INTERFACIAL TENSION AND CONTACT ANGLE TESTS OF RHAMNOLIPIDS AND SOPHOROLIPIDS IN SANDSTONE FOR ENHANCED OIL RECOVERY

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Abstract

In the context of the development of enhanced oil recovery technology, biotechnology can be used to develop new methods that are more environmentally friendly and sustainable, one of which is the use of biosurfactants. Rhamnolipids and sophorolipids are non-toxic biosurfactants, biodegradable, and environmentally favorable substances. This study compares the results of laboratory studies on fluid-to-fluid interactions, including the interfacial tension and rock wetness tests at various concentrations and salinities, which represent high salinity, medium salinity, and low salinity. The mechanism of these two biosurfactants is based on reducing the interfacial tension (IFT) between the fluids and changing the wettability of the rock to a more water-wet state. In the interfacial tension test, the reduction in the interfacial tension of rhamnolipids reached 10^{-2} mN/m, while that of sophorolipids only reached 10^{-1} mN/m. Then, the sandstone wettability test showed that the two biosurfactants were classified as water-wet (<75°). This happens because of its chemical structure, which has two parts: the hydrophobic part and the hydrophilic part. Within the salinity range of 8,000-32,000 ppm, the rhamnolipids work better in the higher salinity to reduce interfacial tension, and the salinity has a subtle effect on the contact angle. On the other hand, sophorolipids were not affected by salinity for both interfacial tension and contact angle. These two biosurfactants have the potential to increase oil recovery in sandstone so that they can contribute to environmentally friendly biotechnology.

Keywords: **Contact angle; Interfacial tension; Rhamnolipids; Sophorolipids**

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Introduction

Biosurfactants are metabolic products generated as extracellular products or as part of the cell wall by bacteria, yeast, and fungi. Biosurfactants are amphipathic, surface-active compounds composed of hydrophilic and hydrophobic components. A polar molecule's hydrophilic portion can be a carbohydrate, amino acid, or phosphate group. The hydrophobic portion is typically along carbon chain or a fatty acid's hydrocarbon chain. Therefore, biosurfactants can bind to hydrocarbon molecules, reduce interfacial tension in the space between water and oil, and produce microemulsions that aid in hydrocarbon degradation (Georgiou *et al*., 1992; Banat, 1995; 2000; Desai and Banat, 1997; Mulligan, 2005). Geetha *et al*. (2018) demonstrate that the use of biosurfactants in conventional reservoirs can alter the wettability of rocks, making them more water-wet.

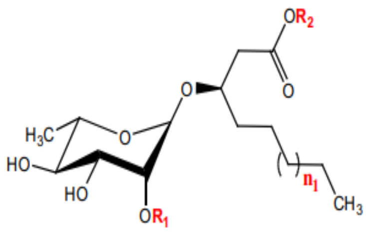
The biosurfactant rhamnolipids is produced by Pseudomonas aeruginosa. Rhamnolipids consist of one or two rhamnose molecules bonded to one or two b-hydroxydecanoic acid molecules. Rhamnolipids have the ability to reduce interfacial tension, allowing them to be utilized as bioremediation to increase hydrocarbon recovery. Rhamnolipids are anionic and contain carbonyl and hydroxyl groups that can be exchanged for cations, so they can be utilized to adsorb heavy metals (Ron and Rosenberg, 2001). Sophorolipids is a biosurfactant derived from Torulopsis bombicola, Torulopsis petrophilium, and Torulopsis apicola yeasts. Long chains of hydroxy fatty acids connect dimeric carbohydrates.

Biosurfactants are capable of dissolving enzymes, receptors, and proteins in cells. Biosurfactants differ from synthetic surfactants in several ways, including low toxicity, high ability to biodegrade, and more stable emulsifier properties, so that the degradation process is more optimal, more environmentally friendly, produces a great deal of foam, has a specific molecular group for a reaction, and has specific activity in extreme environmental conditions (temperature, pH, and salinity) (Kitamoto *et al*., 2002; Rodrigues *et al*., 2006; Rudakova *et al*., 2021; Sarubbo *et al*., 2022; Nagtode *et al*., 2023).

