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Research Article

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Drop Size Distribution in a Standard Twin-Impeller Batch Mixer at High Dispersed-Phase Volume Fraction

The preparation of concentrated aqueous silicone oil emulsions has been investigated with particular attention to the effect of the dispersed-phase volume fraction ϕ from 0.01 to 0.5 for a wide range of oil viscosities (50 to 1000 cSt). Oil was added on the top surface of a 6-L vessel. Drop size distribution and Sauter mean diameter, d_{32} , measurements were carried out over 24 h mixing time. Emulsification was found to be relatively sensitive to the oil phase viscosity, μ_d , for the same ϕ yielding a narrower drop size distribution for low oil viscosity (50 cSt) and a wider drop size distribution for the highly viscous oil (1000 cSt). For the same ϕ , increasing μ_d resulted in increasing d_{32} . The equilibrium d_{32} was found to be well correlated to the viscosity number by $\frac{d_{32}}{D} = 0.026 * V_i^{0.204}$ for $\phi = 0.5$. For the same oil viscosity, d_{32} was found to increase with increasing ϕ . A multiregression of d_{32} with both ϕ and V_i for various silicone oil viscosity grades was successfully correlated by $d_{32} = 9.6\phi^{0.069} V_i^{0.216}$ with a regression coefficient (R^2) of 0.975. This shows a very weak dependence of the equilibrium d_{32} on ϕ .

Keywords: Dispersed-phase volume fraction, Drop size distribution, Liquid-liquid dispersion, Silicone oil, Surfactant

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1 Introduction

Liquid–liquid dispersion is one of the most complex of all mixing operations. Agitating two immiscible liquids results in the dispersion of one phase in the other in the form of small droplets with drop size distributions whose characteristics depend on the equipment and the operating conditions. It is virtually impossible to make dispersions of uniform drop size, because of the wide range of properties and flow conditions.

The knowledge of the resulting drop size distribution characteristics or, more exactly, the evolution of this distribution with changes of external energy input is of major importance. A large amount of work can be found in the literature concerning the prediction of drop size distributions in turbulent liquid-liquid dispersions in stirred vessels. Most of them use the concept of a turbulent energy cascade to predict the maximum stable diameter, referring to the Hinze-Kolmogorov theory [1–6].

Nonstabilized agitated dispersions have been studied in the past and a literature review of this important area has been given by Tavlarides and Stamatoudis [7]. Although the use of surfactants as stabilizing agents has wide industrial applications, it is not given that much attention in the open literature. The presence of surfactant results in lowering of the coalescence rate and thus the dynamic equilibrium between drop breakage and coalescence is reached at smaller drop sizes.

Most of the experimental data on average drop sizes exist for low-dispersed phase concentrations where a variety of measuring techniques were used [8]. Calabrese et al. [9] studied extensively the effect of the low-volume fraction, ϕ , of the dispersed-phase viscosity on drop breakup and correlate the Sauter mean diameter, d_{32} , to both the dimensionless groups, the Weber number, W_e , and the viscosity numbers, V_i . The Weber number $\frac{\rho_c N^2 D^3}{\sigma}$ represents the effects of turbulence intensity and the physicochemical properties of the system. The V_i is an effective way of describing the effect of variation in dispersed-phase viscosity. It represents the ratio of dispersed-phase viscosity to surface forces. Most of the work published in the literature (except [9]), is for a low-viscosity dispersed phase and surfactant-free systems. Koshy et al. [10] and Kumar et al. [11]

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investigated the effect of surfactants on drop breakage in turbulent liquid dispersions of low viscosity.

Few studies dealt with high-dispersed phase concentration but only [6, 12] obtained drop sizes in unstable dispersions; in all other cases surfactant-stabilized emulsions were used. Shinar [13] applied the theory of isotropic turbulence to predict the maximum drop size in a turbulently stirred vessel with dilute noncoalescing liquid-liquid dispersion.

For drops of low viscosity, whose diameters fall within the inertial subrange of turbulence, Eq. (1) was verified with a large number of liquid-liquid dispersions for a noncoalescing system:

$$\frac{d_{\max}}{D} = c_1 W_e^{-0.6} \quad (1)$$

Reported values of the constant “ c ” vary and fall in the range of 0.053 to 0.15 [14]. d_{32} and d_{\max} are correlated by $d_{32} = c_2 d_{\max}$ where c_2 is found to vary from 0.37 to 0.7.

