

RESEARCH PAPER

The phase behaviour and stability of an environmentally friendly oil-in-water emulsion produced using *Balanites aegyptiaca* oil and green emulsifiers

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Abstract

Due to environmental concerns about extensively using chemical emulsifiers and hydrocarbon oil in emulsion systems formation, recent research has shifted to using natural emulsifiers and oil. This study produced green emulsion using *Balanites aegyptiaca* oil and various bio-emulsifiers (such as sodium alginate, carboxymethylcellulose, guar gum, and Saponin). Phase behavior and stability of the emulsion were tested at different NaCl concentrations over one month of ageing. The emulsion was also produced using *Balanites aegyptiaca* oil and synthetic emulsifiers (Tween 80 and Triton X-100). The critical micelle concentrations of emulsifiers were determined and used as the fixed concentration for the green emulsion preparation. In addition, the properties of aqueous emulsifiers, particularly interfacial tension and contact angle, were studied for solid-liquid and liquid-liquid systems. Phase behaviour stability test confirmed the potential of sodium alginate and carboxymethylcellulose in the formation of emulsions with 5.5 ml volume, Winsor type II phase behavior similar to synthetic emulsifier Triton X-100 at different NaCl concentrations was obtained. Furthermore, the stability of the formed emulsions is independent of the NaCl concentration ranging from 0.5 to 3.5 g/100 ml. The equilibrium time for all the as-prepared green emulsion systems was one month. Depending on the type of emulsifier, the phase stability of the formulated emulsion systems prepared in this study ranks from (Sodium Alginate ~ Carboxymethylcellulose ~ Triton X-100~Tween 80~Saponin ~ Guar Gum). The findings revealed the potential to produce green emulsion using *Balanites aegyptiaca* oil and bio-emulsifier. Such an environmentally friendly emulsion system can be utilized in enhanced oil recovery.

Keywords: Bio-emulsifier, Enhanced oil recovery, Green emulsion, Phase behavior, Sonication

1. Introduction

Enhanced oil recovery (EOR), a tertiary recovery, is a process to extract crude oil from the reservoir that the primary and secondary recovery methods could not achieve unless an external force or a substance—not initially available in the reservoir—were applied.¹ It was reported that a proper EOR might further recover 30–60% of the remaining oil

in the reservoir.² In particular, EOR via chemical injection produces oil more efficiently than gas and thermal injection.³ The typical chemical substances that are being injected into the reservoirs are polymer,⁴ surfactant,⁵ and alkali.⁶ However, due to the elevated cost of polymer and surfactant, another method, such as a pre-prepared emulsion for EOR, has been developed to achieve the required objective.

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The pre-prepared emulsion creates an emulsion with ultra-low interfacial tension via the mixing of surfactant, co-surfactant, oil, water, and brine; the emulsion is prepared and ready before being injected into the reservoir. Researchers have tested the use of different types of oil under various conditions, such as high salinity ranges, high pressures, and high temperatures, to produce a low interfacial tension solution.^{7,8} Noteworthy that there is significant proof that micro-emulsions with higher water content are more efficient at reservoir conditions than those with higher oil content. Therefore, the oil-in-water type of micro-emulsion is preferred in EOR applications. Oil-water IFT, emulsion quality, stability, droplet size, and oil phase viscosity could further characterize emulsion properties. These properties of emulsion have been observed to affect both displacement and sweep efficiencies mechanisms.^{9–11}

Despite numerous research on emulsion flooding, it is still in the experimental laboratory stage. With very few emulsion flooding implementations reported in the literature, the Midway-Sunset Oil Field's Section 5 K field test is the most successful example of emulsion flooding implemented in field tests. Environmental pollution, in general, has a more significant impact on the development of chemical flooding technology. Future chemical flooding research should prioritize environmental protection with minimal oil pollution and damage over incremental recovery. Several studies have achieved stable emulsion formulation using a mixed system of different types of hydrocarbon oils with different emulsifiers, such as gear oil without surfactant,¹² crude oil with Tween 80 and Span 60 emulsifiers,¹³ mineral oil with Carboxybetaine-based zwitterionic emulsifier, and gasoline combined with synthesized sea water and sodium dodecyl sulphate (SDS).¹⁴ Most of the experimental test results for characterization, mechanisms, and emulsion stability showed promising potential for application in chemical EOR. Unfortunately, the main environmental and cost concerns widely limit the implementation of these hydrocarbon-based emulsions in oil fields. Therefore, formulating an environmentally friendly emulsion system for EOR is necessary; particularly, it is essential to speed up the development and production of emulsifiers with high efficiency, green, low cost, and good emulsifying properties.¹⁵ Researchers are now looking into the feasibility of producing emulsions using bio-emulsifier.

The emulsion can be generated in-situ in the reservoir or pre-generated before being injected into the reservoir to enhance the extent of oil recovery. The commonly used oil phase for emulsion generation is light hydrocarbon oil, then mixed with

diluted surfactant water phase. Recently, different types of bio-oils, such as pine oil,^{16,17} neem oil,^{18,19} palm oil,^{20,21} and jatropha oil²² have been used to generate emulsions. The availability of various edible and non-edible oils can be employed as the oil phase to replace the commonly used light hydrocarbon oil exploited to reduce the operational cost of the chemical EOR process.

Due to environmental concerns about chemical surfactants, numerous studies have been attended to increase the implementation of environmentally friendly surfactants.^{23–25} Using bio-emulsifier is significant in the performance of sustainable industrial processes as they are obtained from renewable resources. Biodegradability and low toxicity have prompted researchers to focus on biosurfactant applications in the petroleum, bioremediation, agricultural, cosmetic, chemical, health, and food processing industries.

This study investigates the potential of utilizing *Balanites aegyptiaca* oil, an underutilized non-edible oil extracted from desert date plants, as the oleic phase for emulsion preparation. The desert date plants which commonly found in African and Middle Eastern countries are emphasized as a sustainable source for the oleic phase. The study aimed to assess the emulsifying capabilities of bio-oil when combined with various bio-emulsifiers which include sodium alginate (SA), carboxymethylcellulose (CarboxMC), guar gum (GG), and Saponin, under varying NaCl concentrations. The study goes further to compare the stability of the formulated bio-emulsion with emulsions formed using *Balanites aegyptiaca* oil and synthetic emulsifiers, namely Tween 80 and Triton X-100 (TX100), providing practical insights for industries seeking eco-friendly alternatives. Here, the emulsification capability of *Balanites aegyptiaca* oil/various surfactants/aqueous systems were investigated utilizing stability tests (i.e., phase behavior) at various time intervals. Surface tension studies were carried out to evaluate the critical micelle concentration (CMC) of surfactants in aqueous media. In addition, the properties of emulsifiers aqueous were studied, particularly the interfacial tension between the emulsifier aqueous solution and mineral oil, and the contact angle for solid-liquid and liquid-liquid systems.

2. Methodology

2.1. Materials

Four bio-emulsifiers (CarboxMC, SA, GG, and Saponin) and two synthetic emulsifiers (TX-100 and Tween 80) were utilized, and their properties are

shown in (Table 1). This study used distilled water as the aqueous phase for all solution and emulsion preparation. Furthermore, the oil of *Balanites aegyptiaca* was collected from a Sudanese locality (Omdurman) a local market and used as the oleic phase.

2.2. Critical micelle concentration (CMC) measurement

In order to know the emulsifier concentration at which micelle formation is first seen for all of the bio-emulsifier, the surface tension of the bio-emulsifiers was measured using the platinum ring detachment method on a Du Nouy ring tensiometer (SEO DST 30 Surface tension meter, Korea). Here, emulsifier solutions with concentrations ranging from 0.0001 g/ml to 0.01 g/ml were prepared for the measurement. Worth noting that this value is lower than the range reported by Jeirani *et al.*,²⁰ which was in the field of 0.005–0.04 g/ml of emulsifier.

