Fundamentals of reservoir surface energy as related to surface properties, wettability, capillary action, and oil recovery from fractured reservoirs by spontaneous imbibition

DE-FC26-03NT15408

Quarterly Report
10/1/2003 – 12/30/03

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February 2004
Re-Submitted May 2004

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PUBLIC ABSTRACT

The objective of this project is to increase oil recovery from fractured reservoirs through improved fundamental understanding of the process of spontaneous imbibition by which oil is displaced from the rock matrix into the fractures. Spontaneous imbibition is fundamentally dependent on the reservoir surface free energy but this has never been investigated for rocks. In this project, the surface free energy of rocks will be determined by using liquids that can be solidified within the rock pore space at selected saturations. Thin sections of the rock then provide a two-dimensional view of the rock minerals and the occupant phases. Saturations and oil/rock, water/rock, and oil/water surface areas will be determined by advanced petrographic analysis and the surface free energy which drives spontaneous imbibition will be determined as a function of increase in wetting phase saturation. The inherent loss in surface free energy resulting from capillary instabilities at the microscopic (pore level) scale will be distinguished from the decrease in surface free energy that drives spontaneous imbibition.

A mathematical network/numerical model will be developed and tested against experimental results of recovery versus time over broad variation of key factors such as rock properties, fluid phase viscosities, sample size, shape and boundary conditions. Two fundamentally important, but not previously considered, parameters of spontaneous imbibition, the capillary pressure acting to oppose production of oil at the outflow face and the pressure in the nonwetting phase at the no-flow boundary versus time, will also be measured and modeled. Simulation and network models will also be tested against special case solutions provided by analytic models.

In the second stage of the project, application of the fundamental concepts developed in the first stage of the project will be demonstrated. The fundamental ideas, measurements, and analytic/numerical modeling will be applied to mixed-wet rocks. Imbibition measurements will include novel sensitive pressure measurements designed to elucidate the basic mechanisms that determine induction time and drive the very slow rate of spontaneous imbibition commonly observed for mixed-wet rocks. In further demonstration of concepts, three approaches to improved oil recovery from fractured reservoirs will be tested; use of surfactants to promote imbibition in oil wet rocks by wettability alteration: manipulation of injection brine composition: reduction of the capillary back pressure which opposes production of oil at the fracture face.

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INTRODUCTION

Objectives

The long-range objective of this project is to improve oil recovery from fractured reservoirs through improved fundamental understanding of the process of spontaneous imbibition by which oil is displaced from the rock matrix into the fractures. Spontaneous imbibition is fundamentally dependent on the surface energy. An initial objective is to determine the surface energy and relate the dissipation of surface energy to the mechanism of spontaneous imbibition. A parallel objective is to model the mechanism of spontaneous imbibition by a combination of network analysis and numerical modeling. Also fundamentally important, but not previously considered, parameters of spontaneous imbibition, the capillary pressure acting to oppose production of oil at the outflow face and the pressure in the nonwetting phase at the no-flow boundary (in effect within oil in the non-invaded zone of the rock matrix) versus time, will also be measured and compared with values predicted by the mathematical model. The next objective is to measure surface energy and related spontaneous imbibition phenomena for mixed-wettability rocks prepared by adsorption from crude oil. The dissipation of surface free energy must then be related to oil production at mixed-wet conditions. The final objective is to apply the results of the project to improved oil recovery from fractured reservoirs in three ways: reduction of the capillary force that opposes oil production at the fracture face; change in wettability towards increased water wetness; identification of conditions where choice of invading brine composition can give improved recovery.

TASKS

Budget period 1, July 1, 2003 through June 30, 2005 – Ideas and Concept development: Fundamentals of Spontaneous Imbibition

Task 1. *Work of displacement and surface free energy.* Obtain complementary sets of capillary pressure drainage and imbibition data and data on changes in rock/brine, rock/oil, and oil/brine interfacial areas with change in saturation for drainage and imbibition for at least two rock types (sandstone and carbonate). Determine free-energy/work-of-displacement efficiency parameters for drainage and imbibition for at least two rock types so that changes in rock/wetting phase/nonwetting phase surface areas can be closely estimated from capillary pressure measurements.

