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## Enhancing Oil Recovery from Oil-Wet Carbonate Rock by Wettability Alteration and interfacial tension Reduction Using Hawthorn Leaves Extract as a New Natural Surfactant

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### Highlights

- Increasing the Hawthorn leaves extract (HLE) natural surfactant concentration from 0 to 10 wt % reduced the interfacial tension (IFT) from 35.2 to 10.98 mN/m.
- HLE changed the oil-wet carbonate rock toward water-wet compared to the other chemical surfactant, SDS and DTAB.
- The oil recovery significantly increased from 37% water flooding to 54.7% at 1.3 pore volume of the HLE flooding.

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### Abstract

Oil recovery from oil-wet carbonate rock is a significant challenge in the oil industry. The present study investigates the influence of the natural surfactant Hawthorn leaves extract (HLE) on oil recovery from carbonate rock. Two chemical surfactants, sodium dodecyl sulfate (SDS) and dodecyl tri methyl ammonium bromide (DTAB), were used to validate and compare oil recovery with the new natural surfactant HLE. A wettability alteration test using the contact angle method, an interfacial test (IFT) using pendant drop, and core flooding were employed to investigate the behavior of the surfactants on oil recovery. The experimental results show that the critical micellar concentration (CMC) point of different concentrations of HLE, SDS, and DTAB solution occurs at 3.25, 3.00, and 4.06 wt %, respectively. In wettability alteration, the natural surfactant HLE is more effective than other chemical surfactants (SDS and DTAB) at the CMC point. As observed, the contact angle of the carbonate pellet and the HLE at the CMC point is 86°, and this angle for SDS and DTAB is 112° and 92°, respectively. The core flooding results show that the oil recovery factor improves from 37% with water flooding to 47.6% with SDS, 56.2% with DTAB, and 54.7% with HLE. The results prove that this new natural surfactant (HLE) can be used as a novel surfactant for the chemically enhanced oil recovery process in carbonate oil reservoirs. HLE has beneficial effects in oil recovery because of its environment friendly compared to SDS and DTAB.

**Keywords:** Contact Angle, EOR, Hawthorn leaves extract, IFT, Wettability alteration.

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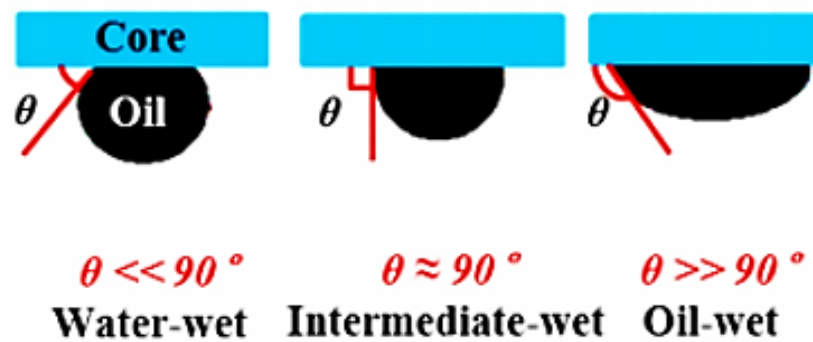
## 1. Introduction

More oil production from mature reservoirs using oil recovery techniques is significant in worldwide energy demands. Carbonate reservoirs include almost half of the world's discovered oil reserves, and it has been proven that many of these reservoirs are naturally fractured (Roehl and Choquette, 1985). Heterogeneity is one of the inseparable parameters of carbonate reservoirs. These reservoirs often have a high degree of heterogeneity and a complex pore structure (Standes, 2001; Schramm, 2000). Water flooding in carbonate reservoirs has poor efficiency due to wettability and interfacial tension (IFT) at scales of microscopic and macroscopic oil trapping (Manrique et al. 2006). Because of the oil-wet wettability and low permeability of these reservoirs, enhanced oil recovery (EOR) is a great challenge. Wettability alteration can improve the recovery from oil-wet carbonate reservoirs. The positive zeta potential of the rock surface, the hardness of the brine, and the presence of asphaltenes and organic acids in the oil change the wettability of carbonate rocks from mixed-wet to oil-wet (Sharma et al. 2014). The microscopic distribution of fluids in the pore network and fractures strongly depends on the wettability of the reservoir rock (Jadhunandan and Morrow, 1995). As shown in Figure 1, the wettability condition on a solid–liquid system is often distinguished using the contact angle of the liquid phase on a solid surface (Sun et al. 2017). The wettability alteration and IFT reduction help reduce residual oil saturation in the reservoir. The residual oil saturation in porous media strongly depends on capillary number (defined as the dimensionless ratio of viscous force to capillary force), which is related to IFT. By decreasing IFT, the capillary number increases, and by increasing the capillary number, the residual oil saturation is reduced (Mohsenatabar et al., 2018).

