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Studies on Droplet Size Distribution of Oil-in-Water Emulsion in SMX Static Mixer

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ABSTRACT

Oil droplet size distribution of an emulsion produced by Sulzer Chemtech's static SMX static mixer under flow condition was experimentally studied and reported. The dispersed phase of vegetable oil-in-water (O/W) emulsion produced through static mixer by varying the concentration from 1 to 4 vol % oil in water, flowrate of dispersed and continuous phase and operating time. The effect of run time on oil drop sizes is characterized using the spectra obtained from the particle size analyser. The static mixer with 9 perpendicular elements made of teflon is stacked against each other had a void fraction of 0.93. The sauter mean diameter of oil droplet decreases from 8 µm to 4 µm with an increase in Reynolds number. The emulsion droplets of mean sauter diameter in the range 4.1 µm to 4.7 µm were produced by increasing the concentration of the dispersed phase from 1:100 to 1:25, within a span value of between 30 to 240 sec, at atmospheric pressure and room temperature. Performance equation for sauter mean oil droplet diameter is developed based on the experimental data has ±0.2 rms deviation.

Keywords: SMX Static mixer; Dispersed phase; Emulsion; Sauter mean oil droplet.

1. INTRODUCTION

Emulsions are metastable colloids of minimum two immiscible fluids, one being dispersed in the other by applying a shear force, in the presence of a surface active agent, an emulsifier like detergent. Nature of fluids, emulsifier, flow rates of dispersed and continuous phases and the concentration of the dispersed phase have critical effect on an emulsion (Caubet, Le Guer *et al.* 2010). Emulsion droplets exhibit the behavior of metastable colloids such as Brownian motion, reversible and irreversible phase transitions (Leal-Calderon, Bibette *et al.* 2007). Further, the emulsifier (eg. food additives) keeps the parts of an emulsion mixed together and helps to maintain the structure, quality, freshness, texture and sometimes prevent the growth of moulds in many foods (Friberg, Larsson *et al.* 2003). Nowadays nanoparticles are synthesized based on emulsion techniques. An attempt is made in the present study to prepare emulsion solution with the help of static mixers. Static mixers are motionless mixers (eg. Kenics mixer, SMX Sulzer mixer), employed inline in a once-through process or in fed batch systems or in a recycle loop where they supplement or even replace a conventional agitator (Berkman and Calabrese 1988), (Lemenand, Della Valle *et al.* 2003, Lemenand, Dupont *et al.* 2005, Lobry, Theron *et al.* 2011). Static mixer consumes less energy, offer better-controlled rate of dilution, enhanced heat transfer, provide homogenization of feed streams, occupies less space, narrow residence time distribution and reduced maintenance cost. (Middleman 1974, Montante, Coroneo *et al.* 2015, Soman 2016).

The SMX static mixer consists of multiple X-shaped repeated cross-bars in an axial or tangential direction and placed in a circular tube (Hirschberg, Koubek *et al.* 2009, Simpson, Dawson *et al.* 2015, Soman 2016) has wide applications includes dilution of hair sprays, moisturizers, waste water treatment and chemical processing. (Thakur, Vial *et al.* 2003), (Jones, Sotiropoulos *et al.* 2002).

The oil droplet size distribution in a static mixer is influenced by viscous shear force, frictional force which tends to deform the drops; inertial force and surface tension forces will stabilize the oil droplets. The relative magnitude of these forces on the droplet fragmentation is described by Reynolds number, Weber number and friction factor (Yamamoto, Kawasaki *et al.* 2007). Generally, the characteristic of oil-in-water (O/W) emulsion produced by a static mixer modeled by considering the behavior of sauter mean oil drop diameter (SMODD) (d_{32}) of liquid–liquid dispersions (Legrand, Morançais *et al.* 2001)

