18th International Conference on
TRANSPORT AND SEDIMENTATION OF SOLID PARTICLES
11-15 September 2017, Prague, Czech Republic
ISSN 0867-7964 ISBN 978-83-7717-269-8

IN-SITU MEASUREMENT OF PARTICLE SIZE DISTRIBUTION IN AN AGITATED VESSEL

Bohuš Kysela[1], Jiří Konfršt[1], Zdeněk Chára[1], Radek Šulc[2], Pavel Ditl[2]

1 Czech Academy of Sciences, Institute of Hydrodynamics, Pod Paťankou 30/5, 166 12 Prague, Czech Republic,
2 Czech Technical University in Prague, Faculty of Mechanical Engineering, Department of Process Engineering, Technická 4, 166 07 Prague, Czech Republic

Agitation of solid-liquid suspension or two immiscible liquids is a frequent operation in chemical and metallurgical industries (suspension/emulsion polymerization, catalytic chemical reaction, hydrometallurgical solvent extraction). The product quality, yield and economy of the processes are significantly affected by a mixing process. Prediction of mean particle/drop size and particle/drop size distribution (PSD) during the agitation is fundamental for emulsification, suspension polymerization, solid particle dispersion or crystallization.
The aim of this contribution is to propose a simple method of in-situ measurement of particle size distribution. The particle size measurement is based on an image analysis performed on raw image records. Evaluation method based on the best focused particles with sharp detected boundaries enhanced by the analysis of particle circularity was developed. Precise spherical mono-disperse steel and plastic particles were used to verify accuracy of evaluation method. The method has been proposed for the measurement of the time evolution of the drop size distribution in liquid-liquid dispersion in an agitated tank. The effect of droplet size distribution on the impeller speed in water-oil dispersion in agitated vessel was obtained.

KEY WORDS: particles, droplets, PSD, image analysis, agitated vessel.

1. INTRODUCTION

Particle size distribution is a crucial property in particulate processes and it affects a lot of quantities - flow of suspensions, dispersions, eventual separation processes, mass transfer etc. This contribution is focused on in-situ measurements and evaluation of particle size distribution of suspended solid spherical particles and spherical droplets of oil in water-oil dispersion. The dispersions were processed in an agitated vessel. These investigated processes are commonly used in chemical or metallurgical industry (e.g. extraction, catalytic reactions, polymerization etc.). The dynamic evolution of droplet size distribution in time inside an agitated vessel has been investigated by many researchers

[1,2,3,4]. They tried to develop a simple model of droplet size distribution developing in time and depending on parameters of the mixing system. Several models based on population balances [5] or various models of distribution curves were used. The agitated vessel geometries or impeller types were also examined [6,7]. The improvement in that field is permanent and is related to the development of experimental equipment and computing power.

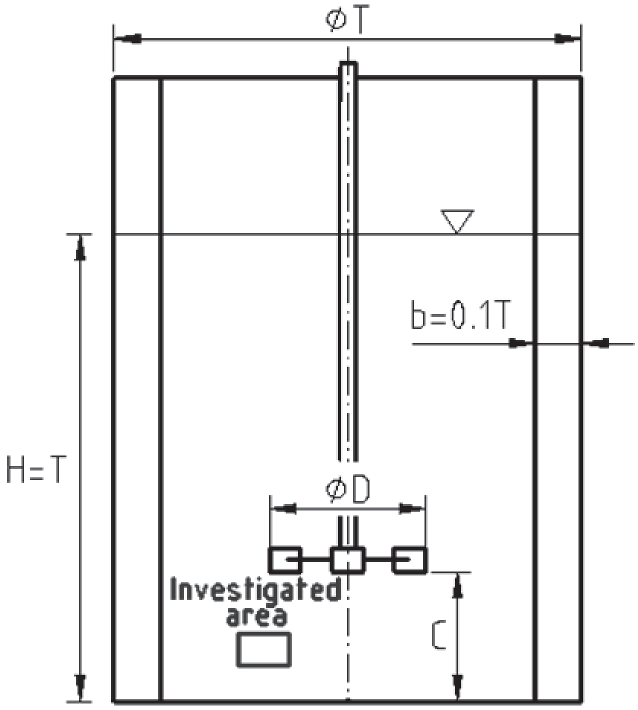

Fig.1. Description and geometry of an agitated vessel
(H/T = 1; D/T = 1/3; C/D = 4/3; b/T = 1/10; four baffles).