Kraume et al. [15] showed that for a small dispersed-phase volume fraction ϕ and thereby the relatively small probability of coalescence the breakage process is dominant so that the preconditions applied for the derivation of $\frac{d_{32}}{D} = C W_e^{-0.6}$ are fulfilled. With increasing ϕ , the probability of coalescence becomes more and more important and the power on W_e becomes larger than -0.6 . The power reaches a value of -0.32 for $\phi = 0.5$.

To account for the effect of increasing ϕ (which can cause coalescence but also decrease turbulence intensity), correlations in the form of Eq. (2) have been suggested by [16]:

$$\frac{d_{\max}}{D} = c_3(1 + c_4\phi)W_e^{-0.6} \quad (2)$$

where the term $(1 + c_4\phi)$ reflects the influence of the dispersed phase in damping down the overall level of turbulence in the system, while ϕ is the volume fraction of the dispersed phase. The effect of the volume fraction was found to be negligible

for $\phi < 0.015$ and linear for $\phi > 0.15$ [17–19]. Values of c_4 were reported to be a function of the dispersed phase fraction. For $\phi < 0.3$, a value of 3.14 for c_4 was reported by [6], whereas [18] found $c_4 = 5.4$ for $\phi < 0.2$. For a low-viscosity dispersed-phase system, these researchers reported values for c_3 as 0.051 to 0.06.

It is the main aim of this work to examine the mechanisms and behavior involved in the emulsification of a high volume of dispersed silicone oil in water surfactant solution. Correlations of d_{32} as a function of ϕ and V_i are of concern in this work. To the knowledge of the author, the case of a high-volume fraction of silicone oil water dispersion has been poorly investigated in the open literature. Therefore, measurements of the drop size distribution of moderately low- to high-viscosity silicone oil (50 to 1000 cSt) were carried out during a 24 h mixing time.

2 Materials and Procedure

2.1 Stirred Tank

Experiments were carried out in a standard R&D ESCO mixer unit (ESCO-Labor AG). The unit has several advantages, such as easy recording and saving the mixing process on a PC through its data logging system, easy control of the temperature of the vessel, exact reproduction of the mixing process, and the possible use of a multi-impeller etc. Therefore, it is typically used by the health care industry for prototyping structured liquid products, such as toothpaste, shampoo, or hair conditioner. The vessel has a working volume of 6 L with 20 cm internal diameter with a standard geometry scraped wall anchor blade inside (see Fig. 1). In addition, a high-shear impeller for drop size reduction and dispersion can be fitted to one side of the main shaft and can be operated in conjunction with the anchor or by itself. A 5-cm diameter high-shear Sawtooth impeller was used in this study (see Fig. 1). The power number of the impeller was measured at turbulent Reynolds

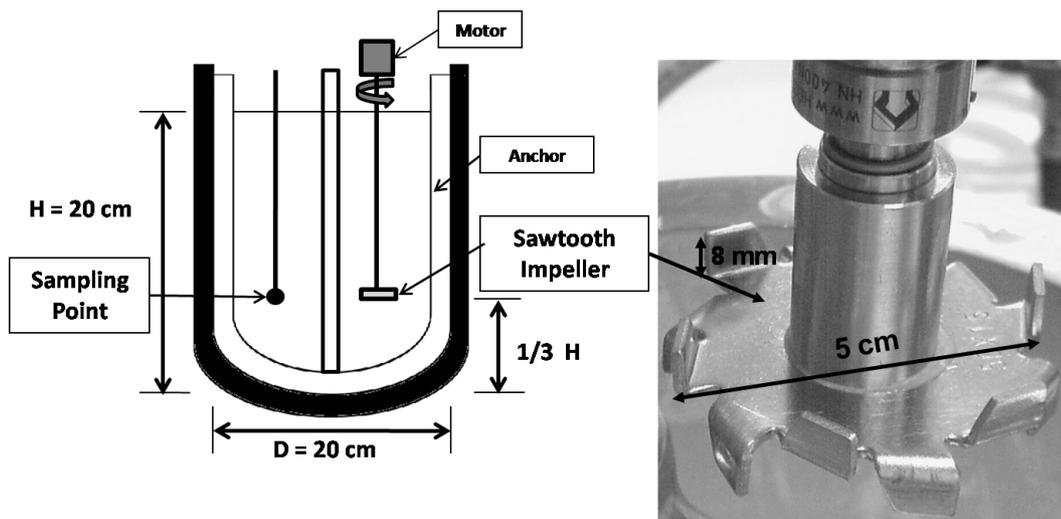


Figure 1. ESCO 6-L mixer and Sawtooth impeller.

numbers, $\frac{\rho ND^2}{\mu_c}$, using a calibrated torque strain gauge connected to an ASTECH telemetry system. The measured power number was found to be 0.32.