In detail, a sample of 100 ml distilled water was measured and transferred to other beakers, and a magnetic bar was inserted into the beakers containing the distilled water and placed on a stirrer at 700 rpm. Emulsifier concentrations (0.0001, 0.0005, 0.001, 0.002, 0.005, and 0.01) g/ml were prepared by dissolving a known amount of the emulsifier in the beakers containing 100 ml distilled water. The solution was stirred for 30 min at a room temperature of 25 °C to complete the dissolution. This procedure was followed for the bio-emulsifiers solution (Carboxymethylcellulose, Sodium Alginate, Saponin). Unlike the other emulsifiers investigated in this study, the maximum concentration of Guar Gum was set at 0.007 g/ml instead of 0.01 g/ml due to the formation of a highly viscous solution in the later concentration. Then, a beaker with 50 ml of the as-prepared emulsifier solution for each surface tension measurement was allocated in the Du Nouy tensiometer instrument. The Du Nouy ring was slowly inserted into the liquid surface and pulled slowly from the solution to determine the surface tension. The average value was determined by three surface tension measurement readings for each emulsifier solution sample. The CMC was then determined by plotting the surface tension breakpoint against the logarithm of the concentration curve.²⁸

2.3. Contact angle measurement

The wetting capacity of utilized emulsifiers solutions was determined by measuring static contact angle of sessile droplets of each emulsifier solution onto a glass surface using The Phoenix 150 Contact Angle Analyzer Operated under Surfaceware 8 at

Table 1. The properties of the emulsifiers used in this study.

Emulsifier name	Type	Natural/ Chemical Origin	CMC, g/100 ml	Hydrophilic lipophilic balance (HLB)	Surface tension (mN/m)	MW, g/mol	Hydrophobic/ Hydrophilic/ Amphiphilic
CarboxMc	Anionic (anionic polyelectrolyte)	Natural	Well be Determined in this study	–	Well be Determined in this study	250 000	Hydrophilic
SA	Anionic (anionic polyelectrolyte)	Natural	Well be Determined in this study	–	Well be Determined in this study	216.12	Hydrophilic
GG	Non-ionic and hydrocolloidal	Natural	Well be Determined in this study	–	Well be Determined in this study	535.15	Hydrophilic
Saponin	Non-ionic	Natural	Well be Determined in this study	12.7–19.9	Well be Determined in this study	–	Amphiphilic
Triton X-100	Non-ionic	Synthetic	0.0194	13.5	30 ²⁶	625	Amphiphilic
Tween 80	Non-ionic	Synthetic	0.0025	15	35 ²⁷	1310	Amphiphilic

25 °C. The same experiment was repeated to study the contact angle between the emulsifier's solutions and oil-wet glass surface. The oil-wet glass surface was produced by painting the glass slide with light mineral oil. To calculate the contact angle, 2.5 ml of emulsifier solution was withdrawn into a syringe and carefully dropped onto the surface of glass and oil wet glass. As baseline reference to analyse the achieved contact angles results between the distilled water glass, and oil wet glass was conducted. All tests were conducted automatically five times, with the average value calculated and the process repeated three times to ensure consistency in the results for individual samples.

2.4. Interfacial tension measurements

Interfacial tension (IFT) significantly affect the development of the interfacial area between the dispersed and continuous phases in a sheared emulsion, In this study, IFT was measured by Du Nouy ring tensiometer (SEO DST 30 Surface tension meter, Korea). Light mineral oil was used as the outer phase and various aqueous solution (CarboxMC, SA, GG, Saponin, TX-100, and Tween 80) was used as the inner phase. All the aqueous solutions were prepared by dissolving the optimized NaCl and the different emulsifiers mixtures at CMC concentration in distilled water. In addition, one aqueous solution was prepared by dissolving the optimized NaCl without emulsifiers for the purpose to compare IFT results before and after and the addition of an emulsifier. Each sample was measured three times. Finally, the IFT results presented in this study are an average of three measurements for each of the liquid/liquid IFT test systems evaluated.

2.5. Preparation of emulsifier solution

A sample of 100 ml of distilled water was measured and transferred to other beakers. A

known amount of NaCl was weighed using a beam balance and dissolved in 100 ml of distilled water to prepare a salt solution of 0.005, 0.015, 0.02, 0.025, 0.03, and 0.035 g/ml concentrations. The solution was magnetically stirred at 700 rpm for 60 min to complete the NaCl dissolution. The emulsifiers were weighted at CMC concentrations because it is efficient and economical to use emulsifiers at CMC concentrations during EOR application.²⁹ The emulsifier was then added to the prepared salinity water and thoroughly mixed at 1500 rpm for 60 min at room temperature (25 °C).

2.6. Preparation of emulsion and time-lapse phase separation evaluation

The as-prepared emulsifier solution at different salinity ranges was added to *Balanites aegyptiaca* oil at a constant 5:5 (ml) volume ratio. The mixture was then sonicated for 3 min using the Hielscher UP200S ultrasonic device at one cycle and 75% amplitude to create a homogeneous mixture or emulsion. Then, the formed emulsions were observed for 1 h, 5 h, three days, and one month at room temperature (25 °C) to determine phase separation or equilibrium time (i.e., stability time of the emulsion phase at different NaCl concentrations). The observed emulsion phases were photographed and subjected to further phase behavior analysis.

3. Results and discussion

3.1. Fatty acids profile of *Balanites aegyptiaca* oil

Table 2 shows the summary of the free fatty acid (FFA) and fatty acid methyl esters (FAME) content of the *Balanites aegyptiaca* oil used in this study. The FFA and FAME were tested using AOCCS official method Ca 5a-40 and AOAC 996.06 methods, respectively. FAME major compositions found from the test were methyl *cis*- 10 pentadecenoate (C15:1),

Table 2. FFA and FAME content of the *Balanites aegyptiaca* oil used in this study.

Parameters	Composition analysis results, %	Test method
FFA (as Oleic Acid)	1.97	AOCS Official Method Ca 5a-40
FAME Profile		
Methyl <i>cis</i> - 10 pentadecenoate (C15:1)	7.36	In-House based on
Methyl <i>cis</i> - 10 heptadecenoate (C17:1)	6.07	AOAC 996.06
Methyl stearate (C18:0)	0.20	
Methyl <i>trans</i> -9 eladiate (C18:1)	23.33	
Methyl linolelaidate and linoleate (C18:2)	20.02	
Methyl <i>cis</i> -4, 7, 10, 13, 16, 19-docosahexaenoate (C22:6)	0.92	
Total	57.9	

methyl *cis*- 10 heptadecenoate (C17:1), methyl stearate (C18:0), methyl *trans*-9 eladiate (C18:1, the most abundant one), methyl linolelaidate and linoleate (C18:2), and methyl *cis*-4, 7, 10, 13, 16, 19-docosa-hexaenoate (C22:6). In total the *Balanites aegyptiaca* oil contained 57.9% methyl ester and has a low acidity of 1.97% the low acidity indicates that this bio-oil can be used as a potential green material to produce an emulsion system for EOR, as previous studies successfully used various edible and non-edible oils in emulsion-EOR experimental scale applications (such as *Jatropha* oil, *Neem* oil, *Palm* oil, and *Pine* oil) with FFA% (2.23 ± 0.02), (1.22 ± 0.029), (2.5) and (89.1 ± 1.9), respectively.^{30–33} Therefore, *Balanites aegyptiaca* oil deserves research attention to investigate its feasibility in EOR.