2. EXPERIMENTAL

The experimental work was divided into two parts: the first case was the measurement with mono-disperse solid precise particles with known diameter. The second case was the measurement with two immiscible liquids - water and silicone oil. Both cases were performed in an agitated system schematically described in Fig.1. The particle sizes were evaluated from raw image records by the image analysis.

2.1. AGITATED VESSEL

The experiments were performed in the agitated vessel with standard cylindrical geometry and with flat bottom. The vessel was equipped with four radial baffles (width b = 0.1 T). The diameter of the vessel was T = 300 mm. The vessel was filled with water and the liquid level was H = T. The standard Rushton impeller with ratio T/D = 3 and off-bottom clearance C = H/2 (see Fig.1) was used. The impeller speed was 150; 200; 250; 300 rpm. The model liquids were distilled water and silicone oil AP 200 (density = 1070 kg/m^3, kinematic viscosity = $2\cdot10^{-4}$ m^2/s at 25 °C). Used dispersed oil phase fraction was 0.0047, where the oil was added in before measurements by syringe injection. Firstly, the measurement limits of water-oil dispersion were tested. The following procedure was suggested - each record was 1000 images (total time 33.3 s) and time of 10 min. was taken between impeller speed changes to stabilise the droplets. The investigated area (Fig.1) was approximately 15 x 12 mm. The area was placed 40 mm from impeller axis and off-bottom clearance was 30 mm. The focused plane was approximately 50 mm from vessel wall. This place was chosen according to the circulation loop shape generated by the radial impeller. Dispersion in that place was moved uniformly.

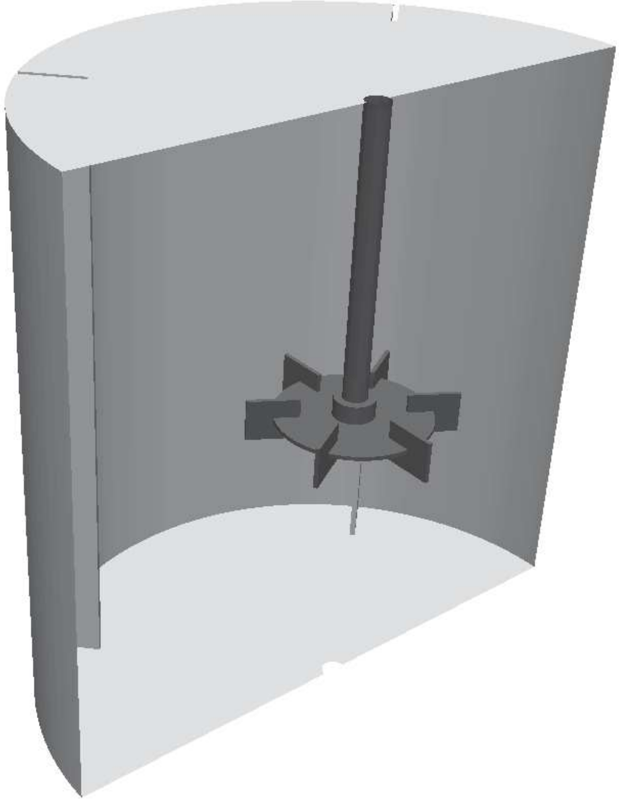

Fig.2. Sketch of the agitated system with Rushton turbine impeller.