(Zhang and Xu 2016). Numerous mathematical models have been proposed for the prediction of SMODD in the literature (Lang, Drtina *et al.* 1995) (Kiss, Brenn *et al.* 2011). Usually, the turbulent flow regime has been examined and explained based on Kolmogorov's theory of isotropic turbulence, the most preferred droplet size correlation is proposed by (Middleman 1974):

$$\frac{d_{32}}{d} = C_1 f^{\frac{2}{5}} We^{-3/5} \quad (1)$$

$$We = \frac{du^2\rho}{\sigma} \quad (2)$$

d_{32} is a sauter mean diameter, d is the pipe diameter and C_1 is constant which would vary based on static mixer design. *We* is the Weber number, and *f* is the friction factor determines the pressure loss across the mixer,

$$f = \frac{d\Delta P}{2\rho u^2 L} \quad (3)$$

Where ΔP/L is the pressure drop per unit length in the static mixer. The friction factor for turbulent flow in pipes, $f \approx Re^{-1/4}$ the Eq.(1) reduces to

$$\frac{d_{32}}{d} = C_2 Re^{-1/10} We^{-3/5} \quad (4)$$

Where, C_2 is constant, applicable for the range given by (Haas 1987).

Spectral size distribution is done to characterize O/W emulsions produced by SMX static mixer. The concentration of the dispersed phase and flow rates of the dispersed and continuous phase in the turbulent flow regime were taken into consideration for droplet size distribution. The present study further demonstrates the effect of run time on oil droplet size distribution of SMODD ($\mathbf{d_{32}}$) measured by (Lobry, Theron *et al.* 2011).

$$\mathbf{d_{32}} = \frac{\sum iN_i d_i^3}{\sum iN_i d_i^2} \quad (5)$$

Where N_i is the number of drops with the diameter d_i. Subsequently, the pressure drop in the column was calculated using Burke-Plummer equation (Bird, Stewart *et al.* 2002).

$$\frac{P_o - P_L}{L} = \frac{7(\rho u_o^2)(1-\varepsilon)}{4(D_p)\varepsilon^3} \quad (6)$$

Where, $\frac{P_o - P_L}{L}$ is the pressure drop in the column, ρ is the density of the fluid, u_o is flow velocity, D_p is the particle diameter and ϵ is a void fraction. In the present study, the pressure loss per unit length was found to be in the range 11.55 Pa/m - 11.18 Pa/m. The factors which affect the stability of an emulsion were examined. The model equation was derived specifically to the static mixer valid for Weber's number and Reynolds number within the region of 30,000<We<3,00,000 and 1500<Re<2500, respectively.

2. EXPERIMENTAL METHODS

The vegetable oil used to produce emulsion was procured from Kachi Ghani, India and act as dispersed phase, the normal treated water as continuous phase. The physical properties of fluids (O/W) which form the emulsion at different concentrations have been determined as they have a profound impact on the emulsion quality. The specifications of the static mixer used in the study were given in Table 1.

Table 1 Specifications of the static mixer and fluid property

Geometric Characteristics		Values
Mixer Design		SMX
Mixer's alignment		Horizontal
Length(cm)		71.4
Breadth(cm)		8.4
Thickness(cm)		9.4
Sphericity of packing Materials		0.13
Void fraction		0.93
Number of elements		9
Void volume(m^3)		$5.64*10^{-3}$
Fluid Properties		
Fluid homogeneous	Density	Kinematic viscosity, 10^{-4} m^2/s
oil	920	65.03
water	1000	1.32
O/W, 1:100	992	1.76
O/W, 1:50	986	1.97
O/W, 1:25	971	2.14

2.1 Experimental Set-Up and Process Description

A schematic diagram of the experimental set up used in the present study is shown in Fig. 1, consists of a horizontal static mixer filled with SMX packing elements with two rotameters for adjusting the inlet flow connected with two inlet pumps. The O/W with the following concentration ratios were considered: 1:100, 1:50 and 1:25 (vol%:vol%). The various flow rates studied includes: 1250 LPH, 1000 LPH and a combined flow rates of oil and water at 750 LPH. The effect of o different runtimes is carried out in continuous process with complete recycle at 30, 60, 120, 180 and 240 seconds.

