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Oil-water interfacial tension, wettability alteration and foaming studies of natural surfactant extracted from *Vernonia Amygdalina*

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ABSTRACT

Surfactant flooding is an enhanced oil recovery (EOR) method for recovering residual oil in the reservoir through mechanism of interfacial tension (IFT) reduction and wettability alteration. Due to toxicity and high cost associated with conventional surfactants, recent research has focussed on developing low-cost and environmentally benign surfactants. Herein, a low-cost green surfactant is extracted from *Vernonia Amygdalina* (VA) and appraised for EOR applications. The extracted surfactant was characterized using Fourier Transform Infrared (FTIR) and High-Pressure Liquid Chromatography (HPLC). The IFT of the synthesized surfactant at the oil-water interface was determined using Kruss tensiometer. Additionally, the foam stability of the synthesized surfactant was examined. Moreover, the wettability of the saponin based natural surfactant (SBNS) at the rock-fluid interface was analysed using Dataphysics drop shape analyser. Experimental result revealed that SBNS (1 wt% concentration) stabilized foam for longer periods with half-life of 1100 min. Furthermore, the synthesized surfactant was effective in lowering the IFT of oil-water interface from 18 mN/m to 0.97 mN/m. Finally, SBNS altered the wettability of sandstone cores to water-wetting condition by reducing the contact angle from 118.5 ° to 45.7 °. Overall, SBNS exhibit excellent properties desirable for EOR and thereby recommended as supplementary alternative to conventional surfactants.

1. Introduction

Huge volumes of oil remain in the reservoir after primary recovery (Thomas, 2007). Literature suggest about two-third of oil is left in the reservoir due to capillary trapping phenomenon (Abbas et al., 2018). Hence, numerous enhanced oil recovery (EOR) methods have been devised. The EOR methods are broadly classified into thermal and non-thermal EOR (Gbadamosi et al., 2018). Thermal EOR methods such as steam flooding, in situ combustion and steam assisted gravity drainage are deemed unsuitable for reservoirs with thin pay zone and huge depth. Moreover, they emit large amount of carbon dioxide (CO_2) and associated greenhouse gases which are sources of environmental concern (Guo et al., 2016). Thus, non-thermal EOR methods such as chemical and microbial oil recovery are more coveted for EOR (Agi et al., 2020).

Surfactant flooding, a chemical EOR method has received prodigious attention due to its ability to increase the pore scale displacement efficiency in reservoirs (Tumba et al., 2019; Elhag et al., 2020). Numerous applications of surfactant for EOR have been explored for conventional (sandstone and carbonates) and unconventional reservoirs such as ultra-tight shale formations. By lowering the interfacial tension at the fluid—fluid interface, the capillary number increases (see Equation (1)). Therefore, oil trapping force is lowered and the oil flows towards the production well (Sheng, 2014). Moreover, surfactant also alters the wettability of the rock-fluid interface to water-wetting condition desired to increase oil productivity (Gbadamosi et al., 2019a).

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$$N_c = \frac{\mu \cdot v}{\sigma \cos \theta} \qquad 1$$

Where N_c is the dimensionless capillary number, μ is the viscosity of the aqueous phase, v is the velocity, σ is the interfacial tension between the oil and water, and θ is the contact angle.

Numerous conventional surfactants have been explored for surfactant flooding. These include anionic surfactant, cationic surfactant, zwitterionic surfactant, non-ionic surfactant, gemini surfactant, viscoeleastic surfactant, and polymeric surfactant (Kamal et al., 2017). The use of conventional surfactants has resulted in recovering capillary trapped oil with varying degree of efficiency (Tumba et al., 2019; Abbas et al., 2021). Several field trials show that the use of surfactant can boost the overall oil recovery efficiency (Abbas et al., 2018; Olajire, 2014). Nonetheless, the use of conventional surfactant has some limitations. Firstly, the high cost of conventional surfactants increases the operational cost of oil production. More importantly, the toxicity of conventional surfactants poses serious environmental issues (Olajire, 2014).

