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STUDY OF PARAMETERS AFFECTING THE COALESCENCE OF DIMETHYL DISULFIDE DROPS IN A MEROX UNIT

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Abstract - This study focuses on the coalescence of dimethyl disulfide drops with the mother phase at a flat aqueous-organic interface between dimethyl disulfide and different sodium hydroxide solutions. Drop coalescence is an important part of the Merox process for regenerating the solvent. A digital high-frame rate camera was used for recording drops coalescence and duration time. Drops of dimethyl disulfide were directed in different sodium hydroxide solutions as the continuous phase. Applying the experimental design method, the influences of independent variables of drop size and physical properties on coalescence time were investigated. Computational fluid dynamics (CFD) was employed to simulate the drops released from a nozzle, moving toward the interface, and the CFD results were validated by experimental data. The maximum deviation between the predicted and experimental coalescence times was 18.7%. It was found that, among the physical properties, interfacial tension plays the most important role on the coalescence time. Based on the results, a correlation for coalescence time was proposed.

Keywords: Merox process; Coalescence time; Liquid/liquid interface; Volume of fluid method; Interfacial tension.

INTRODUCTION

Coalescence of drops with their mother phase has been studied widely over the past decades (Kavehpour, 2015). Focus on gravity separators with the aim of separation of two immiscible liquid phases and the study of the coalescence time of drops with the mother phase are of high importance, particularly in the oil industry (Paul, Atiemo-Obeng et al., 2004). One special relevant matter is the ease of disulfide separation in Merox units (Liu, Xia et al., 2005). The major portion of the coalescence process takes place at the interface and most of the experimental research in this area has been conducted for a single drop coalescing at the planar interface of phases. Therefore, the effect of drop diameter and physical properties on drop-interface coalescence have to be taken into account.

The drop-interface coalescence process has been the subject of several works and considerable theoretical and experimental studies have been performed in this regard. The early works corresponds to Charles and Mason (1960), who investigated the coalescence time and reported that, in an oil/water system, stability increases with increasing drop size and with decreasing temperature. Davis et al. (1971) investigated the symbolic design of the stages of coalescence and their corresponding times. They found that the coalescence takes place in four consecutive stages: collision of liquid bodies, drainage of the narrow film trapped between them, rupturing of the film and eventual coalescence. Mohamed-Kassim and Longmire (2004) focused on drop coalescence and the rupturing stage by using PIV (partial imaging velocimetry) and also investigated the effect of viscosity at similar densities for low Reynolds number on drop coalescence. Chen and Mandre (2006)

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examined the effect of fluid properties, drop size, viscosity and gravity on the partial coalescence process prior to full coalescence. They found that partial coalescence occurs for an intermediate range of drop sizes. In another investigation, Aryafar and Kavehpour (2006) studied a wide range of fluids and presented the parameters affecting full coalescence and partial coalescence and also examined the behavior of fluid drops as they coalesce across planar interfaces. Blanchette and Bigioni (2009) combined high speed video imaging with numerical simulations to find conditions of partial coalescence. Bozzano and Dente (2011, 2013) proposed a model for the mechanism of coalescence of single drops at the planar interface and they investigated the physical properties that influence the coalescence time. In another attempt, Bart and Singh (2015) studied drops coalescence at the interface of two immiscible liquids using numerical simulation. Based on the proposed model, they examined the effect of physical properties and drop size.

Extensive experimental research has also been performed on modeling the drop coalescence with a mother phase at planar surfaces. In this regard, CFD (computational fluid dynamic) simulation based on volume of fluid (VOF) for the drops coalesced at the interface was introduced by Bonhomme et al. (2012). They used different densities, viscosities and drop sizes over a wide range in the simulation. Recently, Behjati et al. (2017) modeled the immiscible liquid-liquid systems by the Smoothed Particle Hydrodynamics method. The validity of the model was demonstrated by applying it to a range of model problems. In the same year, Politova et al. (2017) studied the effects of drop size and viscosity of the oil phase on the stability of water drops moving towards a planar oil-water interface. They found small drops coalescence to occur before formation of a planar film at the interface. More recently, Wang et al. (2018) studied the dynamic behavior of drop coalescence at different temperatures.

Despite a rich history of drop coalescence and interface study, there is a lack of consistent investigation on the effect of physical properties on coalescence. Use of digital imaging technology has created a turning point for investigating the unknown aspects of this phenomenon (Kavehpour, 2015). Based on this fact, an attempt was made here to study the effect of parameters influencing the coalescence time using a digital high-frame rate camera for recording drop coalescence steps as well as the coalescence time. Also, in this work CFD simulation was performed in order to investigate the coalescence process from theoretical aspects. The proposed model has been evaluated by comparing with experimental results. Finally based on experimental results, a correlation has been proposed for the coalescence time in terms of influencing parameters.