3.2. Determination of critical micelle concentration (CMC)

A surfactant will spontaneously form micelles at concentrations above CMC, causing two phenomena: (1) solubilization and (2) emulsification. The formation of micelles with concentration above the CMC is undesired as it will compromise the function of the surfactant in stabilizing an emulsion. Accordingly, it's critical to determine or comply with the CMC value for each surfactant employed in emulsion formation for EOR application. According to Nourafkan *et al.*,³⁴ highly concentrated micelles result in a clouded and unstable surfactant slug during enhanced oil recovery process, which is not recommended for injection. Pal *et al.*³⁵ reported that surfactant concentration at (CMC) causes smaller droplet size of the formed emulsion, whereas higher concentrations lead to larger droplets due to micelle aggregation in the emulsion phase. Furthermore, Pal *et al.*³⁶ presented the major difference between

small and large emulsion droplet sizes in terms of emulsion stability; the smaller emulsion droplet size is thermodynamically and kinetically stable, whereas a larger emulsion is thermodynamically unstable and weakly kinetically stable. As a result, the larger emulsion droplet size caused by the formation of micelles with concentrations above the CMC could yield an unstable emulsion system, potentially leading to an undesirable emulsion-EOR agent. More importantly, CMC identification will assist in obtaining the minimum concentration that may be utilized to minimize the consumption of surfactants from economic, technical, and environmental perspectives. Moreover, the CMC is to be identified in the current study because it is a vital characteristic of surfactants, an indicator of surfactant efficiency, emulsion formation, and stability.

In this respect, the CMC of CarboxMC, SA, GG, and Saponin emulsifiers were determined through surface tension analysis; for comparison purposes. Fig. 1 shows the surface tension and concentration of the surfactant relationship curve of the investigated emulsifiers. As shown in Fig. 1, GG, SA, and Saponin experienced a decrement in the magnitude of surface tension when their concentrations were increased. Upon reaching a critical point, there was negligible change in the surface tension value. On the other hand, CarboxMC showed the opposite trend.

Due to emulsifier dispersion onto the distilled water, a rapid reduction in surface tension can be observed at low emulsifier concentrations, 0.001–0.002 g/ml. The surface tension magnitude of GG and SA emulsifiers dropped from 61 to 48 mN/m and 49 to 42 mN/m, respectively. This rapid degree for both emulsifiers stopped at 0.002 g/ml, implying that the CMC concentration is approximately 0.0025 g/ml.

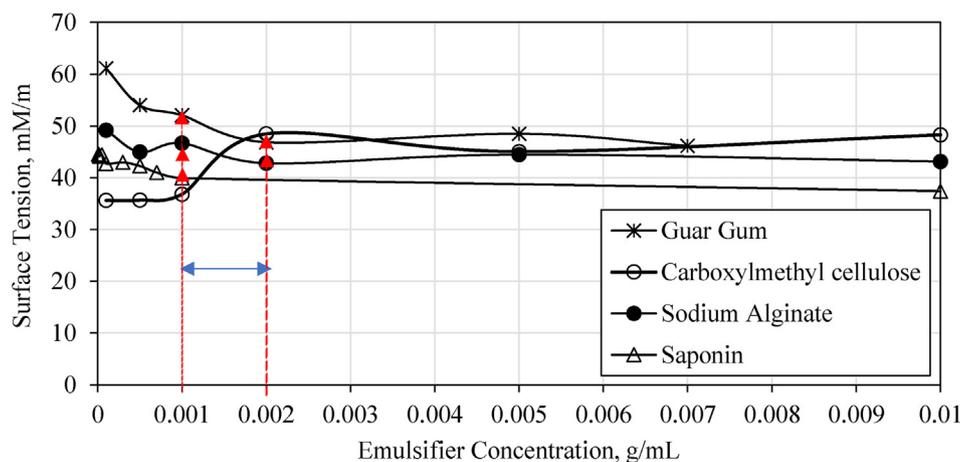


Fig. 1. Surface tension measurements of CarboxMC, GG, SA, and saponin.

A similar trend was observed for the Saponin emulsifier. Here, it was noticed that the surface tension rapidly decreased from 44 to 41 mN/m, with a lower value observed at 0.001 g/ml concentration, and the CMC value was estimated to be around 0.0007 g/ml. Our result agrees with the CMC values Alpandi *et al.*³⁷ reported, ranging from 0.003 to 0.072 wt% or 0.00003–0.00072 g/ml.

Noteworthy that the CarboxMC emulsifier demonstrated an opposite trend whereby the surface tension of CarboxMC showed an increment when the concentration was increased from 0.0001 g/ml to 0.002 g/ml; after that, there were not many changes in the surface tension value even though the concentration of CarboxMC was increased to 0.01 g/ml. This opposite trend as compared to GG, SA, and Saponin can be ascribed to polymeric structure (polysaccharide) and high molecular weight of CarboxMC. According to Traibelsi and Langevin,³⁸ previous investigations have shown that the CarboxMC emulsifier (when utilized alone) is not surface-active below a concentration of 7 g/l (0.007 g/ml). This may also explain why the

surface tension of CarboxMC decreases at low concentrations. However, the CarboxMC emulsifier's surface tension was assumed to be constant at 0.002 g/ml concentration, and the CMC was estimated to be approximately 0.0025 g/ml.

The partial similarity of the three studied bio-emulsifiers' structures and physical properties may explain why their CMC values are near each other. Fig. 2 shows the chemical structure of GG, SA, Saponin, and CarboxMC.

3.3. Determination of contact angle

Two contact angle measurements were performed in this study: First, consider the contact angle between the glass surface and distilled water with and without emulsifiers. Second, the contact angle of mineral oil and distilled water with and without the presence of an emulsifier solution. Firstly, the contact angle between the aqueous solution containing emulsifiers and the solid glass surface is shown in (Table 3). As can be seen from (Table 3), all emulsifiers show lower contact angles compared to

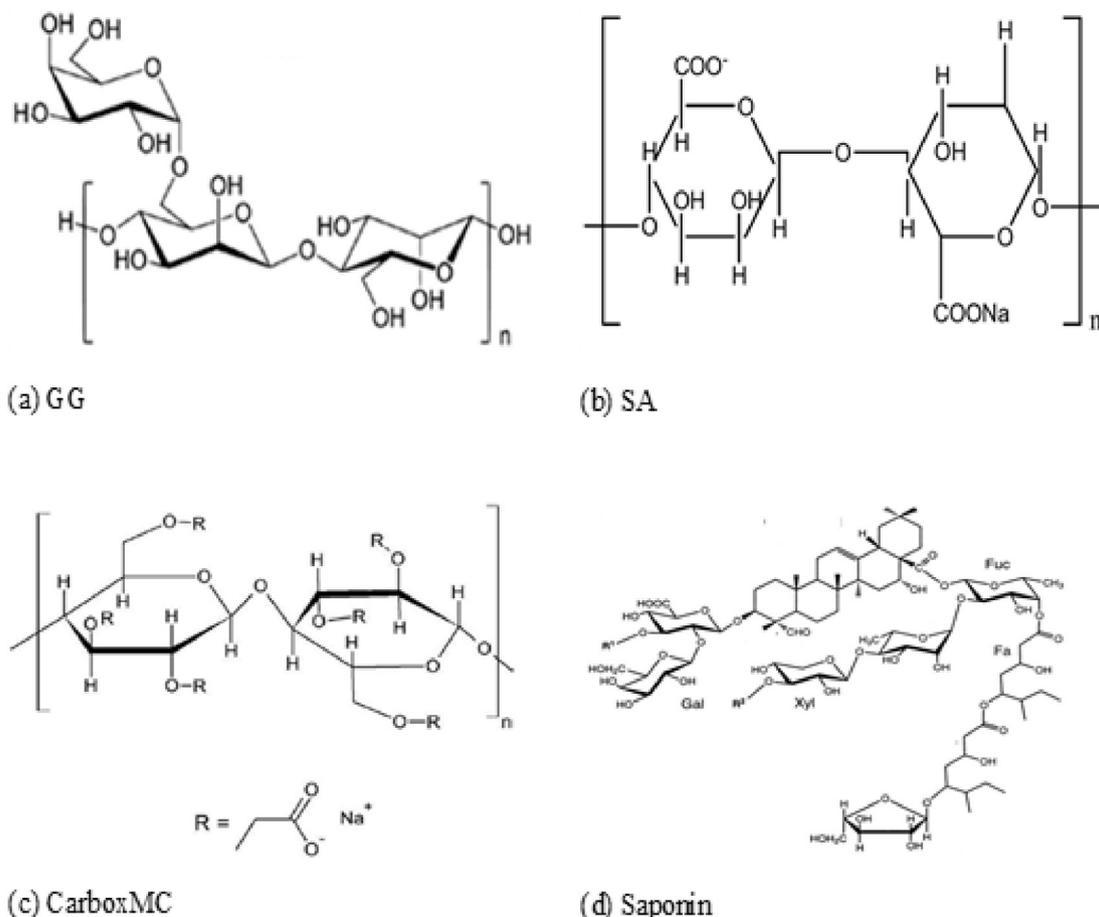


Fig. 2. Chemical structure of (a) GG, (b) SA, (c) CarboxMC, and (d) saponin.

