

Experimental Investigation of Temperature on Interfacial Tension and its Relation to Alterations of Hydrocarbon Properties in a Carbonated Water/ Hydrocarbon System

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Abstract—The study addresses interfacial tension (IFT), and mass transfer in carbonated water (CW)/hydrocarbon systems, using the axisymmetric drop shape analysis (ADSA). Experiments and a mathematical model was used for CW+n-decane system of non-isobaric (10-70 bar) and non-isothermal conditions (25°C, 35°C, and 45°C) using the axisymmetric drop shape analysis (ADSA). Further, the study addresses the inconsistencies of the reported in results reported in the literature between temperature and IFT.

The experimental results (volume changes) and analytical equations have been used to develop a compositional model to estimate the mass of CO₂ diffused into hydrocarbon and the density of n-decane+ CO₂ as a function of time, pressure, and temperature. The obtained densities are then used to estimate real-time and the equilibrium IFT from the pendant drop experiments. The results indicated that for CW-n-decane system equilibrium IFT increases as pressure increases (10–60 bar), depicting an opposite trend to that observed for the CO₂-n-decane system. This was shown to be related to the density difference between the hydrocarbon and the carbonated water, and CO₂ solubility. Further, it was observed that the IFT was inversely related to temperature, which was credited to the changes in the kinetic energy and entropy. Among the three chosen temperatures, it was observed that the IFT at 35°C did not display the same behaviour as that at 25°C and 45°C with pressure and temperature. Up to a certain pressure the IFT at 35°C was lower than at 45°C, and beyond this pressure, the IFT at 35°C was greater than at 45°C.

The present article takes a step in resolving the controversy of IFT vs temperature and building the knowledge by carrying out a non-isothermal and non-isobaric study on the influence of temperature on the IFT in a system containing carbonated water and hydrocarbon. Further, the study gives an insight into the feasibility of carbonated water injection as a successful recovery process.

Index Terms—Carbonated water injection, interfacial tension, pressure, temperature.

I. INTRODUCTION

Carbonated water injection (CWI) is gradually gaining significance as an effective enhanced oil recovery (EOR) method and a solution for long-term or permanent sequestration of CO₂. The increased interest is due to the fact that CWI overcomes the problems associated with the widely applied CO₂ EOR. The mobility of the carbonated water in

contrast with oil is more favorable than CO₂ in the gas-oil system as it forms a single phase, this helps in overcoming the poor sweep efficiency and early breakthrough associated with CO₂ injection [1], [2]. Carbonated water injection is particularly beneficial in watered-out reservoirs in which high water saturation adversely affects the CO₂ injection performance [3]. In the majority of the cases, CO₂ is not available readily, and the actual cost of capture, compression and transport to the reservoir it incurs is high. Therefore, making CO₂-EOR infeasible especially in the fields that are far away from the source. The Carbonated Water Injection forms an alternative CO₂ injection strategy that uses a smaller amount of CO₂ (available from nearby oil and gas fields) and yet can lead to an increase in oil recovery.

Whether it is CO₂-EOR or CWI the swelling and the resulting hydrocarbon mobility are the primary physics governing the oil recovery. The degree of swelling and the mobility depends on the mass transfer of the CO₂ into the displaced fluid (hydrocarbon) from the displacing fluid (CO₂ or CW). The interfacial tension between the displacing fluid and displaced fluid is a major parameter that controls the mass transfer and hence, the extent of oil recovery. Therefore, it is critical to study and understand the interfacial tension between CW and hydrocarbon and further identify the parameters influencing the IFT for optimising CWI.