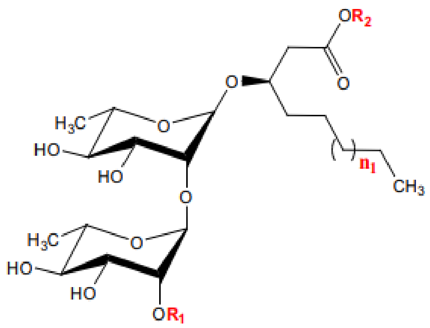
The Atomic Composition of a Biosurfactants

Rhamnolipids

Rhamnolipids is an anionic compound with surfactant properties. In aqueous solutions, these compounds can form micelles in which the fatty acid chains serve as hydrophobic centers and the rhamnose sugars serve as hydrophilic heads (Figure 1).



(a) Monorhamnolipids



(b) Dirhamnolipids

Figure 1. General structure of the rhamnolipids molecule (Abdel-Mawgoud *et al*., 2010)

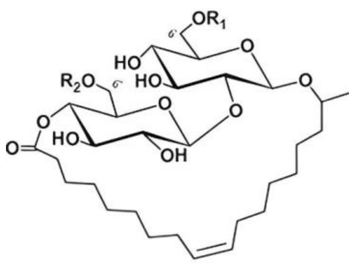
According to a study published in the Journal of Industrial Microbiology and Biotechnology, rhamnolipids produced by Pseudomonas aeruginosa have a mono-rhamnolipid structure, which means that they contain one molecule of rhamnose sugar. On the other hand, the rhamnolipids produced by Burkholderia thailandensis have a di-rhamnolipid structure, meaning that they contain two rhamnose sugar molecules (Ławniczak *et al*., 2013).

Another study published in the Journal of Applied Microbiology compared the molecular structures of rhamnolipids produced by various strains of bacteria, including Pseudomonas aeruginosa, Burkholderia thailandensis, and Burkholderia pseudomallei. The study found that the rhamnolipids produced by Pseudomonas aeruginosa and Burkholderia thailandensis have a similar molecular structure, while the rhamnolipids produced by Burkholderia pseudomallei have a more complex structure (Sotirova *et al*., 2008).

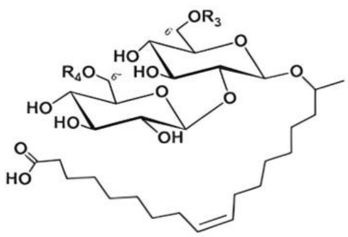
Sophorolipids

Sophorolipids are a type of amphoteric surfactant, meaning they can function anionically or nonionically depending on their environment.

When the pH is low (acidic), sophorolipids act as anionic surfactants, whereas nonionic surfactants act when the pH is high (alkaline) (Van Bogaert *et al.*, 2007). Sophorolipids are a type of biosurfactant produced by several species of yeast, such as Candida bombicola and Candida apicola. The molecular structure of sophorolipids consists of two parts, namely the hydrophobic part, which consists of fatty acids, and the hydrophilic part, which consists of sophorose sugar (Figure 2). The hydrophobic and hydrophilic parts are connected by a glycosidic bond. The molecular structure of sophorolipids can vary depending on the source and conditions of production, such as the length of the fatty acid chain, the degree of saturation of the fatty acid chains, the position of the sophorose sugar, and the amount of sophorose sugar in the molecule (Elshafie *et al*., 2015; Akbari *et al*., 2018; Cho *et al*., 2022).



(a) Lactonic sophorolipid



(b) Acidic sophorolipids

Figure 2. General structure of the sophorolipids molecule (Nguyen and Sabatini, 2011)

In surfactant injection, there are two main limitations: reservoir temperature and high salinity, and secondly, excess surfactant adsorption on the rock surface, which can reduce the recovery of trapped oil from the pores in the rock. A number of studies have identified various reasons for surfactant adsorption, which include ion exchange, ion association, hydrophobic bonds, dispersion forces, electrostatic attraction, van der Waals forces, and electron polarization π (Paria and Khilar, 2004; Zhang and Somasundaran, 2006; Kamal *et al*., 2019). Thus, this study aims to carry out a series of static tests consisting of IFT and contact angle tests to determine the ratio of biosurfactants in increasing oil recovery in Berea sandstone.

The objective of this research is to compare the relative efficacy of sophorolipids and rhamnolipid biosurfactants in sandstones for enhancing oil recovery, given the growing interest of exploration and production businesses in employing surfactant injection in sandstones with high temperatures. Consequently, it is hoped that this research's findings would be able to significantly progress both science and the petroleum sector

Materials and Methods

Materials

The following materials will be used in this study:

1. Rhamnolipids and Sophorolipids Biosurfactants
2. Chemical beakers, spatulas, measuring cups, pipettes, electric magnetic stirrers, and hotplates are examples of laboratory equipment. For various compatibility testing applications, standard laboratory equipment such as beakers, spatulas, measuring cups, dropper pipettes, electric magnetic stirrers, hotplates, and electric heaters is required.
3. The salinity of synthetic saline NaCl solutions ranged from low to high (8,000 ppm, 16,000 ppm, 24,000 ppm, and 32,000 ppm).
4. The used oil sample is light oil with a reservoir temperature of 68.3 degrees Celsius.