Table 3. Contact angles of emulsifier aqueous solution: comparisons on solid-liquid and liquid-liquid systems.

Emulsifier at CMC	CarboxMC	SA	GG	Saponin	TX-100	Tween 80	Baseline reference
Contact angle (°), for Solid-Liquid system	26.2	31.0	26.76	26.82	20.9	34.9	37.6 (Distilled water)
Contact angle (°), for Liquid-Liquid system	34.2	34.4	29.2	28.9	12.4	26.3	30.9 (Mineral oil/Water)

distilled water which measured as 37.6° , this result is in line with Al-Sulaimani *et al.*,³⁹ whom reported that the contact angle of distilled water on a smooth surface ranges from $25.32^\circ \pm 0.06^\circ$ – $70.6^\circ \pm 0.3^\circ$. The results of the phase contact angle of the emulsifiers used in this study range from 20.9° for TX-100 to 26.2° for CarboxMC, 26.67° for GG, 26.82° for Saponin, 31° for SA, and 34.9° for Tween 80. The contact angle between the aqueous solution containing emulsifiers was significantly lower when the emulsifier was added in aqueous solutions. This indicates that potential of the emulsifiers used to modify wetting behavior has significant wetting properties. Secondly, the contact angle between the liquid-liquid (Mineral oil-aqueous with/without emulsifier) is measured to investigate the ability of the used emulsifier to change wettability from oil-wet to water-wet, which is an effective mechanism of emulsifiers in EOR; the results of this contact angle measurement are reported in (Table 3). The non-ionic emulsifier type had a lower contact angle (TX-100 with 20.9° , Tween 80 with 34.9° , Saponin with 26.82° s, and GG with 26.67°) compared to the measured distilled water-mineral oil surface contact angle (30.09°). However, the contact angle for anionic emulsifiers is higher for CarboxMC with and SA, with value of 34.2° and 34.4° , respectively. According to Hosseini *et al.*,⁴⁰ this finding indicates non-ionic emulsifiers have more effect on changing wettability towards water-wet than anionic emulsifiers.

3.4. Determination of IFT

Emulsifiers have both hydrophobic tails and hydrophilic heads, which allows them to adsorb to the interface of two fluids and effectively reduce the interfacial tension between oil and water. In this study, the equilibrium IFT between the aqueous solution (with and without emulsifiers) and mineral

oil was measured for the six emulsifiers at their CMC with 3.5 g/ml and 2.5 g/ml of NaCl. Table 4 shows the analysis results. TX-100 (-0.22 mN/m), Tween 80 (0.52 mN/m), and Saponin (0.88 mN/m) emulsifiers showed a significantly reduced equilibrium IFT with mineral oil than GG (2.16 mN/m), SA (2.21 mN/m), and CarboxMC (2.94 mN/m). The superior performance of TX-100, Tween 80, and Saponin in lowering IFT is can be attributed to their amphiphilic properties. In addition, in (Table 4) the negative value of IFT resulted in this study between mineral oil and TX-100 with NaCl. In practice, negative interfacial tension is uncommon in many systems since it contrasts the natural tendency of phases to minimize their contact areas. However, there may be specialized conditions or systems where such an instance is possible. Zabar *et al.*⁴¹ attributed negative interfacial tension to the following mechanisms. (i) interfacial turbulence, and (iii) diffusion-stranding transient passive interfacial tension expansion. Furthermore,⁴² reported that a negative interfacial tension exists since a two-phase solution to mixed completely. According to Zabar *et al.*⁴¹ and Della Volpe,⁴² an unstable thin layer formed between the interfaces of TX-100 emulsifier molecules and mineral oil phase may explain the instantaneous negative IFT between mineral oil and Triton X100 aqueous. However, confirming the negative value of the IFT for mineral oil/TX-100 aqueous with existing data is difficult, and requires further investigation.

Interesting to note that the carboxMC IFT for mineral oil-distilled water with an emulsifier is slightly higher than that for mineral oil-distilled water without an emulsifier. Such observation can be attributed to the polymeric nature and the maximum molecular weight ($250\ 000$ g/mol) of carboxMC solution which made it tend to have a higher viscosity than distilled water. This increased

Table 4. Interfacial tension analysis of mineral oil/aqueous systems with and without emulsifier.

	Mineral oil/aqueous system	GG	SA	CarboxMC	Saponin	TX-100	Tween 80
Emulsifier IFT (mN/m) at CMC and optimized NaCl (mN/m)	Aqueous solution without emulsifier	3.49	2.39	2.39	2.39	2.39	2.39
	Aqueous solution with emulsifier	2.16	2.21	2.94	0.88	-0.22	0.52

viscosity may affect the spreading and contact dynamics at the interface, perhaps increasing interfacial tension with mineral oil slightly.

3.5. Phase behavior study for bio-emulsifiers

During the injection of the generated emulsion for EOR, the emulsion will reduce the amount of water breakthrough present, alter the pore level, and change the microscopic channels. Therefore, the following sections are focused on evaluating the feasibility and stability of an emulsion system with three bio-emulsifiers, namely CarboxMC, SA, GG, and Saponin, at various salt concentrations and equilibrium times at room temperature.

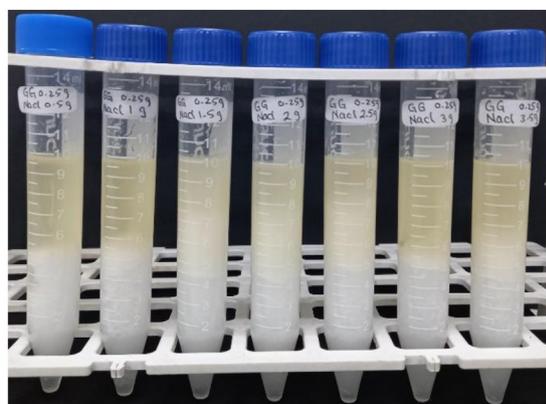
3.5.1. Guar gum (GG) as bio-emulsifier

The GG emulsifier system was evaluated at various salt concentrations at room temperature to evaluate the feasibility of emulsion formation by mixing *balanites* oil with GG emulsifier in an

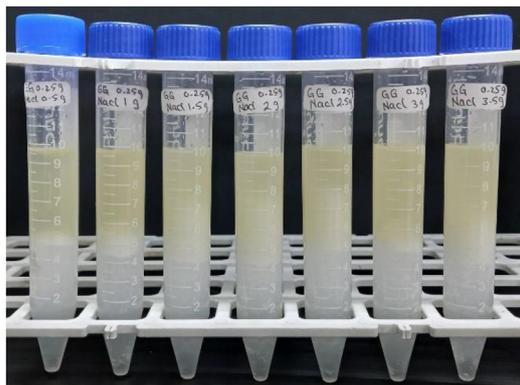
aqueous solution containing salt. In addition, to examine the stability of generated emulsion that can be used to improve the performance of the emulsion EOR process. Fig. 3 delineates the time-lapse phase behavior of the emulsion formed using 0.25 g/100 ml of GG in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml. Winsor type III emulsion was successfully formed at different salt concentrations because three solution layers (i.e., oleic (*balanites* oil) phase, separated emulsion phase, and water phase) were observed. The milky color emulsion dissolved gradually after 1 h, 5 h, and three days of equilibrium time, as shown in (Fig. 3a–c). The emulsion almost entirely turned into a clear oil and water phase after one month of equilibrium, as shown in (Fig. 3d); less than 50% of emulsion remained in the emulsion prepared in (1.5, 2, and 2.5) g/100 ml NaCl. In short, GG bio-emulsifier can form Winsor type III emulsion with *balanites* oil. However, the stability of the emulsion is relatively poor.