There are commercial ionic and nonionic surfactants for wettability alteration and IFT reduction. Today, the new interest in wettability alteration using surfactants focuses on natural, environmentally friendly surfactants. Pordel et al. (2012) used saponin as a new natural surfactant in EOR. The IFT obtained by saponin was the same as other cationic and anionic surfactants used in chemical flooding processes for enhanced oil recovery. Adding salt and alcohol can decrease the IFT obtained from saponin to ultralow IFT (Pordel et al., 2012; Ahmadi and Shadizadeh, 2013; Amirpour and Shadizadeh, 2013). Ahmadi and Shadizadeh (2013) extracted a natural-based surfactant from the leaves of the mulberry tree. The results demonstrated that a micro-fluid containing only 1 wt % of micro-sized mulberry leaf particles could lower the IFT of a system consisting of distilled water and kerosene by 60%. Saran (2017) experimentally investigated spontaneous imbibition in enhanced oil recovery by extracted Glyrrhiza Globra Linn (GGL). In order to investigate the effect of GGL on oil recovery, two series of imbibition experiments were conducted on water-wet carbonate and sandstone rock plugs and oil-wet carbonate samples. Oil recovery was about 27.22% of the original oil in place (OOIP) for water-wet sandstone plugs using GGL. In the same test, the oil recovery was about 23.56% OOIP for the water-wet carbonate plug. Adding GGL increased the recovery in oil-wet carbonate plugs due to reduced interfacial tension from 17.83% OOIP to 20.76% OOIP. It was concluded that if GGL was added to the injection water, the imbibition recovery was higher than in the case of imbibition with water (Saran, 2017).

Various experimental works have been used to research the mechanism of wettability change of oil-wet rock by plant surfactants (Emadi et al., 2019; Bassir and Shadizadeh, 2020; Nowrouzi et al., 2020; Shadizadeh et al., 2021; Esfandyari et al. 2021). This paper presents the effect of Hawthorn leaves extract (HLE) as a new environmentally friendly surfactant on wettability alteration and oil recovery in carbonate oil reservoirs. In this work, some tests were performed to investigate the effectivity of HLE on wettability alteration and IFT reduction and to find critical micellar concentration (CMC). Core flooding in critical micelle concentrations was conducted to perform oil recovery. Wettability alteration, IFT reduction, and core flooding were conducted to compare the performance of the HLE with

commercial surfactants, sodium dodecyl sulfate (SDS, an anionic surfactant) and dodecyl tri methyl ammonium bromide (DTAB, a cationic surfactant).



**Figure 1**

A schematic of rock wettability conditions [7].

## 2. Experimental methodology

### 2.1. Materials

#### a) Hawthorn berry (*Crataegus oxycanthus*)

Hawthorn is commonly used for curing heart disease around the world. It is used to control and promote the circulatory system. As shown in Figure 2, the tree, fruits, and leaves of the Hawthorn berry are found in the jungle. Table 1 lists the chemical, physical, and microbial properties of Hawthorn berry. In this study, the Soxhlet extraction method using deionized water for 12 h was performed to extract the solution from the leaves of Hawthorn.



**Figure 2**

The a) tree, b) fruits, and c) leaf of Hawthorn berry.

**Table 1**

The physical, chemical, and microbial properties of the extract powder of the Hawthorn berry leaf.

Product	Total extract powder of Hawthorn berry
Used	Part leaves
Preparation	Spray dryer
Description	Fine powder
Color	Light to dark brown
Solubility in cold water	Soluble
Density (g/ml)	1.0–1.05
pH	4.5–6.5
The dried residue (w/w %)	3.0–5.0
Odor	Specific odor
Identification	Plant drug analysis
Clarity	Clear liquid
Salmonella species	Negative
E. coli	Negative
Microbial count (cfu/g)	More than 500

#### b) Carbonate core and pellet samples

Carbonate cores were taken from the South Pars Oil Field in Iran. Some pellets (flat polished surface) have been taken from these cores for contact angle measurement. Figure 3 shows the carbonate core and pellets prepared for this study, and Table 2 lists carbonate rock characterization.

**Figure 3**

The photos of carbonate core and pellets.

**Table 2**

Carbonate core characterization.

