

Wettability Effects on Bandera Gray Sandstone using Biosurfactants

Sunday Ukwungwu¹, Abubakar Abbas¹, Ghasem Nasr¹, Heather Allison², Sean Goodman²

¹Petroleum Technology Group, School of Computing Science and Engineering, University of Salford, Manchester, M5 4WT, United Kingdom.

²Department of Functional and Comparative Genomics, Institute of Integrative Biology, Biosciences Building, Crown Street, University of Liverpool, Liverpool L69 7ZB, United Kingdom.

Corresponding Author Email address: s.v.ukwungwu@edu.salford.ac.uk,

Phone no: +447918554877

ABSTRACT

Despite the growing demand for sustainable technologies that enhance residual oil recovery, the environmental and cost benefits of Microbial Enhanced Oil Recovery (MEOR) have favoured an increased research that is utilising a biosurfactant for oil recovery. In this study, three produced biosurfactants were investigated, and compared, which resulted in different impacts on the IFT and wettability. A secreted, extracellular biosurfactant produced by *BS 2* reduced the IFT from 56.95 to 4.49 mN/m, *BS 3* to 6.69 mN/m and *BS 1* to 10.94 mN/m. The spent culture medium changed the wettability of the grains to both water-wet and intermediate-wet state. The findings prove the suitability of the biosurfactants for an effective EOR and as a biosurfactant preference in sandstone reservoirs.

Keywords – Bacillus, Biosurfactant, Enhanced Oil Recovery, Sandstone, Wettability.

1. INTRODUCTION

Environmental impact, surfactant cost and oil price are the three main parameters that impact upon the robustness of the surfactant flooding in oil reservoirs. These have encouraged current research for better strategy in Enhanced Oil Recovery (EOR). Interfacial tension (IFT) reduction and wettability alteration of the reservoir rocks are the two-main mechanisms of oil recovery that are induced by the surfactant flooding of a reservoir. The potential of microbial secondary metabolites, in the form of biosurfactants, used to degrade heavy crude oil to reduce viscosity in depleted oil reservoirs, is considered to be very effective in MEOR. Biotechnology

has long played a significant role as a tool to increase stagnant petroleum production, going back to the earlier studies of MEOR (Kuznetsov, 1950, Davis and Updegraff, 1954, Updegraff and Wren, 1954), which were based on three broad areas: injection, dispersion, and propagation of microorganisms in petroleum reservoirs; selective degradation of oil components to improve flow characteristics; and production of metabolites by microorganisms and their effects (Shibulal et al., 2014).

Emphasis has been given to the ecological impacts and effects (Dusseault, 2001, Venhuis and Mehrvar, 2004, Ying, 2006, Ivanković and Hrenović, 2010) caused by chemical surfactants, due to their toxicity and recalcitrance to degradation in the environment. Increasing ecological concerns, developments in biotechnology and the rise of stringent laws have prompted the search for biosurfactants that are naturally degraded in the environment by other microorganisms, and can be used to replace the synthetic surfactants that are available in the market. For as long as we want to keep producing oil, we have to adopt an environmentally friendly technique of enhancing effective oil recovery.

Specifically, this work focused on isolating a pure culture, and investigating the strain of bacteria that can be effectively used to produce a biosurfactant, through the addition of a LB broth and culturing on a LB agar plate, for the purposes of enhancing oil recovery. Further investigation was conducted on the surface reaction of these biosurfactants in reducing the interfacial tension of heavy oil, and also in altering wettability. The positive outcomes of these biosurfactants can improve the hydrocarbon mobilisation of trapped oil in the reservoir, which may in turn influence the well productivity, and hence the ultimate oil recovery from reservoirs. This research utilises a thermophilic spore-forming bacteria, growing on sucrose to produce natural biosurfactants. Changing the wettability of the rock, and the way the well is brought into a flowing condition, can affect the pore spaces and subsequently prolong the life of mature fields.

2. MATERIALS AND METHODS

2.1.1 Crude oil and brine

Crude oil, which is a heavy crude from the Niger Delta oil field, with a density of 0.9322 g/cm³ and a viscosity of 134.828 (cSt) at 25 °C, was used in all the experiments. The composition of the synthetic brine that was used was 4 % (w/v) sodium chloride in deionised water. The synthetic brine used in this study was reconstructed by Exova, which was based on a specific formation water analysis from the Niger Delta oil field.

2.1.2 Microbes

The bacteria strains, that were used in this study were *B. subtilis* (DSM 3256), *B. licheniformis* (DSM 1913), and *Paenibacillus polymyxa* (DSM 740), and these were obtained from DSMZ, Deutsche Sammlung von Mikroorganismen und Zellkulturen.