Recently, natural surfactant has been synthesized from leaves and oil extract of plants and exploited for EOR (Emadi et al., 2019; Dashtaki et al., 2020; Nowrouzi et al., 2020a). The phytochemical properties show they are surface active due to the saponins and sapogenin contents of the leaves. Due to the nature of their hydrophilic head and hydrophobic tail, saponins are classified as non-ionic surfactants. Fig. 1 depicts the structure of saponin based natural surfactants. As compared to conventional surfactants, the newly devised surfactants are renewable, non-toxic and environmentally benign. Besides, due to their synthesis and production from waste and cheaply available raw materials, the newly derived natural surfactants are low-cost and make the overall EOR process efficient and cost-effective.

The extraction of surfactant from oils have been explored. Alsabagh et al. (2021) synthesized non-ionic surfactants from waste cooking oil using anhydrous sodium sulphate and sorbitan. The synthesized surfactant lowered the IFT up to 0.06 mN/m while contact angle reduced up to 27.5 °. An incremental oil recovery of 24.3% and 11.7% was recorded for the non-ionic surfactants from palm kernel oil and palm oil, respectively. Nowrouzi et al. (2020b) evaluated the use of waste chicken fat as a renewable primary source for surfactant via esterification and sulphonation processes. At critical micelle concentration, the surfactant lowered interfacial tension of the oil-water interface to 0.043 mN/m and altered the wettability of carbonate rock from oil-wet to water-wetting condition. Approximately 18% incremental oil recovery was achieved during EOR with alkaline-surfactant-polymer slug. Similarly, Nowrouzi et al. (2021b) explored the use of surfactant synthesized from rapseed oil and recorded 14.6–25.7% incremental oil recovery for various injection scenarios.

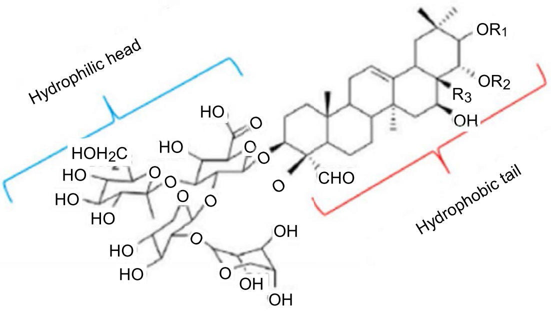

Fig. 1. Molecular structure of Saponin-based Surfactant, adapted from (Nowrouzi et al., 2020a).

More recently, researchers have synthesized and evaluated surfactant from saponin-inherent leafy substances. Nowrouzi et al., (2021) synthesized a double-chain single-head modified saponin from *Anabasis Setifera* plant and exploited its application in a carbonate reservoir. At the critical micelle concentration (CMC), the surfactant lowered the interfacial tension of the oil-water interface and achieved moderate wettability of the carbonate cores. Furthermore, Yekeen et al. (2020) extracted saponin based non-ionic surfactant from *Sapindus Mukorossi* and explored their IFT, foaming and wettability properties at ambient and typical reservoir conditions. At 0.2 wt% concentration of the surfactant, the interfacial tension (IFT) of the crude oil-water system was reduced from 23.24 mN/m to 1.59 mN/m. Moreover, the surfactant altered the wettability of the shale rock to a water-wetting condition. Moreover, Emadi et al. (2019) appraised the application of natural surfactant synthesized from *Zyziphus Spina Christi* for IFT and foam flooding application. The natural surfactant increased oil recovery by 15% over water flooding and showed good foam stability and IFT performance. Nowrouzi et al. (2021c) synthesized natural surfactant from *Myrtus communis* and evaluated its application for EOR in carbonate core plugs. The synthesized surfactant reduced IFT to 0.861 mN/m and an incremental oil recovery of 14.3%.