EXPERIMENTAL SET UP

Experiments were conducted in a simple glass cylindrical column with an inner diameter of 8 cm and a height of 40 cm (Fig. 1a and 1b). The dispersed phase was light organic phase and the setup was designed in the manner that drops were moving upward to the interface. For this purpose, a dispenser device injected dispersed phase by using a syringe connected to a needle. Three different needles for producing different drop sizes were used: Gauge 15 (nominal OD of 1.89 mm), Gauge 18 (nominal OD of 1.27 mm) Gauge 22 (nominal OD of 0.718 mm). The distance between the nozzle tip and the interface (release height) was adjusted at three different levels of 0.5, 2 and 3.5 cm from interface.

A high-frame speed camera (fps1000, Kickstarter, England) was used to capture the images of the coalescence. The frame rate was between 500 to 1000 frames per second at a resolution of 1920×1080 pixels. The test cell was illuminated by a background light (Yemao zoom LED Flashlight) to improve representation. The dimension of the column was far greater than that of the drop, which ensured that the effect of the walls on the dynamics of droplets was minimal.

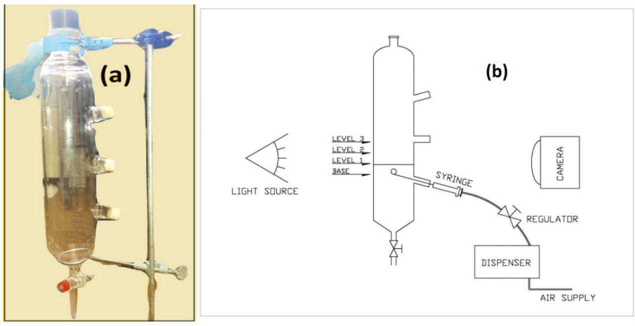

Figure 1. The coalescence cell (a) and the schematic of the device for obtaining coalescence images (b).

CHEMICALS AND OPERATING CONDITIONS

The chemicals used for this study were those involved in a Merox unit and were all Merck products, including sodium hydroxide flakes, dimethyl disulfide and deionized water for making solutions. All chemicals had a purity of more than 99.9%. In this process, three sodium hydroxide solutions of 10, 12 and 14% were used, which are close to those in a real Merox process (Abdolkarimi and Ganji, 2014).

Table 1 lists the measured physical properties of the liquid phases used. Density measurements were done via a DA-100M density-meter (METLER TOLEDO, Germany), which operates based on a controlled electromagnetic pulse. Viscosities were determined by using a viscometer (Schot and CT, Germany) with

Table 1. Physical properties of all the chemical systems used.

Solution/Material	Density (g/L)			Viscosity (mPa.s)			Interfacial tension (mN/m)		
	20 °C	40 °C	60 °C	20 °C	40 °C	60 °C	20 °C	40 °C	60 °C
10% sodium hydroxide solution	1.109	1.099	1.089	1.88	1.25	0.82	8.771	9.243	9.578
12% sodium hydroxide solution	1.131	1.121	1.111	2.06	1.32	0. 86	9.192	9.670	10.123
14% sodium hydroxide solution	1.153	1.143	1.132	2.46	1.41	0. 91	9.671	10.144	10.712
Dimethyl disulfide	1.062	1.041	1.018	0.62	0. 49	0. 37	-----	------	------

a suitable capillary. The interfacial tension between sodium hydroxide solutions and dimethyl disulfide was measured using a tensiometer (IFT-700, France) based on the pendant drop method.

PROCEDURE AND DESIGN OF EXPERIMENTS

In performing the experiments, at first, a sodium hydroxide solution with appropriate concentration was prepared and transferred to the glass cylinder and then dimethyl disulfide as the mother phase was added above the aqueous phase (see Fig. 1). Drops were moved upward from the nozzle tip through the aqueous phase toward the interface of the phases under buoyancy force. The time interval to reach the interface for this phenomenon was considered to be the ascension time. Also, the coalescence time was measured from the moment of drop release from the nozzle tip up to the moment of perfect coalescence with the mother phase and subtracting the ascension times (Bozzano, 2013). The final drainage continues until the interfacial film breaks up, allowing the drop and the mother phase to completely join together. These times were determined by following the frames of the high-speed camera. Fig. 2 shows different frames obtained during the coalescence process for a typical experimental run. When a drop gets very close to the interface, the viscous resistance, created by the displacement of the interposed fluid, quickly dissipates the kinetic energy of the drop and also the velocity quite immediately reduces from several centimeters per second to very low values (Bozzano, 2013).

Response surface methodology (RSM) was used as a statistical method through which it is possible to consider simultaneously the impact of several factors and obtaining optimal conditions by running a limited number of tests (Myers, Montgomery et al. 1995). In this study by applying RSM method, the required tests were reduced. The main factors considered to have influence in the field were:

- drop diameter
- concentration of sodium hydroxide
- temperature
- distance between the release point and interface

Surely, continuous and dispersed phase viscosities and densities will be changed by the change in temperature and these were taken into account in the calculations. Each of these factors were considered at

