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DROP SIZE AND DROP SIZE DISTRIBUTION IN A PULSED SIEVE-PLATE EXTRACTION COLUMN

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Abstract: Drop size and drop size distribution in a pulsed sieve-plate extraction column for the system acetic acid-water-ethyl acetate were studied. Different pulsation intensities were maintained for the same flow rates of dispersed and continuous phases. Mean drop size and distribution of drop size were significantly affected by the variation in pulsation intensity. Height of transfer units were observed to be decreased with the decrease in the drop diameter. Based on the results of experimentation a semi-empirical correlation for the estimation of mean drop diameter is established which proves to be in good agreement to the experimental findings.

***Keywords**:* Drop size distribution, mean drop size, pulsation intensity, semi-empirical correlation, height of transfer units

Introduction

Liquid-liquid extraction is an important separation technique that finds its applications in the chemical, petroleum, metallurgy, biotechnology, nuclear, and waste management related industries [1]. A large list of liquid extraction equipments has been developed over the years. All such equipments either require energy input or not. Energy input is an added advantage as it becomes another parameter that can control the degree of turbulence, drop size and dispersed phase hold-up which in turn measure the extraction efficiency [2] and hence the product quality. The pulsed sieve-plate extraction column is among those that require energy inputs. The energy addition is used to generate pulses and the rate of pulsation (frequency) and amplitude of pulsation measure the performance of the pulsed column.

Almost all the parameters that influence mass transfer and hydrodynamics of the pulsed sieve-plate columns are affected by the drop size formation [3]. Mean drop size and drop size distribution together determine the rates of transport phenomena [4]. Drop size not only influences the dispersed phase holdup and residence time of the dispersed phase but it also affects the allowable throughputs and along with dispersed phase holdup it controls the interfacial area required for necessary mass transfer [5]. Many investigators investigated the drop formation under different operating conditions and column geometries. Boyadzhiev and Spassov [6] used a column having 0.05 m diameter, and operational height of 1.0 m with 21 perforated plates and studied for the water-kerosene-CCl_4 system. They measured the drop size formation and developed a correlation for drop size measurement. They found the developed correlation within ± 20% agreement. Pietzsch and Pilhofer [3] presented a new method for the calculation of drop size and developed an equation that was applicable for the wide range of pulsation intensities. Garg and Pratt [7] used a column with 1 m equilibration

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height, with 7.245 m internal diameter and 16 sieve plates. They observed the effect of drop breakage and coalescence on the size distribution of the drops. Tung and Luecke [8] related mass transfer with drop diameter. Based on previous data Kumar and Hartland [9] developed a correlation for drop diameter that was applicable in mixer-settler, transition, and emulsion regimes Pietzsch and Blass [2] worked on a column of 0.072 m diameter and active height of 3.5 m with 70 plates and discussed the effect of pulsation intensity on the drop size and hold up. They also developed a model based on their own and previous data for the determination of hold-up and drop diameter. Lorenz *et al.* [10] experimented with two pulsed sieve plate columns with 0.072 m and 0.215 m diameter respectively with same active height of 4.3 m having 43 and 44 no. of sieve plates, respectively. They studied toluene-water, n-butyl acetate-water, and n-butanol-water systems. They related Sauter mean diameter and drop size distribution with the pulsation intensity and column height. They discovered that the drop size distribution became narrower and shifted to the smaller diameter with an increase in pulsation intensity. Mohanty and Vogelpohl [11] also worked on two pulsed sieve plate columns with same effective height, 4.3 m but different diameters 0.08 and 0.225 m. They used butyl acetate-water system without mass transfer and developed a hydrodynamic model based on drop population balance. Sreenivasulu *et al*. [12] studied n-butyric acid-water-kerosene system with mass transfer and water-kerosene system without mass transfer on a column with 0.043 m internal diameter and operational height 2 m. They found that the drop size distribution was not affected by the phasesí flow rates and the drop size distribution for dispersion and emulsion regimes followed log-normal distribution. They also developed a correlation for drop size measurement and found Sauter mean diameter was a function of -0.8 th power to the pulsation intensity. Luo *et al*. [13] modified the pulsed sieve plate extraction column having coalescence-dispersion plates between the sieve plates and studied the mean drop size and its distribution.