2.2 Continuous Process with Complete Recycle

In a continuous process, the oil present in tank 1 and water present in tank 4, both the fluids were pumped (2 and 5) through rotameters (3 and 6) to the inlet point of the static mixer (7) at a required flow rate (Fig. 1). Subsequently, fluids would pass through the static mixer packed with 9 SMX elements continuously and undergo flow division and radial mixing. At the end of each cycle, the emulsion formed would be collected in the tank. To achieve the continuous process with complete recycle, the emulsion tank was connected to the tank 4. Initially,

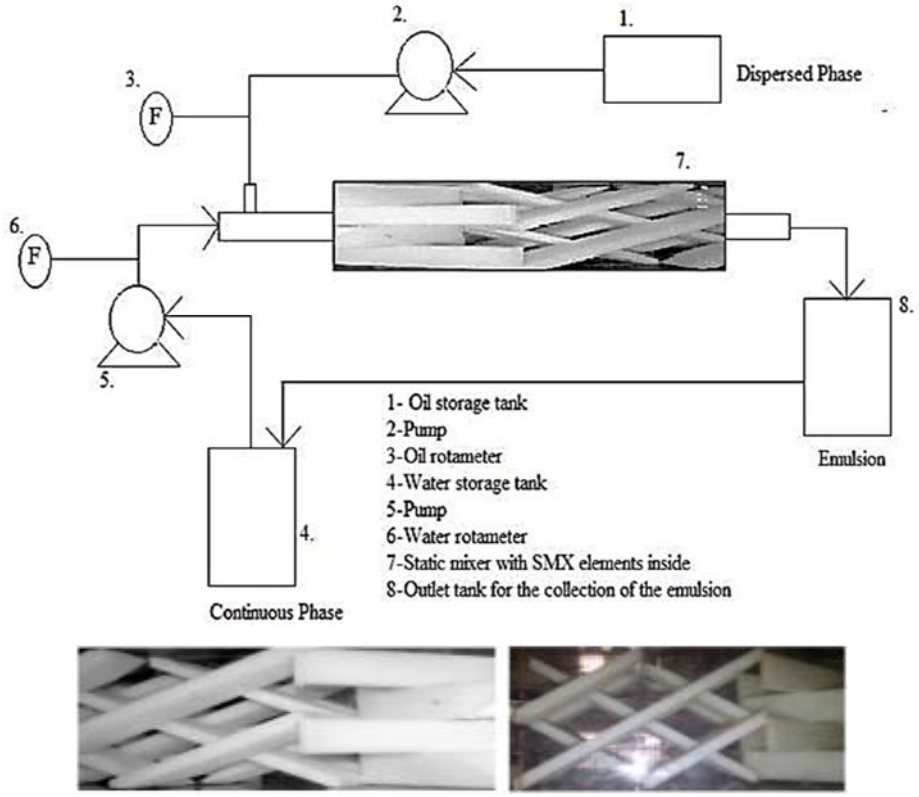


Fig. 1. (a) Schematic diagram of the experimental setup, (b) SMX static mixer.

the oil has flowed out from tank 1, pump 2 was closed and continued the operation with pump 5 in a single cycle. Overall it is represented in the following sequence: 4-5-6-7-8-4 (Fig. 1). Care must be taken to keep the total flow rate equal as before by adjusting the flow rate by rotameter. The samples were collected from the outlet pipe at the required time intervals (30, 60, 120,180 and 240 seconds) and used for analysis.

2.3 Batch Process

The batch process operation was the same as continuous with only difference being that the recycle line between tank 8 and tank 4 was blocked (Fig. 1). Therefore, mixer runs just once and no time dependence. In a single batch process cycle, only one sample has been collected and used for analysis. All the experiments were carried out in triplicates and the mean value reported.