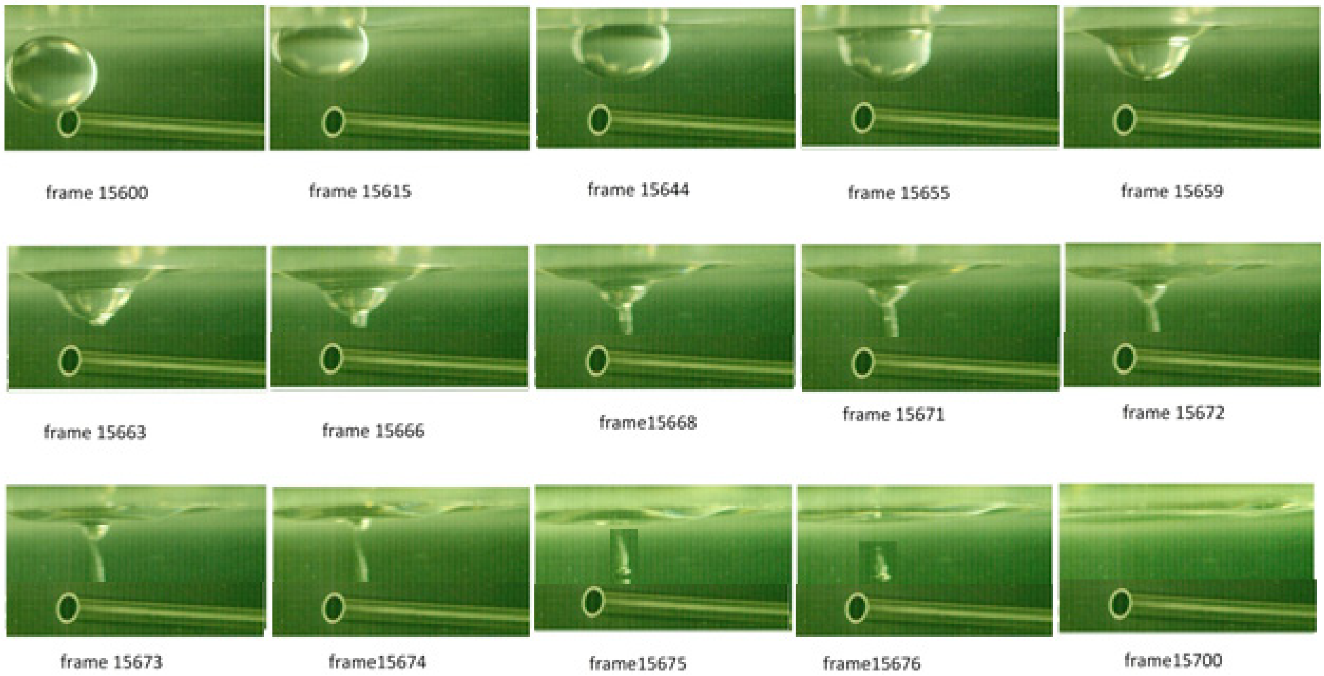

Figure 2. Different frames obtained during the coalescence time by the high-speed camera; sodium hydroxide concentration of 12% and $T = 40$ °C.

Table 2. Factors and levels in the test plan.

Level	Specified factors			
	Nozzle size (mm)	Temperature (°C)	Caustic concentration (wt%)	Distance (mm)
1	1.89	20	10	5
2	1.27	40	12	20
3	0.72	60	14	35

three levels. Table 2 shows the effective variables and their levels, used in the RSM.

One important parameter is drop size, which was accurately determined from the given images. As was mentioned, the external diameter of the nozzles was known and, based on the images, it was possible to obtain the real drop size in comparison with the exact nozzle diameter. In the set of these experiments, an influence due to the effect of glass and liquid diffraction of the light was not necessary to consider due to excellent contrast. A focused part of the images captured by the camera is presented in Fig. 3.

Based on the considered factors, thirty tests were required to obtain data and find the effect of independent variables on the target parameter of coalescence time. According to the results obtained by some researchers such as Jeffreys and Hawksley (1965), Khadiv-Parsi (2004) and Kamp et al. (2014), major parameters influencing coalescence time were the density and viscosity of the two phases, drop size and the interfacial tension. Changes in viscosity and density occur due to temperature variation in operating conditions or changes in the concentration of the continuous phase.

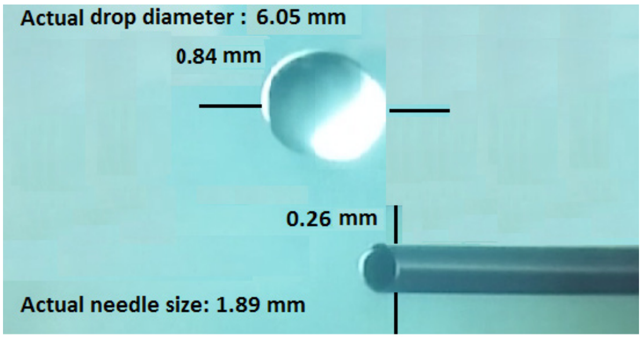

Figure 3. Drop diameter measuring based on nozzle diameter in the provided images.