2.1.3 Core Samples

Bandera Gray Sandstone (porosity: 21 %, and permeability 20 – 40 mD) was used for the qualitative wettability tests. The core plug was 7.62 cm in length and 2.52 cm in diameter and was obtained from Kocurek Industries.

2.1.4 Culture/Preparation of the biosurfactants

As all oil reservoirs are essentially devoid of oxygen, anaerobic bacteria are generally chosen for MEOR field applications. Members of the genus *Bacillus* are aerobes or facultative anaerobes and can grow on nutrient agar. Biosurfactants were produced by culturing bacteria in a minimal salt medium: Sucrose (30 g L⁻¹), KNO₃ (5 g L⁻¹), KH₂PO₄·2H₂O (1 g L⁻¹), K₂HPO₄·2H₂O (1 g L⁻¹), NaCl (3 g L⁻¹), MgSO₄·7H₂O (0.2 g L⁻¹), CaCl₂·H₂O (0.2 g L⁻¹) made up to 1 L with brine. The dry constituents were stirred for 30 minutes to mix thoroughly, and the pH was adjusted to 7.0. The medium was filter sterilised using a filter with a pore diameter of 0.22 µm, and dispensed into sterile containers at a volume of 13 mL using an electronic stripette. LB agar was prepared in a 1 litre bottle and stirred for 30 minutes. Freeze dried strains from DSMZ were carefully removed from the glass tubes, and 1000 µL of LB broth was added to each ampule, and the resuscitated strains were cultured on individual LB agar plates at temperatures of 30 °C and 37 °C for 48 hours.

The protocol for a biosurfactant precipitation is thus: a 400 ml of prepared LB broth was inoculated at Temperature, 0 to 0.1 Abs at 600 nm with the selected bacterial culture. After a 24-hour growth, the cell-free supernatant from the culture broth was obtained by centrifugation at 10,000 x g for 20 min at 4 °C. The remaining cells were removed by filtration using a 0.2 µM filter. The cell free supernatant underwent acid precipitation by adjusting the pH 2 using a 6 M HCl and placed at 4 °C overnight. The precipitate was separated by centrifugation at 10,000 x g for 25 min at 4 °C, and extracted two times with methanol and solvent, which was evaporated using a rotary evaporator at 50 °C (Pathak et al., 2012). Finally, the crude biosurfactant was collected and weighed for total surfactant extraction concentration. The produced biosurfactants, used in these investigations, are represented below with the following abbreviations as follows:

BS 1: *Bacillus subtilis*, **BS 2:** *Bacillus licheniformis*, **BS 3:** *Paneabacillus Polymyxa*

2.1.5 Interfacial tension/contact angle measurements

Measurements were made of the interfacial tension (IFT) between the crude oil-synthetic brine-biosurfactant system covering the ranges of $26\text{ °C} \leq T \leq 75\text{ °C}$ and pressure, $0.15\text{ MPa} \leq P \leq 13.89\text{ MPa}$. The assay used here, for the qualitative screening of the produced biosurfactants, was to check for a possible reduction in IFT. A positive result could be an indication that the produced biosurfactant can possibly be utilised in enhancing oil recovery. Experiments were conducted in real time and lasted between 5.5 hours – 6 hours. The IFT measurements were conducted using the TEMCO pendant drop method, combined with the solution of the Laplace equation for capillarity, for the profile of the oil drop in the oil-brine equilibrium environment, which is located within the Department of Petroleum Engineering at The University of Salford. The experimental set-up is shown in Fig.1 and a total of two hundred and eighty-five (285) IFT measurements were performed.

