

Case Report

Influence of whey protein isolate on CO₂ foams stability in three different types of crude oilMohamed Sasi Said^{a,b,*}, Mohd Zaidi Jaafar^a, Shaziera Omar^a, Sairoz Norazlan Sharbini^a^a Department of Petroleum Engineering, School of Chemical and Energy Engineering Universiti Teknologi Malaysia, 81310, Skudai, Johor Bahru, Malaysia^b Department of Petroleum Engineering, University of Zawia, Zawiya, Libya

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ABSTRACT

The foam performance in the presence of oil plays an essential role in foam application in enhancing oil recovery. This study systematically investigated whey protein isolate (WPI), the effectiveness of foam generation, the effect of three types of crude oil, surfactant concentration on foam performance and oil-water interfacial tension (IFT). The extraction of WPI was compared to sodium dodecyl sulfate (SDS) at the microscopic foam images to have a better understand of foaming in terms of performance. The results showed that the WPI lowers the surface tension, which also tends to disclose and stabilize the interface by forming a viscoelastic network. SDS reduced the surface tension compared to the WPI but did not produce a high modulus interface. WPI generated a more stable foam in oil than SDS foam. The WPI improved foam stability by increasing the gas-water interface dilatational viscoelasticity layers. WPI solution decreases bubble sizes and prevents oil from spreading at the foam lamellae. The WPI foam exhibits higher stability in Libya crude oil when the oil saturation is between 0–20%. WPI foam reduces fluid loss and hinders the movement of oil droplets within the lamella, the significant viscosity contrast between oil and WPI-CO₂. Waxy oil is detrimental to both SDS and WPI foam, while waxy oil can harm WPI foam in some specific cases. The oil-water IFT was reduced to extremely low levels by WPI. As a result, WPI's interfacial tension values with Sharara oil were (21 mN/m) and SDS's were (44.5 mN/m).

1. Introduction

For several decades, significant quantity of crude oil remains unrecovered following the primary and secondary phases of oil recovery. After conventional treatment processes of 70%, the remaining oil still remains untapped in the reservoir [1,2]. For this reason, a tertiary recovery phase, also known as Enhanced oil recovery (EOR) such as gas-injection [3] is employed. Similarly, gas recovery methods such as carbon dioxide have shown some potential on improve oil recovery. The gas injection enhanced oil recovery (EOR-CO₂) process usually results in gravity override, gas segregation, viscous fingering, and channeling through the high permeability streaks [4,5]. Due to low viscosity and density of gas as compared to oil and water, foam colloidal dispersion of gas in liquid helps control gas mobility by increasing the displacing fluid's apparent viscosity and reducing the relative permeability of the gas phase. Foams are thermodynamically unstable and require surface-active agents such as surfactants for continuous generation,

stability, and propagation in porous media. For several decades, surfactants have been used as a conventional method of foam stabilization [6,7]. Surfactants can reduce interfacial tension (IFT) to ultralow levels and improve oil displacement efficiency in chemical EOR processes by adsorbing on the crude oil-aqueous interface [11]. However, at high salinity, temperature, and the presence of the resident, reservoir brines, and oil, surfactant stabilized foams cannot maintain their stability for a long time [8,9]. Thus, under reservoir conditions, surfactant formulations should be optimized before being used in oil recovery applications. This is accomplished using surfactant screening, which remains a difficult process that requires both time and material resources [12]. Surfactant screening is difficult due to two major factors viz: Majority of surfactants are heavily influenced by the conditions in the oil reservoir as well as the type of reservoir rock [13]. Second, there is a diverse range of surfactants with the potential to be used for EOR [14]. Perhaps, surfactant flooding is one of the most effective EOR techniques [10].

The stability of foam generated with crude oil remains a challenge

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and essential in foam EOR. In a kinetic sense, the term stability means “relatively stable”. In essence there are no thermodynamically stable foams. Though the half-life or average lifetime of a generated foam’s is used to determine its stability [15,16]. The presence of oil can negatively affect the foam stability by weakening, thus causing foam destruction [16]. The adverse effect of oil on foam stability and the characterization of the complex interaction between foam and oil is one of the significant challenges to foam’s success in EOR [18]. The findings of bulk foam experiments published in peer-reviewed journals appear to contradict the effect of oil on foam stability. According to some authors, oil, particularly lighter hydrocarbons, destroys or prevents foam formation [19]. On the other hand, other authors have shown that stable foams can be generated in the presence of oil by using an appropriate foaming agent [20]. It has also been demonstrated that foam can be generated effectively in the presence of heavy oil [21]. Other experiments have concluded that oil can improve foam stability [22,23]. Foam stability is critical for effective oil displacement. Many physicochemical properties of the surfactant solution, such as surface tension, surface viscosity, and elasticity, among others, influence foam stability. Oil’s destabilizing effects on the foam are caused by direct surface interactions between oil and foam, which are determined by various physicochemical properties [24]. Foam stability in porous media functions both as the foam film and the petrophysical rock properties. The bulk column tests are used to determine most of the foam oil interaction characteristics. The height of the foam is calculated in the experiments as a representative of the surfactant’s ability to create a stable foam with crude oil [17].

Foamability and foam stability are two main categories of foaming qualities. Foamability is a term that refers to the rate at which foam forms. In other words, it is the ability to easily create bubbles: the more significant the foamability, the more stable the foam films. The amount of foam produced can be used to determine an aqueous surfactant solution’s foamability. The term “foam stability” refers to a foam’s capacity to retain its initial qualities after being created. Changes in surface tension affect the elasticity of the foam film, which in turn affects the type and concentration of surfactants used in foam production [25]. The difference in pressure generated inside the lamella and the plateau borders is one of the most critical factors in the liquid drainage rate for lamella. Three independent mechanisms contribute to the foam’s instability: liquid drainage, bubble coalescence, and disproportionate formation. Drainage is the fluid flow from a foam caused by gravity and capillary forces [26]. A foam becomes dryer because of drainage, and bubbles may become distorted due to the conversion from spherical to polyhedral foams. The merging of two bubbles caused by the rupture of thin films between the bubbles is known as bubble coalescence. In the foam, larger bubbles appear, and the number of bubbles decreases. Surfactant blend solutions improve foam stability by adsorbing onto interfaces and preventing bubble coalescence via steric effects. Inter bubble gas diffusion and Ostwald ripening are other terms for disproportionation. Larger bubbles grow at the expense of smaller bubbles during this process. The smaller bubbles will eventually shrink and disappear. These processes alter the distribution of the liquid and gas phases, thereby altering the properties of the foam [16].

By increasing the hydrophilic chain length of the surfactant, which increases its hydrocarbon solubilization power, the stability of the generated foam with crude oil is improved [27]. A hydrophilic group is a polar group that is water-soluble. The lipophilic chain length is illustrated by the surfactant’s hydrophilic head (the length of the carbon chain). Non-polar and insoluble in water, the hydrophobic group. Surfactants, in general, have a lipophilic chain of 8–20 carbon atoms. Short-chain surfactants dissolve quickly in water, while long-chain surfactants are less soluble [28]. Increased length of the hydrophobic group may improve surfactant efficiency [29]. Foams are commonly created using anionic, non-ionic, and cationic surfactants. The foam’s stability is affected by the surfactant concentration. The orientation of the surfactant molecules at the interface controls the foam stability and

interfacial tension on a molecular level. Electrostatic interactions absorb molecules at the interface. It absorbs quickly and completely [30].

Proteins are a group of Zwitterionic surfactants widely used in EOR due to their remarkable ability to withstand harsh reservoir temperature and salinity conditions [31]. When used in EOR, proteins surfactants have several advantageous properties. Proteins are salt-tolerant, which reduces interfacial tension (IFT) and increases viscosity. The effective viscosity of the gas increases as the number of bubbles in the foam increases and the surface fluid is adsorbed in a larger contact surface between the two fluids, increasing the thickness of the lamella. As a result, liquid can be drain from the lamella at a slower rate. Furthermore, lowering the IFT improves foam stability by reducing capillary forces [32]. Sodium dodecyl sulfate (SDS) is an anionic surfactant that is found in a variety of products. In the presence of potassium and sodium chloride salts, the performance of SDS can be significantly affected and easily precipitable [31].

Surfactants, proteins usually stabilize foam structures, and their half-life characterizes the stability of foams. Long-life foams normally last 50 min or longer [33]. Proteins represent the major category of natural foaming agents in EOR [31]. Proteins typically used for foaming purposes include whey protein isolate [34]. However, some proteins have certain limitations either in foam capacity or foam stability, which cannot meet the requirements of the increasingly fastidious food industry. It has been observed that protein can significantly improve the stability of foams and potentially reduce the destabilizing effect of oil on foam [35]. For example, adding protein enhances the foam stability due to the formation of a viscous surface layer, which raises the interfacial viscosity of the foam, thereby reducing the rate of film thinning. Proteins are known for their foam-enhancing properties [31]. Protein behavior at the interface influences foam formation and stabilization. The formation and structural, rheological, and mechanical properties of the interfacial film have a significant impact on foam stabilization [33]. In simple model systems, factors that ensure optimal film properties may retard film formation or cause destabilization in foams. Thus, environmental and processing factors such as temperature, pH, protein concentration, salts, the continuous phase composition, and so on, which alter protein configuration and stability, have a significant impact on film formation and film properties [36]. Protein-based foams are also influenced by intrinsic molecular properties, such as the nature and extent of protein-protein interactions, which can differ between proteins.