2.2. IMAGE RECORDING AND IMAGE ANALYSIS

The image recording was realised by a high speed camera system SpeedSence MK III with frame rate lowered to 30 fps, shutter time 0.1 ms, full resolution 1280 x 1024 pixels. The frames were captured at the region under the impeller (see Fig. 1), where the particle velocities were below 1 m/s. The image resolution was 0.0118 mm/pixel in this configuration.

The objects evaluation was investigated by several approaches. At first the illumination of the captured area was tested. The light sheets as well as various light sources were used.

It was shown that the standard evaluation by a threshold value was not quite accurate. The results of this approach are shown in Fig. 3. The boundary of particles was not resolved properly and particles outside the focused plane were also evaluated. Hence, the new approach based on sharp boundary was developed.

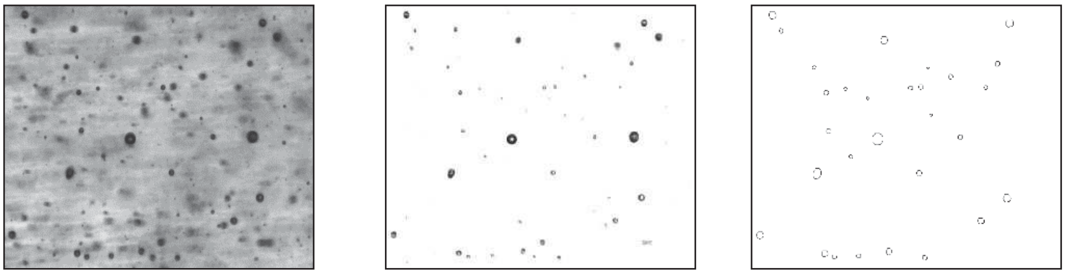

Fig.3. Evaluation by standard threshold.

The back lighting combined with new approach of image evaluation based on finding edges of spherical particles was found as the best final configuration. The three basic conditions had to be fulfilled:

a) Only different particles should be detected on the subsequent frames (that is why frame rate was only 30 fps).

b) The moving particles should have sharp boundary (very short shutter time is necessary as well as high-power light source)

c) Using precise spherical particles or provided minimal deformation of droplets.

The final approach for the image analysis was: 1) edges finding (high gradients), 2) threshold, 3) evaluation of particles based on circularity.

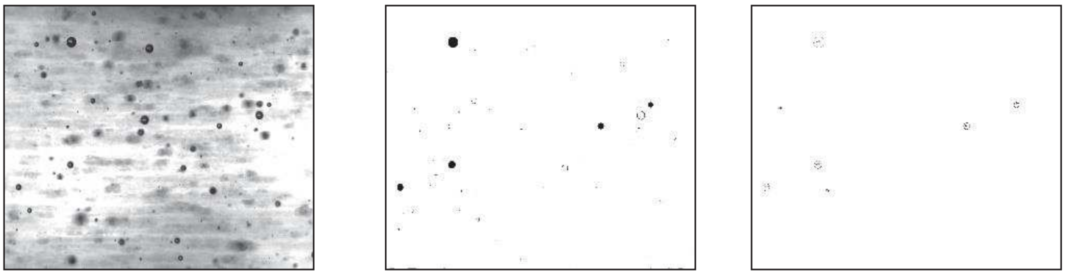

Fig.4. Evaluation by new approach using edges finding and particle circularity.

Although acquired number of particles unsubtly decreased the accuracy of evaluated diameters increased. It is also characterised with very high degree of reproducibility.

2.3. VERIFICATION ON SPHERICAL SOLID PARTICLES

Precise spherical mono-dispersed particles were used to verify the measuring method prospectively the obtained results were used for corrections. Two types of precise spherical particles were used. Stainless steel particles 0.635 ± 0.001 mm in diameter and plastic particles 3.00 ± 0.01 mm in diameter. The concentration was given by number of used particles approx. $2\cdot10^5$ (dispersion fraction $4\cdot10^{-4}$), or approx. $5\cdot10^3$ (dispersion fraction 10^{-3}), for the steel and plastic particles respectively. An example of steel particle size distribution result is depicted in Fig. 5. The results obtained for steel particles were 0.640 ±0.020 mm and for plastic particles 3.05 ±0.05 mm. Attained accuracy of particle size measurement by the proposed method for the steel and the plastic particles was under 2% and 3%, respectively.