Task 2. *Imbibition in simple laboratory and mathematical network models.* Study imbibition in at least three simple tube networks that can be modeled analytically to establish and/or confirm fundamental aspects of the pore scale mechanism of dynamic spontaneous imbibition with special emphasis on determining how spontaneous imbibition is initiated and the key factors in how the saturation profile develops with time. Incorporate rules developed from laboratory measurements on relatively simple networks into the design of a computational network model. Use the network model to obtain an account of the mechanism by which imbibition is initiated, the saturation profile is developed, and the rate of spontaneous imbibition in terms of the dissipation of surface free energy that accompanies change in saturation.

Task 3. *Novel observations on fluid pressures during imbibition and the mechanism of non-wetting phase production at the imbibition face.* Make novel observations on the imbibition mechanism including details of the mechanism of oil production at the outflow rock face and the change in the nonwetting phase pressure at the no-flow boundary of the core during the course of spontaneous imbibition for at least 16 distinct combinations of rock/ fluid properties.

Task 4. *Network/numerical model and new imbibition data.* Develop a numerical simulator specifically designed for spontaneous imbibition. Incorporate the network model to obtain a network/numerical model that includes matching the measured pressure in the nonwetting phase at the no-flow boundary, and the pressure that opposes production of oil at the open rock face. Imbibition data will be obtained for at least 10 rocks with over six-fold variation in permeability, and at least 6 orders of magnitude variation in viscosity ratio, and at least 10 variations in sample size, shape, and boundary conditions.

Task 5. *Comparison with similarity solutions.* Compare results given by simulation with special case analytic results given by similarity solutions for spontaneous imbibition for at least five distinct cases of rock and fluid properties.

Budget Period 2, July 1, 2005 through June 30, 2008 - Demonstration of concept: Application to mixed wettability rocks and improved oil recovery from fractures reservoirs.

Task 6. *Rock preparation and Work of displacement and surface areas*
Obtain a range of rock types and identify and obtain crude oils that induce stable mixed wettability. Prepare at least 25 rocks with mixed wettability through crude oil/brine/rock interactions.
Determine work of displacement for drainage and imbibition and measure the variation in rock/brine, rock oil, and oil/brine interfacial areas during the course of drainage and imbibition for at least two examples of mixed wettability.

Task 7. *Novel imbibition measurements on mixed-wet rock and network models.* Obtain, for at least six mixed-wet rocks, spontaneous imbibition data that includes measurements of the nonwetting phase pressure at the no-flow boundary, observations on the capillary pressure that resists production at the open rock face.

Task 8. *Application of network/numerical model to mixed wet rocks.* Use network models to relate dissipation of surface energy to rate of spontaneous imbibition and to account for the frequently observed induction time prior to onset of spontaneous imbibition into mixed wettability rocks.

Task 9. *Increased oil recovery by spontaneous imbibition.* The mechanism of increased recovery from mixed wet rocks by use of surfactants that promote spontaneous imbibition by favorable wettability alteration will be investigated for at least four distinct examples of crude oil/brine/rock/surfactant combinations.
The mechanism of increased recovery by manipulation of brine composition will be investigated for at least four crude oil/brine/rock combinations.

Addition of very low concentrations surfactants to the imbibing aqueous phase will be explored as a means of increasing the rate of oil recovery by reducing the capillary forces which resist production of oil at the fracture face. At least twelve combinations of rock and fluid properties including both very strongly wetted and mixed wet rocks will be tested.

EXECUTIVE SUMMARY

Good progress has been made during the second quarter of this project. Experiments designed to measure saturations and surface areas are reported. Experiments on simple tube networks will be extended to consolidated bead packs. A novel method of measuring the dead-end non-wetting phase saturation pressure has been applied to recovery of oil by imbibition of water and air by imbibition of oil. Surface tensions and interfacial tensions of glycerol/brine mixtures are reported. An analytic solution for spontaneous countercurrent imbibition is being developed.