2.2 Materials

The model system is an oil-in-water emulsion where the oil phase is silicone oil (Dow Corning Oil 200 Fluid). Various grades of oil were used with viscosities varying from 50 to 1000 mPa·s. The surfactant stabilizer is sodium laureth sulfate (SLES) which is a mixed alkyl ether sulfate (C₁₂₋₁₄) with EO sodium salt. The grade used is a commercial sample (from Cognis UK Ltd, Hertfordshire, UK) and is obtained as a viscous yellow liquid of a specific gravity of 1.03–1.04. SLES is widely used in many household and personal care products because it is a good stabilizer for a wide range of oils. Our choice of a commercial grade of SLES rather than a purified analytical grade is because in subsequent work we would like to use it at much larger scales (56 and 2000 L) where the costs of purchasing sufficient pure surfactant would be prohibitive.

Our principle interest is to examine the effect of ϕ (1 to 50 % w/w) on the drop size distribution, Sauter mean diameter (d_{32}), and equilibrium time. A surfactant concentration well above the critical micelle concentration (CMC) was used in order to have only a breakup of drops and no coalescence.

2.3 Experimental Procedure

Fluid physical properties and the ranges of parameters used in the experiments are listed in Tabs. 1 and 2, respectively. The experimental procedure used here is the same as that described in [20] except that the silicone fluid oil equivalent to 1–50 % w/w was slowly poured only onto the top surface of the water surfactant continuous phase with the aid of a conical funnel.

A sample of 20 mL of the suspension was taken through the sampling point which was set at 2 cm from the outside of the high-shear impeller at 2 cm above the level of the impeller (see Fig. 1). Samples were aspirated using a 5-mL pocket. The sample was then taken to the Malvern 2000 instrument to measure the drop size distribution. It was found that samples measured directly after withdrawal gave the same results for the same samples measured after one day. In fact, no significant difference in the distribution was noted even after one month pro-

Table 1. Fluid physical properties at 25 °C.

Dispersed phase, silicone oils ^a		
Viscosity μ_d [mPa·s]	Density ρ_d [kg·m ⁻³]	Interfacial tension ^b σ [mN·m ⁻¹]
48	957	13.0
340	967	13.1
1000	973	13.1

^a Dow Corning 200 fluids

^b With continuous phase

Table 2. Experimental parameters used.

Parameter	Value
Dispersed-oil viscosity [cSt]	50, 350, 1000
Impeller type	High-shear Sawtooth
Impeller diameter	0.05 m
Tank diameter	0.2 m
Impeller speed [rpm]	2000
Injection position	Surface
Operating temperature [°C]	25
SLES concentration [ppm]	7090 ^c

^c Very well above CMC

viding the sample. This stability was a result of the presence of the SLES surfactant. Sampling was carried out after 2, 8, 15, 30, and 60 min and then after 2, 4, 6 h and finally after 24 h. In all experimental work, the impeller speed was kept at 2000 rpm at a given Reynolds number in the turbulent and fully turbulent regions.

3 Results

Many factors may play a role in determining the drop size distribution in agitated dispersions, e.g., impeller rotational speed, dispersed phase concentration, which is usually expressed as dispersed-phase volume fraction ϕ , agitation time, viscosity ratio of dispersed phase to continuous phase, and the interfacial tension. For the system under consideration, the effect of rotational speed [20] and the interfacial tension [21] on drop size distribution were studied and published both at low ϕ (0.01 v/v). The amount of information available and the means by which we approach the design process depend significantly on the drop phase concentration. It is common to categorize liquid–liquid systems with respect to their dispersed-phase concentration as dilute systems where $\phi < 0.01$, moderately concentrated systems with $\phi < 0.2$, and more concentrated systems where $\phi > 0.2$ [15].

To demonstrate the effect of ϕ on drop size distribution and mean droplet size, four ϕ -values were selected in this paper, i.e., 0.01, 0.05, 0.25, and 0.5. An agitated speed of 2000 rpm was chosen. All experiments were carried out for three oil viscosity grades ranging from low-viscosity oil (50 cSt) to high-viscosity oil (1000 cSt).

3.1 Effect of the Dispersed-Phase Volume Fraction ϕ

Coalescence, dispersion, and settling are all affected by the dispersed-phase concentration, e.g., coalescence rates increase with increasing ϕ . This

is due to both an increase in collision frequency and rheological changes that enable longer contact intervals to be obtained. A high dispersed-phase concentration also affects small-scale turbulent eddies, reducing their intensity and making them less able to disperse drops. A set of experiments was carried out to measure the drop size distribution and Sauter mean diameter, d_{32} , of emulsions of various dispersed-phase viscosity grades at different values of ϕ . All experiments were carried out at a fixed rotational speed, namely 2000 rpm.