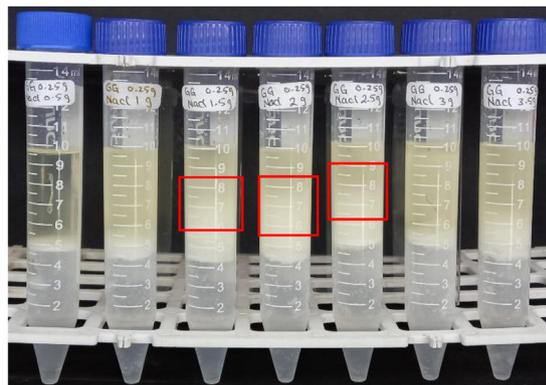
(a) After 1 hour



(b) After 5 hours



(c) After three days



(d) After one month

Fig. 3. Phase behavior of emulsion formed using 0.25 g/100 ml of GG emulsifier in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml.

3.5.2. Sodium alginate (SA) as bio-emulsifier

Next, a phase behavior study was conducted on the emulsion formed using 0.25 g/100 ml of SA as an emulsifier. Similarly, the study was conducted in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml. As shown in (Fig. 4a), the emulsions were present in full milky color within the first hour after they were prepared. The cloudiness of the emulsion gradually reduced after 5 h, three days, and one month of storage under room conditions, as shown in (Fig. 4b–d), respectively. Such observation indicates that emulsion stability decreased over time due to oil droplet coalescence due to surfactant destabilization, which will lead to the formation of two separated phases (*i.e.*, emulsion and water) when the equilibrium time is reached. After one month, the emulsion phase reached equilibrium, and Winsor type II emulsion (water-in-oil emulsion) was observed as in (Fig. 4d). The salinity screening test showed no effect amount of formed emulsion phase across the different tubes. Thus, it is not

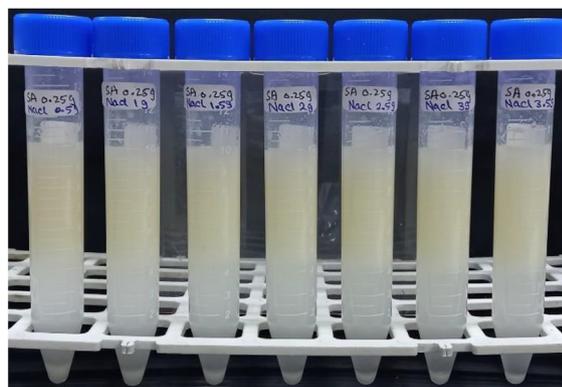
possible to identify the optimal salinity value. According to Hernández and Moreno,⁴³ W/O micro-emulsions (Winsor type II) were present in most of the surfactant concentrations studied with low salt content, which is what is observed in (Fig. 4).

3.6. Carboxymethylcellulose (CarboxMC) as bio-emulsifier

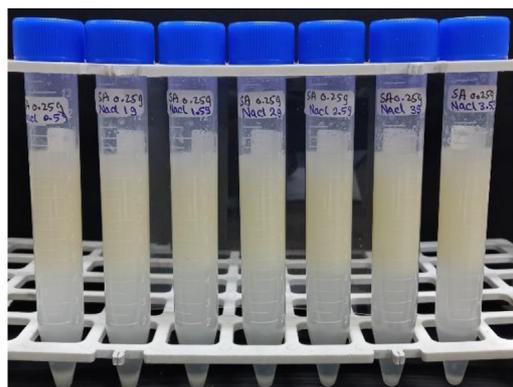
Fig. 5 shows the phase behaviour of the emulsion formed using CarboxMC as an emulsifier. Similar to SA, a Winsor type II emulsion was successfully formed because the emulsion phase remains at the top part of the solution after one month of storage,⁴⁴ as shown in (Fig. 5d). At tested equilibrium times of 1–5 h to 3 days, the emulsion was not completed equilibrium, as seen in (Fig. 5a–c). After one month, the emulsion phase reached equilibrium with (5.5 ml) volume equally distributed at different NaCl concentrations, as shown in (Fig. 5d). However, the identified water phase is cloudy water. The reason for not getting a clear aqueous phase can be



(a) After 1 hour



(b) After 5 hours

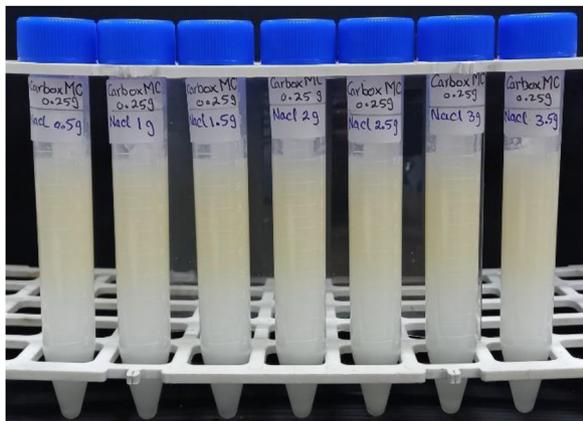


(c) After three days

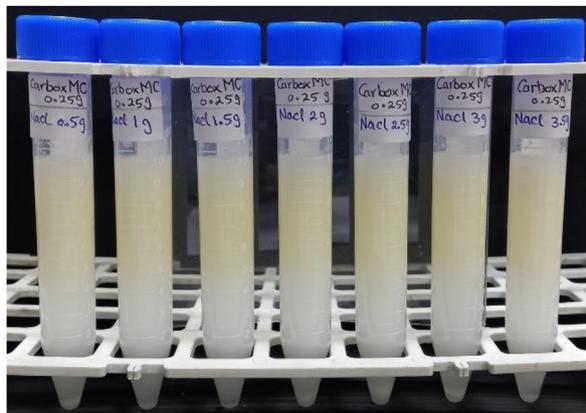


(d) After one month

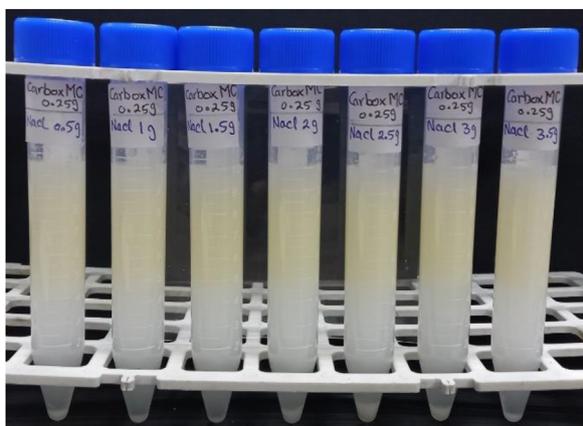
Fig. 4. Phase behavior of emulsion formed using 0.25 g/100 ml of SA emulsifier in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml.



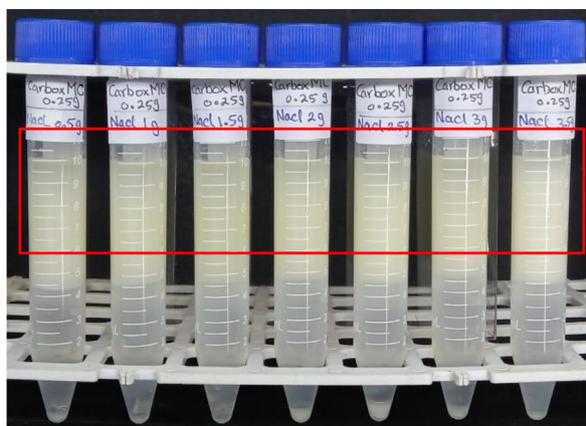
(a) After 1 hour



(b) After 5 hours



(c) After three days



(d) After one month

Fig. 5. Phase behavior of emulsion formed using 0.25 g/100 ml of CarboxMC emulsifier in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml.

ascribed to the polymeric and high molecular structure of CarboxMC.

