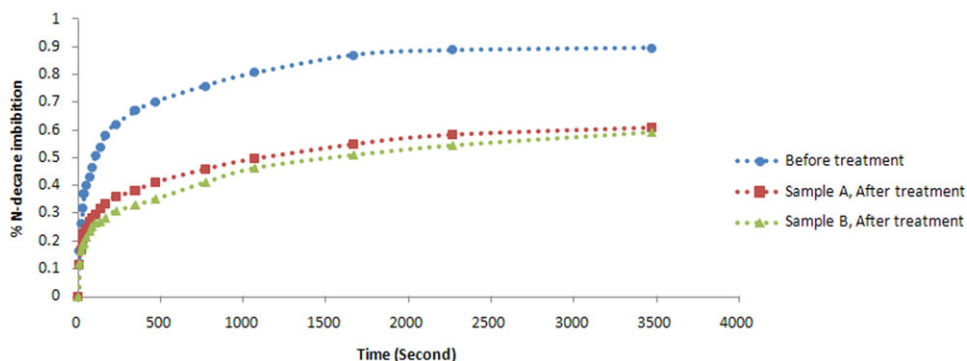


Figure 11. N-decane imbibition on sample A and B before and after treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Spontaneous Imbibitions

The main factor that affects gas production by spontaneous imbibition is wettability. In gas condensate reservoirs, the change in capillary force can control spontaneous imbibition. When the wettability of the rock is altered to gas-wetting, capillary force, and condensate build-up are reduced. This phenom-

enon leads to lower rate of imbibition and increasing in gas recovery. In this section, spontaneous imbibition tests before and after treatments are run at room temperature to ascertain that whether the sol-gel modification could alter the wettability of Sarkhun rock. The percentage of water imbibed is defined as follows⁶:

$$\% \text{ Water imbibition} = \frac{\text{Volume of water imbibed}}{\text{Pore volume of the core}} \times 100 \quad (5)$$

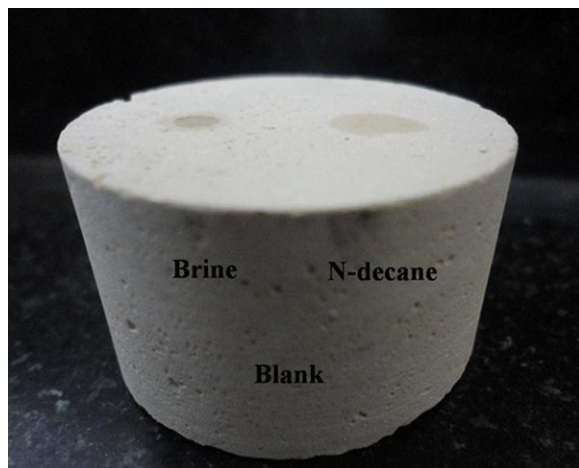


Figure 12. Brine and n-decane droplets on the surface of the air saturated before sol-gel treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

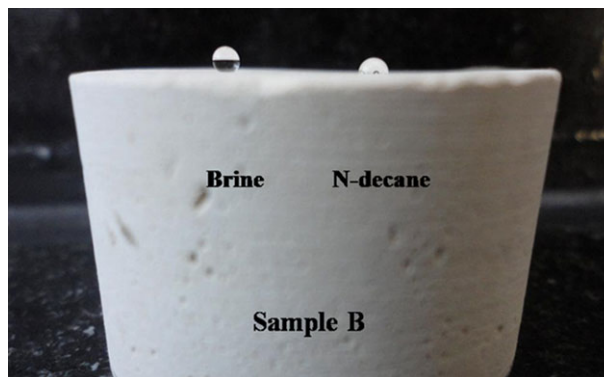


Figure 13. Brine and n-decane droplets on the surface of the air saturated after sol-gel treatment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