The present study is a continuation of our work [14] in which mass transfer performance in a pulsed sieve plate extractor was studied and an effort was made towards contribution for the sum of the data required for pulsed sieve plate extractors, on the basis of which such column can be better modeled and optimized for design purposes. In this study, the influence of pulsation intensity (the product of pulsation frequency and pulsation amplitude) on the drop size and drop size distribution is presented for the aqueous acetic acid solution with ethyl acetate as the solvent. Also the effect of drop size on the height of transfer unit, a measure of the mass transfer in the mass exchanger unit, is studied. The data collected are used to develop a correlation for the measurement of drop size. For the conditions of experimentation, no similar data have previously been found in the published literature.

Materials and Methods

Apparatus and chemicals

Experiments were carried out in a glass pulsed sieve plate extraction (PSE) column of 0.05 m internal diameter and 4.1 m active height. There were 80, 0.001 m thick and 0.05 m in diameter perforated stainless steel plates in the column. Each plate contained 36 circular holes of 0.003 m diameter laid on triangular pitch of 0.005 m. The distance between the two consecutive plates was 0.05 m. The plates were placed in the column in the form of a long cartridge. Separate pumps were used for the dispersed and continuous phases. These pumps were calibrated against different stroke lengths of the metering pumps. The pulsations were produced by mechanical piston type pump which was also calibrated for the rate of pulsations or frequency of pulsations. The details of the column geometry and apparatus assembly can be found elsewhere [14].

The chemicals, acetic acid and ethyl acetate, were of commercial grade and manufactured by International Petrochemicals. Water used was tap water. All these chemicals were used as received without further purification. The temperature during experimentation was kept between 21±1∞C. The drop diameters were measured by photographic method. A region of the 6th plate, counting form the bottom, was selected for photography and for calculating the mean drop diameter.

Analytic procedure

Initially, the whole column was filled with the solution of acetic acid in water which formed the continuous phase. The aqueous solution being heavier flowed from top to the bottom of the column. The dispersed and lighter phase, ethyl acetate, was then introduced from the bottom of the tower. The stroke length, the dispersed phase and the continuous phase flow rates were kept constant, while the pulsation frequency was varied. Each time the pulsation intensity was varied, sufficient time was provided for the drops to be fully developed. The drop diameters were measured by photographic method, and the region of the 6th plate counting form the bottom was selected for taking the photographs and calculating the mean drop diameter.

Results

The drop size in the pulsed perforated column depends upon the drops breakage to smaller drops and drops coalescence to bigger drops. The breaking of drops may be caused by turbulence produced by the pulses, the drops flow through the sieve holes, and the hitting of drops with the wall of the columns and the sieve plates [12]. When drops strike each other coalescence of the drops is expected. The breakage and coalescence of the drops produces a range of drop sizes, thus a mean value of drop sizes and the way these drop sizes are distributed is meaningful in such circumstances. The variation of mean drop diameter with pulsation intensity, Af (mm/s) for constant dispersed phase superficial velocity, u_d (mm/s) and continuous phase velocity, u_c (mm/s) is shown in Fig. 1. Pulsation intensity is defined as the product of pulsation amplitude, A (mm) and pulsation frequency, f (s^{-1}). The mean diameter is taken as the Sauter mean or volume-surface mean diameter, d_{32} defined by the expression [6].

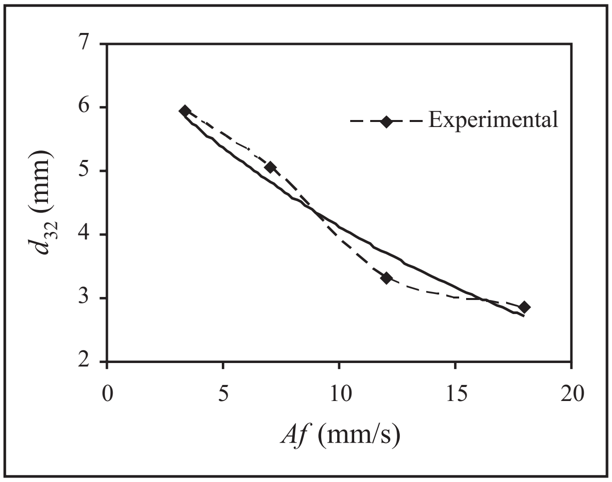

Fig. 1. Influence of pulsation intensity on Sauter mean drop diameter.

$$d_{32} = \sum_i^n d_i^3 \Big/ \sum_i^n d_i^2 \qquad (1)$$

where d_{32} (mm) is the Sauter or volume-surface mean diameter, d_i (mm) is the ith drop diameter, and n and i *represent* nth and ith values respectively.