3. ANALYTICAL METHODS

The density and viscosity of the fluids have been calculated by specific gravity flask and Redwood Viscometer. respectively. The depth of the cylinder and diameter of the heating coil of viscometer was 9 cm and 4.75 cm respectively. The interfacial area was calculated with the help of a tensiometer (K100, range: 1-2000 mN/m, resolution: ±0.001 mN/m,). The oil droplet size distribution of the emulsion was characterized by using a particle size analyzer (model: Malvern Mastersizer 2000, Malvern Instruments Limited, UK). In order to prevent erroneous results, the emulsion was analyzed immediately after the emulsification process. The experimental data plotted against volume (%) and particle size (µm) for further investigation.

4. RESULTS AND DISCUSSION

4.1 Effect of Concentration on Oil Droplet Size

The effect of concentration on the oil droplet size was carried out with Reynolds number in the range (1500<Re<2500). In the first series of emulsification studies, the concentration (vol%) of the dispersed phase was varied and flow rate in both the phases kept constant at 1250 LPH. Continuous process with complete recycle was performed for 4 minutes of run time and observed that as the concentration of O/W increases, the sauter mean diameter also increases. The oil droplet size reached in its stable form within 3 minutes of run time and after that no considerable variation was observed as shown in Fig. 2a (O/W at 1:100 emulsion), Fig. 2b (O/W at 1:50 emulsion) and Fig. 2c (O/W at 1:25 emulsion).

From graphs, observed a primary peak followed by a smaller secondary peak, may be attributed to the instant coalescence of some of the oil droplets. Similar experiments carried out by (Kiss, Brenn *et al.* 2011) reported that the mean droplet size is obtained between 160 - 190 µm, infers that the emulsion quality was influenced by dispersed phase

(polymer) concentration (0.06 - 0.14 g/L) with respect to a constant continuous phase (organic phase) corresponding to a flow velocity of 0.0755 m/s and dispersed phase hold-up of 0.14 vol% . Further observed that as the concentration of O/W reduces the plots become flat and the peaks become more pronounced as run time of the operation increases from 30 seconds to 3 minutes after which stability was attained. In fact, as the emulsion starts flowing through the static mixer more number of times, the bigger oil droplets keep on breaking into smaller oil droplets till they attained a stable size based on process parameters. The increase in volume percentage was observed corresponding to increase in run time, may be due to more number of oil droplets attains similar smaller sizes with respect to break down of bigger oil droplets.

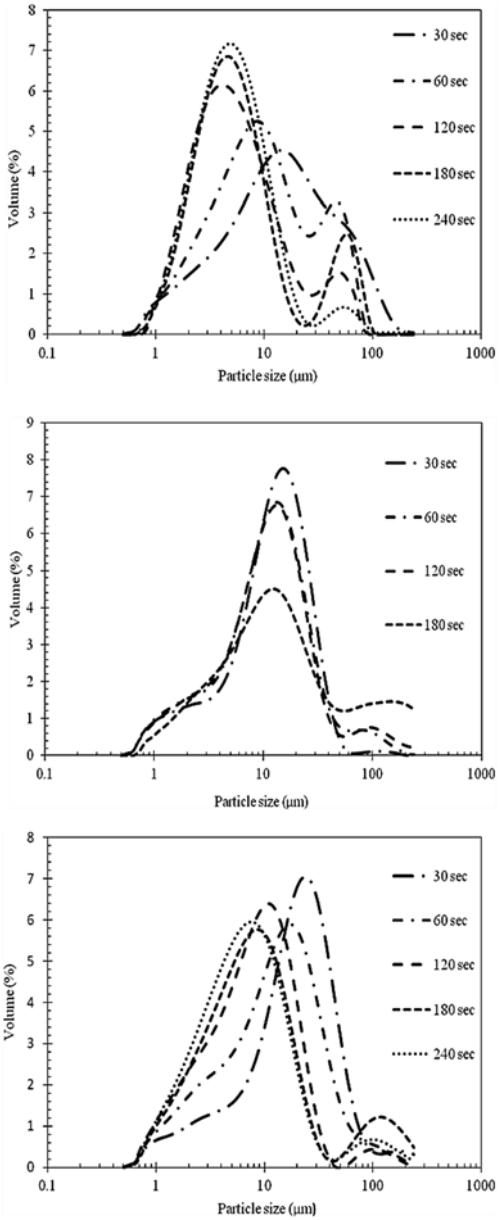


Fig. 2. Particle size distribution, Vol % (a) 1:100; (b) 1:50; (c) 1:25, at 1250 LPH flow rate.