Although, many studies have been carried out on IFT of CO₂-hydrocarbon system few studies have focused on CW-hydrocarbon system. Further, most of the studies on CO₂ and CW-hydrocarbon system, have overlooked the influence of temperature on the IFT due to its unpredictable relation. At present, there is a lot of discrepancies associated with IFT vs temperature relationship, Mackay and Hossain [4] has reported an inverse relation of IFT with temperature, while Yang, *et al.* [5] has reported a direct relation. Hence, it is important to try to remove or understand why there is a discrepancy when it comes to the influence of temperature on IFT of CW-hydrocarbon system. Apart from the calculation of IFT, it is important to identify the parameters that are influencing and are influenced by the IFT, to optimise the process by possible alteration of these parameters.

The present study through experiments and mathematical model addresses the influence of temperature on the interfacial tension (IFT) for a system consisting of carbonated water (CW)-n-decane at non-isothermal and non-isobaric conditions. Further, the study addresses the inconsistencies in results reported in the literature between temperature and IFT.

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The experiment was designed to resemble the contact between CW and residual oil, as CW is injected, and to successfully use the results in flooding experiments. An Axisymmetric Drop Shape Analysis (ADSA) pendant drop technique has been used to carry out IFT analysis for a pressure range of 10–70 bar and at 25°C, 35°C, and 45°C. IFT during the diffusion (dynamic) and at the end of diffusion (equilibrium IFT) has been estimated. The experimental result (volume variations) has been used in the mathematical model to estimate the mass of CO₂ diffused into hydrocarbon from CW, and hydrocarbon density at different time intervals. The obtained densities of CW and hydrocarbon are then used to extract real-time IFT from the experimental data.

II. PROCEDURE

A. Materials

The *n*-decane was used as the hydrocarbon sample for the drop phase, manufactured by Merck KGaA (purity 99%). CO₂ with the purity of 99.9% (PRAXAIR) was used to prepare the CW. NIST Chemistry Web Book [6] is the source of density and viscosity measurements at various pressures and temperatures of *n*-decane and CO₂. The CO₂ solubility, and hence, the density of 100% saturated CW was calculated using model presented by [7].

B. Experimental Setup and Procedure

A high-pressure pendant drop apparatus (PD-E1700 LL-H) manufactured by EUROTECHNICA and KRUSS was utilised to carry out axisymmetric pendant drop experiments. Schematics of the experimental setup along with its essential components used in the present work is presented in Fig. 1. The important part of the setup is the corrosion resistant cylindrical high-pressure chamber (VC) (diameter of 18 mm), with a maximum working pressure and temperature of 68.9 MPa and 180°C respectively. A thermocouple (NiCr-Ni) controls the temperature inside the VC. A pump (maximum pressure of 32 MPa, GILSON) connected to CO₂ cylinder maintains the required pressure inside the VC. The VC has a see-through window and is placed between a high-resolution camera (CF03) and a light source. The camera acquires digital images of the PD (hydrocarbon pendant drop), as the CO₂ from the surrounding CW, diffuses into PD. A KRUSS DSA 100 software is used to analyse the captured images and compute the IFT at pre-set time intervals. The experimental method used in the present study to measure the IFT of the pendant drop is an automated process and devoid of any human interference.

At the beginning of the experiment, the high-pressure chamber (VC) is filled partially with deionised water (DIW) (20 ml of 25 ml capacity), this is followed by the injection of CO₂ at experimental pressure. The CO₂ is injected into DIW present in the VC by the pump until the water reaches the saturation level of CO₂ (at P and T) and can no more accept CO₂ into it. Further, experiments using flow meter confirmed that the between 95–98% saturation was obtained in the VC when compared with results from the model presented by

Duan and Sun [8], hence, it is safe to assume that water is saturated with CO₂ (saturated CW). When it is made certain that the water is saturated with CO₂, a hydrocarbon pendant drop (PD) is generated in the VC. The experimental setup was designed in a way that it replicates the physics occurring during the CWI (secondary). The detailed experimental procedure may be found in Bagalkot and Hamouda [9].