MODELING AND THEORETICAL ASPECTS

Drop coalescence with the mother phase at the interface depends on different forces acting on it. The forces that improve the coalescence at the interface are often referred to as the driving forces (Singh, 2015); however, the forces making an attempt to restrict this phenomenon are often referred to as restraining forces. The main restraining force is the interfacial tension force, which tries to resist against coalescence (Singh, 2015). During drop coalescence at the interface, the kinetic energy of drops dissipates to the required joining energy. Therefore, interfacial tension between two immiscible phases has an important role in the drop coalescence process.

The free moving velocity of drops also depends on the density and viscosity of phases. For a drop rising in a viscous liquid, the velocity is proportional to the viscosity of the continuous fluid and is also inversely proportional to the density difference. These physical properties also have an influence on the drop coalescence time (Kemiha, 2007).

Based on this feature of contact, a numerical modeling was performed by using the commercial software ANSYS Fluent (Version 16) in which the VOF method is employed, in which a single momentum equation with the contribution of both phases, physical properties is defined. In this software, the mass and momentum conservation equations are based on the following equations (Singh, 2015):

$$\nabla.\vec{v} = 0 \tag{1}$$

$$\frac{\partial(\rho\vec{v})}{\partial t} + \vec{\nabla}(\rho\vec{v}\vec{v}) = -\vec{\nabla}_p + \vec{\nabla}\vec{\tau} + \rho\vec{g} + \vec{F}_p \tag{2}$$

where v is the fluid velocity vector, ρ is the effective fluid density, *p* is the static pressure, τ is the viscous stress tensor, g is the acceleration due to gravity and F is the volumetric body force due to surface or interfacial tension. The viscous stress tensor is defined as:

$$\vec{\tau} = \mu\left\{(\vec{\nabla}\vec{v}) + (\vec{\nabla}\vec{v})^T\right\} \tag{3}$$

In this equation, μ is the dynamic fluid viscosity. The density and viscosity are the weighted average of densities and viscosities of the individual phases. The weighted average was obtained from VOF as (Brackbill, 1992):

$$\rho = \sum_{q=0}^{1} \rho_q \alpha_q \tag{4}$$

$$\mu = \sum_{q=0}^{1} \mu_q \alpha_q \tag{5}$$

where q and α denote the phase index and the volume fraction, respectively. The heavier and lighter liquid phases were considered as the primary (q = 0) and the secondary (q = 1) phases, respectively.

The VOF method is used for tracking the interface between dimethyl disulfide and sodium hydroxide solutions. For this purpose, the continuity equation in volume fraction, given by the following equation is solved for the dispersed phase (Singh, 2015):

$$\frac{\partial \alpha_q}{\partial t} + \vec{\nabla}.(\vec{v}\alpha_q) = 0 \tag{6}$$

The volumetric body force, F, related to surface or interfacial tension, is estimated using the gradient of the volume fraction following the continuum surface force (CSF) model proposed by Brackbill et al. (1992). The expression of F, generalized for a binary system, is given as:

$$\vec{F} = \sum_{p<q} \sigma_{pq} \frac{\alpha_p \rho_p \kappa_q \nabla \alpha_q + \alpha_q \rho_q \kappa_p \nabla \alpha_p}{(\rho_p + \rho_q)} \quad (p > q) \tag{7}$$

where σ is the surface/interfacial tension. The condition p > q in eq (7) ensures that a pair of two specific fluids is accounted for only once. Also, κ in the above equation is the interface curvature, evaluated using the following equation:

$$\kappa_q = -\vec{\nabla}.\hat{n}_q \tag{8}$$

where n_q is the unit normal to the interface and is evaluated from the information on the considered cell volume fraction using the following equation:

$$\hat{n}_q = \frac{\vec{\nabla}\alpha_q}{\left|\vec{\nabla}\alpha_q\right|} \tag{9}$$

Eqs. (1-9) represent the complete set of equations that have been used in the VOF numerical modeling.

Using the mentioned software, the volume fraction equation (eq 6) was solved by using an explicit scheme. Semi-implicit method for pressure linked equations (SIMPLE) was accomplished for pressure-velocity coupling. Pressure staggering option (PRESTO) scheme was employed for the pressure interpolation. Geo-Reconstruct option was considered on the volume fraction. The discretization used in the momentum equation was the first order upwind differencing scheme.

Simplicity of the computations allows the use of structured distance grids. Selection of mesh size was done carefully and to find the reasonable mesh size, three mesh sizes of *d*/25, *d*/50 and *d*/100 were examined. Here *d* is the average drop size, i.e., the mean drop diameters in thirty experiments. The obtained results for mesh sizes of *d*/50 and *d*/100 were similar (maximum deviation was less than 2%), but different for *d*/25. Therefore, the corresponding mesh size of *d*/50 was selected in simulations. Further, a 2D computational domain, suitable with respect to the drop size, with dimensions of 10×10 cm was selected. In the work reported by Yuan et al. (2018), the domain size was selected as 10*d*. Thus, the domain used here (10 cm) seems adequate even for the largest examined drop of 6.05 mm. This consideration prevents wall effects on drop coalescence. The Piecewise-linear method (PLIC), which was introduced by Youngs (1998), was used to represent a well-defined interface within the VOF method. In this method, the interface between two immiscible liquids is considered initially as a straight line, and since the volume fraction in each cell can be from 0 to 1 and the size of each drop is much smaller than the total domain, the volume fraction distribution is smooth. Fig. 4 shows a typical computational domain used in the simulations.