4.2 Effect of Flow Rate on Oil Droplet Size

The effect of flow rate on the oil droplet size was performed in the turbulent flow regime at concentration of 1:100 O/W (vol%:vol%) emulsion. The above concentration was chosen due to the formation of smaller

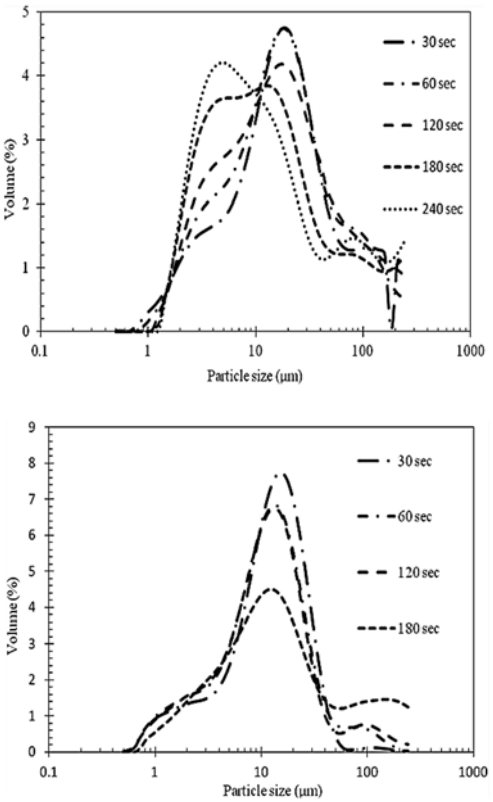


Fig. 3. Particle size distribution, Vol % 1:100 at different flowrate (a) 1000 LPH; (b) 750 LPH.

size of oil droplets as observed from the first series of experiments. The measured density (1032 Kg/m^3) and viscosity (1.7667 x 10^{-6} m^2/sec) of the emulsion is used for calculating the Reynolds number. Continuous process with complete recycle was carried out for 4 minutes of run time and observed that as flow rate increases, the sauter mean diameter decreases. The oil droplet size reached in its stable point nearly a run time of 3 minutes and after that, no considerable variation was observed as depicted in Fig. 2a (emulsion run at 1250 LPH), Fig. 3a (emulsion run at 1000 LPH) and Fig. 3b (emulsion run at 750 LPH). It is noteworthy that the peaks become more pronounced as the run time increases from 30 seconds to 3 minutes and a smaller secondary peak found, may be attributed to the instant coalescence of some of the oil droplets. Therefore, as emulsion starts flowing through the static mixer more number of times, the bigger oil droplets keep on breaking into smaller oil droplets until they attain a stable size. In this regard, a previous study reported that static mixers with 6 mm SMX mixer elements, the droplet size deviated from approximately100 to 250 µm, while for the mixer with 10 mm SMX elements, droplets with 90 to 220