Vernonia Amygdalina (VA) is a shrub found in Africa and Asia continents and often referred to bitter leaf because of their bitter taste. Some of its components are used in manufacturing pharmaceutical products and cosmetics. The phytochemical analysis of its extracts confirms the presence of saponin based natural surfactant (SBNS). Saponin are naturally occurring plant glycosides with strong foamability in aqueous solution. The foamability is due to the non-polar sapogenin and water-soluble side chain, which has a close resemblance in structure to synthetic surfactants. Hitherto, saponin has been extracted from various plants such as *Sapindus Mukorossi*, *Seidlitzia Rosmarinus*, *Gylcyrrhiza glabra, Zizyphus Spina Christi*, and *Anabasis Setifera* plants using conventional methods of extraction. The extraction methods have several shortcomings such as high solvent consumption, and long extraction period. More recently, the use of ultrasonic extraction has been proffered as more economic, effective and a clean extraction process for green plants. The ultrasonic extraction process enhances the extraction efficiency and avoids structural damage and degradation of the polysaccharides.

Herein, ultrasonic assisted extraction of saponin-based natural surfactant (SBNS) from *Vernonia Amygdalina* (VA) and explored for EOR applications. The synthesized surfactant was characterized using FTIR and HPLC. Thereafter, the foaming behaviour of SBNS was studied. The effect of salinity representative of typical reservoir conditions was examined on the IFT of SBNS at oil-water interface. Finally, the wettability alteration effect of the surfactant was estimated on sandstone cores.

2. Laboratory experiment

2.1. Materials and reagents

Matured *V. amygdalina* leaves were harvested in August from Universiti Teknologi Malaysia (UTM) campus Johor Bahru, Malaysia (1° 33′ 19.79″ N and 103° 38′ 17.39″ E). 99% purity sodium chloride (NaCl) with a mol. wt. of 58.44 g/mol was acquired from Sigma Aldrich. For the analyses, an intermediate crude oil gotten from Sarawak oilfield, Malaysia (4° 6° N and 110° 112° 114° E) with a viscosity and density at 25 °C of 10 mPa s and 0.82 g/mL (API 37.7), respectively was applied. Table 1 shows the SARA property of the crude oil. Cores of sandstone of the same outcrop with mid-

Table 1
Crude oil SARA Properties.

Volatiles (%)	Inorganics (%)	Saturates (%)	Aromatics (%)	Resins (%)	Asphaltenes (%)
79.83	0.06	11.02	2.73	6.35	0.01

permeability were applied for the wettability test. Table 2 present the properties of the sandstone cores used in this study. Distilled (DW) and deionized water (DIW) were used as the solvent.

2.2. Surfactant synthesis

About 10 kg of *V. amygdalina* leaves were cleaned, air dried for 5 days and grinded into powder form. The powdered plant material was passed through a 0.5 mm sieve. About 2 g of the powdered sample was dispersed in 100 ml of DIW in a beaker. This was shaken for 2 min on an orbital shaker (Protech Model 720) after which the beaker was positioned in an ultrasonic water bath (40 kHz, 500 W). The characteristics of the ultrasonic power and measurement of the water bath is reported elsewhere (Agi et al., 2019). The extraction was done for 2 h during which the bath temperature was between 27 and 35 °C. After extraction, the liquid samples were transferred into 50 ml conical polypropylene centrifuge tube and centrifuged (Sorval, Wx 100 plus + Ultra Series) at 2000 rpm for 20 min. The liquid extracted (50 ml) was then collected using an ash-less filter paper. It was then placed in a freeze dryer to evaporate to dryness under vacuum at 45 °C.

2.3. Characterization and equipment

The functional groups of the extracted saponin (SBNS) was studied using Tracer 100 Fourier-transform infrared spectroscopy (FTIR-Shimadzu IR). Before placing the dried SBNS in a sample container, it was first introduced into potassium bromide (KBr) before it was placed in a sample bearer and the FTIR spectra were attained from a wave-range of 500–4000 cm^{-1}. The SBNS (0.1 wt%) was diluted 3-fold using DIW and filtered through a 0.45 μm syringe filter before injection. The chromatograph was acquired from the HPLC system using UV–Vis detector run at 0.8 mL/min. The mobile phase is a buffer solution of o-phosphoric acid with a pH of 2.4 for 30 min. The total saponin was detected and quantified at 280 nm.