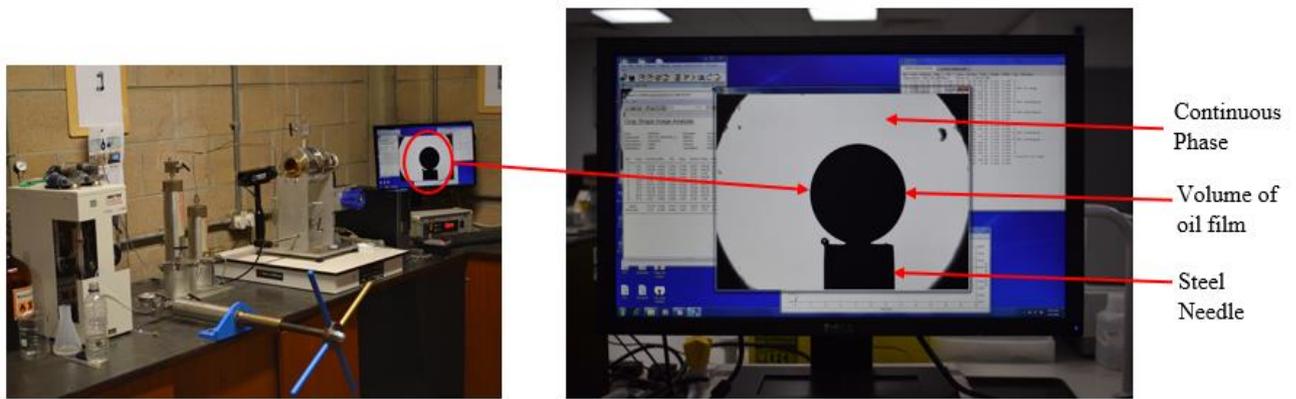


Figure 1: Experimental set-up of the Pendant Drop shape method

It should be noted, that for every set of these experiments, the continuous phase comprised of a serial dilution of cells/cell-free biosurfactants mixed with the synthetic brine and the heavy oil was the drop phase. Accurate density measurements were essential to the pendant drop method, since the IFT calculations depend on the density difference between the two fluids. The volumes of the produced biosurfactants were kept constant at 200 ml, for both the cultures of cells and cell-free supernatant. The biosurfactants were diluted serially with the synthetic brine to a ratio of 1:1, thus the addition of 200 ml of synthetic brine was used, with the aid of sterilised filters, to prevent contamination of the solution, thus making the total solution 400 ml. For gaining the optimum measurements, the calibration of the capillary tube was necessary before the beginning of any experiment. The diameter of the needle was 3.03 mm and measured with the aid of a digital Vernier calliper.

2.1.6 Wettability Tests

Two qualitative tests were performed: a floating test adapted from (Wu et al., 2006) and a two-phase separation test (Somasundaran and Zhang, 2006).

2.1.6.1 Core Sample Preparation for crude oil treatment

The core sample initially was cleaned with toluene in a soxhlet extraction device to remove all contaminants leaving it strongly water-wet. The cleaning process was carried out in a fume cupboard in a continuous process until there was no more discoloration of the solvent with

contact time. The sample was then placed in an oven and dried to a constant weight. To evaluate the effectiveness of the biosurfactants, in meditating wettability, the sample was crushed and meshed with a 225 μm sieve, to check the size of the distribution and remove particles. The crushed sample was submerged in crude oil in the OFTIE aging cell (Fig.2), and stored in an OFITE 5 roller oven at an aging temperature of 62.2 $^{\circ}\text{C}$ for two weeks. At the end of the aging period, the oil that was aged in the core was displaced with kerosene (pure grade paraffin), and the grains were dried again in the oven for a day, and were then ready for the biosurfactant treatment.



Figure 2: Aging of Bandera Gray (grains) in crude oil

2.1.6.2 Core Sample Preparation for biosurfactant treatments

2 g of the crushed rock sample, (Bandera Gray) sandstone, was added into six (6) bottles containing biosurfactant solutions with cells and supernatant cultures (Fig.3), and allowed to saturate for a day in a refrigerator. The biosurfactant was removed by the successive addition of distilled water to the bottles, and grains were retrieved by filtration with the aid of unbleached filter papers, and placed onto petri dishes. The sample was dried again for a day in an oven at 32 $^{\circ}\text{C}$ and was then ready for the qualitative wettability tests. The core sample was then treated separately with the produced biosurfactants and left to saturate. Aging allows the grain sample to imbibe the crude oil under high temperature. Saturation of the biosurfactants and aging indirectly alters the degree of wettability of the grains.