PROGRESS BY TASK - BUDGET PERIOD 1

Task 1. *Work of displacement and surface free energy.*

The aim of this research is to understand the mechanism of the spontaneous imbibition, a process which can be of key importance in oil production from fractured reservoirs. During the process of spontaneous imbibition, the surface free energy of the system is converted to work of displacement, for example in the recovery of oil. The method proposed in this work utilizes thin section analysis from different core samples to determine fluid saturation and surface areas after solidification of epoxy resin. This information relates change in surface free energy to saturation.

Introduction

In order to determine the change in surface free energy a dyed liquid epoxy resin was used as the wetting phase. After establishing a specific saturation the resin is solidified. A second epoxy was then injected at high pressure and solidified before prepration of the thin section. After both the epoxies have been set in place thin sections (28 microns thick) from the samples are made. Then laying a grid over the thin section a point count is done to determine the relative amount of area corresponding to oil-water and oil solid interfaces. The change in surface free energy of the system is given by:

$$\Delta F = \sigma_{ow}(\Delta A_{ow} + \Delta A_{so} \cos\theta)$$

Where,

ΔF is change in the surface free energy

σ_{ow} is the oil – water interfacial tension (measured by drop volume method)

ΔA_{ow} is the oil – water interfacial area

ΔA_{so} is the oil – solid interfacial area

Experimental

Imaging of fluid distribution

As mentioned in the previous quarterly, the change in surface free energy is related to the fluid saturation in the core. To study the fluid distribution of the two phases, to simulate the wetting phase, a colored epoxy resin was used. Araldite 502 (modified Bisphenol A epoxy CAS # 84-74-2) with DDSA (Dodenyl Succinic Anhydraide CAS # 26544-38-7) as the hardener and BDMA (Benzyldimethylamine CAS # 103-83-3) as curing agent was selected for this study. A petrographic microscope, Olympus® (Model BX 51), has been purchased for the petrographic imaging of the thin sections.

Sample Preparation

1. A piece of core after being cut and dried, is then vacuum saturated with an epoxy of a specific color (blue)
2. Saturation is ensured by application of 1000 psi pressure (hydraulic) to make sure that all accessible pore space (macropores & micropores) are filled.
3. The sample is then centrifuged to some intermediate saturation of the added wetting phase
4. The sample is then left at 60^0 C for 36 – 48 hrs to set
5. After cutting off the top and bottom of the core a second epoxy (with a red dye) was introduced under vacuum. The core was then pressurized under liquid epoxy at 1000 psi to ensure filling of the remaining pore space. The second step gives the sample good integrity for the preparation of thin section and identifies the location occupied by the non-wetting phase.
6. The sample is left at 60^0 C for 36 – 48 hrs to set. It is then sent to a thin section preparation laboratory.

The image of the thin section thus obtained can be used to obtain saturation by point counting. Line intersections are counted to obtain areas of interfaces that represent oil – water (wetting – non wetting), oil – solid (non wetting – solid) and water – oil (wetting – solid).

UV fluorescence

After making the thin section, a fluorescent dye of different color can be applied to the thin section for a very short time (2-5 sec) and then wiped clean. This technique reveals very small fractures and small intergranular pores of less than about 1 micron in diameter. The small fractures and spaces at grain boundaries may form the only path of connectivity in case of low permeability rocks.

Task 2. *Imbibition in simple laboratory and mathematical network models.*

Introduction

Task 2 concerns the study imbibition in at least three simple tube networks that can be modeled analytically to establish and/or confirm fundamental aspects of the pore scale mechanism of dynamic spontaneous imbibition with special emphasis on determining how spontaneous imbibition is initiated and the key factors in how the saturation profile develops with time. Rules developed from laboratory measurements on relatively simple networks are to be incorporated into the design of a computational network model. The network model will be used to build an account of the mechanism by which: imbibition is initiated; the saturation profile is developed; and the rate at which imbibition occurs in terms of the dissipation of surface free energy that accompanies change in saturation.