2.4. IFT experiment

IFT in the presence of SBNS (0.1–3 wt%) were measured using the tensiometer of Easy Dyne obtained from Kruss GmbH, Germany. The tensiometer was attached to an ultrasonic bath for temperature control. The ring correction process by Harkins and Jordan was applied to calibrate the equipment. The SBNS critical micelle concentration (CMC) were determined through conductivity measurement. Besides, the CMC was confirmed from IFT-surfactants concentration plot, above which no substantial variation in IFT was measured. Thereafter, the IFT of SBNS at varying brine concentrations (0.9–3.0 wt% of NaCl) was studied.

2.5. Foamability and foam stability test

One of the inherent properties of surfactant is their ability to generate and stabilize foam. The foamability aids fluid diversion from thief zones to low permeable regions in the reservoir. With the presence of the saponin content of the leaves, SBNS extracted from VA leaves was tested for its ability to stabilize foam. The foamability tests were conducted using a measuring cylinder. The surfactant solution at CMC with a quantity of 50 ml was added into a measuring cylinder and shaken vigorously using a vortex mixer for 5 s. Subsequently, the foam height and half-life of the foam was measured as a function of time to ascertain its stability (Nowrouzi et al., 2020a).

2.6. Wettability test

To estimate the contact angle, the core samples were trimmed to a diameter of 5 cm and 2 cm in thickness with a flat surface (see Table 2). DIW and toluene were applied in the washing of the cores to remove impurities and contaminants. The cores were subjected to drying for 24 h afterwards. The samples were put into a beaker and made oil-wet using the method of Giraldo et al. (2013). This was done by completely submerging the core samples in crude oil. The sealing of the beaker was carried out to avoid evaporation and adulteration and oven-dried at 80 °C for 48 h. To measure the efficiency of alteration of wettability of SBNS in a static condition, the oil-wet cores were preserved with varying concentrations of SBNS (0.2–1.2 wt%). To ensure that the samples were oil-wet, the contact angle of the untreated sandstone samples were determined and used as reference (Alveroz-Berrios et al., 2018). Subsequently, the contact angle of the oil-wet samples treated with SBNS was estimated. This was achieved using a DataPhysics drop shape analyser.

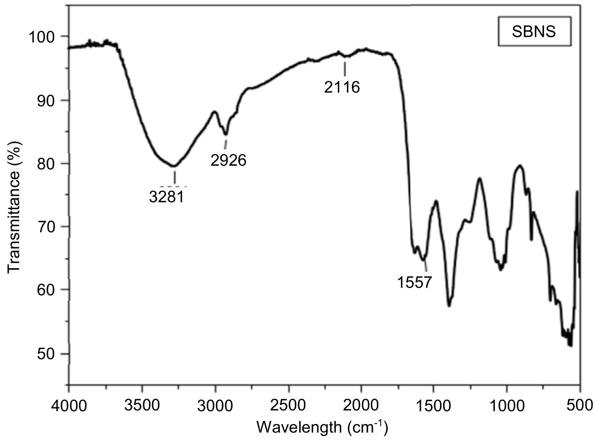

Fig. 2. FTIR characterization of SBNS.

Table 2
Properties of sandstone core used for contact angle test.

