

Dispersion of immiscible liquid-liquid system in a vessel agitated by a Sawtooth impeller: drop size time evolution

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Abstract. Mixing of immiscible liquid-liquid system is the crucial process for many branches of industry. The main purpose of this process is to increase mass transfer efficiency in processes occurring in chemical and metallurgical industry. The knowledge of drop size and surface area of drops is important for the determination of mass transfer time. The aim of this contribution is to study drop size time evolution in immiscible liquid-liquid system agitated by high-shear Sawtooth impeller in baffled cylindrical vessel. The drop size evaluation was investigated in three different regions of interest. The time evolution of drop size was described by the kinetic model proposed by Hong and Lee (1983) for each region of interest. Simultaneously, the drop size distribution (DSD) was determined and compared for each regions of interest. The time evolution of drop sizes was investigated by non-invasive measurement directly in control volume. The images of illuminated region of interest were captured by a high-speed camera. The captured images were processed by the Image Analysis method.

1 Introduction

The knowledge of drop size kinetics is vital for many processes in wide branches of industry. The information about Sauter mean diameter d_{32} in the final stage of the process has been mostly presented in the literature. The drop size distribution (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 different experimental methods for the measurement of drop size exist which may be affected by many factors.

The kinetics of Sauter mean diameter d_{32} and drop size distribution in an agitated vessel has been investigated by many researchers. Hong and Lee [1] investigated unsteady-state in the agitated vessel of a liquid-liquid dispersion system. They proposed a simple model of drop size describing d_{32} time evolution with model parameters depending on the mixing system characteristics. The time evolution of the DSD was investigated by Šulc et al. [2].

In our previous work [3] the time evolution of Sauter mean diameter and DSD and the homogeneity of liquid-liquid dispersion were investigated in a cylindrical baffled vessel agitated by a Rushton turbine. We found that the differences between the three different regions of interest exist. The highest differences were found between the highest placed region and both other lower placed regions of interest. The results obtained in both

lower placed regions of interests were practically the same.

The aims of this contribution are 1) to measure the time evolution of DSD and Sauter mean diameter and 2) to investigate the homogeneity level of liquid-liquid dispersion in baffled vessel agitated by a high-shear Sawtooth impeller. The homogeneity was assessed comparing numbers of evaluated drops and Sauter mean diameters and DSD in three investigation areas placed in different off-bottom distance. The drop size was evaluated by the in-situ measurement with the following Image Analysis (IA).

2 Experimental

The experiments were carried out in a cylindrical baffled vessel of inner diameter $T = 300$ mm with a flat bottom and the vessel was filled with water-oil emulsion. The mixing of the emulsion was provided by a high-shear Sawtooth impeller with diameter $D = T/3$. The impeller was placed with off-bottom clearance $C = T/4$ (see Figure 1). The impeller revolution of 600 rpm was used through all of the performed experiments. The speed of the impeller was set to obtain the turbulent regime and also to prevent the oil settling on the bottom of the vessel due to the higher oil density comparing with water. The impeller Reynolds number was equal to $Re = 107\,262$.

The continuous phase was distilled water and the dispersed phase was silicone oil WACKER AP 200. The physical properties of used silicon oil (dispersed phase) and distilled water (continuous phase) are given in Table

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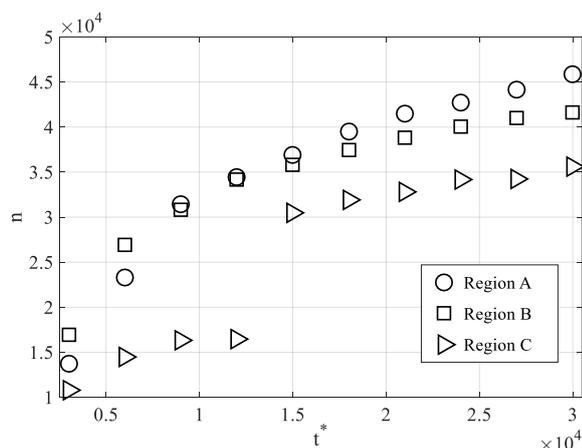


Figure 3. The evolution of the number of evaluated drops.