The mean drop diameter decreases with an increase in pulsation intensity as it is expected because of more turbulence generated at the higher pulsation frequency. This observation is in agreement with other investigators [10,12]. Also, it is observed that this decrease in drop di-

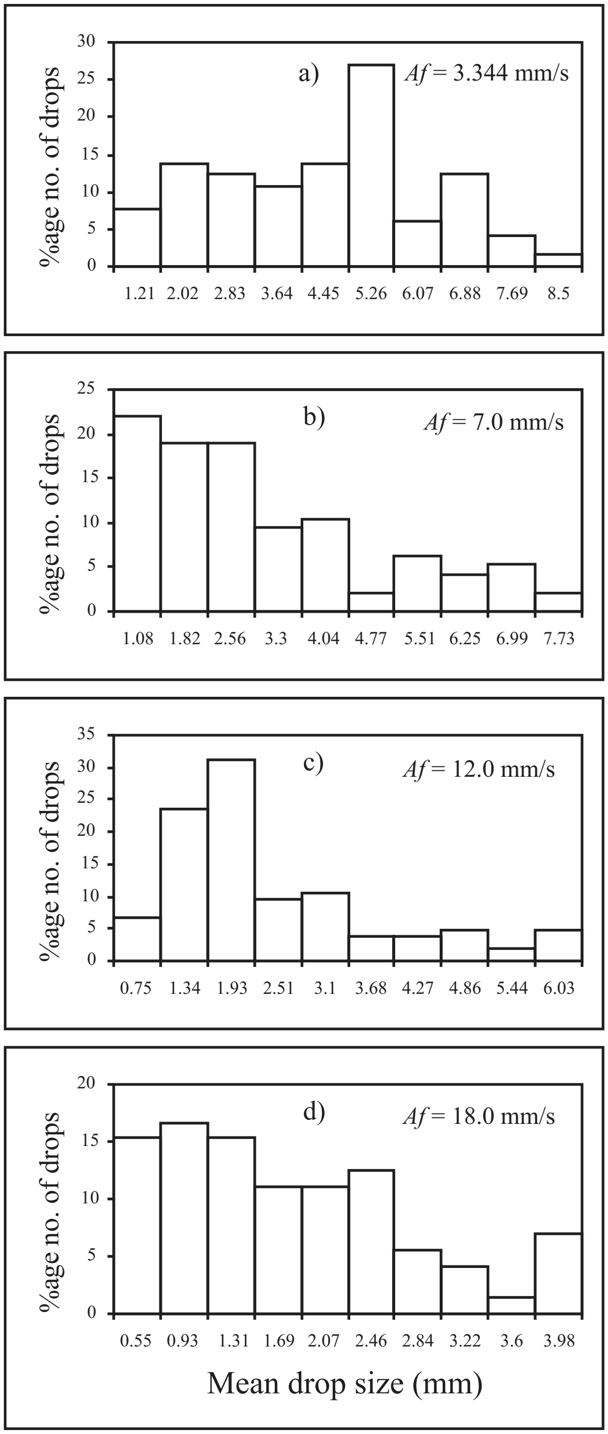

Fig. 2. (a), (b), (c), and (d) Drop size distribution at 6th plate counting from the bottom; u_c=3.022 mm/s and u_d = 3.022 mm/s.

ameter is more pronounced at the lower pulsation intensities and slowly the change diminishes with increase in pulsation intensities. Drop size distributions against different pulsation intensities are shown as histograms in Fig. 2. The drop size spread or range (the difference between the maximum drop size and minimum drop size) reduces with the increase in the pulsation intensity, which means deviation in drop sizes is decreasing and an increased pulsation intensity system tends to produce more uniform product distribution. The same fact is also deducible from Fig. 3 in which standard deviation (σ) is plotted against pulsation intensity. At the lowest value of the pulse intensity, namely 3.334 mm/s, the distribution is moderately skewed to the left i.e. skewness is negative; however at higher pulse velocities the skewness is shifted to the right and positively skewed. This explains that the concentration of the small droplet mass is generally increasing with the increase in the pulsation intensity.