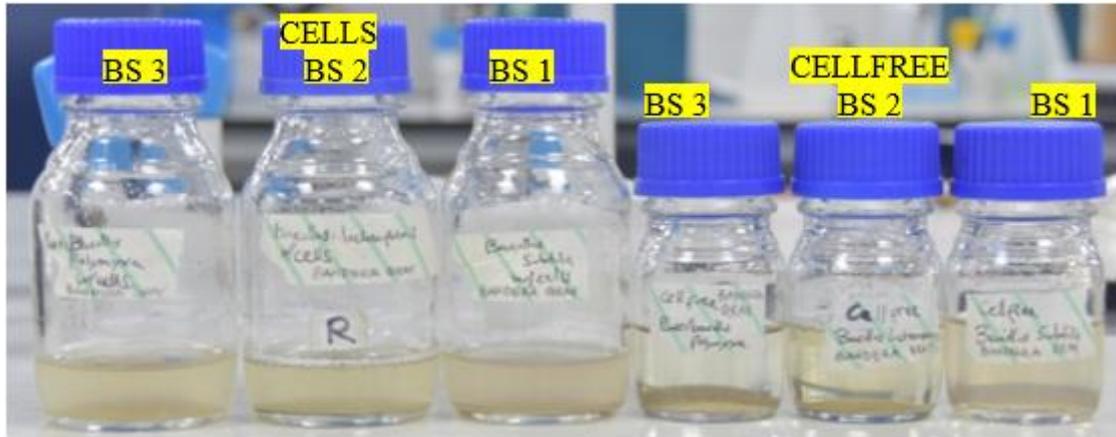


Figure 3: Saturation of Bandera Gray sandstone in *BS 1* *BS 2* and *BS 3*

3. RESULTS AND DISCUSSION

This section will illustrate the isolation of pure cultures and bacteria growth, the effect of the different produced biosurfactants solutions on the interfacial tension properties, and wettability alteration of the Bandera Gray sandstone. These results will confirm the effectiveness of the cultured biosurfactants for microbial enhanced oil recovery.

3.1 Growth and Culture of bacteria.

The strains used were grown on petri dishes for 48 hours at 30°C and 37°C. It was observed that there was faster growth at 37°C in the first 24 hours for all three strains, and more visible growth was seen at both temperatures after 48 hours. However, the growth of type strain DSM 740 was minimal, even after 2 days, and the weak observation of growth was taken as a positive result as shown in Fig. 4 and Fig. 5.



Figure 4: Bacteria growth after 48 hours, stores at 37 °C, (A) DSM 3256 (B) DSM 1931(C) DSM 740



Figure 5: Bacteria growth after 48 hours, stores at 30°C. (A) DSM 3256 (B) DSM 1931(C) DSM 740

In obtaining the required volume needed of the biosurfactant (200 ml), the light scattering technique was used to obtain a fixed amount of the dilute solution from a stock solution, since it cannot measure the cell number nor the colony forming unit (CFU). The dilution equation is given as:

$$C_1 \times V_1 = C_2 \times V_2 \quad (1)$$

3.2 Effect of the Biosurfactants on Interfacial Tension Measurements

The measurement of interfacial tension is an integral part of this investigation, as the application of biosurfactants can aggregate at the interfaces between fluids having different polarities, such as water and oil, leading to the reduction in interfacial tension (Fang et al., 2007, El-Sheshtawy et al., 2015). One of the major considerations, with enhanced oil recovery, is how much the injected bacteria can stimulate the production of additional oil from reservoirs. Usually this is achieved by the addition of nutrients during water injection. The reduction in interfacial tension is possible by stimulating the growth of bacteria at an oil/water interface, which in turn can aid the recovery of oil. From the experimental observations, the graph of IFT against temperature shows that the different biosurfactants used induced reduction in interfacial tension. This could have resulted due to the fact that bacteria growth needs both nutrients from the brine and carbon from the oil, therefore the bacteria is probing through the oil/water

interface to gain access to the carbon (Kowalewski et al., 2004). The effects of temperature and pressure on interfacial activity helps to show the behaviour of liquids during interfacial tension measurements. And with the help of the density difference between liquids, interfacial tension is easily calculated by the pendant drop method.

Previously, (Lin et al., 1995) concluded, after experimenting with air/water and fluid/fluid interfaces, that the measured IFT is drop dependent. In this study, our results demonstrate that there is a reasonable and clear impact of the drop size on the measurement of the IFT. The bigger the drop the more reasonable it is in estimating the IFT by the pendant drop technique, because with this there is higher accuracy in the image edge detection process. This perception concurs with (Yang et al., 2007) who likewise contemplated the impact of drop size on the IFT and adsorption of surfactants, and it demonstrate that the high accuracy, with which the drop coordinates, must be resolved and not just rely upon the numerical treatment of the data, but also on the experimental data itself.