Researchers have recently become interested in foam stabilized protein mixtures for potential applications in EOR. According to previous research, these foams have a high initial foamability as well as long-term stability [31]. This is due to irreversible protein adsorption on their bubbles’ surfaces [37,38]. Proteins that act as foaming agents can also help with oil recovery by lowering the interfacial tension and capillary forces. Because proteins are solids, the foams’ stabilizing components are expected to be stable in porous formation at high salinity, temperatures, and oil [32,39]. To better understand the mechanism of surfactant adsorption, we have to study the effect of crude oil on foamability and stability in two surface-active agents: SDS and whey protein isolate (WPI). This research aims to look at the foaming qualities (stability and foam strength) of various types of crude oil. Proteins are complex molecules that generate highly viscoelastic layers with high surface shear viscosities. Protein adsorbed rigid plateau boundary [33,40]. To the author’s knowledge, this specific feature of protein form steady CO₂ foam for EOR applications has not yet been investigated. As a result, Whey Protein Isolate WPI foam is being investigated as a foaming agent for making CO₂ foam stable for EOR purposes. An investigation into the effects of various crude oils on the foam’s consistency and foamability exemplified the foam. The results obtained will be compared to the performance of the traditional surfactant (SDS) foam.

2. Materials and methods

To investigate foam stability in the presence of oil, we employed two

surfactants in our experiments; SDS ($C_{12}H_{25}NaO_4S$) and WPI which are used as the foaming agents in the experiments. The SDS contains a molecular mass of 288.4 g/mol, supplied by the USA SIGMA-ALDRICH Company. The WPI was exclusively imported and distributed by Lush protein company in Singapore. The surfactant (SDS and WPI) solutions' properties were listed in Table 1. These surfactants were chosen based on their charges, foamability and foam stability with deionized water ($pH = 6.9 \pm 0.1$). The surfactant concentrations are based on the active content and were well at the critical micelle concentration. Carbonyl dioxide gas with a purity of 99.9% and delivered by Mega Mount Company was used to generate foam. Three crude oil models were used to investigate the effect of oil on foam stability. To visualize the foam droplets in the foam column, Hamada crude oil, Sharara crude oil, and Malaysia waxy crude oil (SDS red and WPI green) were mixed into the foam phase. Preliminary tests, including surface tension measurement and foam column, were done to ensure no influence of the dye on the foaming behavior.

2.1. Surfactant solutions

The preferred surfactant concentration was measured on a mass basis after which all the surfactant solution samples were prepared in 500 ml volumetric flasks. Before the final weight of the solution (0.5 kg) was reached, deionized water was added. The concentrations of surfactant solutions were calculated using dynamic content. SDS and WPI were used in the analysis, with 0.23 wt % SDS and 0.06 wt % WPI. To ensure that the surfactant was completely dissolved, each sample was stirred for 20 minutes at room temperature 26 °C.

2.2. Crude oil

Three types of crude oils (Hamada, Al Sharara, and waxy crude oil) each having a different density and composition were examined. The crude oil samples will be used to analyze foam stability and formability. Hamada and Sharara crude oils were collected from the shipping line at the refinery terminal in Zawiya while about 60 L of crude oil samples were collected from Akakous company. The sample collected (Libya's crude oil presence, and porous media) for foam flow processes are in accordance with the standard (ASTM D-4057) method.

Evaluation of Hamada crude and Al Sharara crude oil have been prepared by the National Oil Corporation (NOC). The crude oil sample analysis was carried out using well-recognized standard procedures provided in the methods of ASTM, IP, and UOP. The distillation of crude oil was carried out using an ASTM D-2892 and ASTM D-1160 batch fractionation unit. The crude oil was refined under ambient temperature and vacuum pressure, while the atmospheric residue was further filtered to extract distillate fractions. Fractions of the distillate corresponding to the true boiling point were collected up to 550 °C. Table 1 provides crude oil properties in terms of surface viscosity, density, and crude oil

Table 1
General tests for Hamada and Al Sharara crude oil properties.

Description	Hamada oil	Al Sharara Oil
Density of crude oil @ 15 °C, mg L ⁻¹	0.8304	0.8139
Specific gravity@60/60 °F	0.8311	0.8146
API gravity	38.8	42.2
Flash point (PMCC), °C	-23	-42
Hydrogen sulphide, ppm	17.04	1.02
Sulphur content, wt. %	0.063	1.073
Pour point, °C	-18	-33
Kinematic viscosity. @70 °F, cSt	6.8431	1.1348
Kinematic viscosity. @100 °F, cSt	3.5742	2.2886
Asphaltenes content, wt. %	0.34	0.260
Conradson carbon residue, wt. %	2.05	1.206
Ash content, wt. %	0.004	0.006
Characterisation factor	12.0	11.9
Salt content (as NaCl) mg L ⁻¹	2.14	0.12

composition. The third crude oil used in the experiment was Waxy crude oil obtained from Terengganu crude oil terminal (TCOT), located on Peninsular Malaysia's East Coast. The oil was thermally treated to redissolve wax crystals within the crude. The treatment involved heating the crude oil at 50 °C. Table 2 presents the crude oil's physical properties after dehydration in Universiti Teknologi Malaysia's accredited laboratory (UNIPEM).

3. Methods

3.1. Surface tension and interfacial tension measurements

Surface tension measurements were carried out in a Kruss DSA 25E Drop Shape Analyzer at a temperature of 26 °C by the pendant drop method. Fig. 1 shows the surface/interfacial tension experimental setup. Initially, the instrument was calibrated using deionized water, and the surface tension was determined to be 72.3mN/m at the air-aqueous interface. Each measurement was performed at least three times to ensure results' repeatability. The IFT between different concentrations of WPI, SDS solutions and three types of crude oil were measured. These crude oils are distinguished by their density, viscosity, and surface tension, as given in Tables 1 and 2 to enable investigating the effects of oil viscosity and density on foam stability. The primary water/oil interfacial tension was calculated using the K20 Force Tensiometer (Kruss) and a RI 21 platinum-iridium alloy wire ring employed in the Du Noüy ring method [41]. The concentrations of WPI were 0.06 wt%, while the SDS solution was 0.23 wt%. Details experimental studies are reported in Refs. [20,42].

3.2. Foam stability tests

The preliminary test to determine the foam stability and foamability for the surfactants WPI and SDS were the static test. The surfactant powders were dissolved at WPI, SDS concentration in deionized water. A detailed schematic representation of the bulk foamability experiment was portrayed in Fig. 2. The foam was generated in a tempered glass column 50 cm high and a 5.08 cm outer diameter through gas sparging across a porous disk (40 – 100 μm) at the bottom, which permitted gas introduction through a fritted glass disc (porosity, 4). The bubbling CO₂ air injection spot was located at the blender's base. By stirring 100 ml of surfactant solution and blowing CO₂ gas via the injection spot into the liquid solution. The CO₂ gas employed to generate foam has a purity of 99% and is delivered by Mega Mount Company. At the pump time of 60 s, the flow rate of the gas was fixed at 0.3 L/min [43]. At an ambient temperature (26 ± 1 °C) and atmospheric pressure, test was carried out on every foam bulk stability and formability. The foam height as a function of time at the given time intervals was captured using a high-resolution camera. The tests were executed at least thrice, and each trial went on for 1 min before the gas was shut off, and the generation of foam ended. The average value of the experiment result was used. The appearance of foam and its evolution with time plays an important role in the foam characteristics (stability and strength) [31]. The foam

Table 2
Waxy crude oil's physical properties.

Description	Malaysia waxy crude oil	Method
Dynamic viscosity @ 40 °C (mPa s)	2.22	ASTM D445
Specific gravity@60/60 °F	0.844	Calculate
API gravity °API	36	ASTM D1298
Flash point, °C	130	ASTM D92
WAT °C	24	DSC
Water content vol%	0.05	ASTM D4377
Asphaltene, wt. %	0.071	ASTM D2007-93
Pour point, °C	18	ASTM D97
Saturates (wt%)	65	ASTM D2007-93
Wax content (Wax %)	17	UOP 46

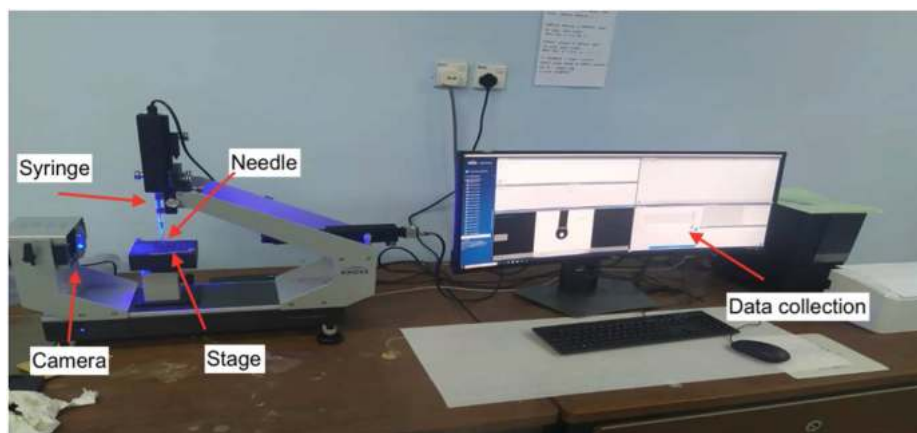


Fig. 1. Experimental set-up showing DSA 25 for surface/interfacial tension.