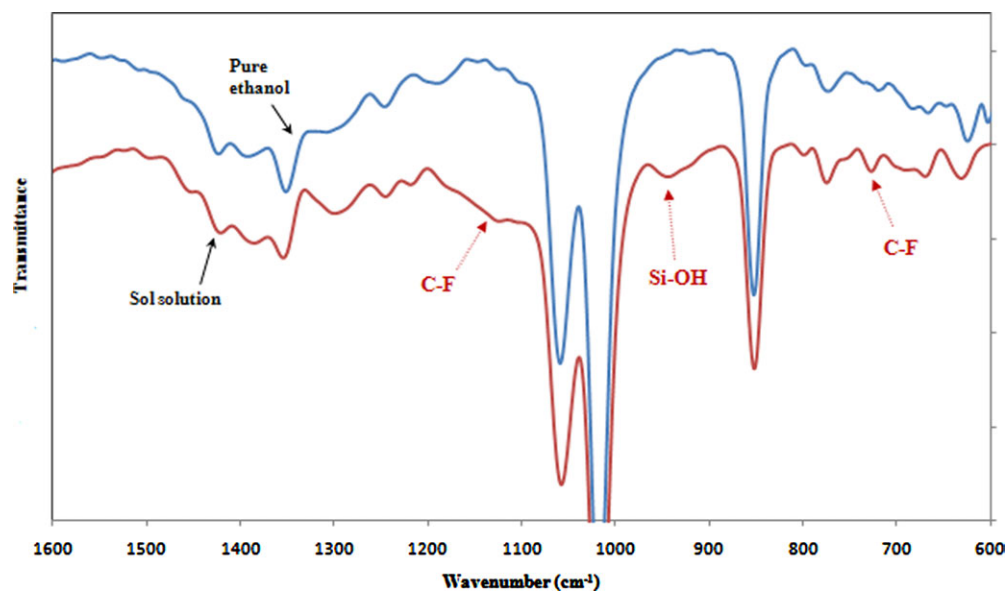


Figure 14. FTIR spectra on pure ethanol and the sol solution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

From Figure 10 it may be observed that the percentage of imbibition, without any chemical treatment, is about 0.8 and a high amount of brine can be imbibed in the first 15 min. After chemical treatment, this percentage is decreased to 0.2 and 0.07 for samples A and B after 60 min of imbibition time, respectively. This indicates that both samples have become hydrophobic after altering the wettability with chemical treatments. The results also show that sample B imbibes almost no brine and compared with sample A, the wettability improvement from

strong water wetness to gas wetness was more effective by the sol-gel process. We also performed spontaneous imbibition for n-decane-air-rock system before and after treatments. The results are shown in Figure 11, which demonstrates that after treatments n-decane imbibition was also reduced but there was no significant difference between n-decane imbibition in both samples. As we can observe in Figure 12, before any treatment the droplets imbibe immediately and wet the core while after modifications there is no brine imbibition occurring into the