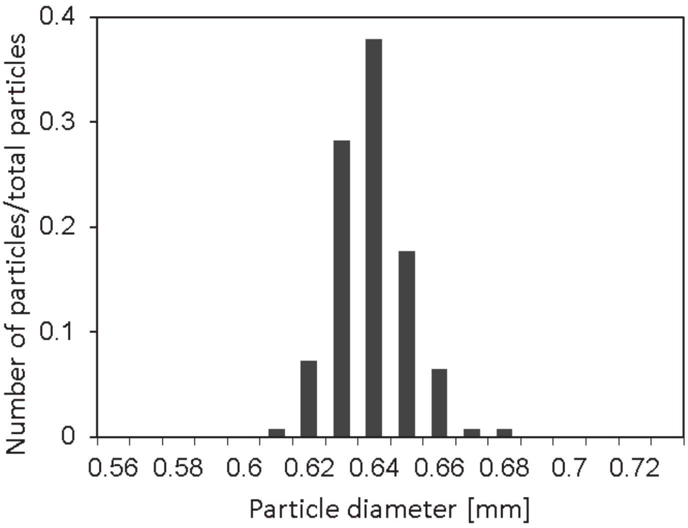

Fig.5. Particle size distribution of steel spheres evaluated by the proposed method.

3. RESULTS

The measurement based on image analysis is limited by a number of evaluated particles. Moreover, the proposed more accurate method this number even decreases. The dependency of mean Sauter droplet diameter on number of droplets was evaluated. The results for all impeller speeds are depicted in Fig. 6. There are visible changes approximately up to 1000 of evaluated droplets for all impeller speeds. The decrease of mean Sauter diameter with number of evaluated droplets for 150 rpm was evidently caused by not absolutely stable state at beginning of the measurement. The big jump in this dependency was induced by random appearing of several big droplets. It confirms opinion of unstable beginning state.

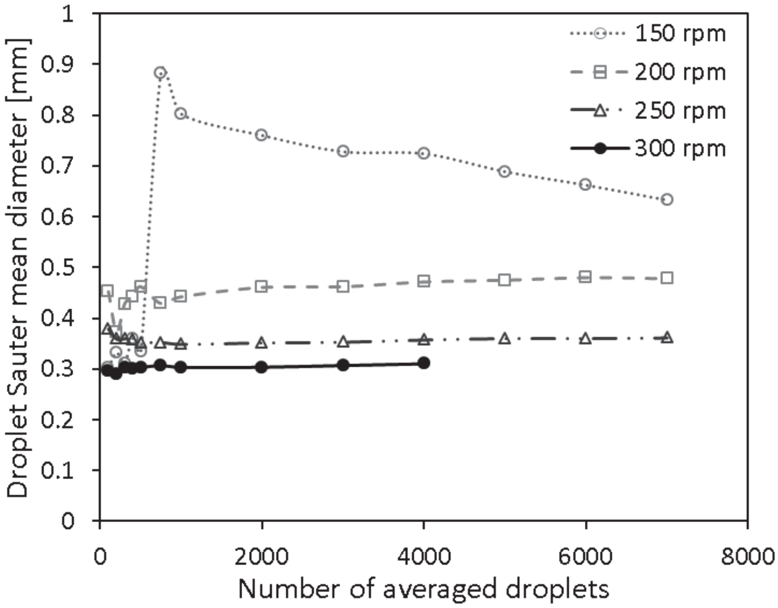

Fig.6. Evolution of droplet mean Sauter diameter with number of droplets. The droplet number was changing by number of evaluated images up to 1000.