- Low-Viscosity Silicone Oil, 50 cSt

Fig. 2a shows the change of d_{32} with agitation time as a function of ϕ for the 50 cSt silicone oil. As ϕ increases, the equilibrium d_{32} increases. The difference between the Sauter mean diameter at 0.01 and 0.5 ϕ -values is about 17 μm at 2 min,

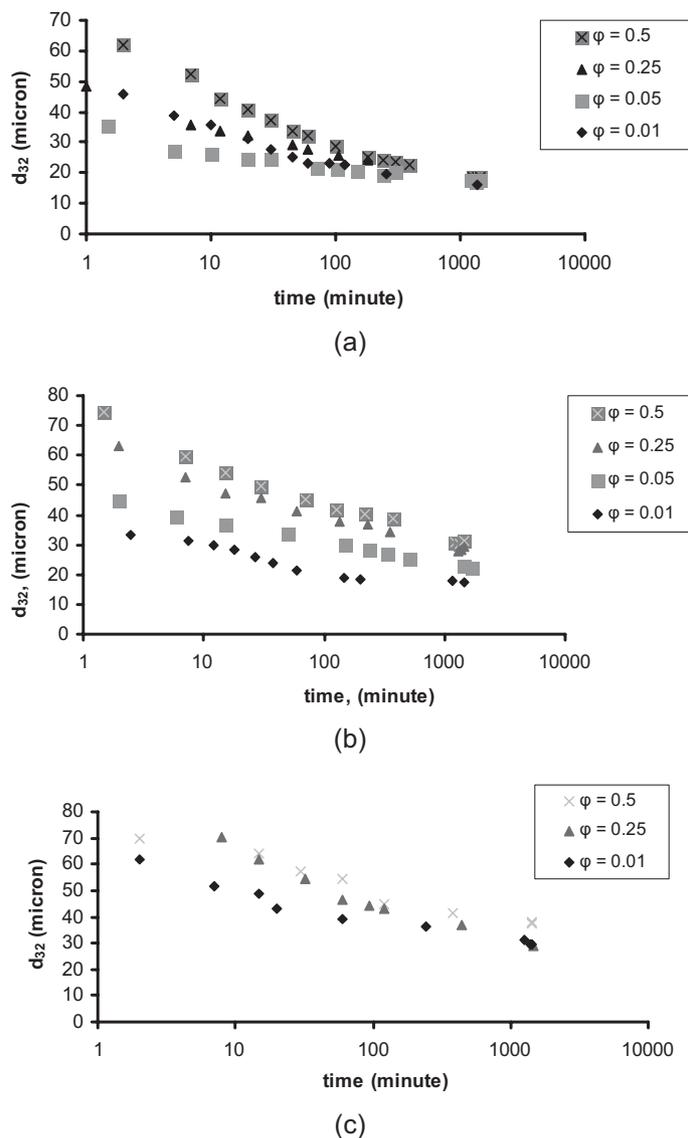


Figure 2. a) Change of d_{32} with time for 50 cSt silicone oil of different ϕ at 2000 rpm; b) Change of d_{32} with time for 350 cSt silicone oil of different ϕ at 2000 rpm; c) Change of d_{32} with time for 1000 cSt silicone oil of different ϕ at 2000 rpm.

which decreased to around 8 μm after 1 h and further to only 2 μm after 24 h.

- Moderate-Viscosity Silicone Oil, 350 cSt

Another set of experiments was carried out using a 350 cSt silicone oil at various ϕ -values (see Fig. 2a). The differences between the d_{32} at various oil phase fractions are more pronounced. At 1% oil fraction, the d_{32} falls from 35 μm at 2 min to 17 μm after 24 h agitation mixing. When ϕ is increased to 0.5, then d_{32} falls from 72 μm to 29.4 μm over the same agitation mixing time interval.

- High-Viscosity Silicone Oil, 1000 cSt

The same qualitative drift of d_{32} with time for the highest oil viscosity, 1000 cSt, is shown in Fig. 2c. Due to the high viscosity of the dispersed phase oil, the difference in the Sauter mean diameter for the three dispersed-phase fractions at the same time is not large. This is due to the fact that the inertia force is relatively low (especially at the high-dispersed phase) compared to the viscosity force between the dispersed phases. Both 0.01 and 0.25 of the dispersed-phase fractions ϕ has a lower d_{32} than that of $\phi = 0.5$. It is obvious in the figure that values of d_{32} vs. time for the dispersed-phase volume fractions of 0.01 and 0.25 were closer together and were separated from the curves for $\phi = 0.5$.

