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Drop Size Evolution Kinetics in a Liquid-Liquid Dispersions System in a Vessel Agitated by a Rushton Turbine

Roman Formánek[a,*], Bohuš Kysela[b], Radek Šulc[a]

[a]Czech Technical University in Prague, Faculty of Mechanical Engineering, Department of Process Engineering, Technická 4, 166 07 Prague, Czech Republic
[b]Czech Academy of Sciences, Institute of Hydrodynamics, Pod Paťankou 30/5, 166 12 Prague, Czech Republic
Roman.Formanek@fs.cvut.cz

Processes in which mixing plays a crucial role in increasing mass transfer, interfacial surface, and homogenization are frequently and widely used throughout the industry. Agitation of immiscible liquids or solid-liquid suspensions is a frequent operation in chemical and metallurgical industry. The aim of this contribution is to study drop size evolution kinetics in a liquid-liquid dispersion system in a vessel agitated by a Rushton turbine and to analyze the homogeneity of the dispersed system in a volume of the agitated vessel. The homogeneity of liquid-liquid dispersion in a volume of a mixed system was determined by comparison of Sauter mean diameters and drop size distribution (DSD) from different measured regions. The sizes of drops were obtained from the images captured by a camera system and were processed by the Image Analysis method.

1. Introduction

Prediction of mean drop/particle size and drop/particle size distribution (DSD) is vital for emulsification, suspension polymerization, solid particle dispersion or crystallization and is affected by many factors. The information about Sauter mean diameter d_{32} in the final stage of the process has been found mostly in the literature. The DSD in a mixing tank has been simulated by means of the population balance approach. Unfortunately, there is a lack of kinetic experimental data to verify the simulation predictions of DSD kinetics.
The drop size was evaluated by the in-situ measurement with the following Image Analysis (IA). The kinetics of the development of drop size distribution in time inside an agitated vessel has been investigated by many researchers. The time evolution of the DSD was investigated by Šulc et al (2018), Baldyga et al. (2001) examined the influence of scale-up on drop size, Hong and Lee (1983) researched unsteady-state in the agitated vessel of a liquid-liquid dispersion system. They tried to develop a simple model of drop size distribution developing in time and depending on parameters of the mixing system. The influence of agitated vessel geometries was also examined by Tang et al. (2005) and the effect of various impellers on the drop size evolution was examined by Zhou et al. (1998). The improvement in that field is continuous and is related to the development of experimental equipment, process conditions, and computing power.
The aim of this contribution is to measure DSD time evolution in a liquid-liquid dispersion in a vessel agitated by a Rushton turbine and to investigate homogeneity of liquid-liquid dispersion in agitated vessel volume. The homogeneity was assessed comparing Sauter mean diameters and DSD in three investigation areas with different off-bottom distance.
The drop size was determined from the images captured directly in vessel volume by the high-speed camera with a powerful light source. In our previous research, we found that the light source quality is crucial for this measurement technique Formánek et al. (2018). As the liquid-liquid system was used the mixture of distilled water and silicone oil. The time evolution of measured DSD data was described using a Log-normal cumulative distribution function and by the evolution of Sauter mean diameters in dependence on dimensionless time.

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2. Experimental

The experiments were performed in the cylindrical vessel of inner diameter T = 300 mm and with a flat bottom. The vessel was equipped with four baffles and the width of baffles was b = 0.1T. The vessel was filled with water-oil emulsion and the liquid level was H = T. Mixing of the emulsion was provided by Rushton turbine with diameter D = T/3 and with off-bottom distance C = T/4. In experiments speed control unit IKA EUROSTAR POWER was used as impeller motor and 200 rpm impellers speed was used through all of the experiments. The speed of the impeller was set to obtain the turbulent regime (Re = 33 333) and also to prevent the oil settling on the bottom of the vessel due to the oil density. The optical box filled distilled water was used for reduction of image distortion. It eliminated the refractive index caused by the cylindrical vessel geometry. The vessel was placed in a center of the optical box (see Figure 1a).