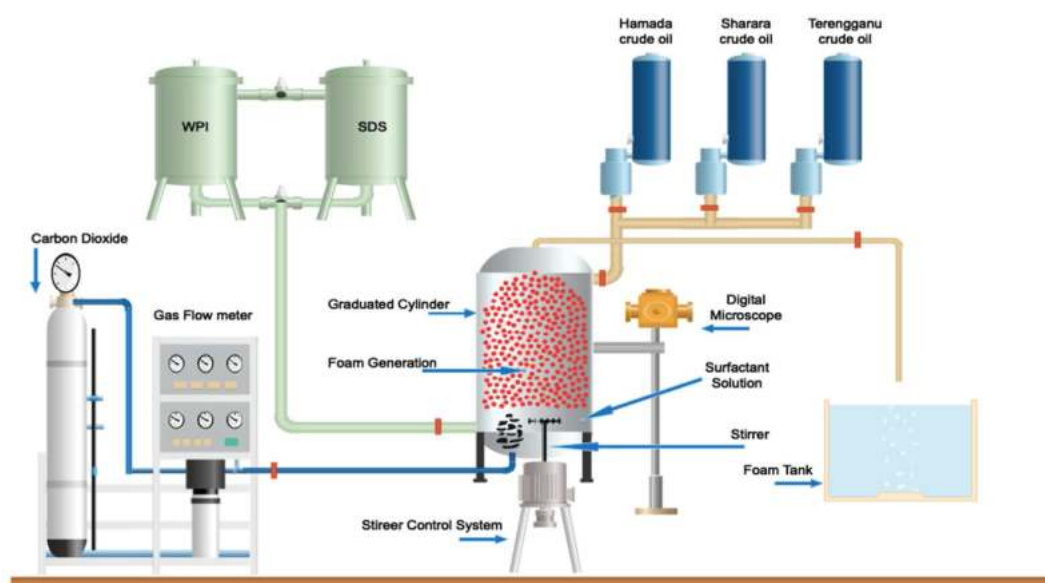


Fig. 2. Experiments for determining the foam stability and foamability of materials.

production and decay processes were watched on a computer screen connected to a fixed digital camera 1600X optical microscope.

This research was divided into two sections: static and dynamic tests. The static foams contained 0.23 wt% SDS and 0.06 wt% WPI as surfactants. For the dynamic part, foams were generated and tested with two Libya crude oil and Malaysia waxy crude oil. Surfactant foams then observed the interaction and foam destruction process in the presence of oil under a microscope. The half-life of foam columns generated with and without crude oil was measured. To comprehend the processes of foam formation and stabilization.

3.3. Determination of foamability, foam stability

A standard simple method similar to the technique previously used by Samin A. M. et al., 2017 [37] use to investigate the bulk foam static stability. The experiments were carried out by dissolving CO₂ into the 100 ml foaming solution. The CO₂ was dispersed into the test solution for a period of 1 minute using a pedal connected to the mixer at a speed of 2100 RPM. The foaming procedure was carried out in a glass cylinder of 500 mm height and of 50.8 mm diameter respectively. After mixing, the glass cylinder was closed at the top to avoid environmental disturbances. The temperature of the generated foam and the experimental setup was kept constant at 26 °C. The foamability was determined by

measuring the height of the foam column (h_0) immediately after foam generation ceased ($t = 0$). A higher foam level improves foamability significantly. The foam stability was defined by the half lifetime ($t_{1/2}$), the time required for the generated foam to be reduced to 50% of its initial height (h_0). A more stable foam has a longer half-life time. The foam stability was determined by plotting normalized foam height versus time [42,44]. The concentrations of WPI and SDS foams ranged from 0.005 to 0.5 wt%. Foamability and foam stability were studied in relation to various concentrations. WPI and SDS had CMCs of 0.06 wt% and 0.23 wt%, respectively. A Leica EZ4 HD stereomicroscope was used to examine the microscopic foam image and the bubble morphology bubble size distribution. The microbubble was placed on a microscope slide, and the image was captured using the microscope's camera. The microscopic image was analyzed using software to determine the bubble's diameter.

4. Results and discussions

4.1. The interfacial tension (IFT) and surface tension

The interfacial tension between oil and water was unique in promoting hydrocarbon recovery because microscopic displacement efficiency increases as the oil-water interfacial tension decreases [45].

Different concentrations of the isolated WPI were investigated on the oil-water interfacial tension. The Surfactant was compared with the performance of the conventional surfactant, sodium dodecyl sulfate (SDS). The surface/interfacial tension was measured via pendant drop techniques (with an outer needle diameter of 1.825 mm). The surfactant CMC was determined from the graph of surface tension against surfactant concentrations as surfactant concentration with insignificant change in surface tension. The CMC value was the surfactant concentration at the point in which the two straight lines crossed.

SDS and WPI surfactant values were 0.23 and 0.06 wt% respectively as presented in Table 1. The results were well supported by the literature [31,46]. The experiments were conducted at room temperature. The results showed that the increasing surfactant concentration generally resulted in a drastic reduction in Hamada crude oil-IFT up to a certain concentration close to the critical micelle concentration. Precisely, the interfacial tension (IFT) at CMC of 0.06 wt% WPI was 11 mNm, 21 mNm, and 35 mNm for Hamada oil, Sharara oil and Malaysia waxy crude oil respectively. While at SDS of 0.23 wt% an IFT of 36.5 mN/m, 44.5 mNm and 40 mNm was recorded for Hamada oil, Sharara oil and Malaysia waxy oil accordingly. It can be observed that, at CMC - WPI 0.06 wt% solution the IFT in Hamada oil decreased to 11mN/m as compared to the sodium dodecyl sulfate 36.5 mN/m. Similarly, at WPI 0.06 wt% the surface tension was 39.73 mNm while the 0.23wt% SDS was 36 mNm. Proteins and other surfactants reduce the tension between these phases because of their affinity with water and oil (amphiphilic nature). The same technique for determining CMC has been reported in Refs. [20,47]. These results showed that the extracted WPI could be considered a supplementary alternative to convention EOR surfactants, such as sodium dodecyl sulfate, to reduce interfacial tension and enhance hydrocarbon recovery [31,48].

Generally, the oil and water interface could be the ideal location for the systematic orientation of the protein's molecules due to their twofold molecular structure and the hydrophobic and hydrophilic elements [45,49]. The change in oil-water IFT can be attributed to the arrangement of surface-active species at the oil-water interface. The hydrophilic head is dissolved in an aqueous solution. At the same time, the hydrophobic tail dissolves in oil [39]. The IFT of the system decreased with the increasing surfactant concentration due to the adsorption of the surfactant molecules at the oil-water interface. The IFT decreases as the surfactant concentration increases, signifying that a rising number of surfactant molecules migrate to the oil/water interface. However, the aggregation of surfactant molecules in the bulk solution prevented their adsorption and accumulation at the oil-water interface to decrease the interfacial tension [31].

4.2. Optimum concentration to generate stable foam

The foam stability test gives an idea of how crude oil and surfactant solutions might interact. Thus, experimental data provide useful basic information on foam generation and its stability as a function of surfactant concentration, oil type, and surfactant type. The purpose of this test is to see if the surfactants can generate a stable foam. This test provides a preliminary and quick evaluation of surfactant foamability. Experiments were conducted at standard room temperature and atmospheric pressure. The initial test in this work is to determine the foam stability and formability (WPI and SDS). Whipping and injection were combined to prepare the static test of foams. Fig. 3 shows the foam generation process using deionized water (WPI, SDS) at different concentrations. The foam generation/stabilization properties of WPI were characterized at room temperature 26 °C. The performance of the WPI foam was compared with the performance of the sodium dodecyl sulfate SDS foam. This is to ascertain if the synthesized WPI can be proposed as a supplementary alternative to conventional foam-stabilization surfactants. The foam generation property was inferred from the variation of foam volume with time. The foam stability was estimated from the decay profile of foam volume versus time.

Fig. 3 shows the optimum concentration levels for both WPI (315 mm) and SDS (300 mm) foam stability with concentrations. This result indicates that the build-up of the WPI stabilized foam during CO₂ was very stable. The foam stability analysis showed that the volume of foam stabilized by a higher WPI concentration of 0.06 wt% was quite stable and eventually attained a fixed foam volume of 315 mm. The critical aggregation concentration (CAC) of the surfactants was WPI 0.06 wt%, it was found to be a good predictor of foamability and foam stability for these small molecule surfactants. These results are in good agreement with values obtained from the previous studies [31,48,49]. While at 0.23 wt% concentration, SDS stabilized foam attained a fixed foam volume value of 300 mm. Therefore, the SDS foam, 0.23 wt% was chosen as the optimum concentration, which is the CMC [22,43]. Thus, the two concentrations, CAC and CMC have been used in further comparison experiments such as effect of crude oil and the foam's stability.

4.3. Impact of increasing surfactant concentration on foamability and stability

Table 4 shows the effect of increasing surfactant concentration (WPI and SDS) on foam generation and stability. The foamability was expressed as a function of foam height and volume. Using (0.02, 0.04, 0.06 wt% WPI), and (0.02, 0.1, 0.23 wt% SDS).