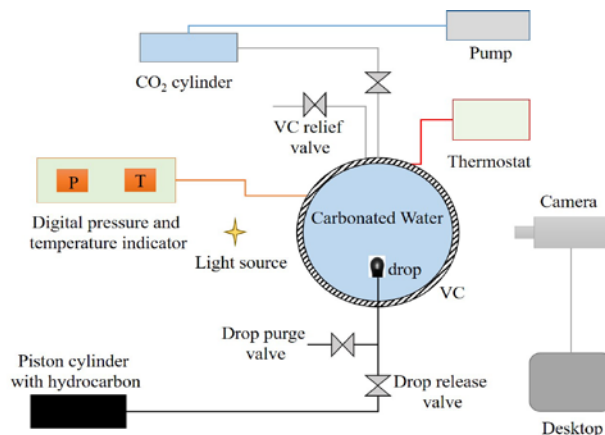


Fig. 1. Schematics of the experimental setup.

C. Mathematical Model

Alterations to the properties of hydrocarbon like density, viscosity, and volume are some of the critical phenomena occurring due the mass transfer of the CO₂ into the hydrocarbon from the displacing fluid (CW). Among these properties the density changes are closely related to the IFT. Studying the changes in the density of the hydrocarbon due to mass transfer of CO₂ would help us to understand the IFT better and gives us a depiction of the impact of the CW as a displacing fluid. To do obtain the density, the mass of CO₂ transferred into the hydrocarbon as a function of time and at equilibrium need to be calculated. A relatively simple mathematical method that utilizes the experimental PD volume as input has been developed to calculate the mass of CO₂ transferred into the PD (*n*-decane).

When the experiment is initiated (just before the diffusion of CO₂ into the PD, time $t=0$ s) the PD consists only of hydrocarbon (100% *n*-decane). As the diffusion of CO₂ starts ($t>0$), the mass transfer of CO₂ into the pendant drop transpires, causing an increase in the volume of the PD and the additional volume is due to the mass of CO₂ that has been diffused into the PD. Hence, the volume of the PD is the summation of the volume of *n*-decane and the increase in volume caused by the CO₂ in the PD.

$$V_{PD}(t) = V_{HC} + V_{CO_2}(t), \quad (1)$$

where V_{PD} (ml) is the volume of the pendant drop; V_{HC} is the volume of the *n*-decane or the initial pendant drop volume, and V_{CO_2} (ml) is the volume of the CO₂ in the pendant drop. The V_{HC} is fixed and is not a function of time. Hence, the volume of CO₂ (V_{CO_2}) is the volume of *n*-decane subtracted from that of total volume at a given instant.

$$V_{CO_2}(t) = V_{PD}(t) - V_{HC} \quad (2)$$

In the equation 2, t is the time. The computed volume of CO_2 (ml) is then multiplied by the CO_2 density (g/ml) at the experimental pressure and temperature to calculate the mass of CO_2 (equation 3).

$$M_{\text{CO}_2}(t) = (\rho_{\text{CO}_2} * V_{\text{CO}_2}(t))_{P,T} \quad (3)$$

In the equation 3 the ρ_{CO_2} is the density of CO_2 and is obtained from NIST web book [6] and M_{CO_2} is the mass of CO_2 in grams. From the computed CO_2 mass, the number of moles, mass fraction, and mole fraction of CO_2 and n-decane in the PD is calculated. Using, the determined mass and mole data of CO_2 and n-decane the density of pendant drop (PD) may be calculated from the equation 4.

$$\rho_{\text{PD}}(t) = (V_{\text{CO}_2}(t) \cdot \rho_{\text{CO}_2} + (V_{\text{HC}}(t) \cdot \rho_{\text{HC}}))_{P,T}, \quad (4)$$

where V_{CO_2} and V_{HC} are the volume fractions of CO_2 and n-decane in the drop respectively at required pressure and temperature, and ρ_{CO_2} and ρ_{HC} are the densities of CO_2 and hydrocarbon respectively at P and T.