Due to the limited production rate of dimethyl disulfide in Merox process, the dispersed phase layer was considered to be a thick layer (5 mm). The middle of the computational domain is introduced as the initial position of the liquid-liquid interface. The solution was saved after every 100 time steps. One advantages of the CFD simulation is that, by changing one parameter and keeping others constant, the effect of that parameter on coalescence time will be revealed. Validation was carried out based on comparison with experimental results.

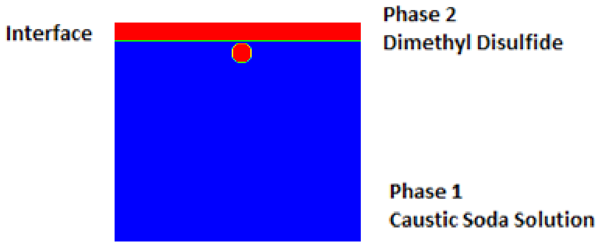

Figure 4. Computational domain and boundary conditions introduced in the simulations.

RESULTS AND DISCUSSION

A typical simulation run is shown in Fig. 5. The model was validated by comparing the simulation with experimental results. Required experimental data for the simulations were those given in Table 2. The same conditions of the experiments were introduced in the simulations.

The coalescence termination was considered when a rather smooth interface was achieved. Comparison between Figs 2 and 5, reveals that there is a nice compatibility between the results of CFD prediction

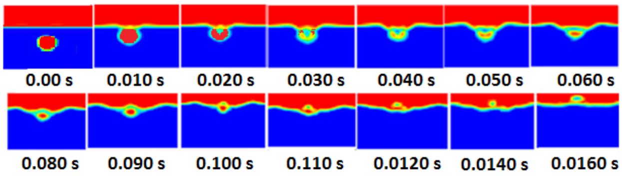

Figure 5. Outputs of drop coalescence simulation at different time intervals. $T = 40$ ˚C, sodium hydroxide concentration: 12 wt%.

and experimental data. The extent of influential independent variables of drop size and physical properties on the simulation and experimental results was investigated as follows:

Effect of interfacial tension

Since the purpose of this study was to find the role of parameters on coalescence time, the effect of interfacial tension on coalescence time was first investigated. The higher the interfacial tension, the more the resistance to coalescence. Simulations were carried out for different interfacial tension values between the liquids while keeping the other physical properties and drop size constant. Fig. 6 (a) shows that, by increasing interfacial tension, the coalescence time is prolonged. The experimental results and simulation outputs are compared in this figure. The average deviation between them was 8.5% and the maximum deviation was 14.7%.

Effect of drop size

Drop size is another significant parameter affecting the coalescence time. In the experiments, a spherical shape of dispersed drops in the continuous phase was observed. Fig. 6(b) shows that, upon reduction of the drop diameter, the coalescence time will decrease. Simulations for different drop sizes from 0.1 to 1 mm were carried out. The comparison between experimental results and simulation outputs shows that the maximum deviation between experimental results and outputs of the simulation was about 15.8% and average deviation was 10.3%.

Effect of continuous phase density

Density difference between two liquids may also affect the drop coalescence time. By keeping the density of the dispersed phase liquid constant, the effect of continuous phase density on drop coalescence was investigated. As the continuous phase density increases the density difference increases. Thus, the ascending velocity of the dispersed phase in the continuous phase will increase. Fig. 6c shows that an increase in the density of the continuous phase, which is a consequence of sodium hydroxide addition, will provide a significant decrease in the coalescence time. In this investigation, the maximum deviation between simulation and experimental coalescence time is 12.7% and the average deviation is 9.6%.