Fig. 4 shows the effect of increasing surfactant concentration (WPI

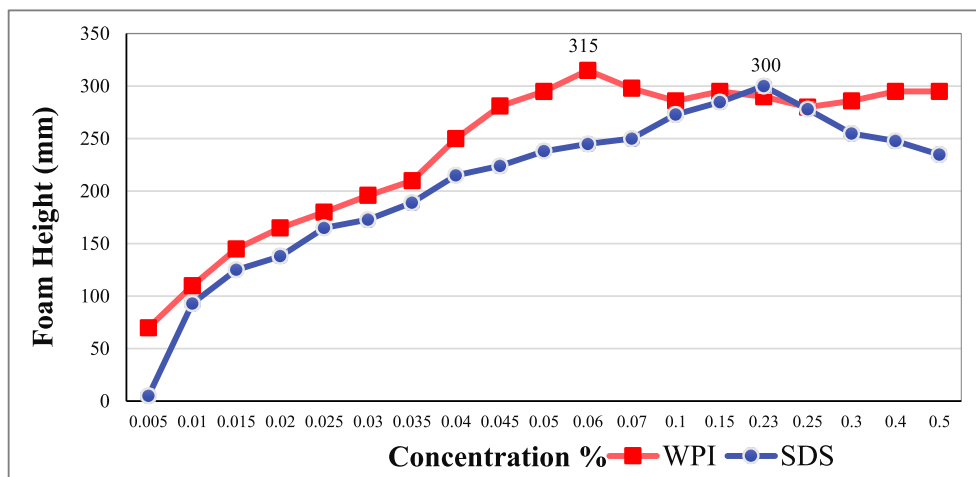


Fig. 3. Maximum foam height generated using WPI and SDS at various concentrations.

Table 3
Surface tension and Interfacial tension measurement WPI and SDS.

Surfactant solution Wt.%	Active content (%)	Charge Anionic	Surface tension (mNm ⁻¹)	Interfacial tension (IFT) (mNm ⁻¹)		
				Hamada crude oil	Sharara crude oil	Terengganu Waxy Crude oil
0.23% SDS	100%	Anionic	36	36.5	44.5	40
0.06% WPI	91%	Zwitterionic	39.73	11	21	35

Table 4
The effect of surfactant concentration on foam generation and stability.

Surfactant concentrations (wt %)	Foam Height (mm)	Foam Volume (ml)	Half-life (seconds)
0.02% WPI	165	334.43	1767
0.04% WPI	250	506.71	2210
0.06% WPI	315	638.45	2449
0.02% SDS	138	279.70	210
0.1% SDS	273	553.32	1010
0.23% SDS	300	608.05	1545

and SDS) on foam generation and stability for the entire concentrations investigated in this study. The foamability was expressed as a function of foam height and volume. Increasing protein concentration leads to increasing foamability and an increase in foam stability. This result indicates that the WPI foam build-up during CO₂ was very stable. We can infer that the WPI foamability and foam stability are likely not to be affected by the three mechanisms of foam destruction: liquid drainage, bubble coalescence, and coarsening [25,52]. The foam stability analysis showed that the volume of foam stabilized by higher WPI concentration (0.02 wt%, 0.04 wt%, 0.06 wt%) was relatively stable and eventually attained a fixed value at a particular time. The 0.02 wt% WPI-stabilized foams reached fixed volume 165 mm and foam half-life 1767s, while 0.04 wt% WPI-stabilized foam volume of 250 mm and half-life 2210 s. The 0.06 wt% WPI -stabilized foam attained a fixed volume value of 315 mm after 2449 s. WPI obtained the most stable foam in the presence of 0.06 wt% WPI solution. The foam volume decay profile was characterized by a slower deterioration in foam volume for a longer time, maintaining a plateau at a specific period. The number of surfactant molecules increased in bulk solution as the surfactant concentration increased from 0.02 to 0.06 wt% WPI. This result showed an optimum concentration for the maximum stability of WPI-stabilized CO₂ foam. This concentration was identified as 0.06 wt% in this study [31,42].

The stability of the WPI foam was compared to that of the SDS foam at the surfactant concentration that produced the strongest WPI foam.

Figs. 3 and 4 illustrate the degradation profile of the SDS foam. The results indicate that the foaming parameters of the SDS foam were less stable. The half-lives of the 0.02, 0.1, and 0.23 wt % SDS-CO₂ foams range between 210 and 1545 seconds. On the other hand, 0.06 wt% WPI at optimum concentration has a higher formability and foam stability. Fig. 3 shows the plotted graphs showing that the formability and foam stability increase as the SDS concentration increases. SDS foams decreased at 0.02 wt % and 0.1 wt % due to a decrease in the number of free surfactant molecules in the bulk aqueous phase. However, at 0.23 wt% SDS, surfactant molecules were more concentrated at the air-water interface to increase foam stability. Therefore, foam stability increased with the increasing concentration. These findings show that WPI can generate more stable foam than SDS foam. Fig. 4 confirmed that the optimal surfactant dosage for maximal foam stability was 0.06 wt% WPI and 0.23 wt% SDS.

The results confirm that, the foam half-life increases as the surfactant solution concentration increases [53]. However, the results show that increasing the surfactant concentration first increased foam stability up to a certain point and then decreased as the surfactant concentration increased further. This points to an optimum surfactant concentration corresponding to excellent foam stability and half-life [48,50]. These findings allow for a more detailed explanation of the effect of surfactant solution concentration. The measure of the impact of all concentrations of WPI and SDS on foam stability is shown in (Figs. 3 and 4). It can be seen that, the foam half-life for concentrations of 0.06 wt% WPI and 0.23 wt % SDS was 41 min, and 25.75 min, respectively. It means that for the range of surfactant concentrations examined. Our studies showed that 0.06 wt % WPI and 0.23 wt % SDS of surfactant were the best concentrations for CO₂ foam stability since increasing the surfactant concentration beyond that did not increase foam stability [31,54].

4.4. Surfactant (WPI, SDS) foam microscopic images

The stabilization was carried out using SDS and WPI surfactants. The absorption and aggregation of surface-active species at the foam's gas-liquid interface typically ensure foam stability [54]. The absorption

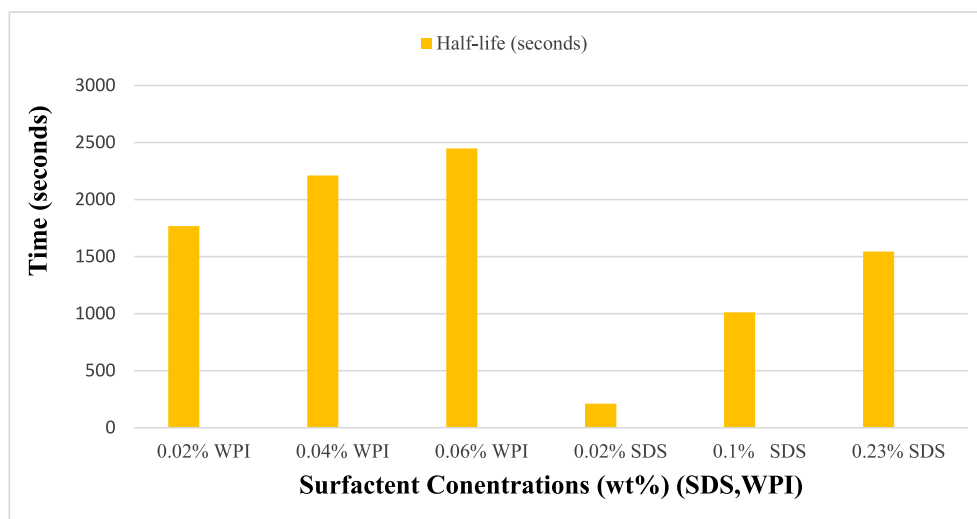


Fig. 4. WPI and SDS half-life at different concentrations.

and accumulation of surfactant molecules at foam lamellae improve oil recovery by lowering the interfacial tension. SDS-foam (0.23 wt%) and WPI-foam (0.06 wt%) were prepared as case studies to capture microscopic images of foam at surfactant concentration. For better understanding of the foam stabilization mechanisms, all bubbles as produced with and without oil are considered. The morphology of surfactant foam (SDS-CO₂ foam) and (WPI-CO₂ foam) were examined using a Leica EZ4 HD stereo microscope. The bubble diameter was estimated using Leica EZ4 HD stereo software. As shown in Fig. 5, the microscopic images of the bubble-size distributions were T = 300 sec, the bubble diameter of SDS foam between 82.55 μm to 205 μm , T = 780 sec was 140.46 μm to 242.39 μm , T = 1140 sec was 141.24 μm to 247.92 μm , T = 1560 sec was 137.98 μm to 259.64 μm , T = 1980 sec was 226.62 μm to 311.78 μm and T = 2400 sec was 305.37 μm to 332.29 μm , respectively. The bubbles' uniformity and homogeneity decreased while the average bubble diameter increased with time [55]. At 0.23 wt% SDS surfactant foam, the bubble diameter increases from 82.55 to 332.29 μm . These changes show that SDS foam has low stability because of coalescence [20]. However, rapid changes were observed for SDS foam size at different times. This foam generation behavior was due to molecular aggregates (micelles) at surfactant CMC. The presence of oil reduces the maximum disjoining pressure of the liquid films and the screening effect of the electrostatic double layer [42,56]. The formation of molecular aggregates (micelles) at surfactant CMC increases the concentration of surfactants in the bulk solution. They resulted in a rapid surfactant

migration to foam lamellae and high surfactant density at the gas-liquid interface of foam [57]. The available surfactant molecules were insufficient to improve foam stability at 0.23 wt% SDS concentration [31,58].