D. IFT Measurement and Data Gathering Procedure

IFT measurements were carried out using the ADSA system. Diffusion of CO_2 into the PD, change the density of the PD, which is directly proportional to the mass of CO_2 transferred into the hydrocarbon (volume or mole fraction) (equation 3). So, for accurate dynamic measurement of the IFT, the density of the drop with CO_2 , is updated in the DSA 100 software to account for the density change. In the present study, the changes of the mole fraction of CO_2 in the drop (from the start to the equilibrium) were calculated (equation 2). The dynamic density of CO_2 (equation 4) was accordingly used to obtain the dynamic IFT of the CW-hydrocarbon system.

III. RESULTS AND DISCUSSION

The influence of pressure on dynamic IFT (IFT as a function of time or function of rate of CO_2 mass transfer), and equilibrium IFT (at the end of mass transfer/diffusion), are presented in Fig. 2. The Fig. 2 shows the variation of IFT with time (0–300 min) for CW-n-decane system at 25°C, 35°C, and 45°C for a pressure range of 10–60 bar. A unique observation of Fig. 2 is the behaviour of IFT with time at various pressures and temperatures. At 25°C for low pressures (10 and 20 bar), the IFT of the system decreases with time and reaches an equilibrium value lower than the initial value, which is similar to the behaviour of the CO_2 -hydrocarbon system. However, above 20 bar there is a transition of the profile of IFT vs time, the IFT increases with time and reaches an equilibrium value greater than the initial. Hence, there exists a “threshold pressure”, above which the IFT increases with time, and below which the IFT decreases. The same is true for other temperature (35°C, and 45°C), except that the transition occurs at different pressures, such that the threshold pressure increases as temperature increases, it is above 40 bar, and 50 bar for 35°C and 45°C, respectively. Hence, there are two phenomena to be addressed. First is the occurrence of the

threshold pressure, and the second is the increase of the threshold pressure with temperature. These phenomena are interrelated and may be explained based on the phase behaviour of the diffused fluid (CO_2) into PD (n-decane). The increment in the IFT with time at higher pressures (30–60 bar) at 25°C may be explained by the effect of CO_2 diffusion on the density of the hydrocarbon. The diffusion of gaseous CO_2 into the hydrocarbon reduces the density of the binary mixture (CO_2 +n-decane), whereas the dissolution of CO_2 into water increases the density of the mixture, this increases the density difference across the interface. Further, the magnitude of IFT is directly proportional to density difference [10], [11], hence, the greater the density difference, the larger will be the IFT. The reduction in the IFT with time at low pressures may be explained by the lack of CO_2 mass transfer into the hydrocarbon from the CW at low pressures, due to low CO_2 solubility in hydrocarbon and water at low pressures. Lower solubility of CO_2 limits the diffusion of CO_2 into the hydrocarbon and hence restricts the reduction in the density of the binary mixture. It may be observed from Table I that at 25°C and 10 bar there is approximately not more than 2.5% reduction in the density of n-decane. Therefore, at low pressure, there is a decrease in IFT. However, as pressure increases the CO_2 solubility increases, leading to enhanced mass transfer and higher reduction in the density (28.4% at 40 bar and 45.5% at 60 bar from Table I) of the n-decane and hence, the observed increase in IFT with time. So far, this may provide an explanation of the occurrence of the threshold pressure, but does not explain the upward shift of the threshold pressure with temperature (20 bar at 35°C to 50 bar at 45°C).