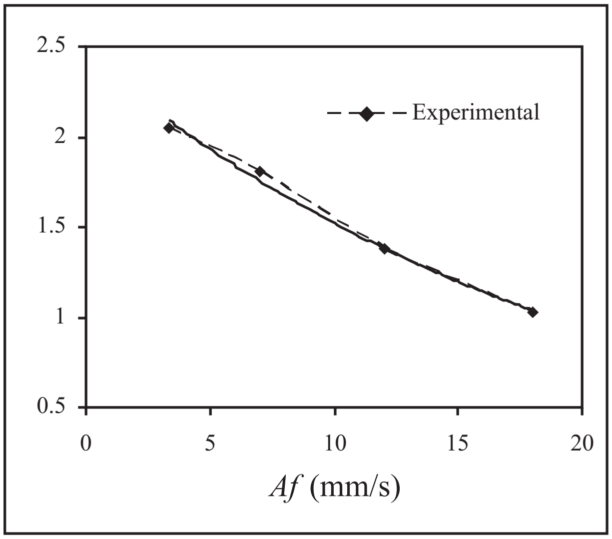

Fig. 3. Measurement of standard deviation for drop sizes with variation in pulsation intensity.

Fig. 4 shows a comparison between the Sauter mean diameters calculated from Eq. 1 with the Sauter mean diameters based on log-normal distribution [4]. The Sauter mean diameter based on log-normal distribution is obtained from the Eq. 2 [4]

$$\ln\,(d_{32}) = \ln\,(d_{ng}) + 2.5 s_g^2 \qquad (2)$$

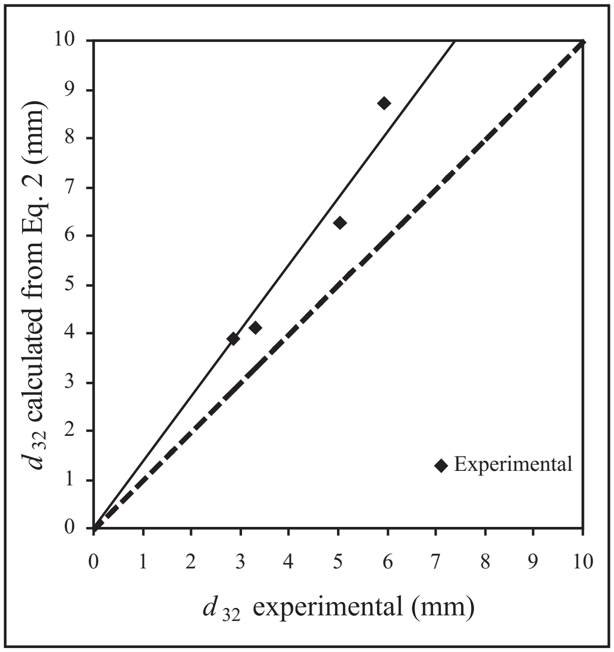

Fig. 4. Comparison of experimental d_{32} with calculated from Eq. 2.