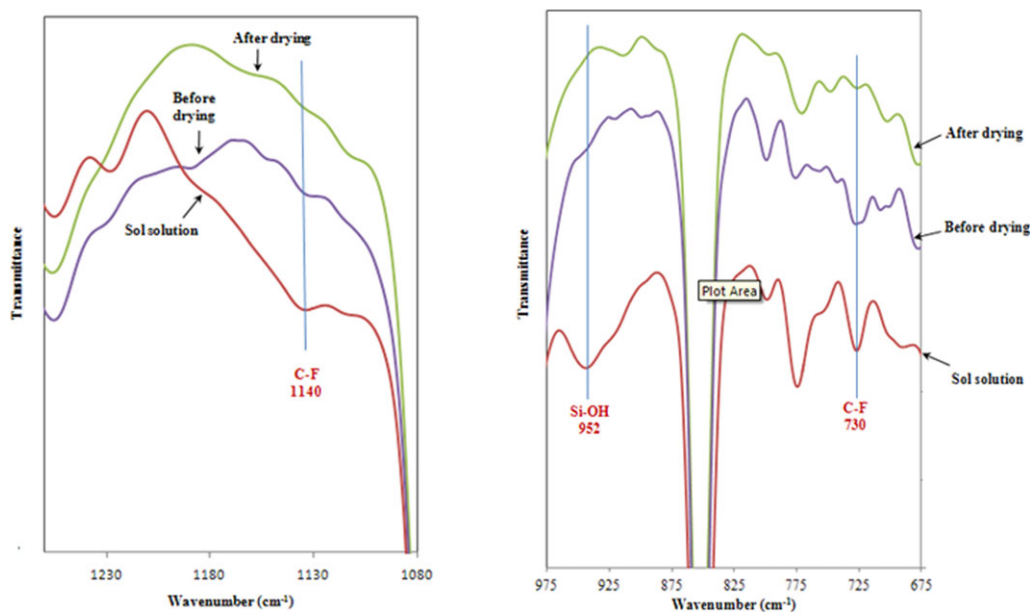


Figure 15. FTIR spectra on the sol solution and sol extractions before and after drying step. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table IV. The Ratio of A_A to A_B for the Absorbance Peaks

	Si—OH (952 cm^{-1})	C—F (730 cm^{-1})	C—F (1140 cm^{-1})
A_A/A_B	0	0.17	0.22

cores. Furthermore, Figure 13 provides a clear schematic of brine and condensate droplets on the surface of the air saturated samples treated with sol–gel process.

Adhesion of the Applied Sol–Gel Coating onto the Limestone Rock

In this section we analyzed the results of FTIR spectra for pure ethanol, sol solution, and extracted liquids from treated samples before and after drying step to demonstrate covalent bond formation of the sol–gel coating with limestone. For this investigation we had to focus on additional peaks in ethanol which were absorbed in the presence of sol extraction. The FTIR spectra of pure ethanol and sol solution are presented in Figure 14. As would be expected, additional peaks in some regions concerning the vibration bonds of C—F ($\sim 1140 \text{ cm}^{-1}$, 730 cm^{-1}) as well as Si—OH (952 cm^{-1}) are emerged, for which we considered the above peaks might be related to sol extraction into ethanol. It should be noted that it was difficult to perform spectroscopic investigation on vibration bonds in $1000\text{--}1100 \text{ cm}^{-1}$, corresponding to the Si—O—Si, due to overlapping with C—O region in ethanol.

Figure 15 depicts the infrared spectra of sol solution and sol extraction before and after drying step. We have also compared the change in C—F and Si—OH vibration peaks of the samples by integration of the area under their absorbance peaks in Table IV. In this analysis, A_B and A_A were represented as integrated values of the peaks related to ethanol sample which was subjected to the treated rock before and after drying step of sol gel process, respectively. The ratio of A_A to A_B for the same peaks (two peaks for C—F and one peak for Si—OH) which are given in Table IV shows that after completion of sol gel coating process, the amount of sol compound extracted from the rock was very low and negligible. This may suggest that by evaporation of the solvent, sol compound chemically bonds to the surface through Si—OH groups. In other words, nearly all Si—OH groups in the product of the eq. (4) are bonded with free —OH groups on the rock surface and the fluorinated siloxane network is attached onto mineral grains.

CONCLUSIONS

Water and condensate banking are causes of gas recovery reductions in gas condensate reservoirs. Wettability alteration to gas wetness is an appropriate method for reducing the capillary pressure due to liquid blockage and increasing the gas permeability. A novel chemical treatment was introduced in this research to change the wettability of Sarkhun rocks from liquid wetness to gas wetness. In our study, PFDS and TEOS were

used to develop polymeric fluorinated siloxane by a sol–gel method. First, we performed the structural characterization of sol–gel powders by using FTIR analysis. The sol–gel system was then applied to the rock samples. To characterize coating film inside the samples we performed SEM and EDX tests at room temperature. Contact angle measurements and imbibition tests were also employed in our experimental tests. On the basis of contact angle results, it can be concluded that both polymeric and monomeric surfactants can increase the hydrophobic/oleophobic characteristics. Treatment with sol–gel has provided more changes in wettability alteration compared with the other treatment. The same as brine contact angles, brine imbibition results revealed that this method of treatment leads to approximately zero imbibition. N-Decane imbibition is also reduced but there is no significant difference between condensate imbibition for both samples. We suggest this sol–gel improvement is due to greater adhesion of solution and more durable siloxane bands onto the rock minerals. As a conclusion, in this project we could protect the limestone rocks of Sarkhun reservoir from water and condensate damage by using PFDS and TEOS. We have also planned further study on this research at reservoir conditions in the next research.

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