Diameter (cm)	3.7
Length (cm)	6.5
Bulk volume (cm <sup>3</sup> )	69.88
Pore volume (cm <sup>3</sup> )	8.4
Porosity (%)	12
Permeability (md)	5.6
<i>S<sub>wi</sub></i> (%)	23.8

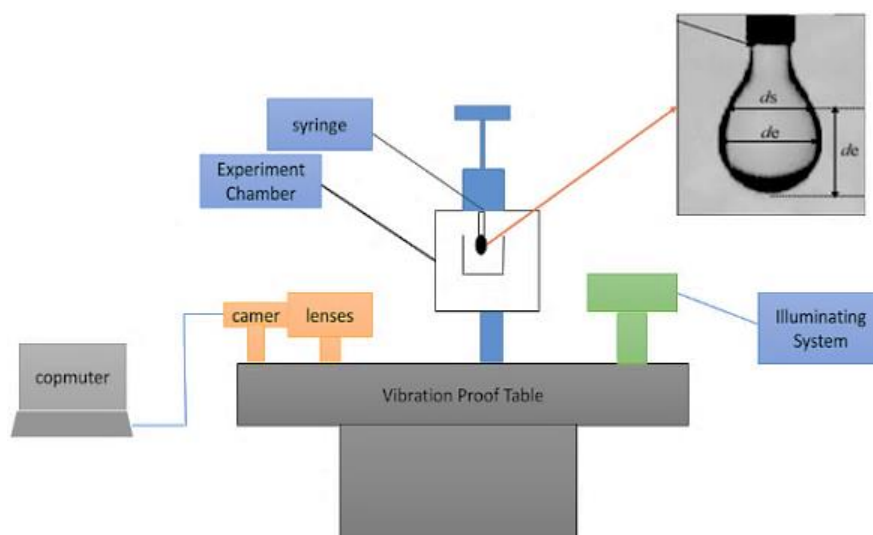
#### c) Crude oil sample and the aqueous phase

Kerosene was chosen as the oil phase in IFT and contact angle measurements because of the cleanness

of kerosene in comparison with crude oil. The density and viscosity of synthetic kerosene at 25 °C were 0.8017 gr/cm<sup>3</sup> and 1.9 cP, respectively. The crude oil used in flooding tests and age were obtained from the Sarvestan oil field in southern Iran. Crude oil has a density of 0.8031 gr/cm<sup>3</sup> and a viscosity of 2.306 cP at 25 °C. Distilled water with different concentrations of HLE, SDS (anionic surfactant) with the formula CH<sub>3</sub>(CH<sub>2</sub>)<sub>11</sub>SO<sub>4</sub>Na, and DTAB (cationic surfactant) with the formula CH<sub>3</sub>(CH<sub>2</sub>)<sub>11</sub>N(CH<sub>3</sub>)<sub>3</sub>Br was used as the aqueous phase.

## 2.2. Experimental procedure

In EOR, surfactants are applied in concentrations close to CMC. Firstly, different approaches are used to determine the CMC of surfactant in the aqueous phase, including pH and interfacial tension methods. These methods were employed to determine the CMC of HLE, SDS, and DTAB. Different solutions of the surfactants in distilled water in the range 1, 2, 3, 4, 5, 6, 8, and 10 wt % were implemented to overcome this issue. Pendant drop apparatus was employed to measure solutions–oil interfacial tension values. Drop shape analysis (pendant drop method) was suitable for measuring IFT. Pendant drop tensiometry, improved by video-image analysis, was considered an exact method for evaluating the IFT of fluid–fluid interface (Figure 4). In this study, as described by Drelich et al. (2002), the pendant drop method was used to calculate the interfacial tension from fluids properties and drop shape. The kerosene was chosen as a bulk fluid and distilled water (DW) and surfactant solutions as the dropping fluid.