The qualitative increase of the droplet size with increase of ϕ for all oil viscosities under study is consistent with published data on model oils. For turbulently agitated model oils the droplet sizes increased at high-volume fractions of the oil-dispersed phase [22–27]. The behavior was generally attributed to coalescence by a higher collision frequency between droplets [28]. In all three silicone oil viscosity grades it seems that the dispersion of dilute systems was affected only by hydrodynamics and each drop is a single entity experiencing continuous phase fluid forces. Coalescence is neglected because few collisions occur. The increased ϕ of oil dampens the turbulent eddies, leaving the eddy length much larger than droplet diameters. As a result, the droplets can not break further. Turbulent damping at a large dispersed-phase ratio is used frequently as an explanation for larger droplet sizes [22]. The droplet sizes with higher ϕ seem to reach more quickly a steady state for all dispersed-phase viscosity oil studies.

3.1.1 Evolution of the Drop Size Distribution with Time

For a better understanding of the effect of the dispersed-phase concentration and the dispersed-phase viscosity range on d_{32} , a measurement of the whole drop size distribution was carried out. For illustration purposes, the evolution of the drop size distribution with time at approximately 2 min, 4 h, and 24 h for 350 cSt silicone oil over the specified ϕ -range is depicted in Figs. 3a–c, respectively. The mean drop diameter of the dispersed oil reduces with time and the distribution width becomes narrower. This is evident from the decrease of the span of the distributions.

Span is a measurement of the width of the distribution, represented mathematically by:

$$\text{span} = \frac{d_{0,9} - d_{0,1}}{d_{0,5}} \quad (3)$$

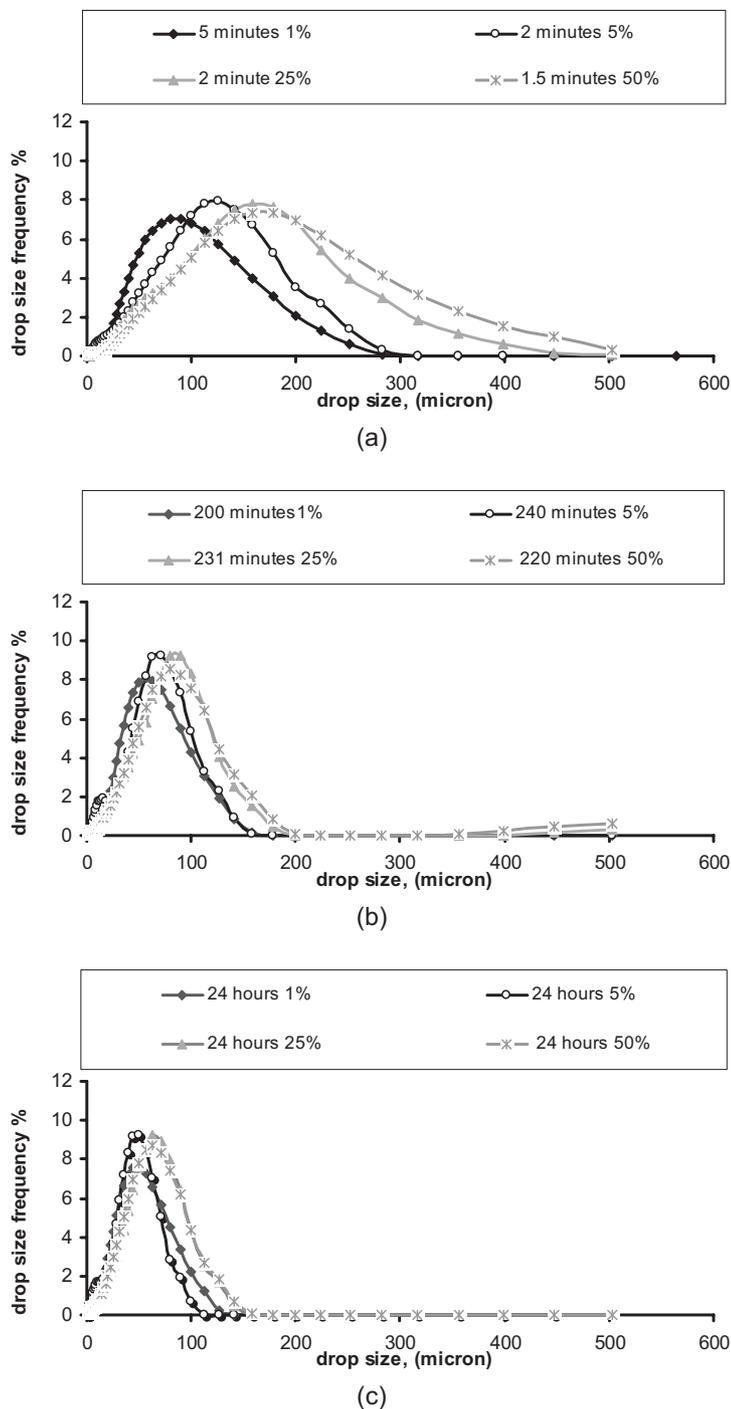


Figure 3. a) Evolution of drop size distribution of 350 cSt silicone oil at various ϕ at approximately 2 min; b) Evolution of drop size distribution of 350 cSt silicone oil at various ϕ at approximately 240 min; c) Evolution of drop size distribution of 350 cSt silicone oil at various ϕ at approximately 24 h.

For the case of $\phi = 0.01$, data were presented after 5 min elapsed mixing time instead of 2 min. From the graph it can be seen that at 2 min the distribution is well separated whereas when the mixing time elapsed, the distributions at various oil fractions ϕ become close to each other. The same qualitative behavior was found for both 50 and 1000 cSt oils.

3.2 Size Distribution and d_{32} versus Time for Fixed ϕ_{oil} of Various Oil Viscosities

Measurements of the d_{32} and drop size distribution of silicone oil of various viscosity grades in water surfactant continuous phase were carried out. At the low dispersed-phase volume fraction ($\phi = 0.01$), the equilibrium d_{32} changes from 13.6 μm for the 50 cSt oil to 29.3 μm for the 1000 cSt oil. The results of increasing ϕ to 0.5 show that for the 50 cSt silicone oil the equilibrium d_{32} is 18.7 μm whereas for the 350 cSt and 1000 cSt silicone oils it is 30.5 and 34 μm , respectively. Trends in the mean size data coincide with those for the drop size distribution which broadens considerably with increasing viscosity. Drop size distributions for the two extreme dispersed-phase concentrations ($\phi = 0.01$ and 0.5) at the end of 24 h mixing time are shown in Figs. 4a and b.

A comparison of the evolution of the drop size distribution with time for various oil viscosity grades (50, 350, and 1000 cSt) at the highest dispersed-phase volume fraction $\phi = 0.5$ is shown in Figs. 5a–c.

From Fig. 5 it can be seen that at the same mixing time the span of the distribution is higher at the high-dispersed phase viscosity than that of the low-dispersed phase viscosity. As the mixing time evolved, the span decreases. At the end of 24 h mixing time, the span values for the 50 cSt and 1000 cSt oils were 0.86 and 2.3, respectively. The same qualitative behavior was found for other studied ϕ -values.

4 Discussion

The influence of dispersed-phase viscosity on drop coalescence in turbulent flow was studied by Podgorska [29] using the population balance equation. The dispersed-phase concentration was only 0.05. Information about the evolution of drop size distribution was obtained by investigating the drop breakup and coalescence processes and solving the population balance equation.

Substituting the values for the material properties in Tab. 1 along with the rotational speed yields viscosity numbers V_i varying from 6 to 130. The viscosity number is defined by:

$$V_i = \frac{\mu_d ND}{\sigma} \left[\frac{\rho_c}{\rho_d} \right]^{0.5} \quad (4)$$

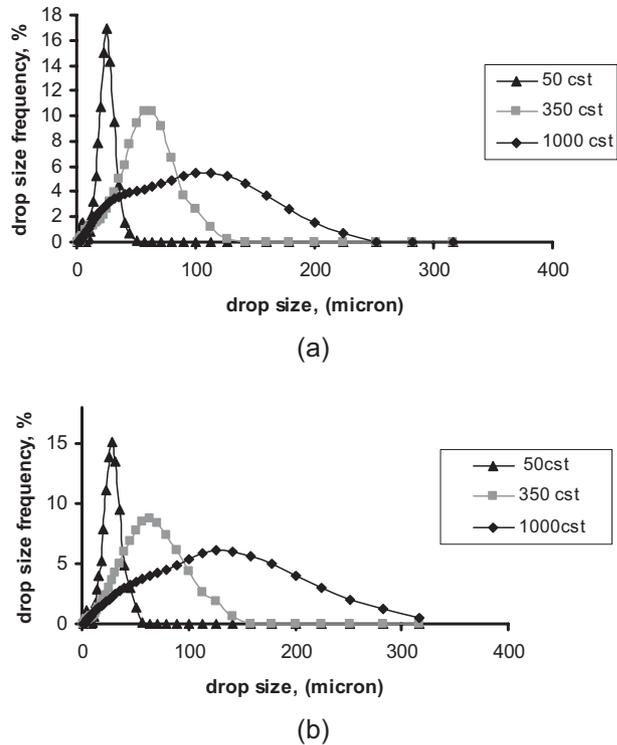


Figure 4. a) Comparison of drop size distribution after 24 h mixing time of different silicone oil viscosities at $\phi = 0.01$; b) Comparison of drop size distribution after 24 h mixing time of different silicone oil viscosities at $\phi = 0.5$.