It was found that the number of evaluated drops increases with increasing dimensionless time in each regions of interest as it was expected. The dimensionless time evolution of a number of evaluated drops is practically the same for region A and B. Unlike this, the number of drops evaluated in region C is significantly lower.

3.2 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 using the following formula:

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

where d_i [mm] is drop diameter in i^{th} class and n_i [-] is a number of the drops in i^{th} class. The d_{32} was evaluated for each time step of measurement and for every region of interest.

Hong and Lee [1] proposed a simple model describing d_{32} kinetics in the following form:

$$d_{32}/d_{32}^* = 1 + \alpha e^{-\beta t^*} \quad (3)$$

where d_{32} [μm] is the value of Sauter mean diameter at time t^* , d_{32}^* [μm] is the value of Sauter mean diameter at steady state, α and β [-] are model parameters.

The model was tested on the measured data. The evaluated parameters of this model for the measured data are presented in Table 2. The comparison of model and experimental data is presented in Figure 4. The correlation is very good.

The differences between d_{32} kinetics and predicted drop diameter at steady state in each regions are evidently significant. The smallest drop diameter at steady-state d_{32}^* was determined in region C. Comparing with this region the drop diameter is twice and three times higher in region A and region B respectively. The parameters α and β vary similarly.

Table 2. Sauter mean diameter kinetics: evaluated parameters of Hong and Lee [1] model for measured data.

Region	d_{32}^* [μm]	α [-]	$\beta \cdot 10^{-5}$ [-]
A	31.639	1.386	-1.469
B	45.076	0.682	-2.918
C	16.874	3.435	-0.726

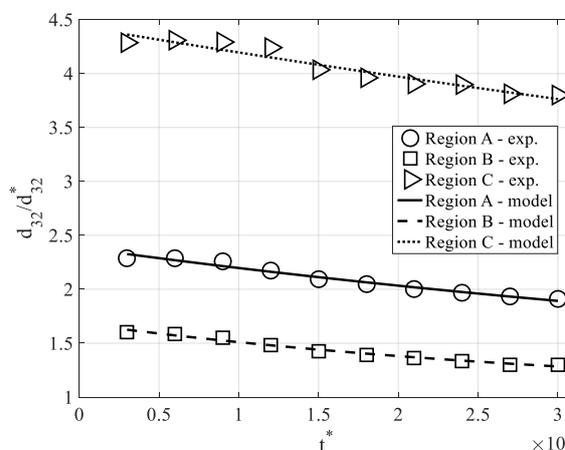


Figure 4. Sauter mean diameter kinetics: comparison of Hong and Lee [1] model and measured data.

3.3 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 diameter. The cumulative volume fraction was determined for each time step and for each region of interest. For illustration, the time evolution of the cumulative volume fraction of region A is shown in Figure 5.

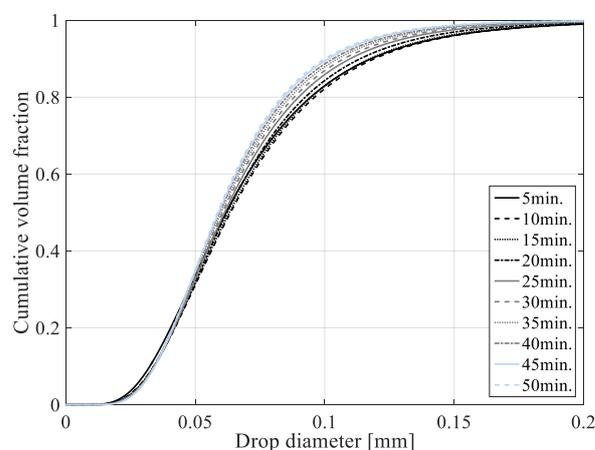


Figure 5. Time evolution of cumulative distribution for measured region A.

It was found that the DSD develops unstably during the first 10 minutes after the oil injection (oil injection at the $t = 0$ min). In this period d_{32} diameter increases and then again decreases. This volatility is probably due to low pumping effect of high-shear Sawtooth impeller. Thus, some “stabilization” time is necessary to overcome this transient state.