Diameter (cm)	Thickness (cm)	Bulk Volume (cm^3)	Pore Volume (cm^3)	Porosity (%)	Permeability (mD)
5	2	100.37	16	15.9	201

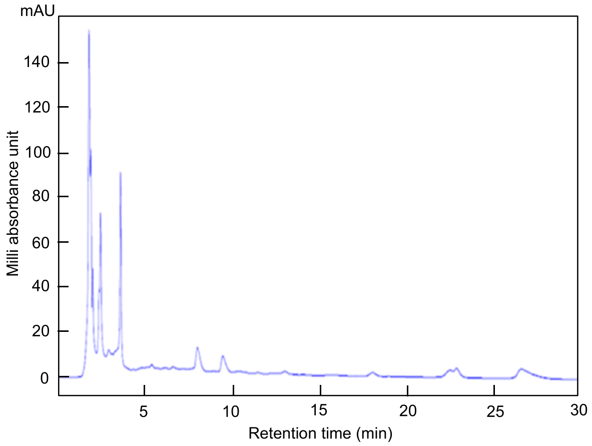

Fig. 3. HPLC characterization of SBNS.

A drop of water was placed on surface of the dried sandstone core (Giraldo et al., 2013), the image of drop was captured by an inbuilt camera and the contact angle was estimated by inbuilt software.

3. Result and discussion

3.1. Characterization of the synthesized surfactant

Fig. 2 illustrate the FTIR result and confirm the presence of saponin in VA plant leaves. A peak was detected at 3281 cm^{-1} which represents the stretching absorbance of the hydroxyl group (-OH) in the side chain of SBNS. This is synonymous to hydroxyl bond functional group reported by Nowrouzi et al. (2020a) for *Anabasis Setifera* plant. Subsequently, an absorption peak was discovered at 2926 cm^{-1} representing the presence of carbon-hydrogen (C–H) aliphatic graft. This agrees with the work of Nowrouzi et al. (2021c) for C–H bond detected on extracted saponin from *Myrtus Communis* plant. Also, the carbonyl stretching vibration (C=O) band was discovered at 2116 cm^{-1} while a weaker peak with an absorbance of 1557 cm^{-1} is characteristics of C=C bond of the saponin. Fig. 3 depicts the chromatogram profile of saponin in VA leaves. SBNS showed multiple retention time of 1.8, 1.9, 2.5, and 3.6 min which corresponds to its saponin constituents.

3.2. Conductivity measurement of the surfactant

Several studies on the utilization of surface-active components have shown that property of the bulk solution is dependent on the concentration of the material used. Besides, these observations have proven that micelles are formed in surfactant solution. Critical micelle concentration (CMC) also referred to as the optimum surfactant concentration is the concentration at which surfactant molecules aggregate in the bulk of the solution due to saturation. Below the CMC, the surfactant molecules are arranged at the liquid interface such that they form a stable emulsion. Above the CMC, desorption of surfactant from the interface occurs resulting in an increase in IFT (Kamal et al., 2017). Herein, conductivity measurement was used to determine the CMC by varying surfactant concentration in the range of 0.01–3.5 wt%. The experimental result of the conductivity test is presented in Fig. 4. The CMC of SBNS was demonstrated to be 1.1 wt%. Accordingly, an approximate value of 1 wt% was considered as the CMC of SBNS in this study.

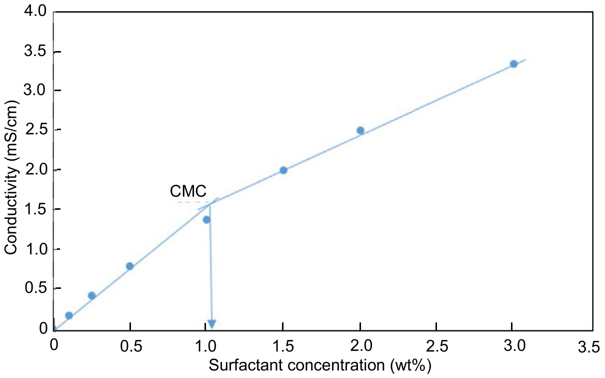

Fig. 4. Conductivity measurement of SBNS.