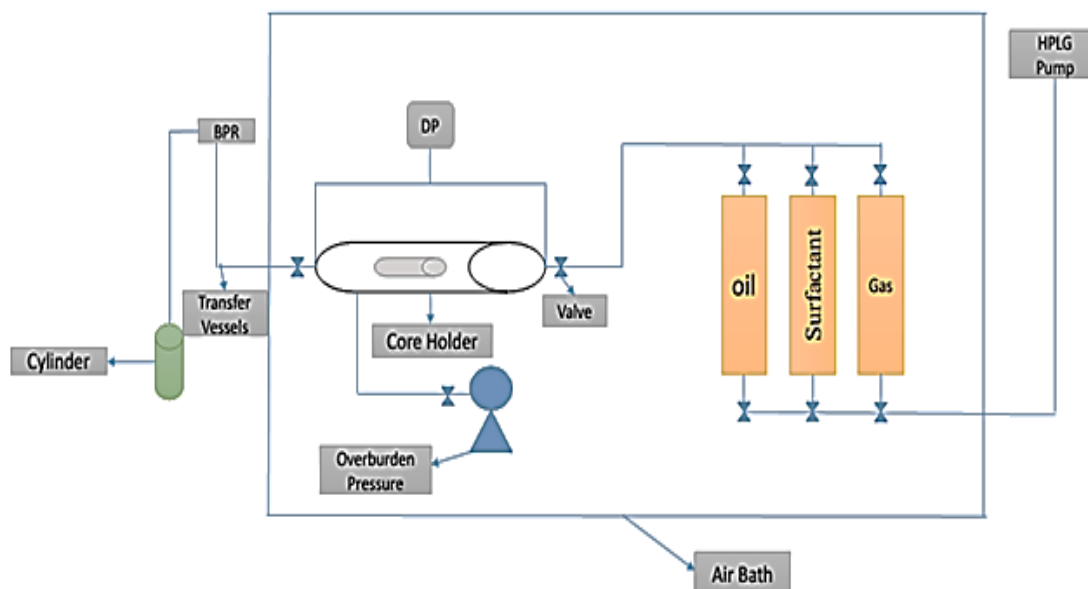


**Figure 3**

A schematic of the IFT measurement apparatus.

Furthermore, the wettability of the carbonate surface was evaluated using the static sessile drop method. The wettability alteration was verified by measuring and comparing the contact angle between the water drop and a single pellet aged at the surfactant concentrations. First, pellets were aged in the crude oil (Sarvestan oil field) for two weeks to become oil wet. Then, one of these plates was selected to determine the wettability condition using DW. For two weeks, the other rock pieces were aged in different concentrations (0–10 wt %) of surfactants solution (HLE, DTAB, and SDS). Finally, the water drop was dispersed on the rock piece, and the contact angle was measured. In the next step, the core flooding apparatus was considered for measuring the initial water saturation, original oil in place, and residual oil saturation (Figure 5). The saturated core by DW was flooded by crude oil to reach irreducible water saturation. The dynamic and static producer was used to have cores oil-wet before water and surfactant flooding. In the dynamic condition, crude oil injection was continued for 72 h to

the core to become oil-wet. Then, the core was removed from the holder and kept static at 60 °C for three weeks. At the end of this period, the core was adjusted in the core holder in ambient conditions. Then, the core flooding apparatus injected the distilled water at the rate of 0.5 ml/min. The oil recovery factor was plotted versus the injected pore volume. Then, the plug was removed from the core holder, and this entire preparation producer was repeated after cleaning by the Soxhlet apparatus. Surfactant flooding (HLE, DTAB, and SDS) was applied at a constant rate of 0.5 ml/min at the CMC of each surfactant.



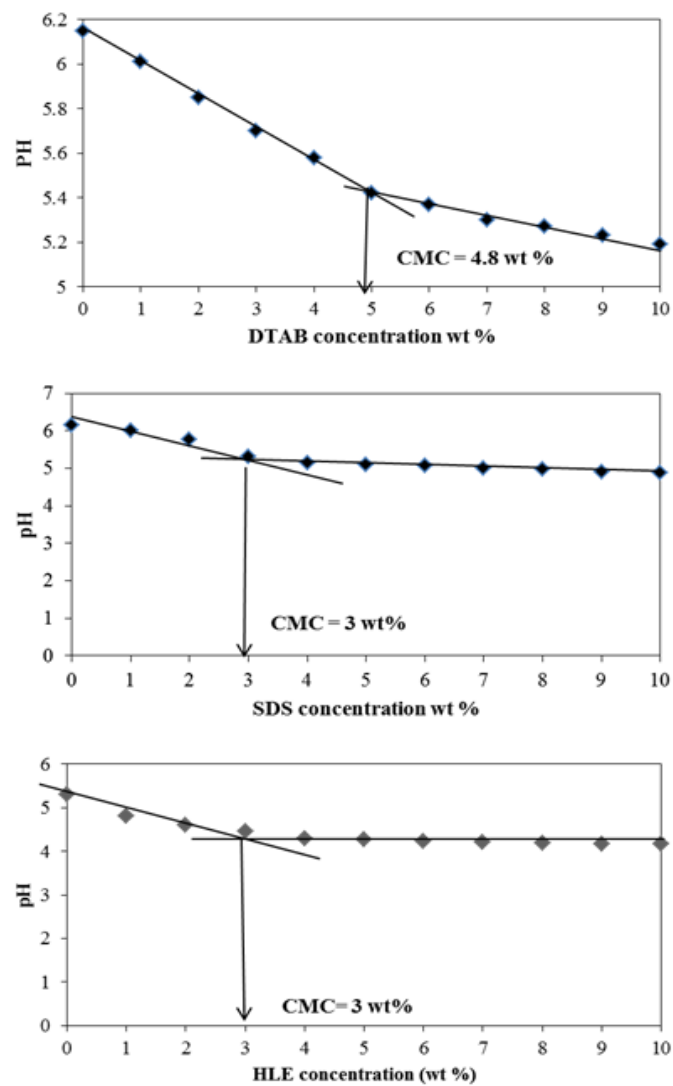
**Figure 5**

A schematic of core flooding apparatus.