The biosurfactants were obtained from Shanghai Yuchuang Chemical Technology Co., Ltd., and and Table 1 below is an analysis of the data from the two biosurfactants.

Table 1. Shows the composition of the two biosurfactants

Items	Test Results Rhamno	Test Results Sophoro
Appearance	Dark brown liquid	Light yellow to brown liquid
Solid content	298.76	51.50%
pH Value	7.67	3.95
Solubility (water)	Soluble	Soluble

Method

Interfacial tension test

The CMC value of each biosurfactant formulation was determined by testing rhamnolipids and sophorolipids biosurfactant solutions with different salinities and oil concentrations. Using a hypodermic, the prepared solution will be injected into the tube, along with 2μL of oil in the form of droplets. The test was then conducted for 30 min at 3,000 rpm using a set of TX 500D interface tension testers with rotating drop tensiometers

Contact Angle test

This test is carried out using Theta Lite Optical Tensiometer TL-100 (One Attention), a set of contact angle testing instruments. Thin sections of the Berea sandstone core to be used in this investigation were sliced, and the testing solution was prepared. A thin incision is made in the instrument, and a solution of rhamnolipids and sophorolipids biosurfactant is then poured onto it. The droplets will form an angle as they spread across the narrow incision. The optical tensiometer will acquire the contact angle measurement.

Results and Discussion

Interfacial Tension Test

An interfacial tension test was conducted on the two biosurfactant formulations to determine their efficacy. The lower the interfacial tension, the simpler it is for the biosurfactant to transport oil through the pores of the rock. Utilizing both polar and nonpolar groups, biosurfactants reduce interfacial tension by adsorbing at the interface between two fluids. Using biosurfactants will increase the capillary force of porous media, thereby enhancing oil recovery (Negin *et al*., 2017).

Biosurfactants possess the same capability as surfactants, which is to disperse the water-oil system and reduce interfacial tension. This biosurfactant disperses oil droplets beginning at the droplet's exterior. Before biosurfactant was added, the oil would appear as round droplets. The oil will become long and thin after the addition of biosurfactants. By elongating and thinning the oil, the droplets will be able to escape from the rock pores (Viades-Trejo and Gracia-Fadrique, 2007).

The structures of rhamnolipids and sophorolipids contain hydrophilic and hydrophobic groups. In rhamnolipids, the hydrophilic group is rhamnose, whereas in sophorolipids, it is glucose. In both cases, the hydrophobic groups are fatty acids.

When rhamnolipids and sophorolipids are applied to the interface between water and rock, the hydrophilic groups interact with water and the hydrophobic groups interact with rock. The interactions between these molecules will result in a decrease in interfacial tension at the interface between rock and water.

Van der Waals forces and electrostatic forces occur in rhamnolipids and sophorolipids when the interfacial tension decreases. Van der Waals forces are those that occur between nonpolar molecules, while electrostatic forces are those that occur

Table 2. Rhamnolipids IFT Value at Salinity: 8,000 ppm

8,000 PPM	
Rhamnolipids Concentration (%)	**IFT (mN/m)**
0	5.201
0.25	2.04×10^{-1}
0.5	2.10×10^{-1}
0.75	2.16×10^{-1}
1	2.22×10^{-1}
5	1.56×10^{-1}
10	1.17×10^{-1}
20	1.25×10^{-1}

Table 3. Rhamnolipids IFT Value at Salinity: 16,000 ppm

16,000 PPM	
Rhamnolipids Concentration (%)	**IFT (mN/m)**
0	4.545
0.25	1.77×10^{-1}
0.5	1.87×10^{-1}
0.75	1.98×10^{-1}
1	2.04×10^{-1}
5	1.09×10^{-1}
10	9.44×10^{-2}
20	1.01×10^{-1}

Table 4. Rhamnolipids IFT Value at Salinity: 16,000 ppm

24,000 PPM	
Rhamnolipids Concentration (%)	**IFT (mN/m)**
0	5.889
0.25	1.71×10^{-1}
0.5	1.71×10^{-1}
0.75	1.77×10^{-1}
1	1.77×10^{-1}
5	7.82×10^{-2}
10	8.44×10^{-2}
20	9.08×10^{-2}