Equilibrium drop sizes for various dispersed-phase viscosities as a function of V_i for various ϕ -values are presented in Fig. 6. The presented data are best correlated by:

$$\frac{d_{32}}{D} = 0.017 * V_i^{0.255} \quad \text{for } \phi = 0.01 \quad (5)$$

and

$$\frac{d_{32}}{D} = 0.026 * V_i^{0.204} \quad \text{for } \phi = 0.5$$

The regression coefficients (R^2) are 0.999 for $\phi = 0.01$ and 0.968 for $\phi = 0.5$.

As the rotational speed and interfacial tension are constant for all experiments carried out, the equilibria d_{32} were correlated as a function of ϕ only. Values of the correlation constants are shown in Fig. 7. The equilibrium d_{32} was found to be very weakly dependent on ϕ for low dispersed-phase viscosity oil (50 cSt). For medium- and high-viscosity oils (350 and 1000 cSt) there was a little increase in the equilibrium d_{32} with increasing ϕ .

The equilibrium data of d_{32} were best correlated to both ϕ and V_i by $d_{32} = 9.6\phi^{0.069}V_i^{0.216}$ with a regression coefficient (R^2) of 0.975. This correlation is valid for high values of V_i and can not be valid for small values of $V_i \ll 1$, as the effect of interfacial tension will be much higher than the effect of dispersed-phase viscosity. All data presented in this work are at constant values of the W_e number and therefore it is not presented in the correlation.

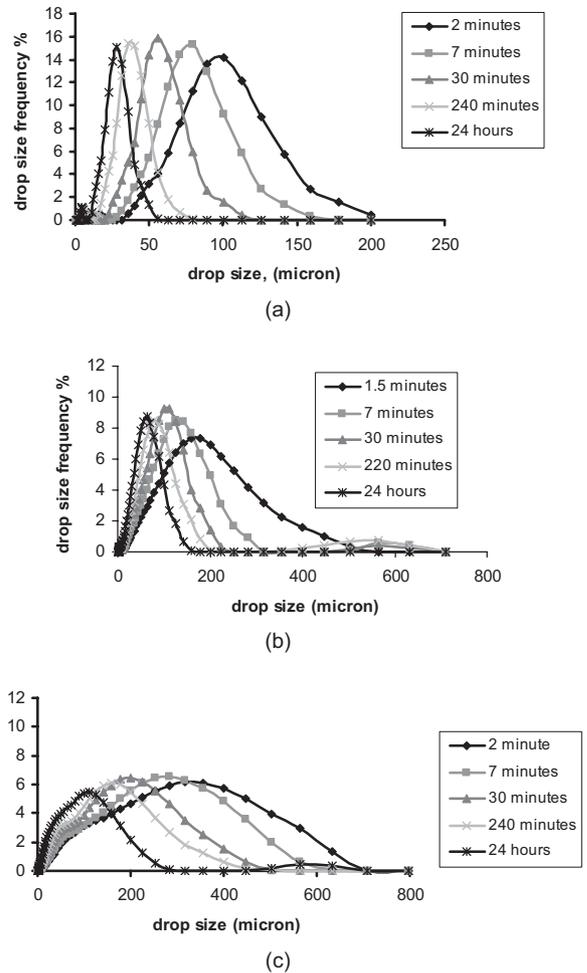


Figure 5. a) Evolution of drop size distribution with time for 50 cSt silicone oil at 2000 rpm; b) Evolution of drop size distribution with time for 350 cSt silicone oil at 2000 rpm; c) Evolution of drop size distribution with time for 1000 cSt silicone oil at 2000 rpm.