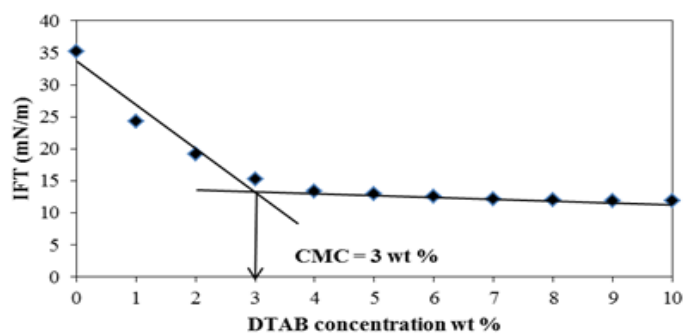
### 3. Results and discussion

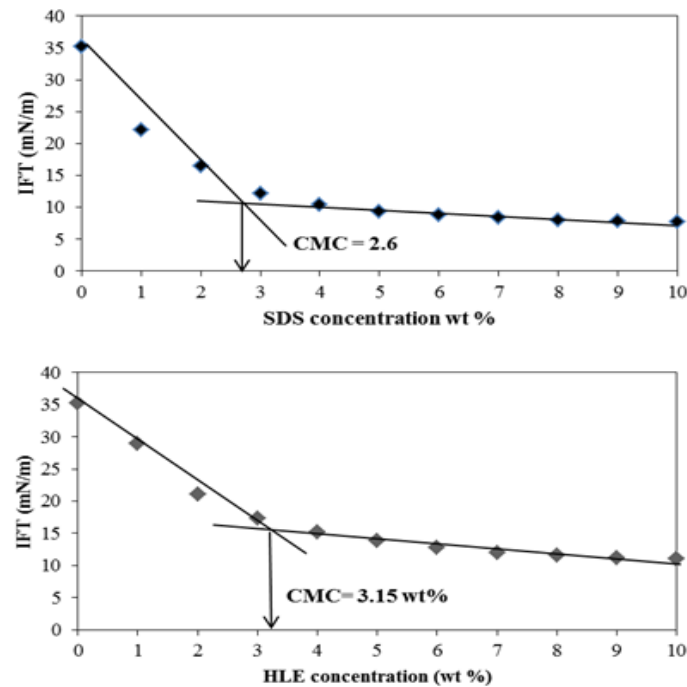
#### 3.1. IFT and CMC

The change of pH and IFT solutions are plotted versus surfactant concentration. As shown in Figure 6, the points after and before the particular concentration lie in two separate lines, indicating the CMC point. The CMC of SDS and HLE is obtained at 3 wt % but at 4.8 wt % for DTAB. These results show that more DTAB is required to obtain the same pH of HLE. Figure 7 shows the increasing surfactant concentration on IFT between surfactant solution and kerosene. The IFT decreases by increasing surfactant concentration. The most efficient IFT reduction will happen at the CMC. At the CMC point, the surfactant monomers in the emulsion have the highest possible concentration, thereby reducing the IFT most. HLE has the same behavior as SDS and DTAB to reduce IFT. The CMC point of HLE, SDS, and DTAB is obtained at 2.6, 3.15, and 3 wt %, respectively. These results show the ability of HLE to be a new natural surfactant. Table 3 presents the CMC value of three surfactants used in this study for core flooding.

**Figure 6**

The CMC determination by the pH measurement method for the used surfactants.



**Figure 7**

The CMC determination by the IFT measurement method for the used surfactants.

**Table 3**

The used CMC value of the surfactants for core flooding.

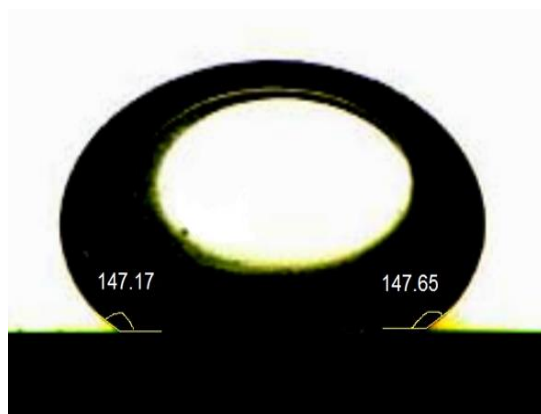
Surfactant	CMC (wt %)
HLE	3.25
SDS	3.00
DTAB	4.06