The microscopic images were captured using concentrations of 0.06 wt% WPI as a case study. The histogram of the bubble-size variations for WPI foams was calculated using a Leica EZ4 HD stereo image, as shown in Fig. 6 the microscopic images of the bubble-size distributions were T = 300 and 780 sec, the bubble diameter of WPI foam less than 18 μm , T = 1140 sec was 18.9 μm to 47.9 μm , T = 1560 sec was 54.2 μm to 82.1 μm , T = 1980 sec was 45.4 μm to 76.2 μm and T = 2400 sec was 39.5 μm to 123.07 μm , respectively. WPI foam bubbles were uniform and homogeneous, the bubble shape remained spherical or ellipsoidal, and the film thickness remained the same at a specific time. The bubble diameter ranged between 18 and 123 μm . We compared the microscopic images of SDS foam in Fig. 5 and WPI surfactant foam in Fig. 6. Found that WPI foam produces finer, more compact, and longer lasting bubbles. Furthermore, WPI foam solutions produced the smallest bubble diameter and the highest density. Whey protein forms a network at the interface with high interfacial elasticity [33,40].

In protein systems, foam stability is mainly due to increase the interfacial viscosity of the protein adsorbed layer appears to be a critical factor in the formation of the foam. In contrast, the interfacial elasticity seems to control both the stability of emulsions and foams [31,59]. The WPI surfactant solutions produced smaller bubble diameters and higher bubble densities. The results showed that 0.06 wt% WPI improves foam

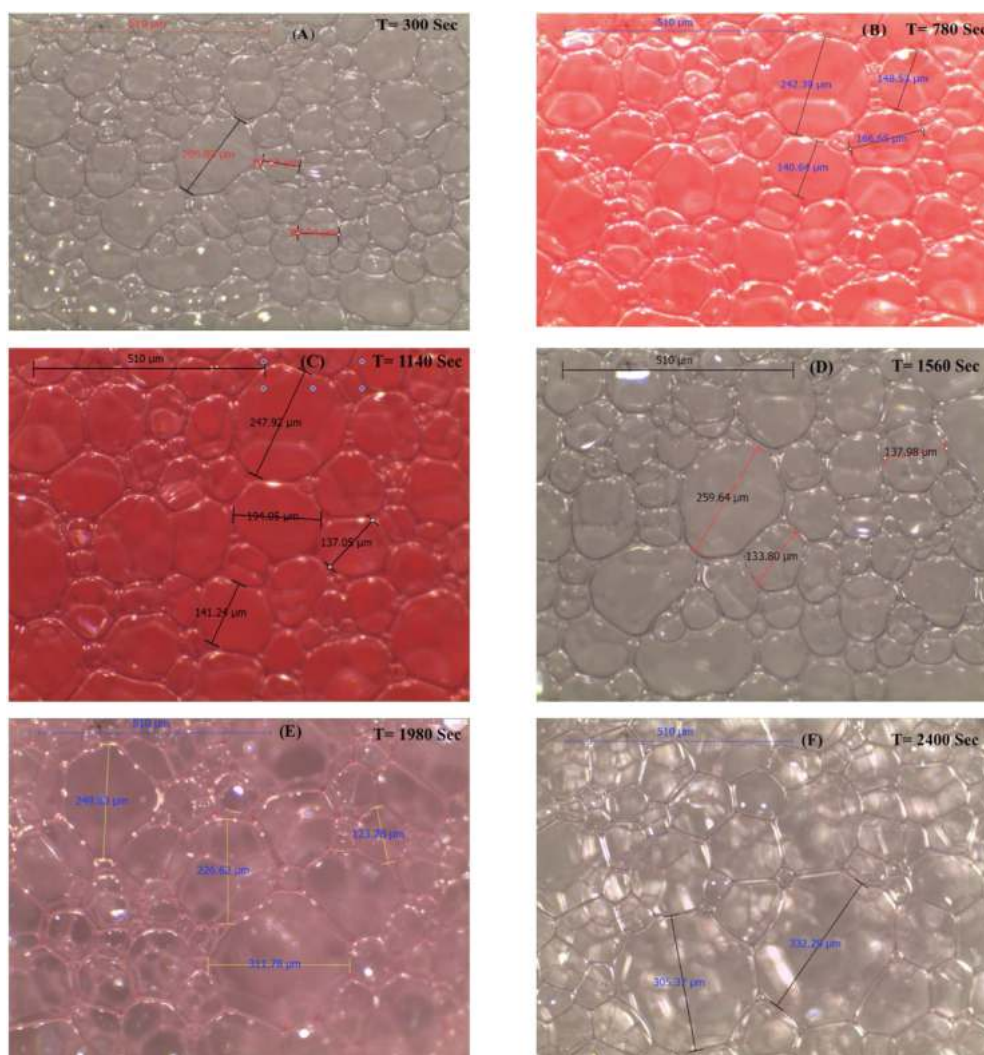


Fig. 5. Microscopic images of 0.23 wt% SDS- CO₂ foam with different time scales at 26 °C.

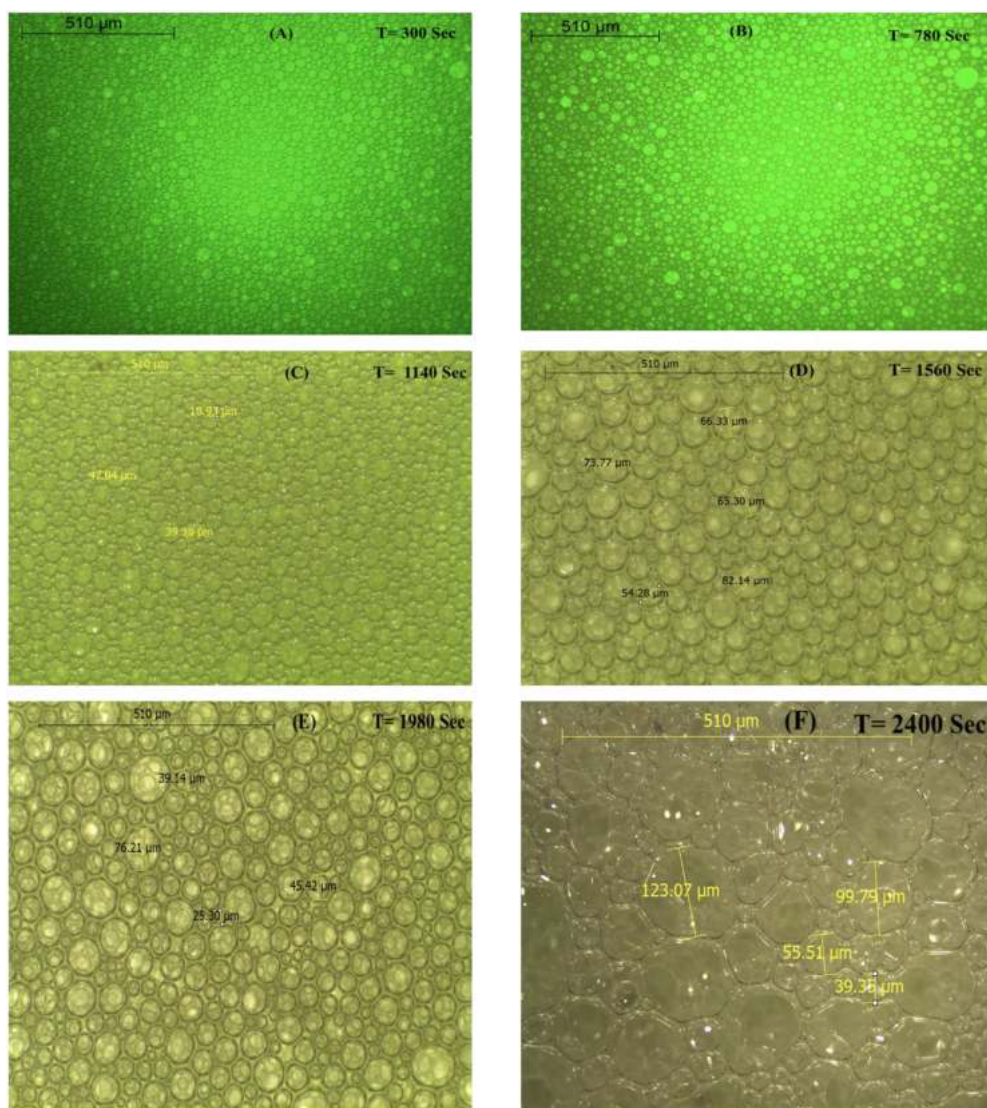


Fig. 6. Microscopic images of 0.06 wt% WPI- CO₂ foam with different time scales at 26 °C.

stability by influencing bubble morphology.

4.5. The effect of oil presence on (WPI, SDS)-CO₂ foam stability

Oils generally destabilize and stabilize a foam system [23]. A preliminary foam stability test was carried out using deionized water and three different types of crude oil. The stability of the generated foam was investigated by tracking changes in the foam height produced in the graduated cylinder over time. As discussed in the methodology section, SDS and WPI are used to calculate the foam height of the surfactant by measuring the distance between the top of the generated foam and the liquid level in the graduated cylinder. Foam stability was investigated at the bulk scale in the presence of Hamada crude, Sharara oil, and Malaysia waxy oil; 0.06 wt % WPI and 0.23 wt % SDS foams were generated. Fig. 7 and Fig. 8 show the foam decay profiles for WPI and SDS surfactants used in this study for Hamada oil, Sharara oil, and Malaysia crude oil, respectively. In other words, 0.06wt% WPI has generated stable foam with Hamada crude oil and aqueous phase as compared to SDS anionic surfactant.

Foam stability was investigated in a series of experiments to investigate the surfactants' ability to generate stable foams in the three different oils. Fig. 7A shows the foam decay profiles for WPI surfactant used in this study for Hamada, Sharara, and waxy crude oil, respectively.