Table 5. Rhamnolipids IFT Value at Salinity: 32,000 ppm

32,000 PPM	
Rhamnolipids Concentration (%)	**IFT (mN/m)**
0	6.283
0.25	1.66×10^{-1}
0.5	1.52×10^{-1}
0.75	1.56×10^{-1}
1	1.38×10^{-1}
5	5.37×10^{-2}
10	6.94×10^{-2}
20	7.61×10^{-2}

between polar molecules. These two forces permit the rhamnolipids and sophorolipids molecules to adhere to the granite surface and form a thin film at the rock-water interface.

Rhamnolipids Biosurfactant

The salinity and concentration of different rhamnolipids biosurfactant were tested. The following is the value of the interfacial tension (IFT) shown in Tables 2-5.

According to the tables above, the average value of the interfacial tension decreases at concentrations of 0.25%, 0.5%, 1%, and 5%. Upon closer inspection, however, the concentration of 5% in various salinities demonstrates a more significant decrease in interfacial tension, particularly at high salinities, 24,000 ppm and 32,000 ppm, where values reach 10^{-2} mN/m. However, the decrease in interfacial tension with each concentration increase has not yet reached the ultra-low IFT of 10^{-3} mN/m or less than 10^{-2} mN/m.

The increase in concentration tends to decrease the interfacial tension, but the lowest point (CMC) has not been attained because the interfacial tension value fluctuates up to 20% concentration. However, the value of the interfacial tension between the biosurfactant solution and crude oil has a direct effect on the decrease in oil saturation, resulting in increased oil recovery. Nevertheless, the lowest IFT does not guarantee the maximum oil recovery, which may not be consistent with current fundamental theory. Therefore, other mechanisms must be examined to determine the efficacy of this Rhamnolipids.

According to the results of the IFT test, the optimal concentrations of Rhamnolipids are 0.25%, 0.5%, 1%, and 5%. So that the concentration value represents the optimal concentration for testing the subsequent mechanism, contact angle. The interfacial tension values were plotted against the biosurfactant concentration for the rhamnolipids

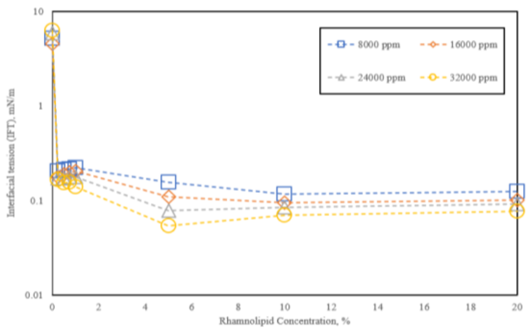


Figure 3. Graph depicting the interfacial tension of rhamnolipids at varying salinities and concentrations

formulation. Figure 3 below illustrates the value of the interfacial tension.

Sophorolipids Biosurfactant

The salinity and concentration of different sophorolipids biosurfactant were tested. The following is the value of the interfacial tension (IFT) shown in Tables 6-11.

In contrast to rhamnolipids, the CMC value of sophorolipids can be determined by plotting the value of the interfacial tension against the concentration of sophorolipids biosurfactant. At various salinities and concentrations, the critical micelle concentration (CMC) value of sophorolipids occurs at a concentration of 0.25% at a salinity of 8,000 ppm, while it occurs at a concentration of 5% at salinities of 16,000 ppm, 24,000 ppm, and 32,000 ppm.

Table 6. Sophorolipids IFT Value at Salinity: 8,000 ppm

8,000 PPM	
Sophorolipids Concentration (%)	**IFT (mN/m)**
0	5.201
0.25	5.05×10^{-1}
0.5	5.32×10^{-1}
0.75	5.70×10^{-1}
1	5.90×10^{-1}
5	6.14×10^{-1}
10	6.24×10^{-1}
20	6.36×10^{-1}

Table 7. Sophorolipids IFT Value at Salinity: 16,000 ppm

16,000 PPM	
Sophorolipids Concentration (%)	**IFT (mN/m)**
0	4.545
0.25	5.28×10^{-1}
0.5	5.17×10^{-1}
0.75	5.82×10^{-1}
1	5.86×10^{-1}
5	5.97×10^{-1}
10	6.18×10^{-1}
20	6.69×10^{-1}