### 3.2. Contact angle

The contact angle measurement for a pellet saturated in crude oil illustrates that the rock surface is oil-wet ( $\theta = 147^\circ$ ) as the distilled water drop does not extend on the rock surface (Figure 8). Then, the contact angle of the treated surface is measured for all concentrations of HLE, SDS, and DTAB. It is necessary to mention that all measurements have been done under ambient conditions ( $P = 14.7$  psi,  $T = 25^\circ\text{C}$ ). As seen in Figure 9, the pellet, saturated in 1 wt % solution, indicates a contact angle of about  $126.25^\circ$ , and as it comes closer to the critical micelle concentration, the contact angle decreases quickly to about  $86.3^\circ$ . The deal of spreading water drops on the rock surface represents the deal of surface water-wetness. For the higher concentration from 3–10 wt %, the water drop is spread on the sample surface, meaning the surface is water-wet. Figure 10 shows the contact angle versus solution concentration of HLE, SDS, and DTAB. This figure shows that the contact angle decreases by increasing the surfactant concentration. According to the results of contact angle tests, the DTAB is more effective than SDS in carbonate pellets. When the DTAB is applied in the solution, the wettability changes from oil-wet to water-wet condition in pellets. The results also show that HLE is more effective than SDS in carbonate pellets. When the HLE is applied in the solution, the wettability changes from oil-wet to water-wet condition at 7 wt % concentration. Table 4 presents the results of wettability alteration in carbonate pellets by implementing one natural (HLE) and two chemical surfactants (SDS

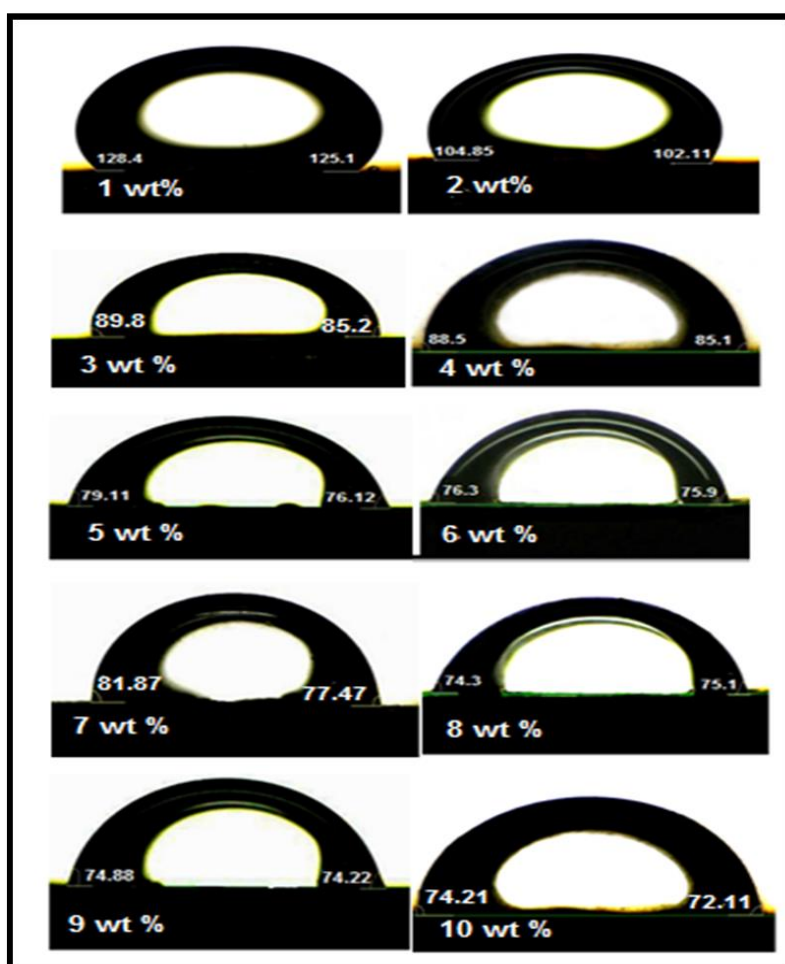


and DTAB). The wettability condition of the rock surface at the CMC point for SDS is oil-wet, but DTAB and HLE tend pellet surface condition to be neutral-wet. These results show that HLE can change the wettability condition of carbonate pellets from intensely oil-wet to neutral-wet at the CMC point.