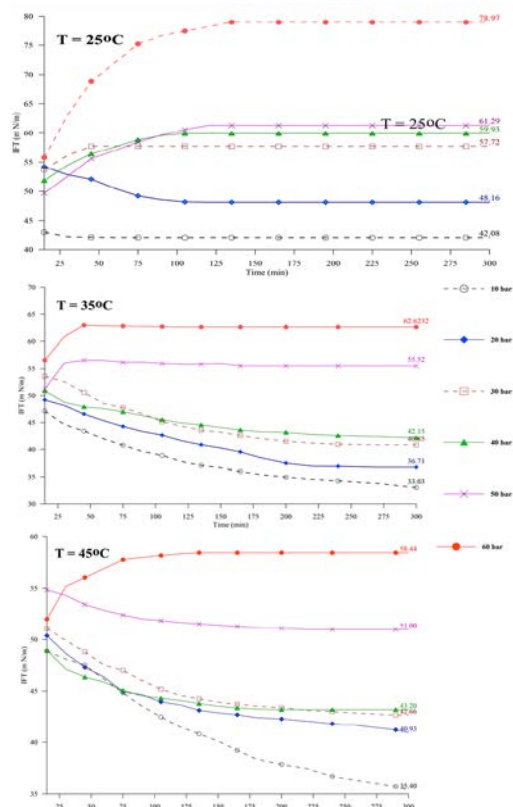


Fig. 2. Dynamic IFT between CW-n-decane system at 25°C, 35°C, and 45°C for a pressure range of 10–60 bar.

Apart from the influence of CO₂ solubility and density changes on IFT, increasing the temperature would cause additional physics that would affect the IFT. The kinetic energy and the entropy (mobility of the molecules) of the binary mixture (CO₂ + n-decane) are increased as the temperature is raised, this increases the total energy of the interface of the CW and binary mixture [10]. The increment in the total energy would reduce the free energy, which evidently results in the reduction of the IFT [10]. Hence, two phenomena having an opposing influence on IFT are present. First, IFT increment due to increment in density difference caused by an increase in CO₂ solubility. Second, is the decrease in IFT (with time) due to increase in kinetic energy and the entropy due to increment in temperature. The behaviour of the IFT with time depends on which of these phenomena is dominant. Increasing the temperature, reduces the solubility of CO₂ in n-decane and CW, however, the kinetic energy increases causing breakage of intermolecular bonds and reduces the free energy consequently reduces the IFT. This only explains the reduced IFT with temperature and not the presence of different threshold pressure at different temperatures. The threshold pressure (IFT trend transition), was demonstrated to increase with temperature. This may be explained by the need for high pressure to reduce the change in the entropy, hence increases the free energy consequently the IFT trend increases with pressure. Apart from an increase in kinetic energy and entropy of the molecules in the binary mixture, the CO₂ solubility in n-decane decreases. The decrease in CO₂ solubility leads to a reduction in the mass transfer of CO₂ into the binary mixture, eventually causing a lower reduction in density of the binary mixture, which may be observed from the data presented in Table I. Therefore, as the temperature is raised the influence of kinetic energy and entropy on IFT is dominant compared to the influence of CO₂ solubility. This explains the observed upward shift in the “threshold pressures” with the increase in temperature. However, at high pressures (50 and 60 bar at 35°C and 60 bar at 45°C), the CO₂ solubility dominates kinetics, causing an increase in IFT with time at these pressures.

TABLE I: THE PERCENTAGE CHANGE IN DENSITY OF THE N-DECANE AND CO₂ MOLE FRACTION IN N-DECANE AT 25°C, 35°C, AND 45°C FOR 10 BAR, 40 BAR, AND 60 BAR

Temperature	25°C	35°C	45°C
Pressure	10 bar		
% density change	-2.258	-1.52	-1.73
CO ₂ Mole fraction	0.0019	0.0012	0.0014
Pressure	40 bar		
% density change	-28.48	-16.02	-9.865
CO ₂ Mole fraction	0.167	0.079	0.044
Pressure	60 bar		
% density change	-45.43	-35.43	-18.60
CO ₂ Mole fraction	0.602	0.346	0.121