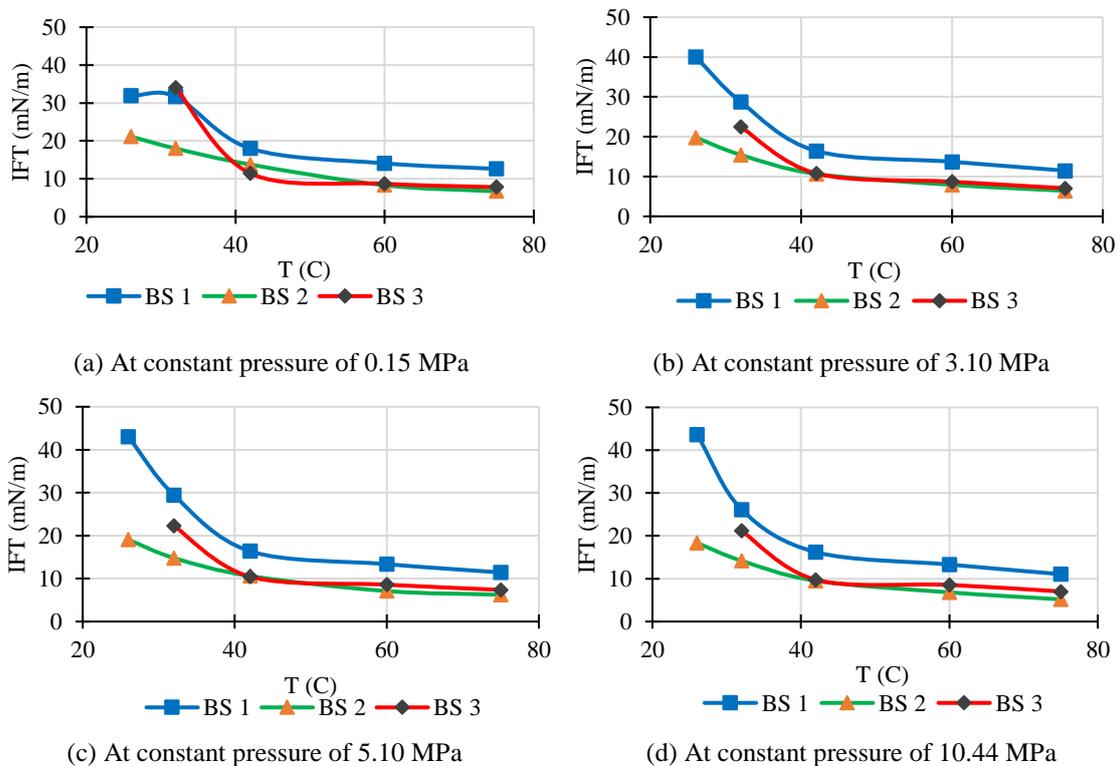
3.2.1 Comparison of the effect of IFT with temperature for supernatant *BS 1*, *BS 2* and *BS 3* biosurfactants

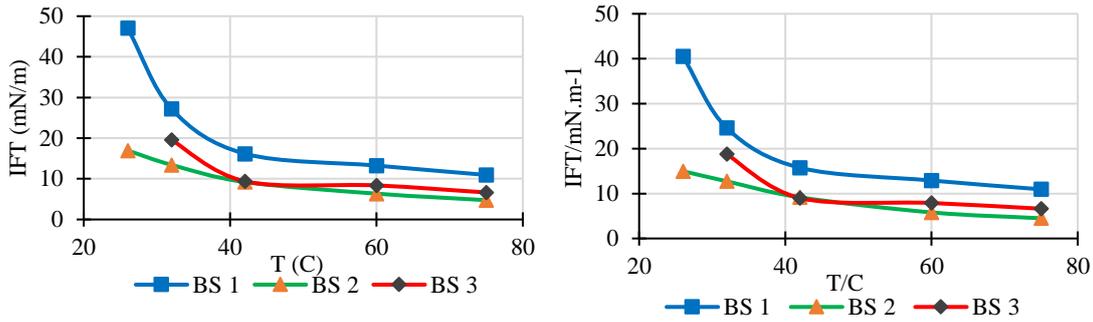
Analysis was carried out in real-time, which lasted about 6 hours, with the densities of the external phase duly calculated. The produced biosurfactants were analysed to estimate which has the greatest impact on IFT reduction. It was observed that the trend in the reduction of interfacial tension followed a similar pattern, as it can be seen from the plot of Fig. 6 (a-f).