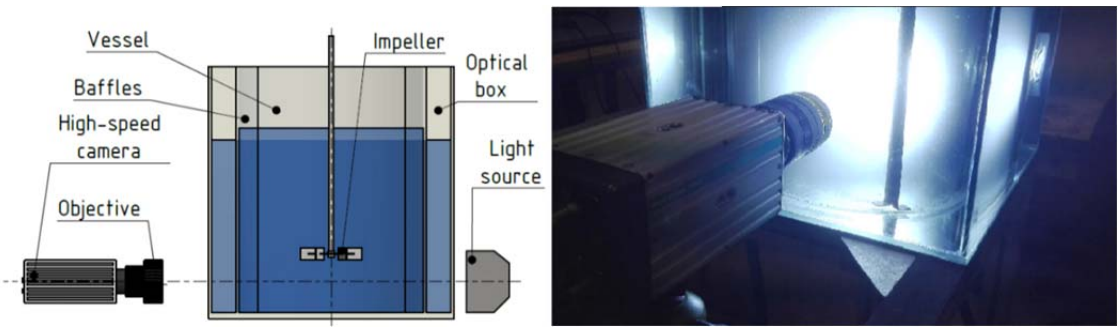

Figure 1: a, Arrangement of measurement equipment b, Demonstration the illumination of light source

The continuous phase was distilled water and dispersed phase was silicone oil WACKER AP 200. The physical properties of WACKER AP 200 were measured in dependence on temperature. The physical properties of used silicon oil (dispersed phase) and distilled water (continuous phase) are given in Table 1 for measured temperature T = 21.8 °C. Dispersed phase volume fraction was 0.00047 v/v (dilute system).

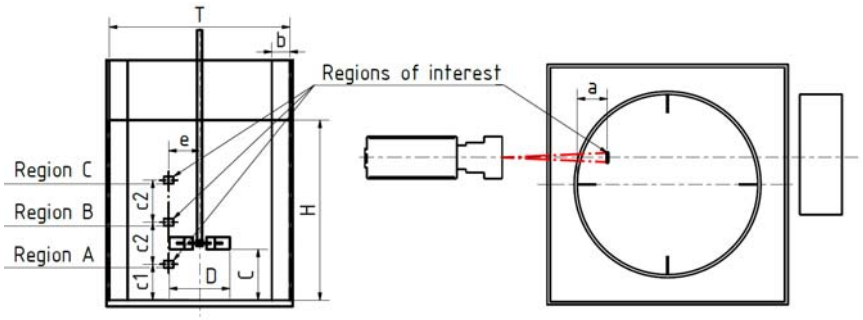

Figure 2: Scheme of the image acquisition system

The image sequences were captured by a high-speed camera SpeedSence MK III with frame rate 30 fps and resolution 1280 x 1024 pixels which was equipped with objective Sigma 105 mm F2.8 EX DG MACRO. This type of objective and its optical properties have no influence on the distortion of the images (see Figure 3c). The used technique of in-situ measurement required a powerful light source (see Figure 1b). The one-point light source was used and was composed of LED chip COB GT-FC90X3-0E, the optical lens (accessories of this chip), and cooler Intel LGA775. The power input of it was 90 W with light flux 6000 lm, radiation angle 20°, and light temperature 8500 K. Both light source and camera were placed perpendicularly on the walls of the optical box and the light source was placed directly against the center of the objective.

Three regions of interest were chosen in which the images were captured (see Figure 2) for research of homogeneity of the dispersed phase in a volume of the agitated vessel. The first region was placed under the impeller with off-bottom distance c1 = 50 mm. Other two regions were placed over the impeller with the same distance between all the regions c2 = 70 mm. All three regions were placed eccentrically from impeller axis with distance e = 55 mm, and with a = 50 mm from the vessel wall. The area of regions was approximately

15 x 12 mm. In these configurations the image resolution was 0.0117 mm/pixel. The image resolution was obtained from scale captured on the image (see Figure 3c).