Fig. 3 shows the images of the PD (n-decane+CO₂) at the equilibrium condition at three pressure (10, 40, and 60 bar) and three temperatures (25°C, 35°C, and 45°C). The objective

of presenting these images is to validate through visual observations the relation between different physics presented in Fig. 2. In the pendant drop method, the interfacial tension is estimated from the shadow of the image of a pendant drop using drop shape analysis. Uniform density region would indicate negligible density difference (gradient) (like the surrounding CW), and such regions have a lighter shadow, while the regions with considerable density difference will have a darker shadow. The images in the first row (Fig. 3) represent the equilibrium n-decane drop at 10 bar and 25°C, 35°C, and 45°C. It may be observed that for all experimental temperatures, the PD has a thick dark region near the interface and then the darkness fades towards the center of the drop, indicating a non-uniform distribution of CO₂ mass within the drop. From the discussion in Fig. 2 this would lead to a reduction in IFT (due to the lower influence of CO₂ solubility), the same has been observed in Fig. 2. Further, the density distribution is similar to 10 bar at all three experimental temperatures, indicating that IFT variations would be similar, which validates with the observed reduction in IFT at 10 bar for all three temperatures (Fig. 2A, 2B, and 2C). From the second row (40 bar at 25°C, 35°C, and 45°C), there is a difference among temperature, n-decane drop at 25°C seems to be lighter and uniform distribution of CO₂ than 35°C and 45°C, a considerable non-uniformity in shadow (CO₂ distribution) at 35°C and 45°C. Going, by this, the IFT variations at 25°C should be different compared to 35°C and 45°C. In Fig. 2, it was observed that at 25°C for 40 bar the IFT increased with time up to equilibrium, whereas 35°C and 45°C showed a decrease in IFT. From the third row (40 bar at 25°C, 35°C, and 45°C), the shadows of the n-decane drop are similar at all the three temperature, indicating a similar variation of IFT with time. In Fig. 2, it was observed that for three temperatures at 60 bar the IFT increased with time up to equilibrium.

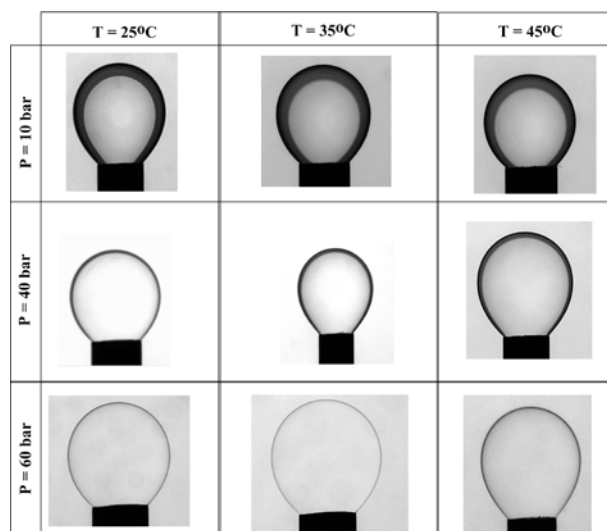


Fig. 3. Images of the n-decane pendant drop at 25°C, 35°C, and 45°C for 10 bar, 40 bar, and 60 bar.

Fig. 4 show the influence of temperature on the equilibrium IFT at three temperatures (25°C, 35°C, and 45°C) and various pressures (10-70 bar). This section discusses the variation of IFT with pressure at 25°C and 45°C. The variation of IFT with