Fig. 6 confirms the destabilizing effect of oil on foams compared to the case where oil was absent (comparing Figs. 3 and 7A and B). The effect of three crude oil and their saturation on WPI foam stability. The crude oil samples were injected into the foam column after injection of surfactant solution. Then, CO₂ has flowed through the column, and foam was generated by passing CO₂ over the mixture of oil and surfactant solution. Fig. 6 shows the results of a series of foam stability tests carried out using 0.06 wt% WPI and CO₂ versus saturation of the three crude oils. As can be seen at 5% in the case of Hamada crude oil, oil has not reduced the formability, but the foam stability was reduced from 2449s to 1221s. The existence (10–20%) of crude oil gradually reduced the stability of the foam, but the reduction was more pronounced when the oil saturation increased to 30%. This indicates that the oil phase reduces foam longevity, at different degrees.

The generated foams enter the decaying regime soon after stopping gas sparging. The overall trend of the foam decay was rather similar for Hamada oils, but it varies considerably in detail depending on the type of oil used. First, at 5% of Sharara oil, foam volume diminishes steeply to 290 mm, slightly lower than the initial volume (315mm). The increased oil concentration presence of WPI foam exhibits the least stability for which oil destroyed the foam column of WPI after 190 s (Fig. 7B). However, for the waxy crude oil, foam volume has a finite value after 477 seconds, while the foam height was decreased to 220 mm [19,31,

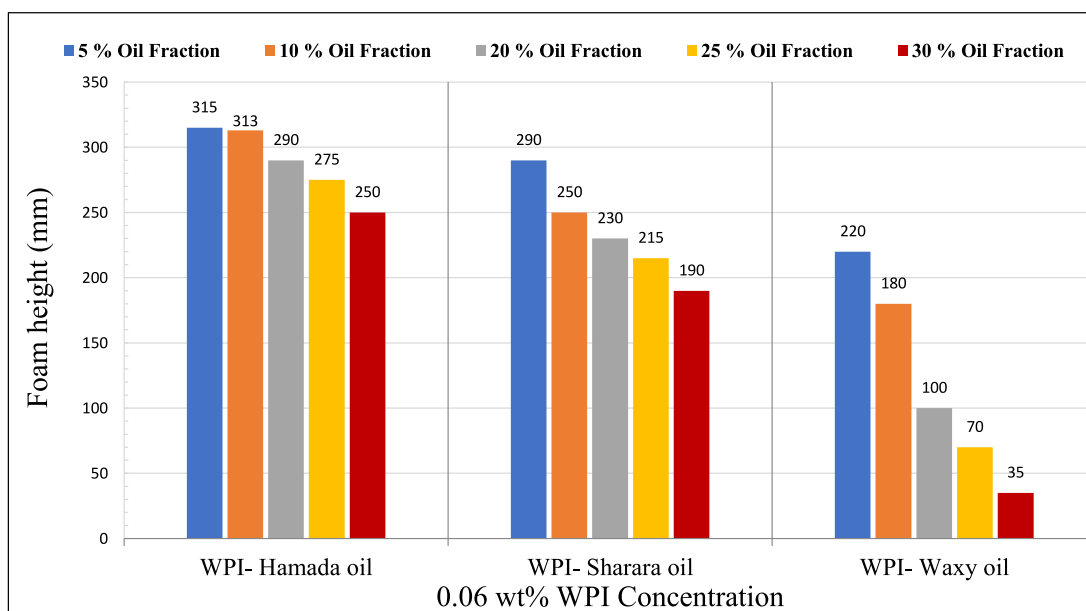


Fig. 7A. The Foam height of 0.06 wt% WPI in different oils.

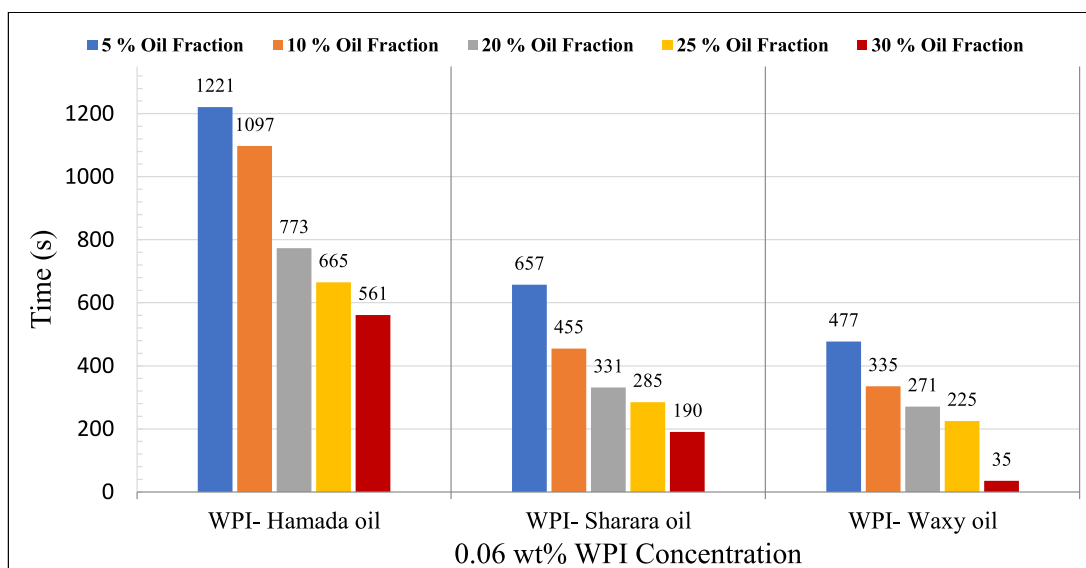


Fig. 7B. The foam half-life time of 0.06 wt% WPI for different oils.

60].

Fig. 8A illustrates the bulk foam stability of 0.23 wt % SDS with different crude oils (5, 10, 20, and 30%) at a temperature of 25 °C. The graph shows that Hamada oil has the least destabilizing effect on foam stability. Sharara oil and waxy crude oil also destabilize SDS foam. At 5%, there was an effect on SDS foam stability for Hamada oil. It decreased from 300mm to 285mm. SDS foam decreased sharply with Sharara and waxy oil. Therefore, the presence of oil reduces the foam's stability [61]. Sharara oil destabilises SDS foam more than Waxy crude oil (150, 180 mm). The results show that increasing the oil concentration from 5 to 30 wt% decreased the SDS foam column height and foam half-life. However, the difference in foam height between 5 and 10% added oil was not significant. Oil was harmful to conventional SDS surfactant foam and can cause it to degrade quickly [31,62,63].

Fig. 8B shows the influence of oil on SDS-CO₂ foam static stability determined through half-life in the stability of CO₂-foam. The results

indicates that the presence of oil has a destabilizing effect on the stability of SDS foams [64]. The higher the foam half-life, the more stable the foam and vice versa. The foam stability decreased with decreasing oil viscosity and density. However, there was no considerable improvement in the stability of the SDS foam [31,64].

The SDS-CO₂-foam half-life times decreased from 285 s to 205 s when Hamada oil was present in the maximum concentration test in this investigation. In Fig. 8A and 8B. It can be seen that, low viscosity and low-density oil are more detrimental to CO₂ foam stability than high viscosity and high-density oils. In addition, the result is less significance in conventional foam stability than oil. The destabilizing influence of oil on the CO₂ foam stability according to the half-life and the normalized foam height is Hamada oil is greater than Sharara crude oil while Sharara is greater than Waxy crude oil. Similarly, a significant influence of oil viscosity and density on foam stability is observed. The more viscous and denser the oil used in our experiments, the more stable the

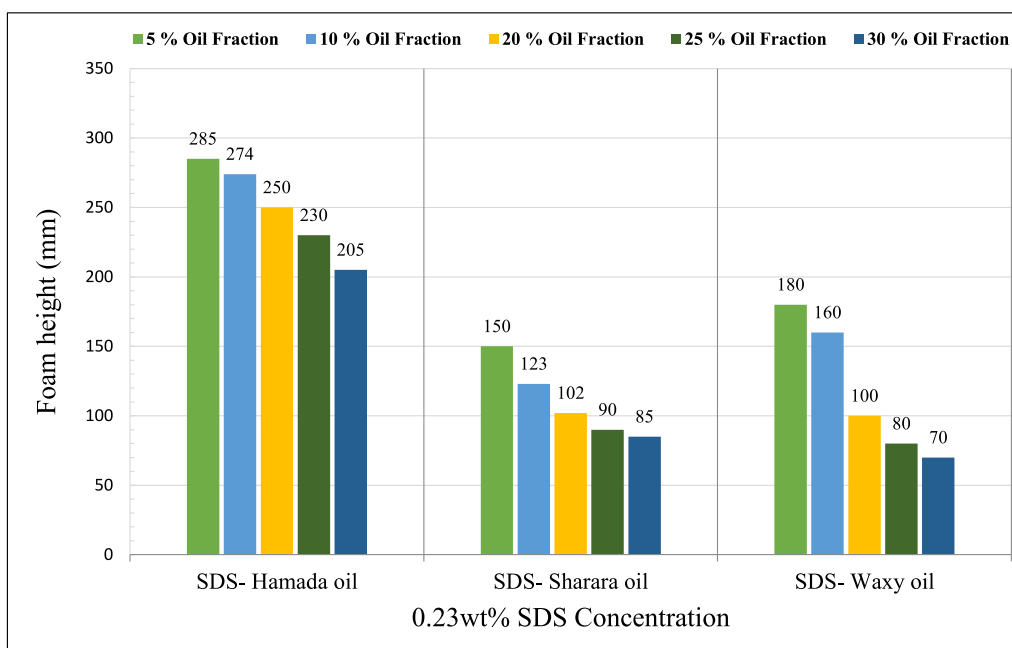


Fig. 8A. The Foam height of 0.23 wt% SDS for different oils.