3.3. Foaming property of SBNS

Due to surfactant inherent properties, they can generate and stabilize foam (Gbadamosi et al., 2019b). The use of foam in oil reservoirs cannot be overestimated. Foams ensure fluid diversion of injectant from high permeability regions to low permeability regions of the reservoir (Yekeen et al., 2018). Asides that, the presence of an aqueous phase and foaming agent creates a favourable mobility ratio in oil reservoir (Rafati et al., 2016). Hence, the foam stability of SBNS (1 wt%) was estimated and illustrated in Fig. 5. SBNS show good characteristic in stabilizing foams for longer period. This can be adduced to the adsorption of the surfactant at the gas-liquid interface of the foam. Initially, SBNS foam volume increases linearly until it reaches its maximum foam height of 32 cm^3. Subsequently, the foam volume decreases with increasing time intervals. Nonetheless, the foam remains quite stable for longer periods due to high viscosity of the foam films. This may be attributed to the smaller foam diameter of SBNS which prevents it from three mechanisms of foam destruction which are coalescence, coarsening and liquid drainage. The half-life of SBNS foam was recorded at 1100 min. With its longer foam stabilizing property, this mechanism may account for the higher oil displacement property when SBNS is utilized as an injectant in oil reservoirs.

3.4. Effect of SBNS on IFT of oil-water interface

The behaviour of surfactant at the oil-water interface is important in estimating the ability of the injectant to lower capillary force between the immiscible fluids, and subsequently improve pore scale displacement efficiency (Agi et al., 2018; Abbas et al., 2021). The IFT behaviour at the oil-water interface of the synthesized SBNS was determined and depicted in Fig. 6. The IFT of the oil-water interface decrease with increasing surfactant concentration up to 1 wt%. Notably, at 1 wt% SBNS concentration, the IFT was reduced from 18 mN/m to 4.1 mN/m. Below the CMC, the amphiphilic dual structure of the surfactant adsorbs at the oil-water interface with the hydrophobic section dissolving in oil and the hydrophilic section dissolving in aqueous layer. Hence, increasing surfactant concentration causes more surfactant molecules to migrate towards the oil-water interface, thereby, causing their adsorption and interaction at the interface. Consequently, IFT reduction occurs at the oil-water interface.

Nonetheless, above 1 wt% surfactant concentration, the IFT reduction was truncated and slightly increases with increasing SBNS concentration. Beyond the CMC, the concentration of surfactant molecules at the interface becomes saturated and micelles are formed. Moreover, the formation of denser surfactant monomers at the interface prevents the further migration of surfactant molecules from the bulk solution. The micelles formation diminishes the surfactant activity at the oil-water interface. Thus, no

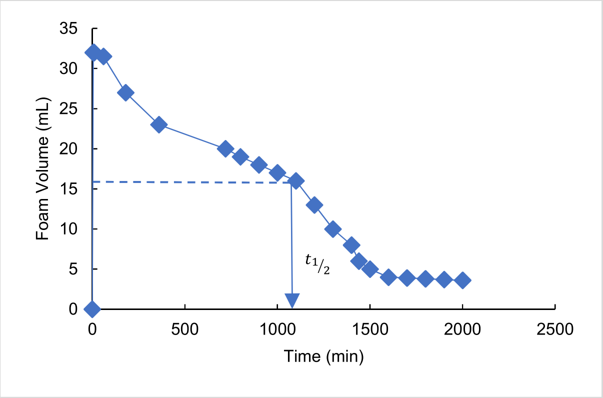

Fig. 5. Foam decay profile of SBNS at 1 wt% concentration.

significant change was observed in the IFT of the SBNS above the CMC. Additionally, the IFT behaviour of the synthesized SBNS was compared to *Zizyphus Spina Christi* (a natural surfactant also referred to as Cedar Extract (CE)) reported by Emadi et al. (2019) and illustrated in Fig. 7. SBNS showed comparatively similar behaviour to CE surfactant at the oil-water interface.