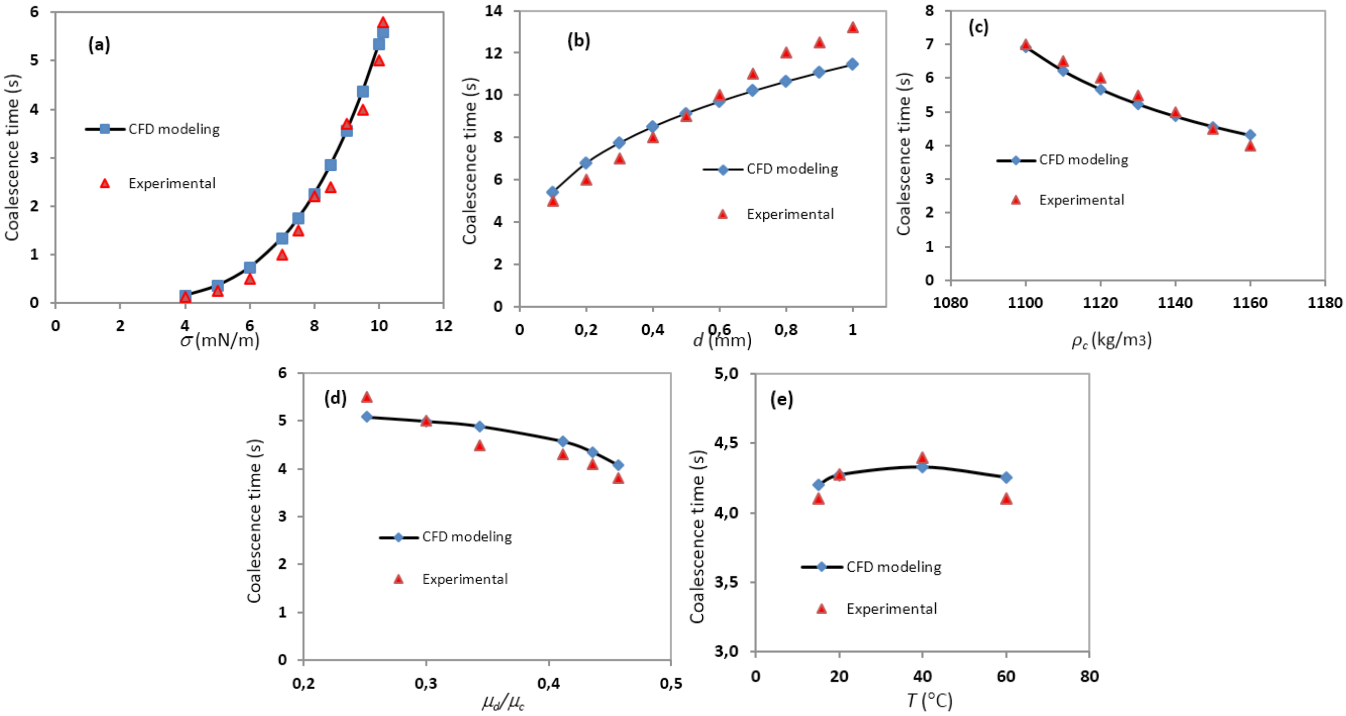

Figure 6. The effect of interfacial tension on coalescence time; $T = 40°$ C, sodium hydroxide concentration: 12% (a). The effect of the drop diameter on the coalescence time; $T = 40$ ˚C, sodium hydroxide concentration: 12% (b). The effect of continuous phase density on the coalescence time; $T = 40$ ˚C (c). Viscosities ratio effect on coalescence time; sodium hydroxide concentration: 12% (d). The effect of temperature on coalescence time; sodium hydroxide concentration: 12% (e). For all cases the distance from the interface was 20 mm.

Effect of the viscosity ratio

Based on the outlined operating conditions of the immiscible phases, the effect of viscosity ratio on coalescence time was investigated. Here the viscosity ratio was the ratio of dispersed phase to continuous phase. Fig. 6d summarizes the results obtained from simulation and experiment. By increasing the viscosity ratio, the movement of drops becomes faster. As a consequence, it can be seen from Fig. 6d that the coalescence time will decrease. The average and maximum deviation between results are about 14.3% and 18.7%, respectively.

Effect of temperature

Fig. 6e depicts the effect of temperature. Temperature affects all physical properties that are involved in the coalescence time. Temperature had a gentle impact on the coalescence time, since density and viscosity of the continuous and dispersed phases were functions of temperature; the continuous phase density increased from 1089.1 to 1153.1 kg/m^3 and the density of the dispersed phase varied from the 1018.2 to 1062.3 kg/m^3. Also the ranges of viscosities of continuous and dispersed phases were within 0.82-2.46 and 0.37-0.62 mPa.s, respectively. In this investigation, the maximum deviation between simulation and experimental results was 14.9% and the average deviation was 10.5%.

RSM predictions

Response surface plots given as coalescence time versus the variables of interfacial tension, continuous phase density and viscosities ratio are shown in Fig. 7. These plots help visualize the response surface shape as a function of two quantitative variables simultaneously. It can be observed that interfacial tension has the strongest effect on coalescence time and minimum coalescence time could be relevant to the lowest interfacial tension. It is also observed that coalescence time decreases with decreasing drop diameter. Further, with increasing continuous phase density and decreasing viscosity ratio, the drop coalescence time is decreased. The optimum operating conditions for drop coalescence time reduction in this process were 10% sodium caustic solution