Table 1: Physical properties of continuous a dispersed phase

Phase	Dynamic viscosity μ [mPa.s]	Density ρ [kg.m^{-3}]	Surface tension σ [mN.m^{-1}]
Continuous (water)	0.959	997.82	72.05
Dispersed (oil)	242	1076.12	26.43

2.1 Experimental procedure

For all regions of interest 8 sets of 1000 images were captured with a shutter speed of 3 μs after 5 min (see Figure 3a). The total time of measurement for all regions was 40 min. At the time t = 0 min silicone oil was added and the first measurement was performed at the time t = 5 min.

Figure 3: a, The original captured image b, The precise calibration spheres "Nylon 6/6 Balls" c, The image with captured scale

The drops sizes were evaluated from captured images by Image Analysis using Image J software. The method developed by Kysela et al. (2017) was used for drop identification. This approach is based on an identification of drop boundary using pixel shade gradient. The method compares the shade of neighbouring pixels. The sharp boundary exhibit high gradient of shade between neighbouring pixels. The computed difference in pixel shade between neighbouring pixels was compared with the set gradient value. If the difference was greater then set gradient value, we assumed that the drop boundary is identified. The drops were identified from images captured and their equivalent drop diameters were calculated according to the projected area for given stirring time.
The IA method was calibrated on images on which captured special polymeric spheres "Nylon 6/6 Balls" with the precise diameter of 3/64'' = 1.19 mm. The photo of these calibration spheres is shown in Figure 3b. The parameters of IA were set up so that to answer evaluated sizes corresponding calibration spheres. Thus set IA was used for evaluation of drops size.

3. Result

3.1 Sauter mean diameter kinetics

The evolution of drop sizes on dimensionless time was described using by Sauter mean diameter d_{32}. The Sauter mean diameter was calculated from the following formula:

$$d_{32} = \frac{\sum_{i=1}^{m} n_i d_i^3}{\sum_{i=1}^{m} n_i d_i^2} \quad (1)$$

where d_i [mm] is drop diameter and n_i [-] is number of the drops. The d_{32} was evaluated for each time step of measurement and for every region of interest. The dimensionless time was defined as follows:

$$t^* = Nt \quad (2)$$

where N [rpm] is a rotation of impeller and t [min] is an agitation time. The evolution of Sauter mean diameter d_{32} on dimensionless time t* is presented in Figure 4.
As seen in Figure 4, the similar Sauter mean diameter kinetics was found in regions A and B which were placed in the same distance from the impeller. The values of d_{32} in last measurement step (at 40 min) were

taken as the steady-state values of a dispersed system for all regions of interest. The d_{32} value of 90 μm at steady state was reached in both regions A and B. Unlike that, in region C which was placed at the highest level the drop breaking rate was two times lower than in both A and B regions. The final Sauter mean diameter in steady state was 1.3 times higher compared to the one in regions A and B, reaching 120 μm.

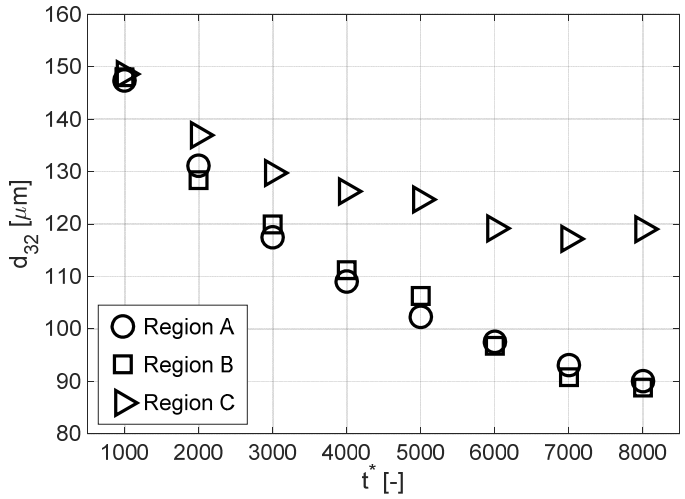

Figure 4: Time evolution of Sauter mean diameter in various regions

The steady-state values of d_{32} were compared with Hinze-Kolmogorov theory (see Figure 5). According to this theory, the dimensionless Sauter mean diameter defined as the ratio d_{32}/D depends on impeller Weber number We as follows:

$$\frac{d_{32}}{D} = KWe^{-3/5} \tag{3}$$

where K is constant of proportionality, D [m] is the impeller diameter and We [-] is the impeller Weber number. Chen and Middleman (1967) reported the value of K = 0.053 for a Rushton turbine and a broad range of physical properties and vessel sizes.