Table 8. Sophorolipids IFT Value at Salinity: 24,000 ppm

24,000 PPM	
Sophorolipids Concentration (%)	**IFT (mN/m)**
0	5.889
0.25	5.23×10^{-1}
0.5	5.14×10^{-1}
0.75	5.70×10^{-1}
1	5.73×10^{-1}
5	5.85×10^{-1}
10	5.87×10^{-1}
20	6.33×10^{-1}

Table 9. Sophorolipids IFT Value at Salinity: 32,000 ppm

32,000 PPM	
Sophorolipids Concentration (%)	**IFT (mN/m)**
0	6.283
0.25	5.10×10^{-1}
0.5	4.83×10^{-1}
0.75	4.89×10^{-1}
1	5.33×10^{-1}
5	5.60×10^{-1}
10	5.85×10^{-1}
20	6.13×10^{-1}

Table 10. Rhamnolipids biosurfactant contact angle values

		Rhamnolipids	
No	**Salinity, ppm**	**Concentration, %**	**Contact Angle, º**
1	8,000	0	53.93
		0.25	32.55
		1	24.5
		5	16.31
2	16,000	0	58.8
		0.25	29.54
		1	21.49
		5	15
3	32,000	0	63.56
		0.25	25.24
		1	24.08
		5	17.70

Table 11. Sophorolipids biosurfactant contact angle values

		Sophorolipids	
No	**Salinity, ppm**	**Concentration, %**	**Contact Angle, º**
1	8,000	0	53.93
		0.25	32.61
		1	27.06
		5	24
2	16,000	0	58.8
		0.25	30.28
		1	26.95
		5	21.89
3	32,000	0	63.56
		0.25	30
		1	25.83
		5	21.24

Increasing the concentration tends to decrease the interfacial tension until the interface is saturated with biosurfactant molecules. The minimum value is the CMC value. After attaining the CMC value, the addition of surfactant concentration tends to cause the interfacial tension to remain constant, with an average value of 10^{-1} mN/m in this sophorolipids. However, the decrease in interfacial tension with each concentration increase has not yet reached the ultra-low IFT of 10^{-3} mN/m or less than 10^{-2} mN/m.

The interfacial tension values for the sophorolipids formulation were plotted against the biosurfactant concentration. Figure 4 below illustrates the value of the interfacial tension

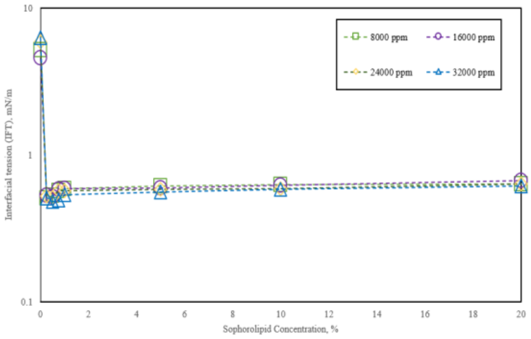


Figure 4. Graph depicting the interfacial tension of sophorolipids at varying salinities and concentrations

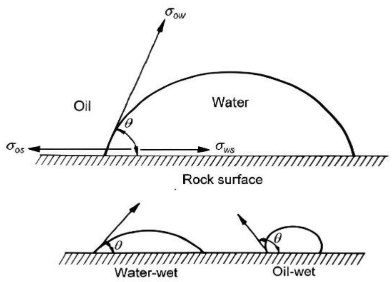


Figure 5. Force balance on water-oil-rock system (Panchal *et al*., 2021)

Contact Angle Test

The effect of the addition of biosurfactant can also be seen on the wettability aspect of the rock. Conventional rocks like sandstone show homogenous wettability as they have uniform mineralogy and significantly less organic content than shales (Wang *et al*., 2011). Wettability of conven tional rocks can be determined by measuring contact angle. These measurements are based on balancing interfacial tension on the surface of water-oil-rock system using Young's equation (Equtation 1), as shown in Figure 5. Rock surface wettability is categorized from 0° to 75° as water-wet, 75° to 105° as intermediate-wet, and 105° to 180° as oil-wet (Anderson, 1986).

$$\sigma_{OS} = \sigma_{WS} + \sigma_{OW} * Cos\theta \quad (1)$$

where σ_{OS} = oil-rock IFT (N/m); σ_{WS} = water-rock IFT (N/m); σ_{OW} = oil-water IFT (N/m); and θ = contact angle (degree).