Experimental

Simple tube networks

Simple tube networks have been run with a radial network model of different sized tubes. Imbibition was observed after submerging the model in brine. However difficulty was experienced in following the simultaneous behavior of the interfaces in the tubes. In order to identify clearly the location of the moving interfaces, a dye was added to the aqueous phase. All the dyes tested to date have changed, to some degree, the wetting of the capillary tube. So to date the methods of making the interface visible for perfectly water wet condition are still being investigated.

Preparation of consolidated cores from glass beads

We have moved a step further from the simple radial tube model to the ideal soil model, a random pack of equal size spheres. Mason (1968, 1971, 1983), an advisor on this project, and his coworkers (Mason and Clark, 1965, 1966; Mason and Yadav, 1983; Mason and Mellor, 1991; Mason and Morrow, 1984, 1986; Yadav and Mason, 1983) have made extensive investigations of immiscible displacements in sphere packings. Different beads sizes allows for the scaling of permeability for a constant value of porosity (~37 % $\pm$ 1 %). In the present work there is special need for preparation of consolidated cores that correspond closely in structure and properties to unconsolidated bead packs. The geometry of the pore structure is fixed in this case and the same core can be used to obtain results for different conditions. Consolidated packs have been prepared by sintering, with little loss in porosity.

Preparation of the cores

Sieving

Unconsolidated Potters Ballotini® impact beads are sieved using Tyler® sieve. Different mesh sizes were experimented with. The higher bead size (60 - 70 mesh) were not found suitable as the resulting permeability was so high that imbibition of the wetting phase was too fast to obtain accurate imbibition data. A lower bead size (200 - 325 mesh), although suitable for imbibition, caused expensive wear and tear on a special quartz core holder that was used in core preparation. Also the consolidated cores produced with this bead size were not always symmetrical. The reason for this asymmetry is still not well understood. Finally a bead size corresponding to 100 – 150 mesh was selected for initial core preparation.

Sintering

A core prepared from glass beads of 100 – 150 mesh was made by fusing the beads in a muffler furnace at a temperature of 540 ^{0}C. The following temperature cycle was employed to get the desired properties of the consolidated core:

Heating rate: 1.5 ^{0}C/min
Final temperature: 540 ^{0}C
Soaking time at 540 ^{0}C: 2 min
Cooling rate: 0.55 ^{0}C/min

These conditions were determined by a process of trial and error. The guiding criteria for the conditions were the porosity and the integrity of the consolidated core. A porosity of ~37 % (due to that of the original unconsolidated bead pack) and structural integrity (to withstand 300 psi confining pressure during permeability measurement) and minor impact when the core was shaken in the imbibiton cell to remove drops of oil before taking a reading. The properties of the consolidated cores prepared so far are shown in Table 2.1

Table 2.1

Glass Beads Size 100 - 150 mesh

	Permeability, Darcy	Porosity %	Pore Volume, ml
Core # 1*	4.4	37.9	21.05
Core # 2*	4.4	36.5	20.77
Core # 3*	4.1	35.9	19.88
Core # 4	3.6	35.9	21.87
Core # 5	3.9	37.3	23.65
Core # 6	3.4	38.0	21.05
Core # 7	3.6	36.9	21.99

* 2.25 inch size core holder was used as compared to 1.75 inch holder for the other cores

The core was cut using a 1.5 inch drill bit, with care to avoid damage of the surface of the core being drilled. The core was then dried at 110 ^{0}C. A high purity quartz holder is used as a mold. It was observed that the size of the holder had an effect on the permeability of the core obtained (Table 2.1). The quartz holder was cut into two pieces that are tied by stainless steel wire to hold the unconsolidated beads in place. At the end of the experiment the wire was cut and the core was removed from the holder. A ceramic tile was placed below the core holder during sintering in the furnace to prevent damage to the base of the core holder.

Task 3. *Novel observations on fluid pressures during imbibition and the mechanism of non-wetting phase production at the imbibition face.*

Introduction

Good progress has been made with respect to the goals of Task 3. The following abstract was submitted for presentation at the Seventh International Symposium on Wettability and Oil recovery. A full manuscript on this work is in preparation.