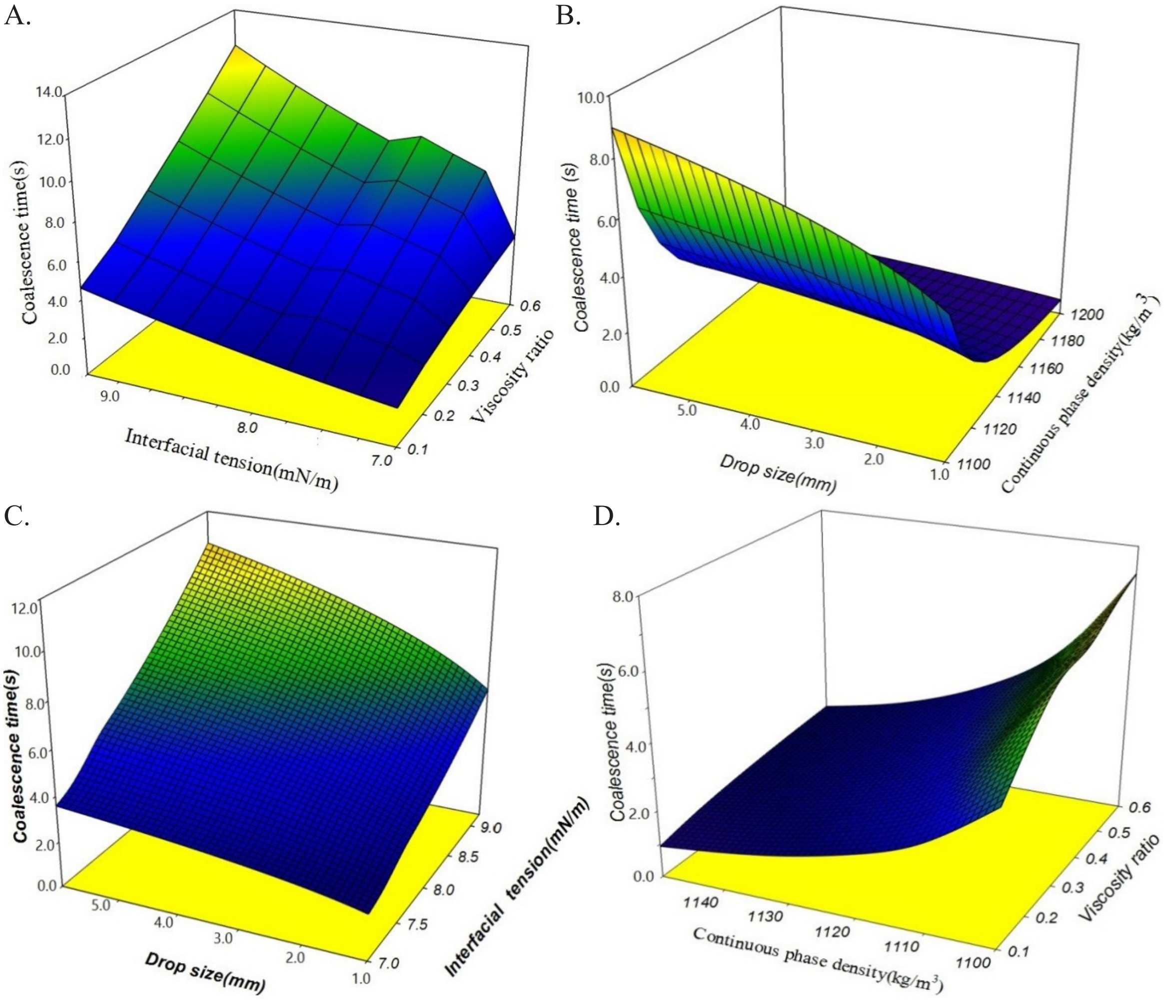

Figure 7. Response surface plots for the coalescence time as a function of drop size, interfacial tension, viscosity ratio and continuous phase density.

concentration and reaction temperature of 45°C at the lowest drop size.

Based on the data, a correlation was proposed in dimensionless parameters which can be used to estimate the coalescence time as:

$$\frac{\sigma t}{\mu_c d} = 2.953\times10^4\left(\frac{\Delta\rho g d^2}{\sigma}\right)^{-0.365}\left(\frac{T}{298.15}\right)^{-0.7}\left(\frac{\mu_c}{\mu_d}\right)^{0.714}\left(\frac{\Delta\rho}{\rho_c}\right)^{1.757}\left(\frac{d}{l}\right)^{1.305} \qquad (10)$$

where t is the coalescence time and T is the temperature in Kelvin and *l* is the distance from the nozzle tip to the interface. The correlation coefficient (R^2) value of 0.9420 for this equation indicates that the model can explain 94.2% of the variability of the response variable, so that it can provide an acceptable prediction.

Two valid similar correlations have already been proposed at T = 25 °C. One is the Smith correlation for coalescence (Davies and Jeffreys 1971, Khadiv-Parsi 2001) as:

$$\frac{\sigma t}{\mu_c d} = 31.0\times10^3\left(\frac{\Delta\rho g d^2}{\sigma}\right)^{-1.24}\left(\frac{\mu_d}{\mu_c}\right)^{1.03} \qquad (11)$$

and another proposed by Khadiv-Parsi (2001) where the simultaneous dependency of the coalescence time with drop size and falling distance was investigated and the correlation time given as

$$\frac{\sigma t}{\mu_c d} = 4.846\times10^3\left(\frac{\Delta\rho g d^2}{\sigma}\right)^{0.512}\left(\frac{L}{d}\right)^{0.651}\left(\frac{\mu_d}{\mu_c}\right)^{0.203}\left(\frac{\Delta\rho}{\rho_c}\right)^{0.406} \qquad (12)$$

Comparison between the model proposed in this work with other models is shown in Fig. 8. A close agreement between models is observed.