3.5. Effect of salinity on IFT of SBNS

The effect of different concentration of NaCl (0.9 wt%, 1.5 wt%, 2.0 wt%, 2.2 wt%, 2.5 wt% and 3.0 wt%) on the IFT behaviour of SBNS was studied and reported in Fig. 8. The presence of NaCl reduces the IFT at the oil-water interface further to 2.9 mN/m, 2.0 mN/m, 1.2 mN/m, 0.97 mN/m, 1.2 mN/m and 1.5 mN/m respectively. This can be adduced to salt-in effects. The inorganic salt breaks the structure of the water, thereby, increasing the solubility of the organic components of the oil in the aqueous phase. Besides, the presence of the ions increases the adsorption of the surfactant at the oil-water interface. Hence, the IFT of the oil-water interface reduces further (Nowrouzi et al., 2021c). The IFT of the oil-water interface was lowest at optimum salinity of 2.2 wt% NaCl where in the surfactant dissolves equally in aqueous and oleic phase resulting in the minimum IFT. Nonetheless, above the optimum salinity concentration, the IFT of the oil-water interface increases with increasing brine concentration. Increasing brine concentration prevent the surfactant from dissolving into the aqueous phase because of formation of repulsive electrostatic double layer and repulsive hydration effect (Arabloo et al., 2016; Bera et al., 2020). This causes the movement of surfactant away from the oil-water interface, thus, resulting in an increase in IFT (Kamal et al., 2017).

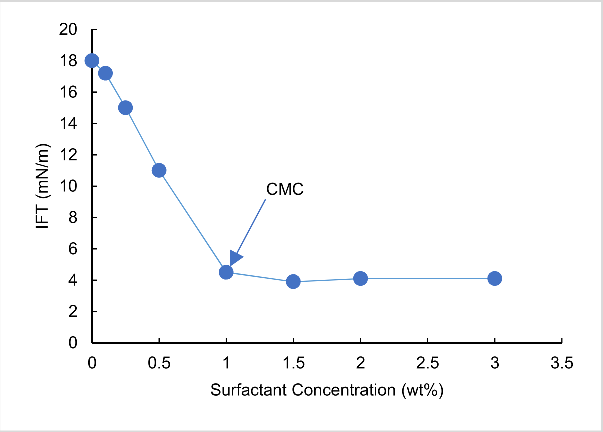

Fig. 6. IFT of SBNS as a function of surfactant concentration.

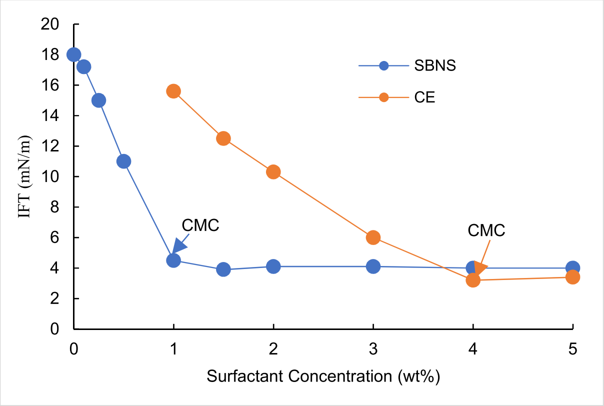

Fig. 7. Comparative analysis of SBNS with Cedar Extract.

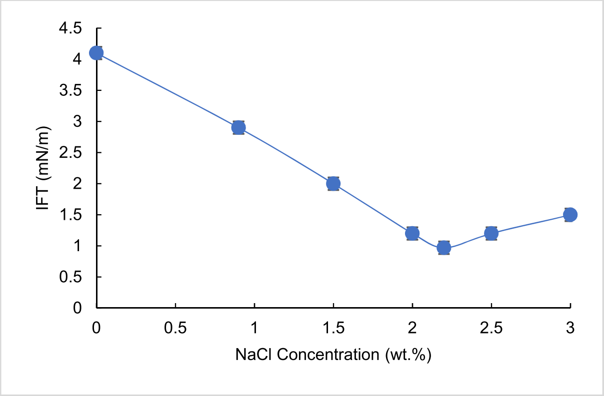

Fig. 8. Effect of salinity on IFT of SBNS.