pressure between 35°C and 45°C is complicated and will be dealt with in detail in the following paragraph and this paragraph the focus will be mainly on 25°C and 45°C. It may be observed from Fig. 4 that regardless of the experimental temperature, equilibrium IFT of CW-PD increases with the pressure, which is unlike the results from the literature for the CO₂-hydrocarbon system [9], [11]. Few important observations may be made in Fig. 4, first, the relation of interfacial tension with pressure for the present system is unlike CO₂-hydrocarbon system [9], where the IFT decrease with increase in pressure. Second, the relation of interfacial tension with pressure observed in Fig. 4 is similar to the water-*n*-decane systems. In water-*n*-decane systems, the IFT linearly increased with pressure. Wiegand and Franck [12] reported the IFT of the water-*n*-decane system to change between 50 to 55 m N/m for a pressure range 0 to 2500 bar at 25°C. However, in the present study the water is carbonated (CW) and the presence of CO₂ in the water has led to an increase in the IFT (25°C) when compared to previously reported IFT values for the water-*n*-decane system, although the trend of IFT with pressure is similar. Hence the carbonation of water increases the IFT of the water-hydrocarbon system. The changes in the density of water and hydrocarbon due to dissolution and diffusion of CO₂ respectively may be credited for the observed increment in equilibrium IFT with pressure. Third, for 25°C and 45°C, at isobaric conditions, the IFT decreases as temperature is increased. Raising the temperature reduces the CO₂ solubility in hydrocarbons and water, leading to an increase in IFT [5]. Further, raising the temperature increases the total entropy and reduces the Gibbs free energy, resulting in an observed reduction in IFT [10] as the temperature is raised from 25°C to 45°C. Additionally, Fig. 5 depicts the density difference between CW and PD at equilibrium (same conditions as that of equilibrium IFT) at 25°C, 35°C, and 45°C and 10-70 bar. It may be observed from Fig. 5 that the density difference increases with increase in pressure at all temperatures. The IFT is directly proportional to the density difference between the two immiscible fluids [10], [11], hence, the greater the density difference, the larger the IFT.

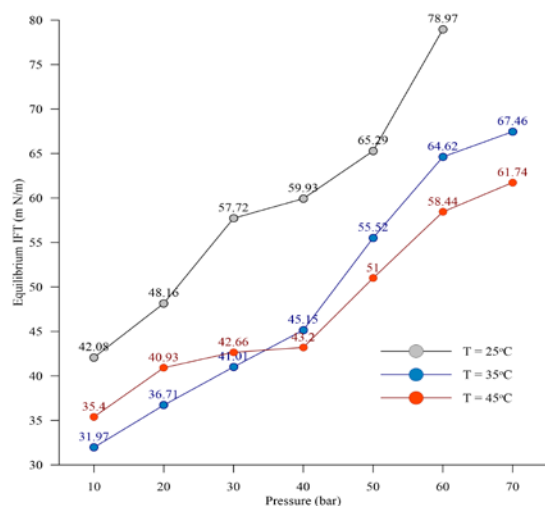


Fig. 4. Equilibrium IFT at 25°C, 35°C, and 45°C for n-decane hydrocarbon drop and at a pressure range of 10–70 bar.

In addition to 25°C and 45°C Fig. 4 shows the equilibrium IFT (m N/m) for CW-*n*-decane system at 35°C and at a pressure range of 10–70 bar. The variation of equilibrium IFT between 35°C and 45°C is not as straightforward as between 25°C and 45°C. It may be observed from Fig. 4 that up to 30 bar the IFT at 35°C is lower than that of 45°C, this is contradicting the theory presented in the above discussion, although IFT at 25°C is the maximum. Above 30 bar (40–70 bar) the IFT vs temperature returns to normal trend (IFT inversely proportional to temperature), with IFT being maximum for 25°C, minimum for 45°C and intermediate for 35°C at isobaric conditions. The proximity of 35°C to the critical temperature of CO₂ (31.1°C), may be credited for the observed behaviour of IFT with pressure. A similar change in slope of equilibrium IFT with temperature has been observed by Zolghadr, et al. [13] (CO₂-hydrocarbon system), Bagalkot and Hamouda [9] (CO₂-hydrocarbon system), and Karnanda, et al. [14] (surfactant flooding). In literature, the increase in IFT with increment in temperature has been credited to decrease in the solubility [5], while the decrease in IFT with the increment in temperature has been credited to increase in the total entropy and hence, reduction in Gibbs free energy [10]. Both of them may be true of the CW-hydrocarbon system, at low pressures (below 40 bar) the decrease in CO₂ solubility plays a major role, thus increasing the IFT. However, at medium and high pressures (above 40 bar) it seems that the reduction in Gibbs free energy due to increase in temperature dominates the effect of reduction in CO₂ solubility, giving rise to a decrease in IFT with an increase in temperature (35°C to 45°C). Apart from these two factors, the density difference between the CW and the hydrocarbon drop plays an important part. It may be observed from Fig. 5 the variation of density difference with temperature follows a similar trend to equilibrium IFT (Fig. 4) between 35°C and 45°C. Thus, confirming the density difference along with the CO₂ solubility and entropy change (reduction in Gibbs free energy) influence the IFT of CW-hydrocarbon system, especially above the critical temperature.