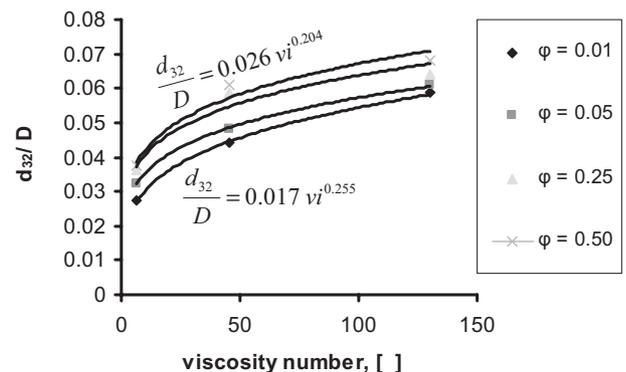


Figure 6. d_{32} as a function of viscosity number for various dispersed-phase ϕ .

Moreover, it has to be stated that for high dispersed-phase viscosity (350 and 1000 cSt) after more than one day of stirring

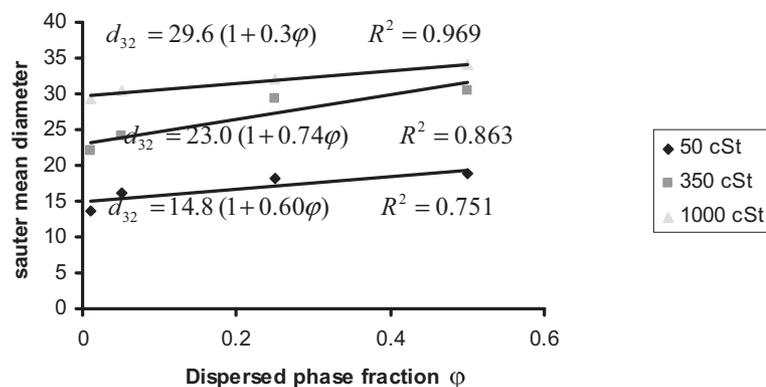


Figure 7. Equilibrium d_{32} as a function of dispersed-phase ϕ for different oil viscosities.

there is little change in drop size but less than that for the low-viscosity oil. Therefore, it can be concluded that breakage and coalescence processes obviously reach equilibrium very fast for low dispersed-phase viscosity compared to that for high dispersed-phase viscosity. In a model presented by [29] the assumption of partially mobile drop interfaces gives quite good results for silicone oil of low viscosity (10 mPa·s). For silicone oil of high viscosity (500 mPa·s), drop size distribution was not precisely predicted by one simple model because drops of different size behave in a completely different way. Drop size distribution data presented in this work can be used to tune the model by [29].

5 Conclusions

Drop size distributions of the high-dispersed volume fraction of silicone oil-in-water emulsions in the presence of surfactant solution are successfully presented for a Sawtooth impeller. From the experimental results and in addition to the conclusions presented in the discussion, the following conclusions can be drawn:

- As the viscosity of the dispersed-phase oil increases, the difference between the equilibrium d_{32} (after 24 h) at the low and high ϕ increases, i.e., for the 50 cSt oil the difference is only 2 μm whereas for the 350 cSt it increases to 10 μm and when the dispersed phase viscosity increases to 1000 cSt, the difference becomes 12 μm .
- In a previous work carried out by the author [20] it was found that the theoretically derived correlation $d_{32} \approx We^{-0.6}$ is valid for a low-phase fraction ϕ (0.01). The exponent on We_c was found to vary with increasing the dispersed-phase viscosity, μ_d . This is due to the complex breakup coalescence mechanism. Therefore, data presented in this work can be used to investigate the breakup mechanism of the low and high dispersed-phase volume fractions. This requires the application of population balance models or any other convenient models.
- This paper enriches the literature with more data on drop size distribution for low and high dispersed-phase volume fractions ($\phi = 0.01$ to 0.5) of silicone oil water surfactant

emulsion. It was found that with increasing ϕ there was a little increase in d_{32} due to coalescence-dominant condition at higher ϕ . This dependence was successfully presented in this work for low to high μ_d (50 to 1000 cSt). The overall effect of both ϕ and We_c on d_{32} is still needed and therefore more work at various rotational speeds at the same ϕ and oil μ_d used in this work would be of great help to draw this dependency.

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Symbols used

μ_d	[Pa·s]	dispersed-phase viscosity
D	[m]	impeller diameter
d_{max}	[m]	maximum stable drop size
N	[s ⁻¹]	impeller speed
P_o	[-]	impeller power number
Re	[-]	Reynolds number
d_{32}	[m]	Sauter mean diameter
V_i	[-]	viscosity number
We_c	[-]	Weber number
ρ_c	[kg·m ⁻³]	continuous-phase viscosity
ρ_d	[kg·m ⁻³]	dispersed-phase viscosity
σ	[Pa·s]	interfacial tension
ϕ	[-]	dispersed-phase volume fraction

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