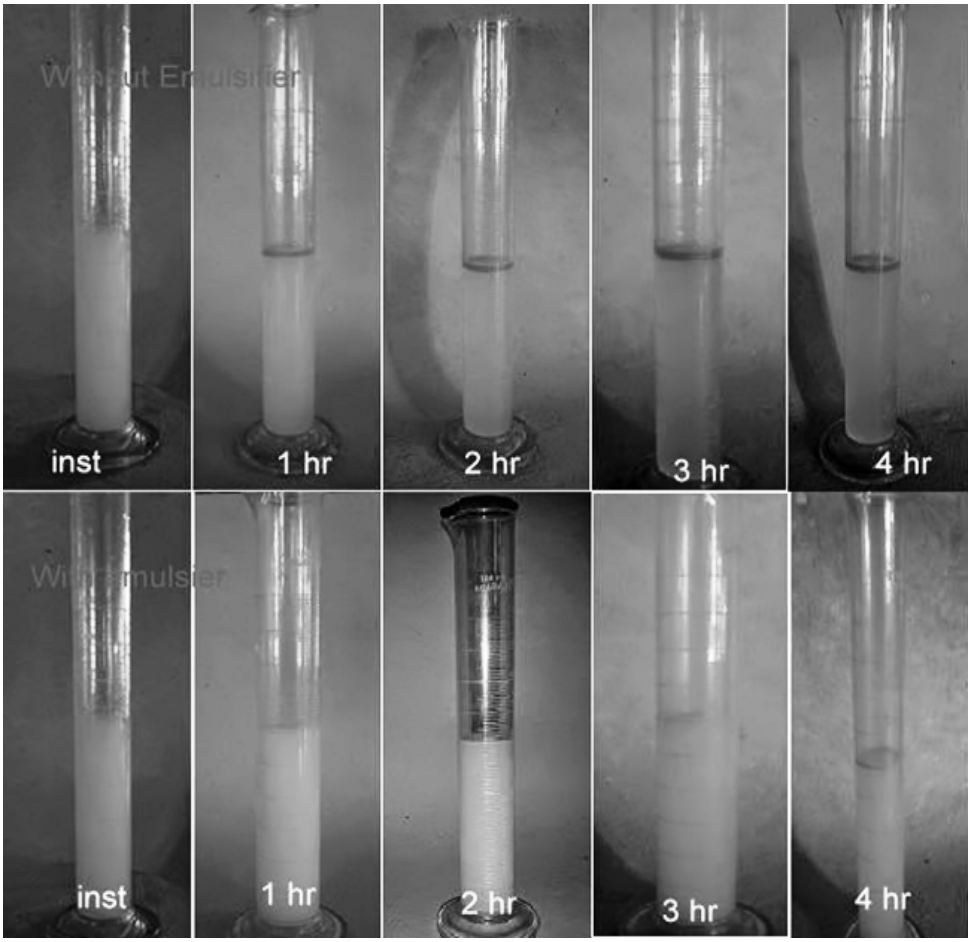


Fig. 4. Stability of the emulsion treated (A) without detergent and (B) with detergent.

μm were produced in the Reynolds number region (7 – 277) with a flow velocity ranges from 0.0426 to 0.2 m/s. Earlier studies carried out by SMX static mixer with 5 elements in the Reynolds number region (10.8 – 291) reported that as the flow rate increases, the sauter mean diameter decreases continuously from 87 μm to 44 μm (Legrand, Morançais *et al.* 2001). The overall increase in volume percentage of the oil droplets was observed and confirms that as run time increases a significant number of bigger size oil droplets break down into smaller oil droplets and attains similar sizes.

4.3 Effect of Creaming on the Stability of Emulsion

The stability of an emulsion is affected by the formation of creaming and coalescence of droplets. As oil is less dense than water, each drop tends to float upwards in the container. Therefore creaming process which allows the buoyant emulsion droplets tend to rise to the top (Robins 2000). In contrast, it is the same process as sedimentation, but in the opposite direction. In the present study, an effort was made to visually analyze two emulsion samples in the same time duration. Samples are prepared with an emulsifier (detergent- 0.5% by weight) and without an emulsifier. Photographs were taken every hour to study the creaming phenomenon in the emulsion (Fig. 4).

It was observed that an oil layer formed on top of the sample treated without detergent in the first hour of study and as time passes it progressively increases till the end of incubation (4^{th} hour) period. Whereas the sample treated with an emulsifier, a thin layer of oil was formed only at the end of incubation (4^{th} hour). It infers that the sample treated without emulsifier more susceptible to creaming phenomenon, hence, the obtained emulsion treated with detergent was more stable. However, creaming could be prevented by adding high molecular weight water soluble polymers (polysaccharides) and it acts as a thickener to increase the viscosity of the aqueous phase and creaming stability(Jeelani, Benoist *et al.* 2005).