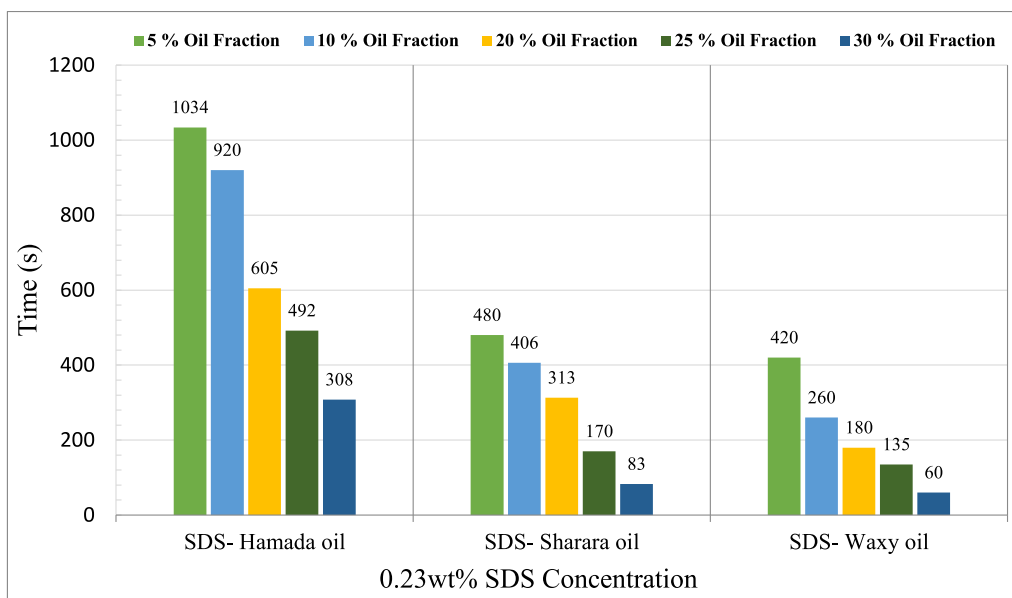


Fig. 8B. The half-decay times of SDS foams in different oils.

CO₂ foam is in the oil.

The results revealed that SDS foam half-life in the three oil samples decreased, indicating weak foam stability. The finding is well supported by other literatures using a wide range of oils viscosities from light to heavy oils [16,64,65]. However, increasing the concentration of WPI in the foam structure substantially enhances stability in the presence of oil. As a result, SDS conventional foam, Waxy oil, Sharara crude oil were more detrimental to foam stability than Hamada oil [66,67]. The results of this study's influence of oil presence on CO₂ foam stability experiments are consistent with previous findings that oils with low viscosity and density are more detrimental to foam stability [19,22]. The low stability of foam in the presence of oil with low viscosity and density can be attributed to the increasing entering and spreading of oil at the foam

lamellae [5,18,68]. The increasing entrance and oil spread on the film interface forces liquid out of the films and into the plateau borders. This action accelerates film thinning, liquid drainage, and foam coalescence; the intensity of oil droplet dispersion in bulk foam increases as oil viscosity and density decrease. As a result of the increased interaction between the oil and the foam at the gas-liquid interface, foam stability decreases [69,70].

4.6. Foam destabilization by oil

Under the Leica EZH4 microscope, the morphology of the generated foam is depicted in Fig. 9. The result showed the microscopic images at 0.06 wt% WPI and 0.23 wt% SDS foam generated in crude oil. This have

provided further insight into the mechanisms of the foam decaying process. When the generated WPI foam bubble size distribution was relatively uniform, as shown in Fig. 6, the WPI foam remained stable with thicker lamellae with time 2449s, as shown in Table 3. However, when the generated SDS foam bubble size distribution was highly dispersed and non-spherical, as illustrated in Fig. 5, the rate of bubble coalescence was faster with time. The foam lamellae become thinner while smaller unstable and inhomogeneous bubbles quickly merge to become larger bubbles. Hence bubble coalescence and Ostwald ripening were principal foam decay processes when the bubble size distribution in the foam structure is highly dispersed and irregular. The bubble coarsening plays a prominent role in the foam decay process if the bubble size distribution in the foam structure was highly dispersed [71–73].

Fig. 9 displays the images of SDS foam crude oil interaction. There was rapid entering and spreading of oil into the thin liquid films of the SDS-foam [72,74]. The oil droplets enter the gas/water interface of the SDS foam, resulting in film rupture and bubble coalesce. In the presence of WPI, the entering and spreading of oil were hindered due to the high stability of the WPI foam compared to the SDS foam. WPI foam can be attributed to hydrophilic sugar part and fat-soluble (hydrophobic). Moreover, Fig. 7 B showed that it could take a longer time for the liquid to drain from the structure of the WPI foam film compared to SDS foam. These results suggest that the surface viscosity and strength of the WPI foam film are high due to the adsorption and aggregation of the molecules of the surface-active species at the foam lamellae. A dense monolayer was formed from the accumulated species at the lamellae, which delays the gas diffusion through the foam film [20]. Moreover, the strength of the foam lamellae was very significant in delaying bubble coalescence and Ostwald ripening.

For the investigated mechanisms of the foam stability improvement by WPI surfactant in the presence of oil, the morphology of the WPI-CO₂ foam micro-bubbles were investigated in the presence of oil under a Leica EZ4 HD microscope. The presence of WPI at the film interface hampered the entry and spread of light oil interactions, as demonstrated in Fig. 10. The oil could not spread at the WPI film because the WPI was a complex molecule that generates highly viscoelastic layers with high surface shear viscosities [75,76]. In addition to adsorption and accumulation of the protein at the lamellae of WPI foam. The significant resistance of WPI-CO₂ foam observed in this study in the presence and absence of oil is due to protein adsorption at the generated foam's gas-liquid interface. WPI forms a thick adsorbed layer with good interfacial rheological properties that retard the rupture of thin films, thus providing stability [77]. The driving force WPI was lowering free energy due to exposure of its hydrophobic groups (surface and interior) to an air/water interface. Consequently, the surface activity of the WPI molecule was dictated by the primary sequence of amino acids [33]. WPI aggregated at the foam lamellas and plateau borders to promote foam stability by delaying the rate of film thinning, liquid drainage, bubble

coalescence, and coarsening [77,78].

These images in Fig. 11 A, B can show the reason for more stable foaming solutions in Libya crude oil than waxy crude oil. Fig. 11 (A) show the dense assembly of droplets trapped and jammed in between the bubbles increased the local viscosity. In addition, it reduced the rate of thinning films and Plateau borders shrinking [79], resulting in a slowing down of the coarsening phenomena [68]. Note that there was enough free surfactant present in water in a higher fraction of oil to improve the foam stability. From Fig. 11 (B) show that the microscopic images of waxy oil emulsion within the foam lamella demonstrated flocculation of several oil droplets within the lamella [51,80,81]. The flocculation was by droplet coalescence and the formation of a larger oil droplet or oil lens, which was detrimental to foam stability [18,48,82].

4.7. Film thickness and strength

Fig. 12 shows the images of WPI (with 0.06 wt% concentration) bubbles with crude oil. As observed under the Leica EZ4 microscope, the film thickness for the WPI foam is between 7.35 μm to 12.61 μm in the crude oil. For WPI foam, the shape of the bubbles remains either spherical or ellipsoidal, and the film thickness remains the same. The increase in film thickness of the WPI foam was adsorption and aggregation of the WPI surfactant at the oil-water interface increases with increasing concentration. At this point, the surfaces of the bubbles are optimally covered protected from coalescence and coarsening [83]. Fig. 12 also demonstrated that the 0.06 wt% -WPI foam was more stable than the 0.23 wt%-SDS foam. WPI foam was uniformly circular or spherical (Fig. 9).

According to the results of previous studies, A smaller initial bubble size can contribute to more extended foam drainage half-life [48]. The WPI foams show higher stability than SDS foams compared to the bubble size. The regular spherical shapes are associated with durable foams, whereas irregular polyhedral shapes are related to unstable foams [63, 85]. Previous research has identified thicker lamellae and increased film interfacial elasticity as mechanisms of foam stabilization due to adsorption and aggregation of at thin liquid films and Plateau borders [42,48,80]. Protein increased the lamellae's thickness and the interfacial film's elasticity in the surfactant solution. On the contrary, a higher liquid viscosity can slow down the foam drainage rate due to a slower liquid flow through the foam network. This has been observed in several studies [55,84,86].