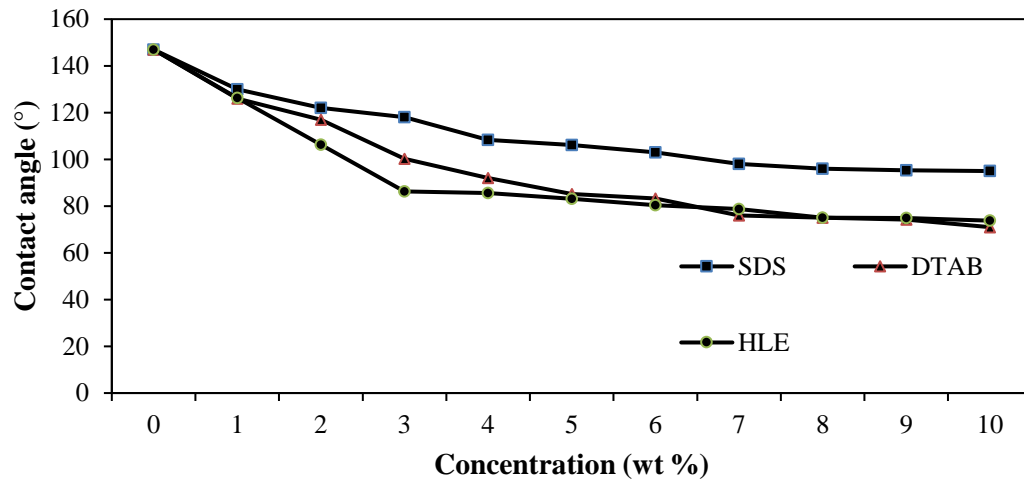
**Figure 8**

The contact angle between the water drop and rock piece saturated in distilled water (0% surfactant concentration).



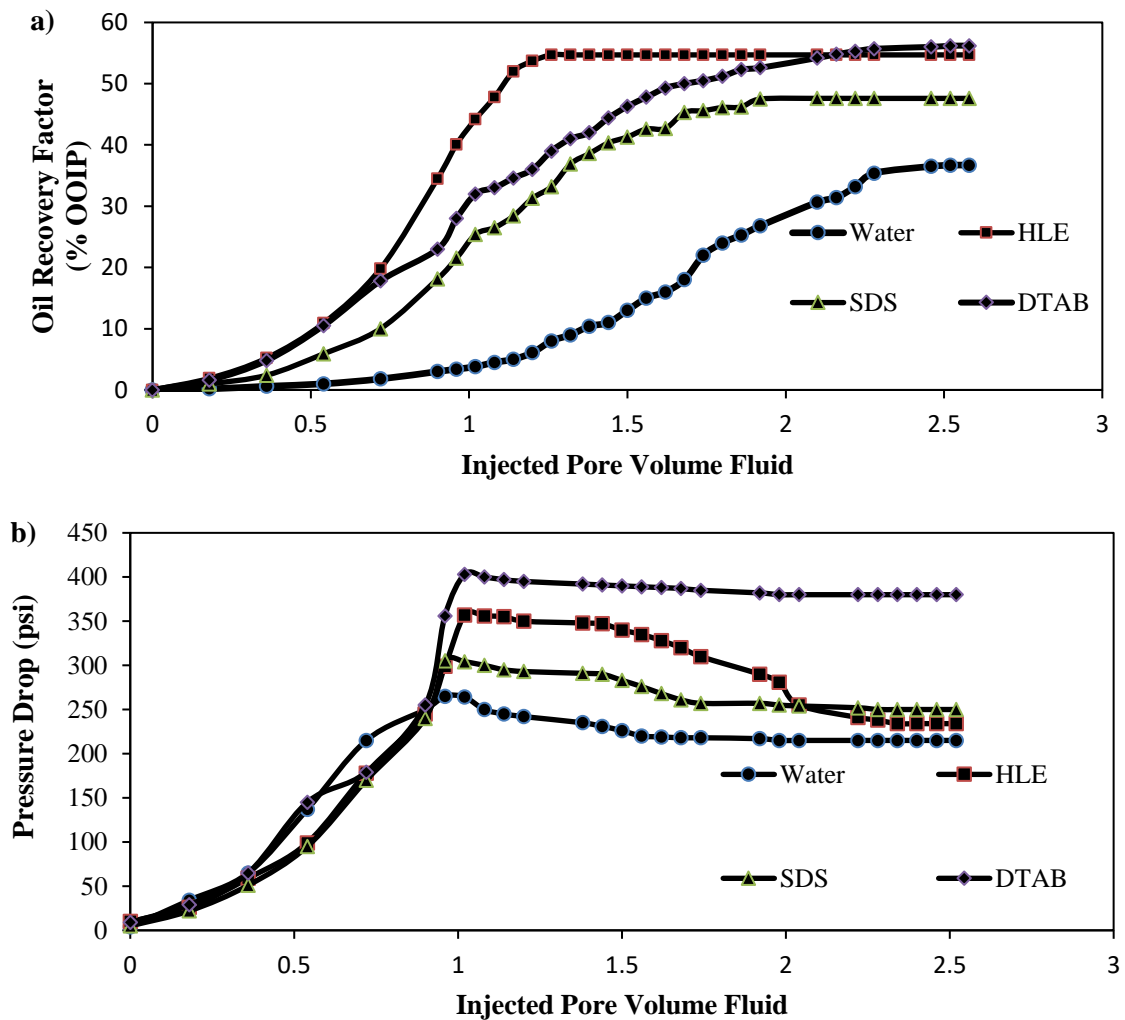
**Figure 9**

The contact angles for an HLE concentration of 1–10 wt %.