4.4 Effect of Coalescing on the Stability of Emulsion

In general, industrial processes required to produce a dispersion of two immiscible liquids one inside another to generate large interfacial tension. The gravitational settlers used to ensure the dispersed phase separated from the surrounding continuous phases. The dispersed phase tends to move to the interface and after resting for some time will coalesce in their reservoir (Bozzano and Dente

2010). In the present study to prevent coalescence, an emulsifier was added (detergent) which coats the surface of each drop and prevents the droplets from coalescing. From Fig. 5a and Fig. 5b, the dotted lines represent the sample which has been analyzed in the particle size analyzer instantaneously after its exit from the static mixer after 180 seconds of run time whereas the continuous line represents the same sample that has been kept outside as an isolated system after its exit from the static mixer (run time 180 sec) for an hour and a half and then whose oil droplet size distribution has been measured. The SMODD of the sample that has been kept for 1.5 h was more than the sample whose oil droplet size has been measured instantaneously. We speculate that the increase in SMODD is due to the phenomenon of coalescence or may be due to creaming action. Sauter mean diameters as displayed by the particle size analyzer for different O/W concentrations (1:100 and 1:50).

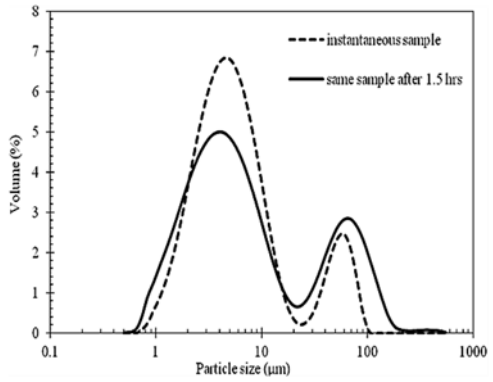
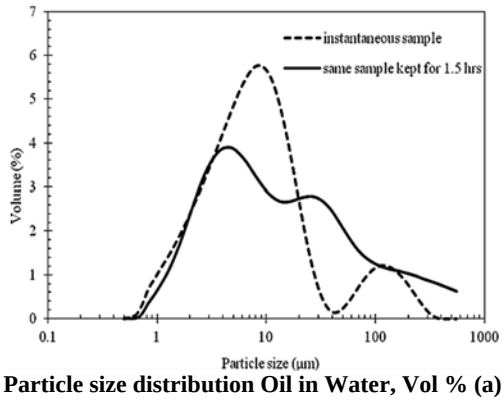


Fig. 5, Coalesce studies,



Particle size distribution Oil in Water, Vol % (a) 1:100; (b) 1:50, at 1250 LPH.

5. STATIC MIXER TURBULENT FLOW REGIME

To examine the Kolmogorov's theory of isotropic turbulence in the turbulent flow regime, the Middleman's relation (Eq. 1), using friction factor was calculated and obtained an average value of 0.235 (constant C_1) for static mixer used in the present study. Subsequently, Middleman's correlation (Eq. 4) using Reynold's number was studied in the turbulent flow regime and found out an average value of 0.249 (constant C_2) for static mixer. Generally, the obtained constants (C_1 and C_2) would vary based on static mixer design.

5.1 Model Equation for Estimation of Sauter Mean Diameter

An empirical relationship was derived for SMODD estimation by analyzing the continuous mode with complete recycle process. In the present study, the emulsion obtained from the static mixer has been assumed to be homogenous in nature. Overall the Sauter mean diameter for an emulsion formed by SMX static mixer depends on the following parameters:

$$d_{32} = f(u, d, L, \mu_d, \mu_c, \rho_d, \rho_c, \Phi, \sigma, t, t_{30}) \quad (7)$$