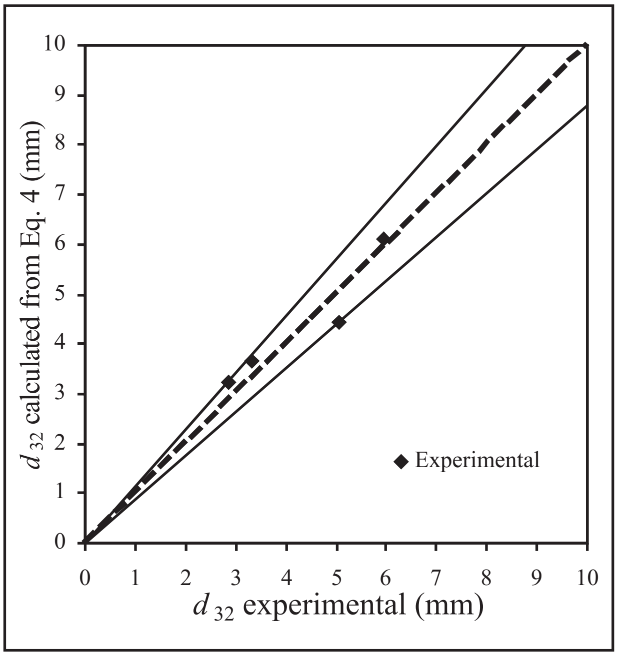

Fig. 5. Comparison of experimental d_{32} with calculated from Eq. 4.

where d_{ng} (mm) is the number geometric mean diameter and s_g is the geometric standard deviation. The d_{ng} is taken as the geometric mean of the set of diameters and s_g is obtained from the Eq. 3

$$\ln s_g = \exp\left(\sqrt{\frac{1}{n}\cdot\sum^{n}(\ln d_i - \ln d_{ng})^2}\right) \tag{3}$$

All the parameters of the Eq. 3 are defined above. There is an average 27% deviation of the data from the log-normal distribution.

Keeping in view the Kolmogorovís theory and the experimental measurements of this study, a semi-empirical correlation as shown in Eq. 4 is developed and the comparison of experimentally measured diameters and that measured from Eq. 4 is shown in the Fig. 5.

$$d_{32} = 11.3 \times (Af)^{-0.463} \tag{4}$$

The correlation predicts the drop diameter very well and maximum error does not exceed beyond ± 9.1%; however this equation should be used with care when applying to other systems with different column dimensions. The correlation developed is in agreement with the other workers, who found the exponent of the product, *Af* varied between ñ0.3 to ñ1.2 [3, 9]. Fig. 6 shows a relationship between the mean drop diameter and the height of the transfer unit, *HTU* (mm). The calculation procedures for the measurement

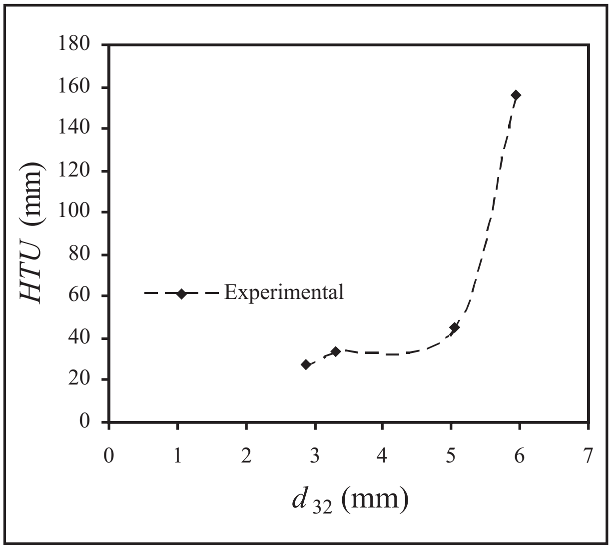

Fig. 6. Variation of *HTU* with mean drop diameter, d_{32}.

of *HTU* from the experimental observations are provided in our previous work on pulsed sieve plate extraction column [14]. It is observed, as expected, that the height of transfer unit decreases significantly with the decrease in drop size and hence the mass transfer is enhanced with the decrease in the drop diameter. However, the change is significant only in the larger drop size region, and with further decrease in drop size the change in *HTU* is not very pronounced.

Conclusion

The drop size decreases with an increase in pulsation intensity, whereas the decrease is less pronounced at the higher pulsation intensities. The difference between the maximum and minimum mean drop sizes decreases with the increase in pulsation intensities and the concentration of smaller drops increases. The height of the transfer unit, a measure of mass transfer, decreases with decrease in drop diameter. These results suggest good mass transfer efficiency at higher pulsation intensities. However, higher pulsation intensities are measures of greater energy consumption. Thus the column conditions are to be sought for the best efficiencies at minimum energy costs. The semi-empirical equation developed on the basis of Kolmogorovís theory is in good agreement with the experimental results. It is found that the developed correlation provides the results within ± 9.1% of the experimental value.

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