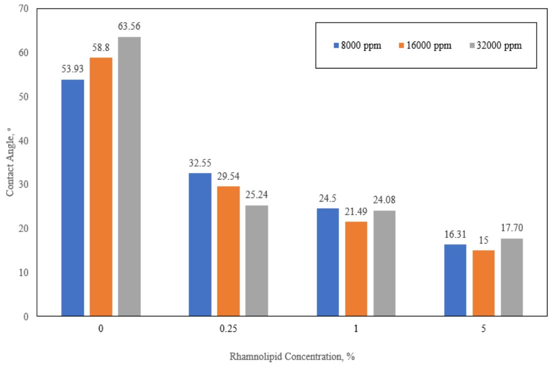


Figure 6. Graph of rhamnolipids contact angle

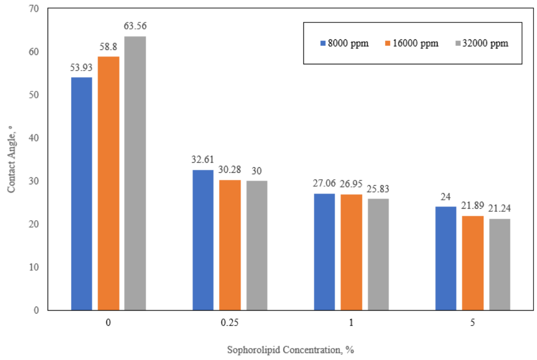


Figure 7. Graph of sophorolipids contact angle

The wettability of the rock plays an important role because it influences reservoir properties such as relative permeability characteristics, fluid distribution in the rock pores, and fluid flow behavior during the recovery process. Furthermore, to determine the effect of the addition of biosurfactant on wettability, contact angle testing was also carried out. This test was carried out at selected concentrations and salinities. The results of the contact angle test for each biosurfactant are as follows:

According to the results of rhamnolipids and sophorolipids biosurfactants, the trend is fairly uniform. The value of the contact angle decreased with increasing concentrations and salinities of the biosurfactant. This indicates that the addition of biosurfactant is able to change the surface properties of the rock in a more water-wet direction and is expected to be able to release the oil that is on the surface of the rock.

Changing wetness to water-wet can affect the effective permeability, resulting in a lower mobility ratio and this can increase area sweep efficiency (Hosseini and Tahmasebi, 2020). The rhamnolipids molecule has a unique structure, with hydrophilic (rhamnose) and hydrophobic (fatty acid) groups distributed unevenly on the surface of the molecule, and the same is true for sophorolipids molecules, which have main components in the form of glucose and fatty acids. Glucose in sophorolipids imparts hydrophilic properties, whereas fatty acids impart hydrophobic properties.

When rhamnolipids are applied to rock surfaces, rhamnolipids molecules adhere to the rock surface and form a thin film at the rock-water interface.The forces that occur when rhamnolipids and sophorolipids attach to the rock surface and form a thin layer are adhesion and cohesion forces. Adhesion forces are the forces that occur between rhamnolipids and sophorolipids molecules and the rock surface, while cohesion forces are the forces that occur between rhamnolipids and sophorolipids molecules themselves.

Rhamnolipids and sophorolipids molecules form a thin film on the rock surface, which reduces the interfacial tension between water and rock. This causes water to spread more easily on the surface of the rock, thereby increasing its ability to penetrate the rock (Amraini *et al*., 2022). As a result, the moisture in the rock becomes more evenly distributed, which causes the rock to be more easily wetted by water. Figures 6 and 7 show the contact angle value plotted versus the biosurfactant concentration.

Conclusions

From this study, the conclusions that can be drawn are:

1. Rhamnolipids and sophorolipids biosurfactants reduce the interfacial tension (IFT) of oil and water. However, both of them have not yet reached the ultra-low IFT of 10^{-3} mN/m or less than 10^{-2} mN/m.
2. Testing the contact angle on rhamnolipids and sophorolipids biosurfactants indicated that there was a change in rock surface wettability to become more water-wet. This phenomenon has the potential to reduce residual oil saturation and eventually aid the increase of oil recovery.
3. Within the salinity of 8,000-32,000 ppm, the rhamnolipids work better in the higher salinity for reducing interfacial tension, and salinity has subtle effect on the contact angle. On the other hand, sophorolipids were not affected by salinity for both interfacial tension and contact angle.
4. Displacement experiments are recommended to further investigate the potential application of these biosurfactants.

Recommendation

Additional compatibility testing, such as adsorption and zeta potential studies, should be performed in order to advance this research.

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