The effectiveness in interfacial tension reduction, by biosurfactants in a liquid-liquid system, can be characterised by the biosurfactants concentration in the entire system, which is required to produce a given interfacial tension (Attwood and Florence, 2012, Möbius et al., 2001). A secreted, extracellular biosurfactant, that was produced by *BS 2*, gave the greatest reduction values of IFT as seen in Fig. 6f. It was assumed that equilibrium between the liquids was reached after five minutes of forming the oil film before the measurements were taken to observe the effect of the biosurfactant. This is in conformity with the reports of (Wang and Gupta, 1995) where oil drops were equilibrated for five minutes to get reliable results. The series of pressure measurements from lowest to highest shows a consistent reduction of the

interfacial tension value, and was more pronounced at higher temperatures, thus decreasing the oil viscosity by reducing the forces of attraction between the molecules of the crude oil. At 75°C the IFT value of 4.49 mN/m was lowest at a constant pressure of 13.89 MPa. This is evidence to demonstrate the importance of screening biosurfactants to isolate the most profitable and versatile amphilic surface compounds to be used for EOR applications, since environmentally friendly biosurfactants of the same family have different surface active strength behaviours. This result may potentially be applicable to an in situ microbial enhanced oil recovery operation.

Although our findings indicate that the endospore forming bacterium has a better effect on interaction with the crude oil if the cells are extracted out from the biosurfactant. The effect of the *BS 1* cell-free biosurfactant, investigated under thermophilic conditions, decreased the IFT from 47.06 mN/m to 10.94 mN/m at 75 °C, which was not expected, since it has the highest concentration that should significantly decrease the IFT even more, and could have resulted in slower interactions between the two interfaces. Although the measurements of *P. polymyxa* declined the IFT to a final reduction of 6.6 mN/m (75 °C).





(e) At constant pressure of 12.51 MPa

(f) At constant pressure of 13.89 MPa

Figure 6: Variation of IFT with temperature for Cell-free biosurfactants

A comparison of the biosurfactants with the cells, in terms of IFT reduction, showed higher values when compared to interactions with cultured cell-free supernatant at constant pressures. A summary of the decreasing effects of the biosurfactants on IFT reduction is presented in the Fig.7.

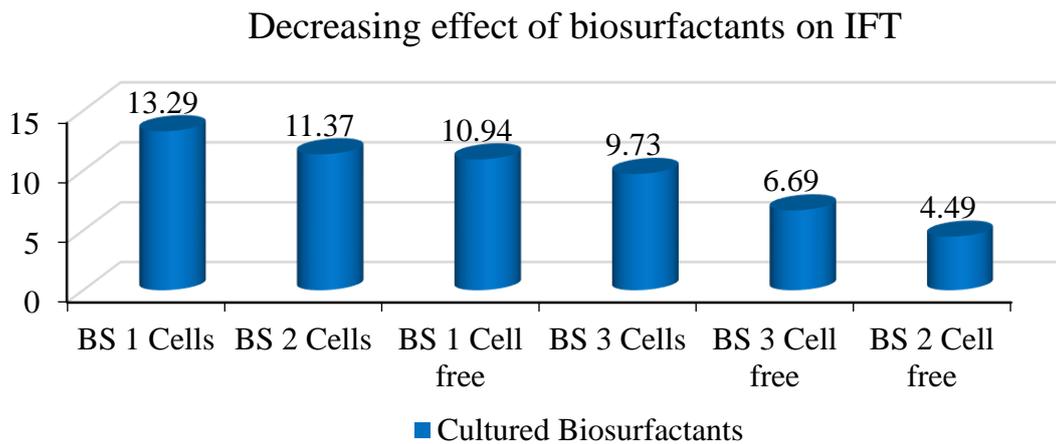


Figure 7: Summary of the decreasing effect of the cultured biosurfactants on interfacial tension

3.3 Qualitative wettability Tests

Qualitative wettability tests were performed on Bandera Gray (crushed grains), to check the effectiveness of the core cleaning and aging procedures, as well as the effectiveness of the biosurfactants in changing the wettability of the crude-oil aged crushed rock. Initial control tests were carried out with the untreated sample by the addition of the grains into distilled

water. In the event that the crushed rock grains sink to the bottom of the tube it is then completely water-wet, otherwise, it is oil-wet if it floats. The outcome of this control test gave a completely oil-wet characteristic (Fig. 8), since the grains were floating and remained that way even after several hours.