where, d_{32} is Sauter mean diameter of the oil droplets, u is flow velocity, d is pipe diameter, L is length of static mixer, μ_d, μ_c indicates viscosity of dispersed and continuous phases, ρ_d, ρ_c represents density of dispersed and continuous phase, Φ is Hold up = $u_d/(u_d+u_c)$, σ is interfacial tension between oil and water, t is time of operation and t_{30} indicates base time (30 sec). Subsequently, through dimensional analysis, the relationship is obtained as given below:

$$\frac{\mu_d}{\sqrt{\sigma\rho_d d_{32}}} = a\left(\frac{\mu_d}{\sqrt{\sigma\rho_d d}}\right)^b \left(\frac{u\mu_d}{\sigma}\right)^c \left(\frac{L}{d}\right)^d \left(\frac{\mu_d}{\mu_c}\right)^e \left(\frac{\rho_d}{\rho_c}\right)^f \left(\frac{t}{t_{30}}\right)^g \Phi^h \quad (8)$$

where, $\frac{\mu_d}{\sqrt{\sigma\rho_d d_{32}}}$, $\frac{\mu_d}{\sqrt{\sigma\rho_d d}}$ indicates Ohnesorge number i.e., $We^{0.5}/Re$, $\frac{u\mu_d}{\sigma}$ represents capillary number for the process, $\frac{L}{d}$ is ratio of characteristic length, $\frac{\mu_d}{\mu_c}$, $\frac{\rho_d}{\rho_c}$ indicates the ratio of viscosity and density of dispersed and continuous fluids respectively, $\frac{t}{t_{30}}$ is time of operation/base time, a, b, c, d, e, f, g and h are constants. Since $(L/d)^d$, $(\mu_d/\mu_c)^e$ and $(\rho_d/\rho_c)^f$ were constants and indicated as a new constant A and the relationship reduced to,

$$\frac{\mu_d}{\sqrt{\sigma\rho_d d_{32}}} = A\left(\frac{\mu_d}{\sqrt{\sigma\rho_d d}}\right)^B \left(\frac{u\mu_d}{\sigma}\right)^C \left(\frac{t}{t_{30}}\right)^D \Phi^E \quad (9)$$

In the above equation A, B, C, D and E are constants and the pattern of emulsification obtained in a single treatment could be readily be subjected to linear regression analysis and the combined data from all replicates used to analyze the data from any one replicate independently. Therefore, using linear regression analysis the following values were obtained: A = 0.296, B = 0.717, C = 0.186, D = -0.151 and E = 0.111. Hence, the final form of model equation derived for SMX static mixer valid within 30,000 < *We* < 3, 00,000 and 1500 < Re < 2500 is given by:

$$\frac{\mu_d}{\sqrt{\sigma\rho_d d_{32}}} = 0.296\left(\frac{\mu_d}{\sqrt{\sigma\rho_d d}}\right)^{0.717} \left(\frac{u\mu_d}{\sigma}\right)^{0.186} \left(\frac{t}{t_{30}}\right)^{-0.151} \Phi^{0.111} \quad (10)$$

6. CONCLUSIONS

The present laboratory-scale study demonstrates the oil droplet size distribution of an emulsion obtained from a SMX static mixer under defined process parameters in the turbulent flow regime. The SMODD was analyzed by varying the concentration of O/W and further determined the effect of flow rate by keeping the concentration constant. The SMODD was analyzed for different run times with particle size analyzer with a good level of accuracy. Under steady state operating condition, the sauter mean diameter of the emulsion was found to be increased corresponding to an increase in the concentration of the dispersed phase and found to decrease as the flow rate increases. The creaming and coalescence were found to be important factors which affect the stability of emulsion and determined that detergent could act as a good emulsifier under stable operating conditions. The experimental data analyzed with numerical model and constants were estimated by linear regression analysis. The kinetic constants were determined for sauter mean diameter of the oil droplet in the turbulent flow regime. Overall results obtained in this study allow developing a model equation prescribed for the static mixer which satisfactorily fit the experimental data in the formulated emulsion with a good degree of accuracy with RMS deviation of ±0.2.

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