3.6.1. Saponin as bio-emulsifier

The potential of forming a stable emulsion using a Saponin emulsifier is presented in (Fig. 6). The emulsion phase separated from the single aqueous phase after 1 h–5 h and reached equilibrium after three days, as shown in (Fig. 6b–d), respectively. The observed emulsion volume after reaching equilibrium is 5 ml; similarly, a type II emulsion was seen. In addition, emulsions volume remained constant after one month for all NaCl tested ranges, as shown in (Fig. 6d).

3.7. Phase behavior study for synthetic emulsifiers

To improve oil recovery, detailed investigations of a wide range of salinity and their impact on

emulsion system formation are recommended by researchers.^{45,46} Various synthetic emulsifiers are suggested in the literature due to their potential for use in industrial and experimental applications.⁵ In this section, two synthetic emulsifiers, including Triton X-100 and Tween 80, were selected for forming an emulsion with the *balanites* oil.

3.7.1. Triton X-100 (TX-100) as synthetic emulsifier

Fig. 7 delineates the emulsion formed using TX-100 as an emulsifier in different salinity ranges. It was found that the study salinity ranges (0.5 g/100 ml–3.5 g/100 ml) have no impact on the volume of the emulsion formed. Fig. 7a and b shows that the tested equilibrium times 1–5 h in which the emulsion system was yet to complete an equilibrium separation. The equilibrium was attained after 5 h to 3 days and completed after one month; Winsor type II emulsion was observed with a total of 5.5 ml

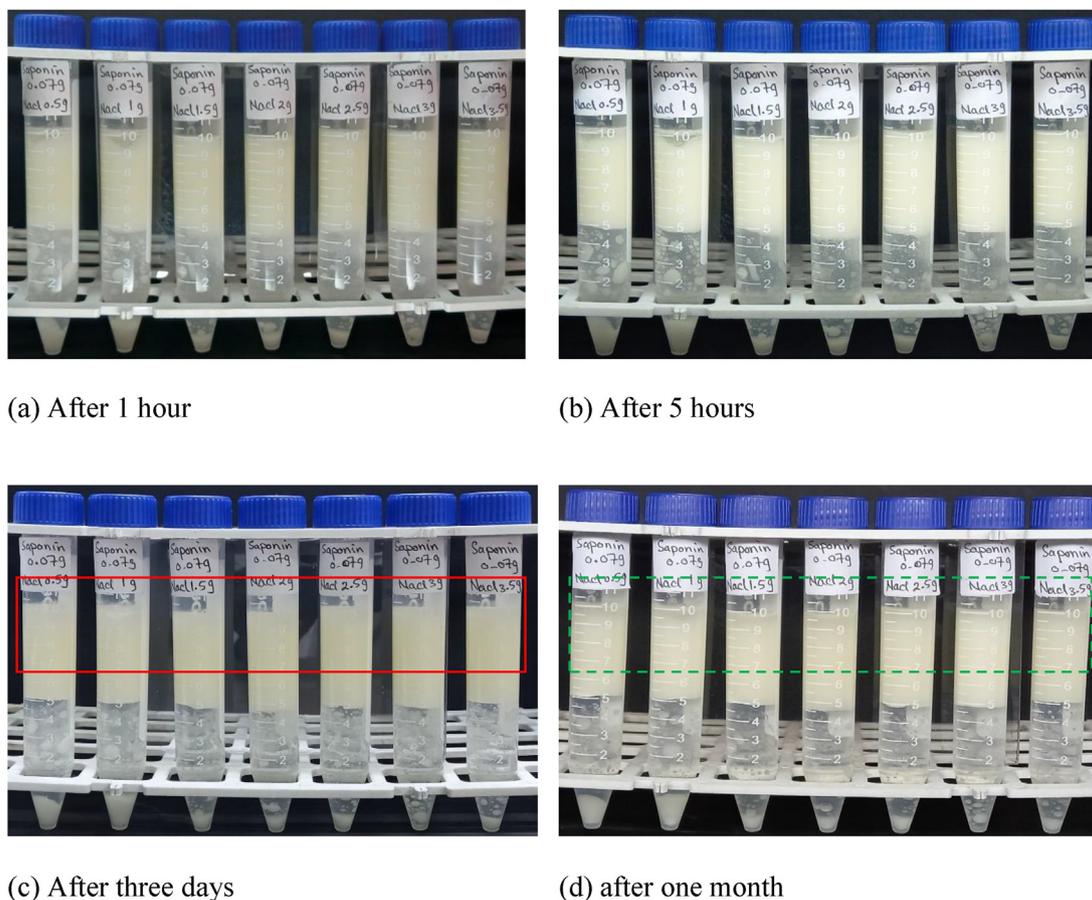


Fig. 6. Phase behavior of emulsion formed using 0.07 g/100 ml of Saponin emulsifier in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml.

emulsion volume. Over time, the observed decrease in the cloudy bottom phase in the testing tube indicates that the emulsion is dissolving in the aqueous phase. However, there is no cloudy phase at the bottom, as shown in (Fig. 7d), because the equilibrium was reached after one month.

3.7.2. Tween 80 as a synthetic emulsifier

Fig. 8 shows the time-lapse phase behaviour of the emulsion formed using Tween 80 as the emulsifier. Similar to TX-100, the investigation of equilibrium time indicated that fast separation of the O/W emulsion into cloudy aqueous and emulsion after 1 h, as seen in (Fig. 8a), after 5 h to 3 days emulsion type II started to be identified with emulsion amount ranged between 6 ml and 5.5 ml. After one month, the type II emulsion phase behavior reached equilibrium with 5.5 ml volume emulsion, which remains constant for all NaCl tested ranges, as shown in (Fig. 8d).

3.8. Comparison

The formulated emulsion is based on phase behavior tests to determine the emulsion volume, separation equilibrium at different times, the effect of varying salinity concentration, and the potential of formation of emulsion results from the sonication of *balanites* oil (as oleic phase) and six different emulsifiers. Worth noting that the test was conducted at room condition. Table 5 provide a detailed comparison of the results obtained.

The present study confirmed the hypothesis that the studied emulsifiers could create stable emulsion phases. Type III emulsions with volumes between 2.5 and 3 ml were successfully produced using GG as the emulsifier. Other emulsifiers (SA, CarboxMC, TX-100, and Tween 80) can achieve a Type II emulsion with (5.5 ml) volume equally distributed at different NaCl concentrations. However, the Saponin emulsifier achieved a Type II emulsion