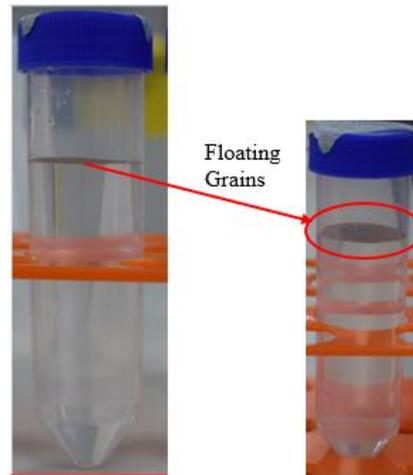
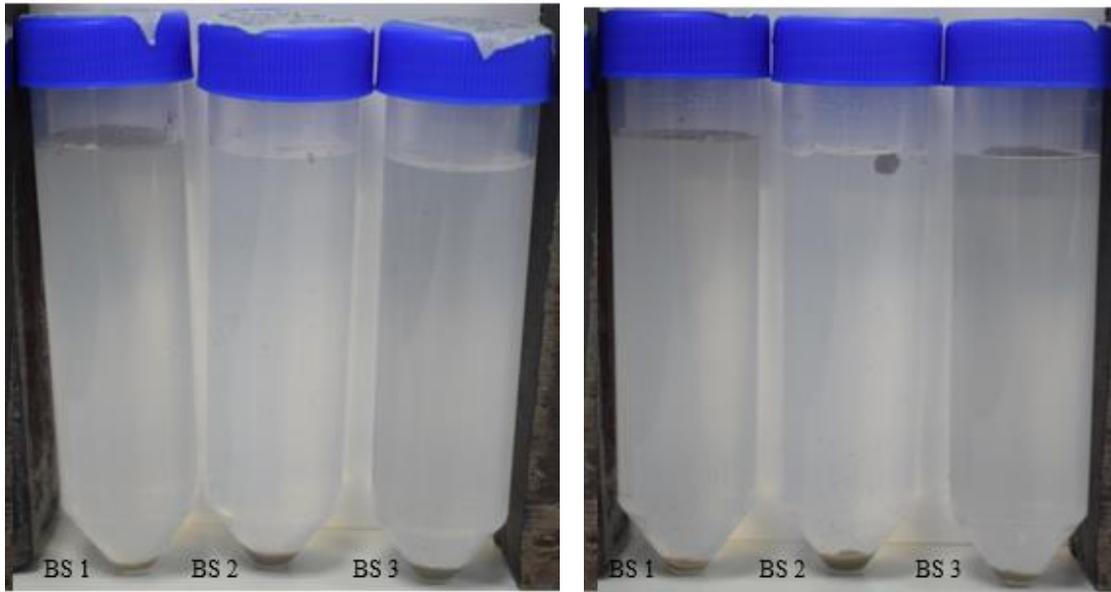


Figure 8: Control test of Bandera Gray with distilled water and untreated grain samples.

3.3.1 Floating Test

The floating test was initiated after treatment with the oil and with the different biosurfactants. 0.2 g of the treated grain sample was weighed and added into six (6) test-tubes for both the cell and cell-free biosurfactants. The impressive performance of the effect of these biosurfactants was observed as the sample was added into distilled water. Interestingly, most of the outcome gave a positive effect in altering the degree of wettability, as can be seen in Fig. 9. These results can be compared to the floatation experiment conducted by (Wu et al., 2006), where different surfactants and naphthenic acids, treated with calcite powder, provided information about the mechanisms responsible for the reversal to a water-wet state. Most of the grains, treated with the biosurfactants, showed alteration in wettability from hydrophobic to a slightly intermediate wet, rather than to a water-wet state, throughout the pore system for this test. A summary of the outcome is presented in Table 1.



(a)

(b)

Figure 9: (a) Distilled water and treated Bandera Gray grain sample in biosurfactant with cells

(b) Distilled water and treated Bandera Gray grain sample in cell-free biosurfactants.

Table 1: Summary of the wettability effect after the floating test

Bio – surfactants	Wettability Effect
	Bandera Gray
<i>BS 1</i> Cells	Intermediate-wet
<i>BS 2</i> Cells	Water -wet
<i>BS 3</i> Cells	Water – wet
<i>BS 1</i> Cell-free	Intermediate-wet
<i>BS 2</i> Cell-free	Water – wet
<i>BS 3</i> Cell-free	Intermediate-wet

3.3.2 Two-phase separation test

In the separation tests, grains were added into a mixture of distilled water, plus sunflower oil, in six (6) test tubes after treatment with biosurfactants. The treated rock grains remained completely in the water phase throughout the experiment with the sunflower oil, signifying a complete water-wet state as shown in Fig.10. The quantity of rock grain remaining in each phase gives a qualitative index of its wettability, and according to (Somasundaran and Zhang, 2006), if the grains remain in the oil phase, it is strongly oil wet, otherwise it is water wet.