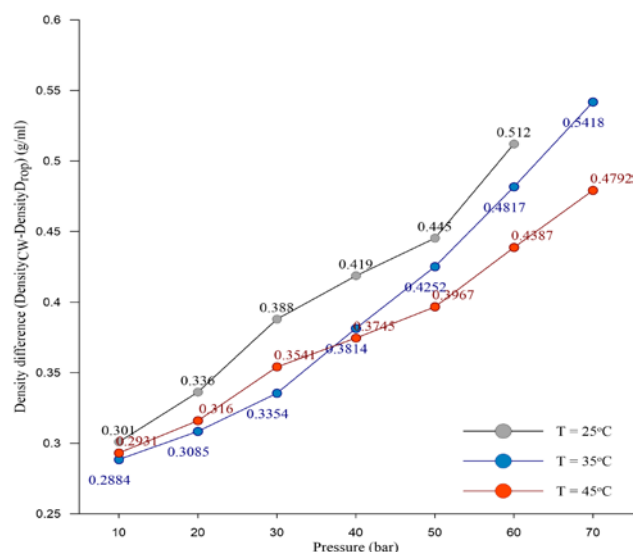


Fig. 5. Density difference at equilibrium at 25°C, 35°C, and 45°C for n-decane hydrocarbon drop and at a pressure range of 10–70 bar.

IV. CONCLUSIONS

The present study through experiments and mathematical model study the influence of temperature on the interfacial tension (IFT) for a system consisting of carbonated water (CW) and hydrocarbon at non-isothermal and non-isobaric conditions. Hence, address and understand the inconsistencies in results reported in the literature between temperature and IFT.

The time-dependent IFT analysis showed the presence of a *threshold pressure* for a given temperature above which the IFT increases with time and below which the IFT decreases. The increase or decrease of IFT with time for different pressure was credited to a complex relationship between the density alterations due to increased CO₂ mass transfer, CO₂ solubility, and kinetic energy and entropy changes controlled by temperature. Depending which of these physics is dominating the IFT increases or decreases with time during the diffusion of CO₂.

For the CW-n-decane system, the equilibrium IFT increases as pressure increases (10–70 bar). The equilibrium value of the IFT depend on density difference influenced by CO₂ mass transfer, and CO₂ solubility at a given temperature. Further, it was observed that the equilibrium IFT at 25°C was higher than at 35°C and 45°C. Increase in kinetic energy and entropy due to increment in temperature was credited to this.

The variation of equilibrium IFT between 35°C and 45°C is not as straightforward as between 25°C and 45°C or 25°C and 35°C. Up to a certain pressure the equilibrium IFT at 35°C is lower than at 45°C, and beyond this pressure, the equilibrium IFT at 35°C is greater than at 45°C. The reduction in Gibbs free energy which reduces the IFT and increases in IFT due to a decrease in solubility as temperature increases, the balance between these physics determine the observed behaviour. Further, it was found that the density difference across the interface showed a similar variation with pressure as equilibrium IFT at 35°C and 45°C, indicating the dependency of the IFT on the density difference.

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