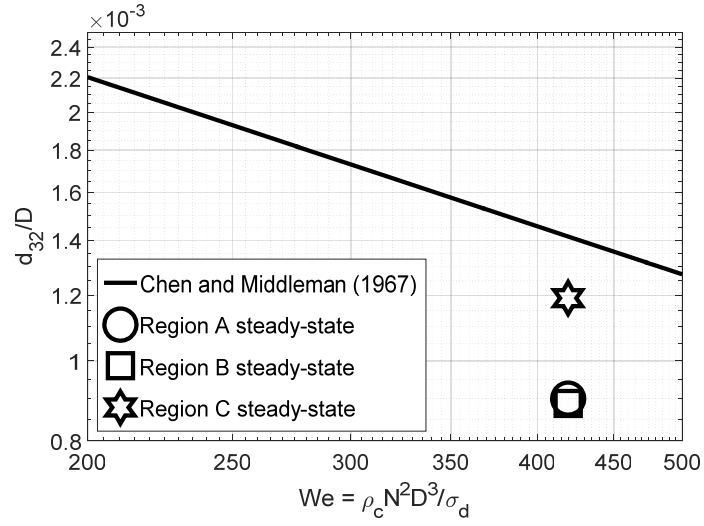

Figure 5: Comparison of steady-state d32 with Chen and Middleman (1967) correlation

The steady d_{32} value predicted by this correlation is 140 μm for given experimental conditions. The steady d_{32} value in region C was closest to prediction given by Eq. (3) and K given by Chen and Middleman (1967). The

steady Sauter mean diameters in regions A and B had practically the same value and were quite distant from given correlation. However, the correlation shown in Figure 5 provides a mean value for the whole system and from this viewpoint could be the difference between evaluated values and correlation and also caused by experimental errors.

3.2 Time evolution of DSD

The drop size distribution was described by Log-normal distribution function plotted as a function of the logarithm of the drop volume. The cumulative volume fraction was determined for each time step and for each region of interest. The time evolution of the cumulative volume fraction of region A is shown in Figure 6. Progress of dispersion is visible between the beginning of measurement and steady state. At the beginning of drop breaking (5 min) 60 % drops had lower volume than 10^{-3} mm^3. At the steady state (40 min) the percentage of drops having a smaller volume than 10^{-3} mm^3 was more than 80 %. The similar DSD evolution was observed in region B. Unlike those, the DSD changes with time in region C were not significantly visible. Therefore, we compared DSD at the beginning and steady-state time steps for each region of interest. The distribution curves are presented in Figure 7.