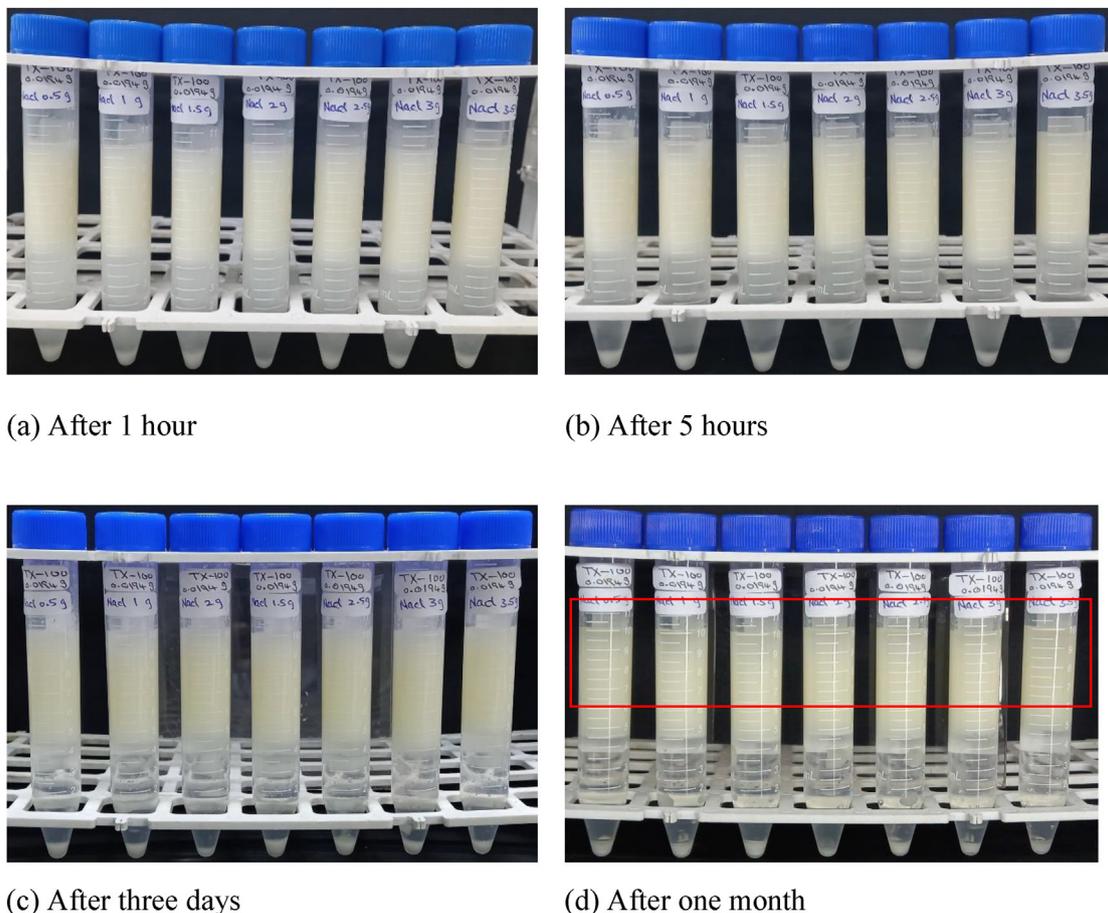


Fig. 7. Phase behavior of emulsion formed using 0.0194 g/100 ml of TX-100 emulsifier in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml.

phase with (5 ml) volume. The observed equilibrium of the emulsion systems phase behavior containing the Saponin emulsifier in 3 days is close to the reported time by Imuetinyan *et al.*,⁴⁷ while the observed equilibrium is stable for more extended periods (5 days).

The equilibrium time for emulsion systems containing (SA, CarboxMC, TX-100, Tween 80, and GG) was one month, and for Saponin emulsion systems were three days. These results show a longer equilibrium time compared to bio-oil-based emulsions reported in the literature; for example, Jeirani *et al.*²⁰ reported that Palm oil-based emulsion has an equilibrium time of one week, and Nordiyana *et al.*²² noted that *Jatropha* oil-based emulsion has an equilibrium time of two weeks.

The effect of the salinity study indicated that no significant change in the phase behavior of the emulsion, particularly in the emulsion's type and volume, was seen for SA, CarboxMC, TX-100, Tween 80, and Saponin. These emulsifiers' phase behavior systems obtained type II emulsions with

constant emulsion volume. Therefore, the optimal salinity could not be identified under the study range. However, phase behavior systems containing GG emulsifier show Type III with different emulsion volumes. The optimal salinity was determined at NaCl (2 g/100 ml) concentration with 3 ml emulsion volume remaining after one month of storage, as shown in (Figs. 3d and 9). The equilibrium time and obtaining of type II (emulsion and water phases) behaviour is achieved after one month for phase behaviour using (SA, CarboxMC, TX-100, and Tween 80) emulsifiers. However, after three days, the saponin emulsifier-formed emulsions reached equilibrium as shows in (Fig. 9). For an emulsion system composed of a GG emulsifier, type III (oil, emulsion, and water phases) behavior was obtained with an equilibrium time of 1 month. The separated water phase was seen in systems with TX-100 or Tween 80 as the emulsifier; slightly clear in systems with SA or CarboxMC as the emulsifier. On the other hand, in the system containing either GG or Saponin as the emulsifier, the water phase

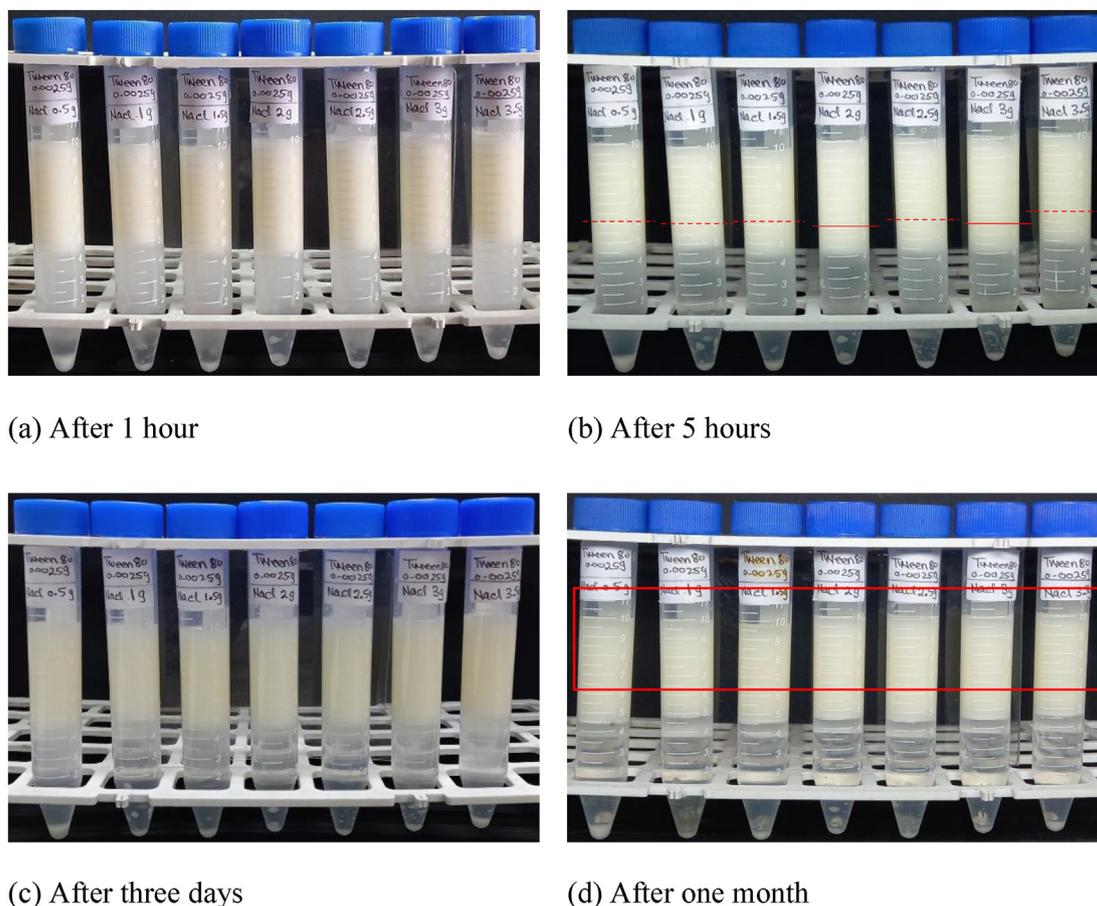


Fig. 8. Phase behavior of emulsion formed using 0.0025 g/100 ml of Tween 80 emulsifier in different concentrations of NaCl (0.5, 1, 1.5, 2, 2.5, 3, and 3.5) g/100 ml.

was cloudy, with small and suspended emulsion droplets. The cloudiness of the separated water phase could be related to the high molecular weight and chemical structures of the GG and Saponin emulsifiers; moreover, this cloudy water phase with suspended emulsion droplets reflects on having lower forming emulsion volumes (3–5 ml) for both GG and Saponin emulsifiers compared to 5.5 ml achieved by other emulsifiers.

Microemulsion-based EOR can be classified into pre-prepared and in-situ prepared. For the pre-prepared emulsion, the emulsion layer can be withdrawn from emulsions of all Winsor type for the EOR application.^{48,49} In this study, it was found that only GG emulsifier leading to the formation of Winsor Type III emulsion. Other bio-emulsifiers and synthetic emulsifiers tested in this study resulted in Winsor Type II emulsion. However, emulsion

Table 5. Comparison of the phase behavior of emulsion formed using bio- and synthetic emulsifiers.