Experimental

Pressures acting in counter-current spontaneous imbibition

Four water/oil and four oil/air counter-current spontaneous imbibition experiments were performed on Berea sandstone cores with permeabilities ranging from 0.065 to 1,094 $\mu m^{2,}$ initially 100% saturated with non-wetting phase and with all faces but one sealed. Such displacements are linear and the experiments showed a clear frontal displacement mechanism. Capillary pressure, at its maximum at the imbibition front, was the driving force of the imbibition process. As well as viscous drag, there is an opposing capillary back pressure associated with production of non-wetting phase at the open face. The location of the imbibition front and the overall changes in core saturation were monitored during the imbibition experiments. The measured pressure in the dead space occupied by non-wetting phase ahead of the imbibition front was essentially constant after a short start-up period. The distance advanced by the imbibition front was proportional to the square root of time. The data obtained were modeled by numerical simulation to determine the saturation and capillary pressure at the imbibition front and the capillary back pressure at the open face. The ratio of the capillary back pressure to the capillary pressure at the imbibition front ranged from approximately 1/3, for oil displacing air, to approximately 1/9, for water displacing oil.

Task 4. *Network/numerical model and new imbibition data.*

Introduction

Good progress has been made in the necessary step of measuring the physical and interfacial properties of glycerol/water or brine solutions. Interfacial tensions against refined oils were very close to linear with concentration and ranged from about 29 to 51[dynes/cm]. Surface tensions were also linear with concentration and ranged from about 64 to 72.5[dynes/cm]. All measurements were made at ambient temperature.

Experimental

Surface Tension

The surface tension was measured by means of the dynamic Wilhelmy plate method (Krüss Processor Tensiometer K12) with a platinum plate as the solid. Before each use, the plate was thoroughly cleaned and subsequently fired in order to remove potential residual organic contaminants. Results of the surface tension over the entire glycerol concentration range from 0 to 100 [%] are displayed in Fig.4.1. The salinity of the brine was not compensated for addition of glycerol. The surface tension decreased continuously from 72.8 [dynes/cm] for pure seawater to 63.5 [dynes/cm] for glycerol. The surface tension for pure liquids is in very close agreement with published values. It affirms that the decrease in ionic strength with addition of glycerol had little effect on surface tension.

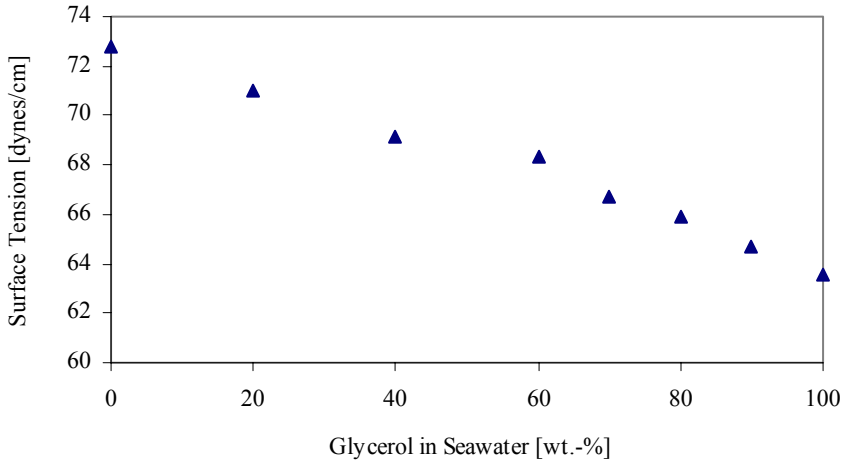

Fig. 4.1: Surface Tension as a function of glycerol concentration

Interfacial Tension

The interfacial tension between the aqueous phase and the oil phase was measured with the Krüss Drop volume Tensiometer DVT-10, whereby the oil phase was dispensed into the aqueous phase. Several observations of time between the subsequent detachments of oil drops were measured in order to obtain consistent results.

Two refined oils were used as the oil phase (Table 4.1), n-decane and mineral oil Soltrol 220. The aqueous phase is composed of seawater plus glycerol.