Finally, this liquid flow provides the resistance against the thinning of liquid film Surfactant blend with the concentration of 0.06 wt% WPI generated foam with small oil droplets due to its low IFT. Oil droplets have little effect on foam stability on foam lamellae and stable bubbles of various sizes. WPI increased the viscosity of the solution while decreasing the rate of gas diffusion [31,77,87]. Sucrose in WPI enhances the air/water interfacial tension of water and thereby influences gas solubility. A high interfacial deletional elasticity reduced the

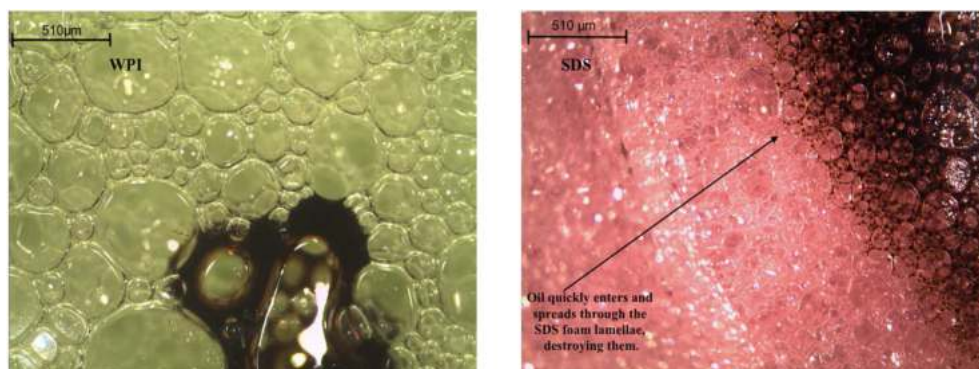


Fig. 9. WPI and SDS foam lamellae oil.

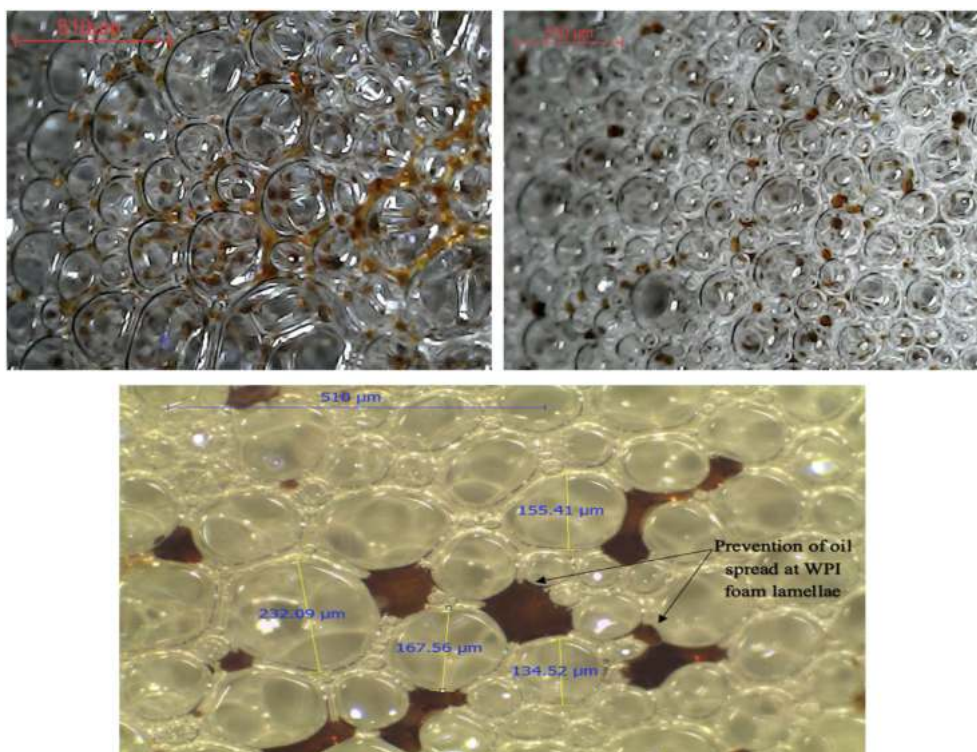


Fig. 10. WPI foam lamellae crude oil entry and spreading.

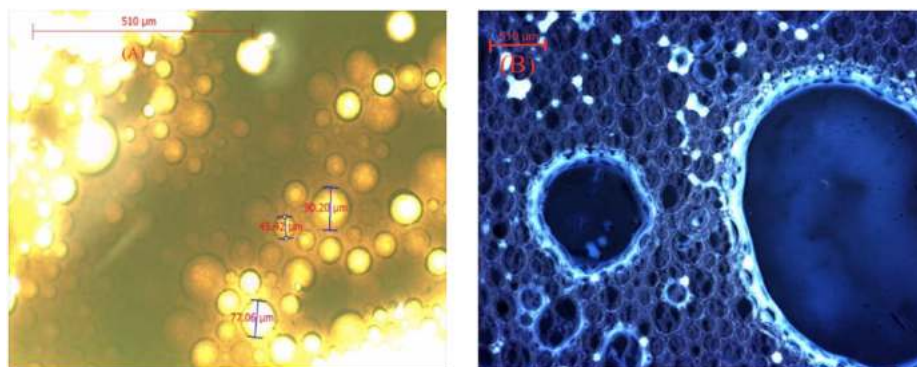


Fig. 11. (a) WPI foam Libya crude oil and (b) WPI foam waxy crude oil.

destabilization of WPI foams caused by disproportionation and coalescence [86,87]. At 0.23 wt% SDS- CO₂ foam with crude oil collapsed due to its high value of IFT compared to the other tested WPI surfactants. This type of surfactant blend generated large oil droplets and collapsed due to the steric effects, in which lamellae become thinner, and foam becomes more fragile. The average oil droplet becomes larger and breaks the foam due to the limited elasticity of the plateau border. When the surface was subjected to liquid film expansion, the local surface concentration decreases with increasing surface area, and the film becomes thinner. The low concentration of surfactant leads to high surface tension. A high concentration of surfactant causes a reduction of the surface to maintain low energy. This surface reduction induces liquid flow in the film from the low-tension region to the higher tension region [16,31,48].

5. Conclusion

A systematic study was performed to examine the bulk foaming characteristics of surfactants. Whey protein isolated (WPI) was

investigated as an enhanced oil recovery agent and as a prospective supplementary alternative to conventional EOR surfactants compared to sodium dodecyl sulfate. WPI and SDS on the static and dynamic stability of CO₂ foams in the absence and presence of Hamada crude oil, Sharara crude oil, and Waxy crude oil with differing viscosity and density. The following conclusions were drawn from the initial findings and analysis: By increasing the surfactant concentration from 0.06 wt% WPI provided the highest foam longevity in the absence of oil and enhanced foam stability considerably in the presence of oil. Liquid volume in the foam, foam stability, oil viscosity and oil density, as well as oil components, all contributed to WPI foam performance. Light oil (Hamada crude oil and Sharara crude oil) positively affects the foam within a certain oil content. Consequently, waxy oil had a higher tendency to destabilize the WPI foam. WPI reduced the oil-water interfacial tension to very low and ultra-low values. However, the IFT values achieved at critical micelle concentration (0.06 wt% WPI) in comparison to the original IFT values of Hamada crude oil (11 mN/m), Sharara oil (21 mN/m), and waxy crude oil (35 mN/m) showed a considerable reduction at room temperature. The SDS foam stability decreases while the size of generated

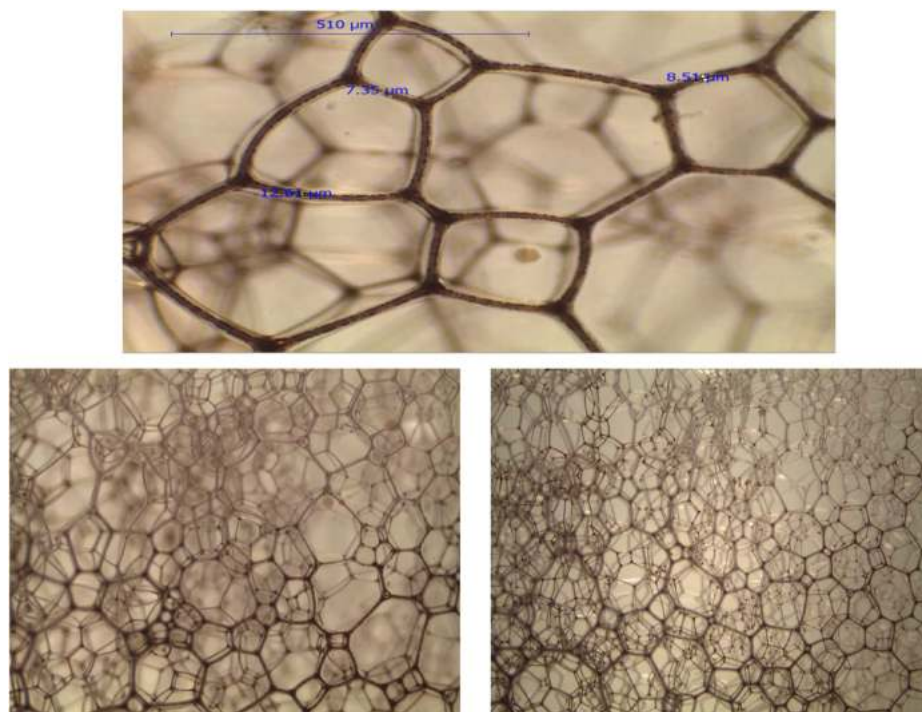


Fig. 12. WPI Lamella thickness with crude oil.

bubbles increases with reducing oil viscosity and density. Furthermore, there was no improvement in the stability of SDS foam in the presence of three types of oil. SDS foam was highly irregular compared to WPI foam, which had a perfectly spherical shape. Due to their non-spherical nature and low dispersion in surfactant solutions, SDS foams will find it difficult to form a network of particle aggregates at the foam lamellae. WPI foam surfactant solution improves the foam half-life and film thickness, decreases bubble sizes, and prevents oil from spreading at the foam lamellae.

Declaration of competing 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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