Emulsifier type	Bio-emulsifiers				Synthetic emulsifiers	
Emulsifier Name	GG	SA	CarboxMC	Saponin	TX-100	Tween 80
Used Concentration (CMC), g/100 mL	0.25	0.25	0.25	0.07	0.019	0.0025
Formed Emulsion Type	Type III	Type II	Type II	Type II	Type II	Type II
Emulsion Phase Volume, ml	2.5–3	5.5	5.5	5	5.5	5.5
Equilibrium Time	One month	One month	One month	Three days	One month	One month
Effect of Different NaCl Concentrations (Yes/No)	Yes	No	No	No	No	No
Separated Water Phase Clarity	Cloudy, containing suspended emulsion in smaller droplets	Slightly cloudy	Slightly cloudy	Clear, containing suspended emulsion in smaller droplets	Clear	Clear

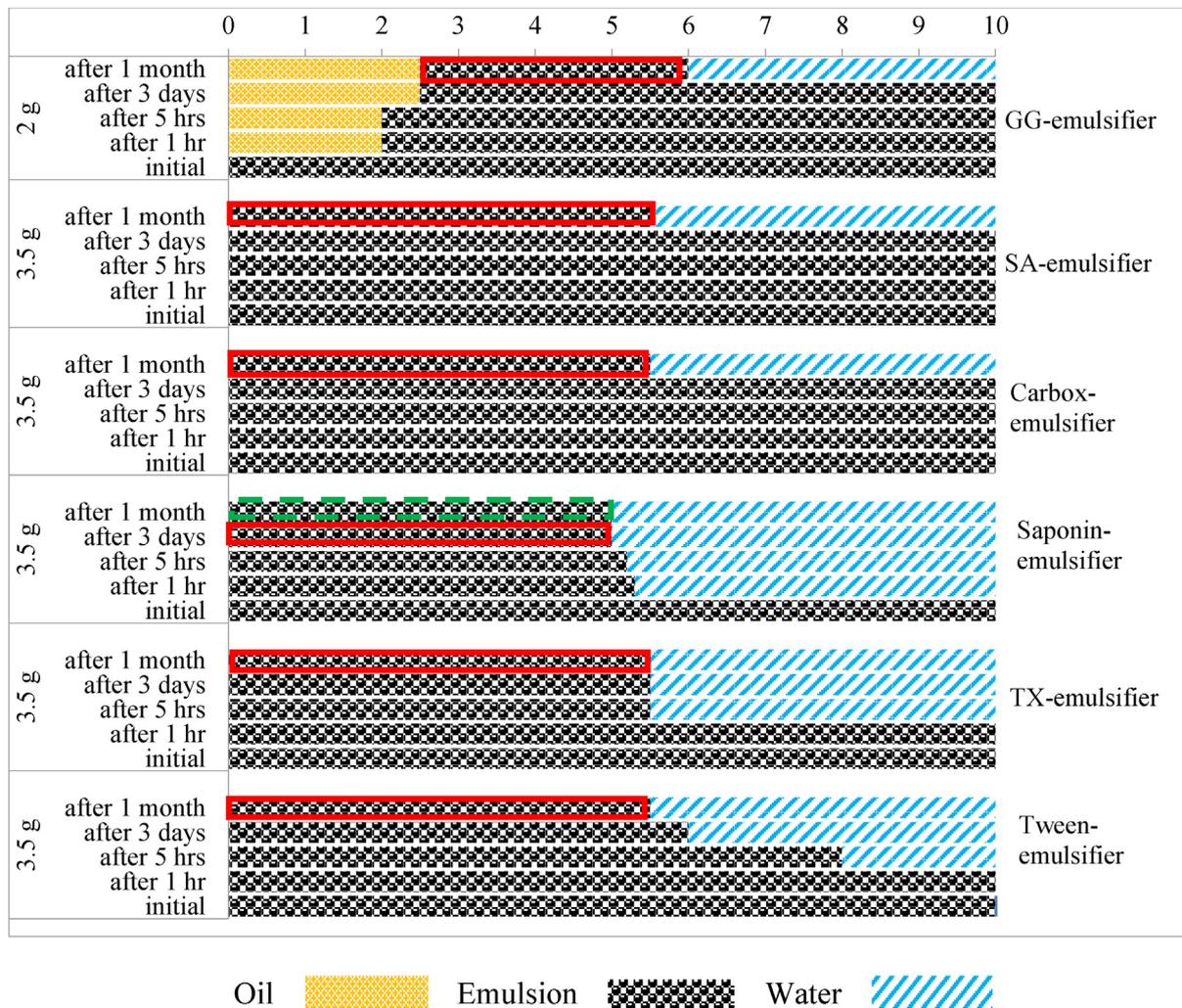


Fig. 9. Quantitative data for the phase volume and equilibrium time of the *Balanites*-oil-based emulsions prepared at optimal NaCl concentrations for the GG-emulsifier, and maximum NaCl concentrations for the emulsifiers (SA, CarboxMC, TX-100, and Tween 80).

formed using GG emulsifier has a lowest volume and hence less suitable for large scale production of such emulsion. In comparison, both CarboxMC and SA emulsifiers gave a larger emulsion volume of 5.5 ml; this amount is similar to the one formed using synthetic emulsifiers. To achieve or increase emulsion phase volume, further work is required to incorporate co-emulsifier into the system.⁴⁸

3.9. Conclusions

This study successfully produced Winsor type II and Winsor type III bio-emulsion using *balanites* oil and bio-emulsifier. For the various salinity systems ranging from 0.0001 to 0.01 g/ml, the CMC value of the GG, SA, and CarboxMC was recorded as 0.0025 g/ml. Meanwhile, the CMC for the Saponin emulsifier is 0.0007 g/ml, consistent with the literature report. The three studied bio-emulsifiers'

partially consistent structures and physical characteristics may explain why their CMC values are the same. Furthermore, due to their high molecular weight and chemical structure, the investigated bio-emulsifiers had a higher CMC than the synthetic emulsifiers. At different NaCl concentrations, bio-emulsifier SA, Saponin, and CarboxMC with *Balanites* oil form type II emulsion, while bio-emulsifier GG with *Balanites* oil form Winsor type III emulsion. SA and CarboxMC emulsifiers form a larger emulsion volume than GG and Saponin emulsifiers. In addition, both SA and CarboxMC emulsifiers reached equilibrium after one month, whereas emulsions formed with Saponin emulsifiers reached equilibrium after three days. These findings indicated the potential feasibility of emulsion formation using the studied bio-emulsifiers. On the other hand, the *balanites* oil also formed Winsor type II emulsions with synthetic emulsifiers (i.e. TX-

100 and Tween 80), reaching equilibrium after one month. The optimal salt concentration for GG bio-emulsifier phase behaviour tests is 2 g/100 ml of NaCl with 0.25 g/100 ml of GG emulsifier, which results in 3 ml emulsion after one month of equilibrium time, and the Winsor type III was observed at this concentration. However, the formed volume of the emulsion is significantly lower when compared to the other emulsifiers used in this study. For other bio- and synthetic emulsifiers, salt concentrations ranging from 0.5 to 3.5 g/100 ml did not affect phase behavior tests, generating a Winsor type II emulsion with a total volume of 5.5 ml. While the uses of *Balanites aegyptiaca* oil and green emulsifier is feasible for green emulsion preparation, more research is needed to apply and test the phase behaviour of this emulsion system at higher salinity water under different temperature ranges. In addition, investigation on the mechanisms and sweep efficiency during its application in EOR are recommended for future study.

Authors contribution

Sami Abdelrahman Musa Yagoub: Conceptualization, Visualization, Resources, Writing – Original Draft. Swee Pin Yeap: Supervision, Conceptualization, Visualization, Resources, Writing – Original Draft. Nurudeen Peter Yekeen: Conceptualization, Supervision, Writing – review & editing. Mohammed Abdalla Ayoub: Writing – review & editing.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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