The evolution of droplet cumulative distribution with increasing impeller speed is shown in Fig. 7. Depicted results indicate similar tendency as transient drop size distribution change obtained by Bak and Podgorska [2] for droplets in liquid-liquid system in an agitated vessel.

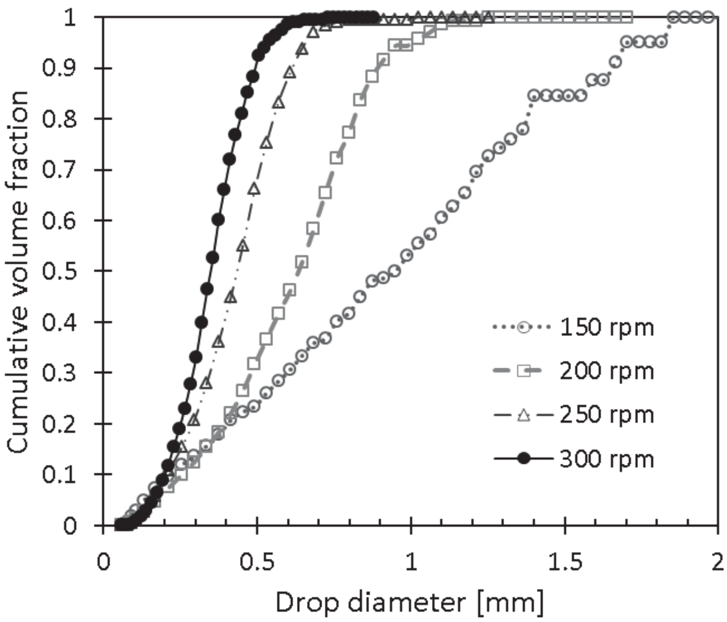

Fig.7. Evolution of drop size distribution with increasing impeller speed.

4. CONCLUSIONS AND DISCUSION

The method of in-situ measurement of particle size distribution based on image analysis was adjusted and evaluated for spherical particles. The method was improved with finding edges (high gradients in images) evaluation enhanced by circularity analysis. The high accuracy and good reproducibility allow applying this method for measurement of drop size distribution. Using a deeper analysis of particle shape than only circularity could bring more accurate results in future.

ACKNOWLEDGEMENTS

This research has been supported by Grant Agency of the Czech Republic project No. 16-20175S and RVO 67985874.

REFERENCES

1. Baldyga, J., Bourne, J.R., Pacek, A.W., Amanullah, A., and Nienow, A.W. (2001) Effects of agitation and scale-up on drop size in turbulent dispersions: allowance for intermittency, Chemical Engineering Science 56(11), volume 56, 3377–3385.
2. Bak, A., Podgorska, W. (2013). Drop breakage and coalescence in the toluene/water dispersions with dissolved surface active polymers PVA 88% and 98%. Chemical Engineering Research and Design, 91(11):2142-2155.
3. Ditl, P., Šulc, R. (2016) Dispersion kinetics modeling, Technical Transactions, iss.1, Mechanics iss. 1-M, 83-94.
4. Hong, P. O., Lee, J. M. (1983) Unsteady-state liquid–liquid dispersions in agitated vessels. Industrial and Engineering Chemistry, Process Design and Development, 1983, vol. 22, 130-135
5. Raikar, N.B., Bhatia, S.R., Malone, M.F., McClements, D.J., Almeida-Rivera, C., Bongers, P., Henson, M.A. (2010) Prediction of emulsion drop size distributions with population balance equation models of multiple drop breakage, Colloids and Surfaces A: Physicochem. Eng. Aspects, Vol. 361, pp. 96-108
6. Tang, H., Wrobel, L. C. (2005) Modelling the interfacial flow of two immiscible liquids in mixing processes, International Journal of Engineering Science 43(15-16), volume 43, 1234-1256.
7. Zhou, G., Kresta, S. M. (1998) Evolution of drop size distribution in liquid-liquid dispersions for various impellers, Chemical Engineering Science 53(11), volume 53, 2099-2113.