Table 4.1	Oil Properties	
	Soltrol 220	n-Decane
Density ρ [g/cm^3]	0.782	0.730
Viscosity μ [cp]	3.9	0.9

The values for the interfacial tension are similar for both oil phases (Fig.4.2). They decrease nearly linearly from around 51.9 [dynes/cm] for n-decane and 50.3[dynes/cm] for Soltrol 220 and pure seawater to around 29 [dynes/cm] for both oil phases and pure glycerol. In all cases the data accuracy and quality is high.

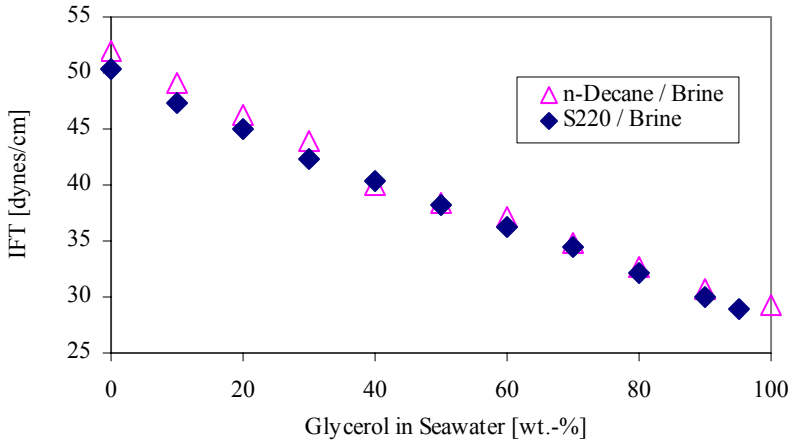

Fig.4.2: Interfacial tension for n-Decane and Soltrol 220 and mixtures of seawater with glycerol

Task 5. *Comparison with similarity solutions.*

Introduction

The objective of Task 5 is to compare results given by simulation with special case analytic results given by similarity solutions for spontaneous imbibition for at least five distinct cases of rock and fluid properties. Good progress has been made in the development of an analytical solution and comparison with numerical solutions.

Experimental

The displacement process in porous media is a notoriously non-linear and complex problem; therefore, it is no surprise that only a few cases have been found to have analytical solutions. In fact, only two such solutions exist in the literature. The Buckley-Leverett (1942) solution predicts the forced displacement of one fluid by another provided that capillary pressure is zero. This is a restrictive condition for most real displacement problems. The other solution was developed by Yortsos and Fokas (1983). Although this solution allows for the existence of capillary pressure, the shapes for the capillary pressure and relative permeability functions are fixed. As such, this solution is severely more restricted than the Buckley-Leverett solution and, again, this solution is not suitable for general displacement problems. Furthermore, it does not appear to include either the Buckley-Leverett or the present spontaneous imbibition solutions as limiting cases.

Li, Morrow and Ruth (2003) have shown that spontaneous displacement of a non-wetting fluid by a wetting fluid in a porous media initially 100% saturated with the non-wetting fluid results in a series of saturation profiles that are self-similar. Furthermore, there are several papers in the literature that show that the profiles scale with the square-root of time (see Morrow and Mason (2001) for a recent review). Self-similarity and squared-root of time scaling allow for an analytical solution of this problem until the penetrating wetting fluid contacts a no-flow boundary.
An analytical solution is provided for counter-current, spontaneous imbibition of a wetting phase (water) into a porous media is being developed. The solution is validated by comparison with numerical simulations.

CONCLUSIONS

1. An epoxy resin solidification technique has been developed which reveals the distribution of wetting phase and nonwetting phase saturations in rocks.

2. A technique has been developed for preparation of consolidated cores from unconsolidated glass beads of 100-150 mesh. The porosities of seven cores ranged from 35.9 to 38.0 %, which is typical of the porosity of the unconsolidated bead packs used in core preparation.

3. Spontaneous imbibition measurements can be made which include the amount of nonwetting phase recovered, the location of the imbibition front, and the dead end pressure in the nonwetting phase.

4. Surface tensions of glycerol/water mixtures against air and interfacial tensions against mineral oil are very close to linear.

5. An analytical solution has been developed for counter current spontaneous imbibition of a wetting phase into porous media.

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