CONCLUSIONS

In a series of experiments the coalescence time for dimethyl disulfide drops was measured. In the experiments, the position of drops and coalescence time was recorded by using a high speed camera. Drop size effects and important physical properties such as density, viscosity and interfacial tension were considered. In order to identify the most suitable conditions for the separation of dimethyl disulfide from sodium hydroxide solutions, a CFD simulation was performed to estimate the drop coalescence time. For tracking the interface between two liquids, the VOF method was employed and validation was carried out based on experimental data. The most important achieved results were:

The interfacial tension between two immiscible liquids was the main variable reducing the coalescence time of the dispersed phase with the mother phase. The results revealed that the coalescence time decreased with a decrease of interfacial tension.

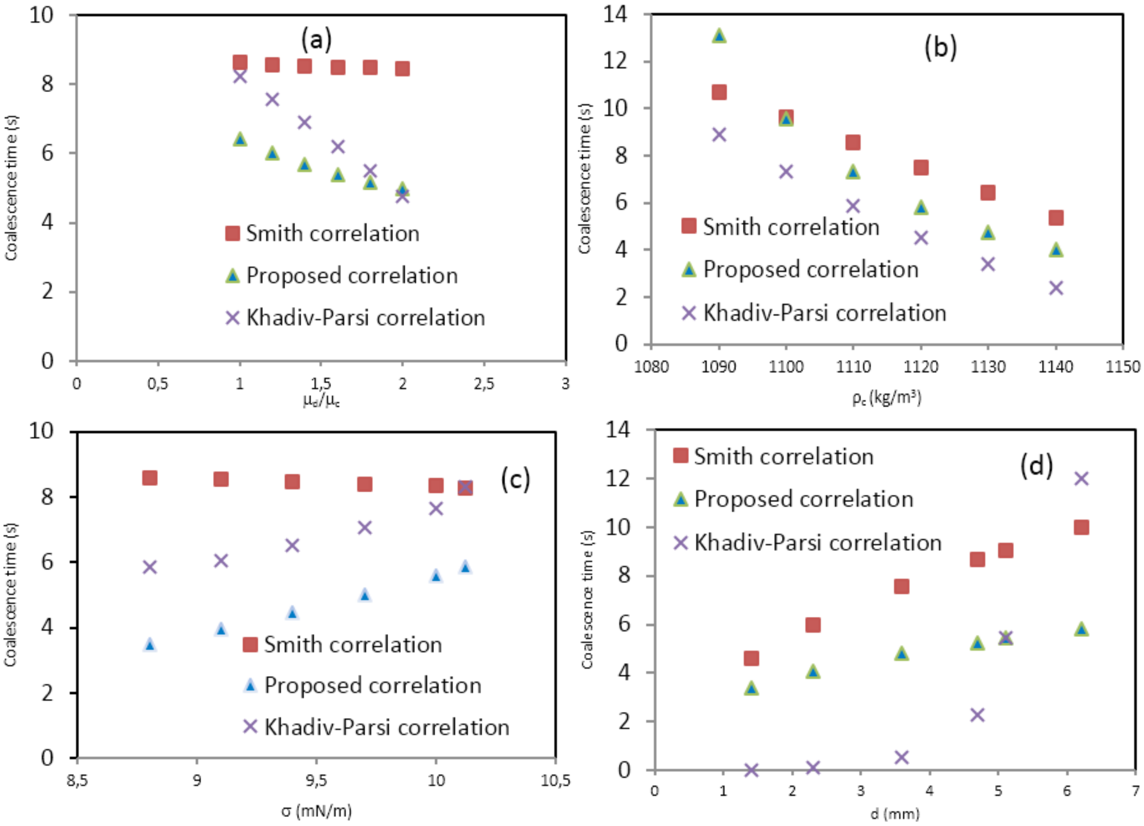

Figure 8. Comparison between predicted values of drop coalescence times by the proposed correlation and the other models; *T*=25 °C, sodium hydroxide concentration: 12% and distance from interface of 20 mm.

With increasing ratio of the dispersed phase to continuous phase viscosities, the drop coalescence becomes faster.

The decrease in drop size leads to a faster coalescence process at the interface.

With increasing continuous phase density, it was found that drop coalescence became faster.

Based on the experimental results, an emperical nonlinear correlation between the coalescence time and the variables involved was obtained. The results indicated that the proposed correlation had an acceptable agreement with experimental results and CFD modeling.

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NOMENCLATURE

d	Average Droplet diameter (mm)
l	Ascent distance
T	Temperature (°C)
p	Static pressure(Bar)
g	Acceleration due to gravity(m/s^2)

Greek Symbols

σ	Interfacial tension (mN/m)
μ_c	Continuous phase viscosity (mPa. s)
μ_d	Dispersed phase viscosity (mPa. s)
ρ_c	Continuous phase density (kg/m^3)
ρ_d	Dispersed phase density (kg/m^3)
Δ	Density differences (kg/m^3)
v	Fluid velocity vector
τ	Viscous stress tensor
F	Volumetric body force due to surface or interfacial tension(N)
α_0	Continuous volumetric fraction
α_1	Dispersed volumetric fraction
κ	Interface curvature
n_q	Unit normal to the interface

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