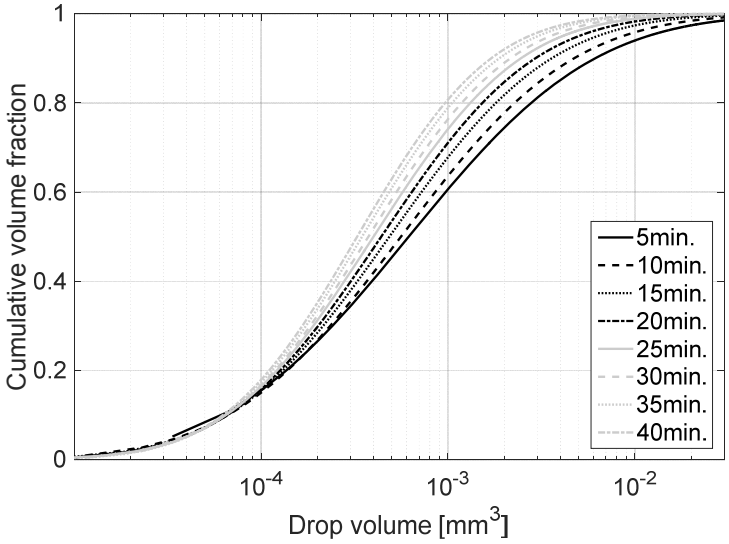

Figure 6: Time evolution of cumulative distribution for measured region A

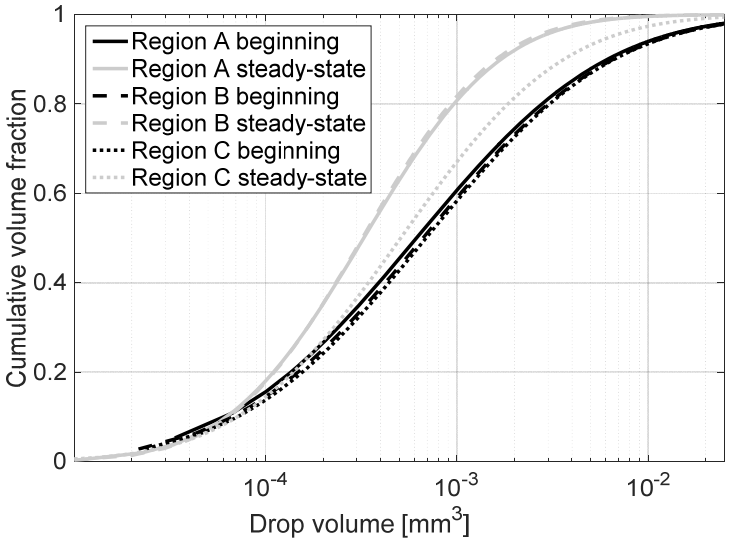

Figure 7: Cumulative distribution at the beginning and a steady-state step for each measured region of interest

The DSD at the beginning state was practically the same in all investigated regions of interest. The difference between beginning states was only several percent and it can be negligible from the viewpoint of the number of evaluated drops which was about 10^4. The DSDs of steady-states were practically the same in regions A and B. The time change of DSD for steady-state in region C is significantly lower comparing with DSD change in regions of interest A and B. It signalizes that the drop size distribution was not homogeneous in the agitated vessel for our experimental conditions. Sprow (1967) found that the drop sizes are strongly dependent on the sampling position in strongly coalescing agitated liquid-liquid systems. For dilute liquid-liquid dispersion the effect of sampling point or investigated area for in-situ measurement is not stated in the literature. The possible effect of measured region location on drop size distribution will be tested for dilute liquid-liquid dispersion in the future experiments.

4. Conclusion

The drop size time evolution and the homogeneity degree of liquid-liquid dispersion were investigated in a vessel agitated by a Rushton turbine. The experiments were executed in a fully baffled vessel with a diameter 300 mm. The tests have been carried out with silicone oil - distilled water dispersion (oil in water) of dispersed phase volume fraction 0.00047. The in-situ measurement based on image analysis was used for determination of Sauter mean diameter and drop size evolution of DSD. The drops were measured in three regions of interest with different off-bottom clearance.

The time evolution of Sauter mean diameter was found practically the same in regions A and B which were placed in the same distance from the impeller. Unlike those, in region C which was placed at the highest level, the drop breaking rate decreasing tendency was two times lower than in both regions A and B. The experimentally determined Sauter mean diameters in steady-state were compared with the correlation proposed by Chen and Middleman (1967). The cumulative volume fraction was determined for each time step and for each region of interest. The increasing proportion of small drops with increasing time was significantly evident in the dispersed system in both regions of interest A and B. Unlike those, the change of smaller drops was not so visible in region C.

It was found that the DSD profiles obtained at the beginning and steady-state time were different for each region of interest. It signalizes that the drop size distribution was not homogeneous in this case. The effect of sampling point is known for strongly coalescing agitated liquid-liquid systems. The possible effect of location on DSD will be tested for dilute liquid-liquid dispersion in future experiments.

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