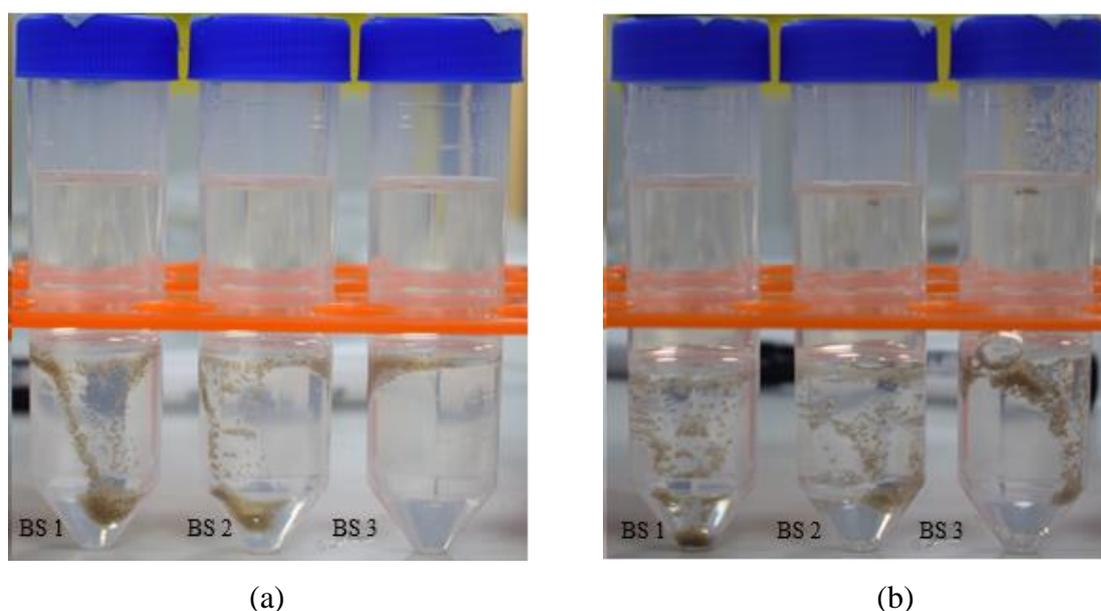


Figure 10: (a) Distilled water with sunflower plus treated grain sample of Bandera Gray in biosurfactants cells.

(b) Distilled water with sunflower oil plus treated grain sample of Bandera Gray in cell-free biosurfactants.

Most sandstone reservoirs in the North Sea are not strongly water-wet, but are recognised generally to exhibit a largely intermediate to slightly oil-wetting behaviour (McPhee et al., 2015). The wettability for Bandera Gray, treated with these biosurfactants, displayed an alteration in wettability from hydrophilic to hydrophobic, characterised by the grains completely remaining in the aqueous phase. The crude oil utilised, while preparing the grains, showed that it is a poor solvent for the biosurfactants, thus having a greater propensity to change wettability (Al-Maamari and Buckley, 2003). Table 2, see below, shows the wettability effects on Bandera Gray grains.

Table 2: Summary of the wettability effect after the two-phase separation test.

Bio – surfactants	Wettability Effect
	Bandera Gray
<i>BS 2 Cells</i>	Water –wet
<i>BS 2 Cell-free</i>	Water – wet
<i>BS 3 Cell-free</i>	Intermediate-wet
<i>BS 3 Cells</i>	Water – wet
<i>BS 1 Cell-free</i>	Intermediate-wet
<i>BS 1 Cells</i>	Intermediate-wet

4. CONCLUSION

Based on the findings from this experimental study, it can be stated that the potential to enhance oil recovery by these microbes is considerable. The experimental findings of decreasing IFT indicated that there was a considerable reaction between the oil-brine-biosurfactant interface, which led to the reduction of intermolecular forces between the oil and brine at increasing temperatures. The produced biosurfactant *BS 2* cell-free supernatant gave the best outcome in IFT reduction against the *BS 1* cells with the least performance. The findings of the qualitative wettability tests revealed that the biosurfactants can decrease the oil wetting of strongly oil-wet Bandera Gray sandstone by changing the wettability of the surface chemistry of the rock grain to being both water-wet and intermediate-wet, respectively. This alteration of wettability is a major factor that could enhance oil recovery, and further experiments of core flooding will prove the viability of these results.

Increased environmental awareness has been the main driver for the search for a replacement to the chemical surfactants. In this study, the effect of the carbon source plays an important role on the biosurfactant production. Sucrose, which is a hydrophobic carbon source, was selected as a suitable carbon nutrient supplement to produce the three biosurfactants. And according to recent research findings the decomposition property of sucrose makes it eco-friendly for biosurfactant production. **REFERENCE**

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