The progress of dispersion can be visibly demonstrated comparing the beginning and last run of measurement. At the beginning of drop breaking (5 min) 60 % drops had a diameter lower than 0.75 mm. At the last measurement run (50 min) the percentage of drops having a smaller diameter than 0.75 mm was approximately 70 %.

For comparison the DSD at the beginning and last time step are presented in Figure 6 for each region of interest. The DSD at the beginning was practically the same in all investigated regions of interest. The difference between beginning states was only several percents and it can be negligible from the viewpoint of the number of evaluated drops. In regions A and B the DSDs at last time steps were practically similar. Unlike those, the DSD changes with time in region C were not significantly visible. It signals that the drop size distribution was not homogeneous in the agitated vessel for our experimental conditions. In our previous work [3] the same results were obtained for dilute oil-in-water system agitated by a Rushton turbine. It seems that the location of the measurement region influences the taken results in dilute systems.

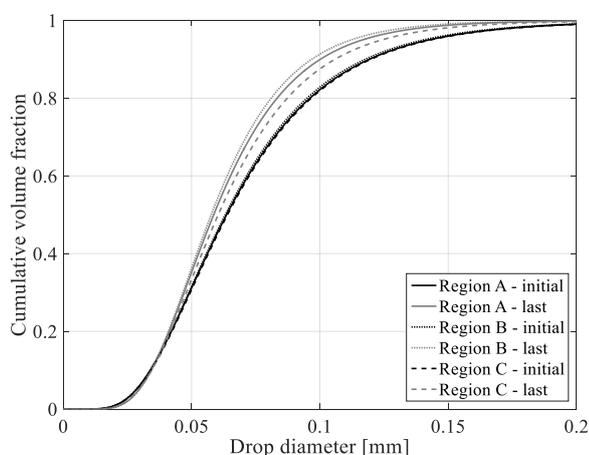


Figure 6. Cumulative distribution at the initial and last time step for each regions of interest.

4 Conclusions

The drop size time evolution and the homogeneity degree of liquid-liquid dispersion were investigated in a vessel agitated by a high-shear Sawtooth impeller. 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 the determination of drop size distribution and Sauter mean diameter. The drop sizes were measured in three regions of interest with different off-bottom clearance. The impeller revolution of 600 rpm was used for all provided experiments.

The number of evaluated drops was found to be increasing with increasing time of measurement in each region of interest as it was expected on the basis of the theory of drop break-up in a dilute system. The number

of evaluated particles was in range from 1 to 1.75×10^4 at the beginning and from 3.5 to 4.5×10^4 at the last time step of measurement.

The time evolution of Sauter mean diameter was found to be different in each regions of interest. The experimental data were treated using the model of d_{32} kinetics proposed by Hong and Lee [1]. The correlation is very good. The model predicts different values of drop diameter at steady-state d_{32}^* in each region of interest. The smallest drop diameter at steady-state d_{32}^* was determined in the region C. Comparing with this region the drop diameter is twice and three times higher in the region A and region B respectively.

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 develops unstably during first 10 minutes after the oil injection (oil injection at the $t = 0$ min). In this period d_{32} diameter increases and then again decreases. Thus, some “stabilization” time is necessary to overcome this transient state. This time period is two times higher comparing with the system agitated by a Rushton turbine [3]. This longer period is caused probably due to the low pumping effect of high-shear Sawtooth impeller.

It was found that the DSD profiles obtained at the beginning (10 min) and at the last time step were different for each region of interest. It signals that the drop size distribution was not homogeneous in the investigated regions. The effect of the sampling point is known for strongly coalescing agitated liquid-liquid systems [4]. The possible effect of location on DSD will be tested for dilute liquid-liquid dispersion and various process conditions in future experiments.

It was found that time that is necessary to achieve steady-state is longer for system agitated by high-shear Sawtooth impeller comparing with the system agitated by Rushton turbine probably due to lower pumping effect. Following it the measuring time period must be prolonged for system agitated by Sawtooth impeller.

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