3.6. Impact of SBNS on wettability of porous media

Wettability is one of the pore scale displacement characteristics of an oil reservoir. An oil reservoir may be oil-wet, water-wet, or

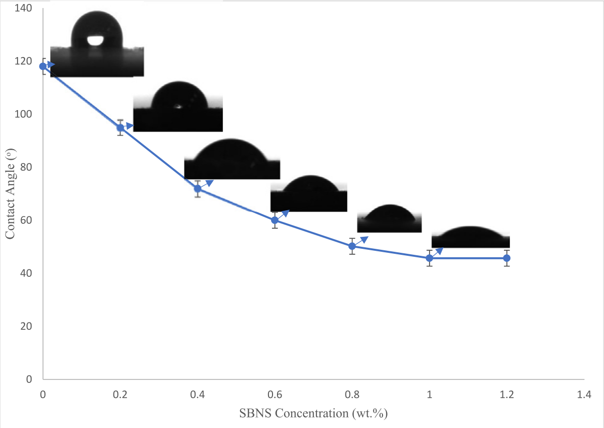

Fig. 9. Effect of SBNS on wettability of sandstone cores.

mixed-wet. A porous media is oil-wet when the contact angle $\theta >$ 90°, mixed wet when $\theta \cong 90^{\circ}$, and water-wet when $\theta <$ 90°. Water-wetting condition have been posited to be desirable and more suitable for oil production. The contact angle of water droplet on the rock was measured as a function of increasing surfactant concentration and illustrated in Fig. 9. In the absence of surfactant, $\theta =$ 118.5° depicting an oil-wet status of the rock. The addition of 0.2 wt% of SBNS lowered the contact angle to 95 °, implying that lower SBNS concentration has little effect of altering the wettability. By increasing the SBNS concentration to 1 wt%, the contact angle $\theta = 45.7^{\circ}$ depicting that increasing surfactant concentration has altered the wettability of the sandstone core to a water-wetting condition. The accumulation of surfactant at the oil-rock interface is accompanied by hydrophobic interaction of the surfactants and oil molecules. This causes the disruption of the upper oil layer on the rock surface. Moreover, the interaction between the hydrogen bond constituent of the non-ionic surfactant and the rock leads to surfactants adsorption on the cores and subsequent oil removal through two major mechanisms of cleaning and coating. Surfactants remove oil molecules from sandstone surface and surfactant molecules adhere on the clean surface (Standnes and Austad, 2003; Hou et al., 2015). Hence, oil is detached from the rock, thereby, creating a water-wetting condition. As the concentration of the surfactant increases, the contact angle decreased further.

4. Conclusion and recommendation

This study investigated the use of SBNS extracted from *Vernonia Amygdalina* (VA) leaves as an EOR agent. Based on the results:

- The foaming, IFT and wettability properties of SBNS were investigated. SBNS exhibited good EOR potentials.
- The foam stabilized using SBNS surfactant lasted for several hours with its half-life estimated as 1100 min.
- The CMC of the surfactant was determined as 1.0 wt% which is lower compared to other natural surfactant reported in previous study. This implies desired efficiency can be achieved with lower quantity of SBNS from *VA* leaves.
- At CMC, the IFT of the fluid-fluid interface reduced from 18.0 mN/m to 0.97 mN/m. Furthermore, SBNS lowered the IFT of oil water interface and showed good stability in the presence of brine.
- Additionally, the use of SBNS altered the wettability of sandstone cores from oil-wetting to water-wetting condition. The contact angle of the fluid-rock interface reduced from 118.5 ° to 45.7 °.
- Future research should compare the behaviour of this green surfactant to commercially available ones. Moreover, oil displacement test and emulsion behaviour of the surfactant synthesized from *VA* leaves should be investigated.
- Overall, this surfactant is recommended as a good substitute for conventional surfactants due to its good efficiency and environmentally benign nature.

Declaration of competing interest

The authors declare no conflict of interest.

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