**Figure 10**

The variation in the contact angle of carbonate pellets with different concentrations of surfactants.



**Figure 11**

Comparing a) oil recovery factor and b) pressure drop versus injected pore volume of water, HLE, SDS, and DTAB flooding.

**Table 4**

The wettability alteration results of contact angle measurements for HLE, SDS, and DTAB.

Surfactant concentration (wt %)		Contact angle (°)			Wettability		
SDS-DTAB	HLE	SDS	DTAB	HLE	SDS	DTAB	HLE
0	0	147	147	147	Oil-wet	Oil-wet	Oil-wet
1	1	130	126	126.25	Oil-wet	Oil-wet	Oil-wet
2	2	122	117	106.3	Oil-wet	Oil-wet	Slightly oil-wet
3	3.2	118	100.3	86.3	Oil-wet	Neutral-wet	Neutral-wet
		CMC		CMC			
4	4	108.3	92	85.6	Slightly oil-wet	Neutral-wet	Neutral-wet
			CMC				
5	5	106.1	85.2	83.1	Slightly oil-wet	Neutral-wet	Neutral-wet
6	6	103	83.3	80.4	Neutral-wet	Neutral-wet	Neutral-wet
7	7	98	76	78.8	Neutral-wet	Slightly water-wet	Slightly water-wet
8	8	96	75	75	Neutral-wet	Water-wet	Water-wet
9	9	95.3	74.2	74.9	Neutral-wet	Water-wet	Water-wet
10	10	95	71	73.77	Neutral-wet	Water-wet	Water-wet

### 3.3. Core flooding

Figure 11 compares the oil recovery factor and pressure drop for water and surfactant flooding. Figure 11 shows that the oil recovery factor by water flooding reaches 37% after 2.5 PV injections and then becomes stable. The pressure drop during water flooding increases from 0 to 268 psi at the breakthrough time, decreases to 210 psi, and becomes stable. Oil recovery of HLE flooding after 1.3 pore volume injection is 54.7%. Oil recovery increases by about 17.7% compared to water flooding at a lower pore volume injected fluid. The pressure drop during HLE flooding increases from 0 psi, at the start time, to 357 psi, at the breakthrough time; it then decreases to 234 psi at the end of the flooding. The pressure drop curve of HLE flooding is sharper than water flooding. The oil recovery from anionic surfactant SDS flooding increases from 37% water flooding to 47.6%. Finally, by flooding cationic surfactant, DTAB oil recovery increases from 37% to 56.2%. Water flooding has the lowest oil recovery compared to other surfactant flooding due to the intensely oil-wet condition of the rock surface and the high IFT between injected water and crude oil. Water flooding in an oil-wet plug has low oil recovery because oil tends to remain on the oil-wet rock surface. Oil wet condition of the rock surface makes a lubricant condition for injected water. Therefore, injected water tends to leave porous media, increasing the water cut. Surfactant flooding can improve oil recovery by changing wetting conditions from oil-wet to water-wet and reducing IFT between the two phases. The ability of HLE to reduce IFT and alternating wettability can improve oil recovery from carbonate oil-wet rock.

#### 4. Conclusions

As a new environmentally friendly surfactant, Hawthorn leaves extract was used for oil recovery in carbonate oil rock. In this work, some tests were performed to investigate the effectivity of HLE on wettability alteration and IFT reduction. To compare the performance of the HLE with commercial surfactants, sodium dodecyl sulfate and dodecyl tri methyl ammonium bromide, we performed wettability alteration, IFT reduction, and core flooding. The following conclusions were made from this work:

1. By increasing the HLE natural surfactant concentration from 0 to 10 wt %, the IFT declined from 35.2 to 10.98 mN/m.
2. Using pH and IFT measurement, the CMC point of different concentrations of HLE, SDS, and DTAB solution was 3.25, 3.0, and 4.06 wt %, respectively.
3. The wettability contact angle of the carbonate pellet at the CMC concentration of HLE, SDS, and DTAB solution was 86°, 112°, and 92°, respectively. Thus, HLE has changed the oil-wet carbonate rock toward water-wet than other surfactants.
4. The oil recovery significantly increased from 37% water flooding to 54.7% at 1.3 pore volume of the HLE flooding. Anionic surfactant SDS increased the oil recovery from 37% water flooding to 47.6% by injecting 2.0 pore volumes. Cationic surfactant DTAB flooding increased oil recovery from 37% water flooding to 56.2% by injecting 2.5 pore volume.
5. The results prove that this new natural surfactant could be used as a novel surfactant for the chemically enhanced oil recovery process in carbonate oil reservoirs.

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