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Drop size distribution and mean drop size in a pulsed packed extraction column

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ABSTRACT

Drop size distribution and mean drop size are used for calculation of interfacial area available for mass transfer. In this study, the drop size distribution and Sauter mean drop diameter (d_{32}) have been investigated using three different liquid systems in the absence of mass transfer in a pilot plant pulsed packed column. The drop size was measured at four different points along the active column height. Three operating variables have been studied including the pulse intensity (*af*) and flow rates of both liquid phases. The effect of liquid properties and height of the active column were also investigated. A combination of the pulse intensity and interfacial tension had the largest effect on the drop size distribution while none of the flow rates were of significance. The height of the column played an important role at the bottom of the active column, but the associated effect was reduced with increase of the height. Finally, a normal probability function of number density was proposed for prediction of the drop size distribution with an Average Absolute Relative Error (AARE) of 8.8% for their optimized constant. Furthermore, two correlations were presented involving height or flow rates of the two phases along with operating variables and physical properties of the liquids. These correlations had AARE values of about 8.5 and 7.8%, respectively.

Keywords: Pulsed packed column; Sauter mean drop diameter; Drop size distribution; Pulse intensity

1. Introduction

Liquid–liquid extraction is one of the useful techniques applied in various separation technologies including chemical, petroleum, food, hydrometallurgy, nuclear, and many other applications (Tsouris et al., 1994). In design of an extraction column, height and diameter of the column have to be specified for a desired mass transfer and allowable flow rates in all phases. Drop size has an important effect on the dispersed phase holdup and the maximum throughput. Furthermore, the interfacial area of mass transfer can be determined using drop size and holdup together (Kumar and Hartland, 1996). The knowledge of drop size distribution is a key parameter to scaleup of the system (Maaß et al., 2011), and obviously contains more information than a mean drop size alone because different drop size distributions might have the same mean drop size while having different interfacial areas.

Pulse intensity can affect the drop size: the drop breakup is enhanced with increasing pulse intensity due to enhaced collisions between the dispersed liquid drops and the internal wall (Ousmane et al., 2011). The drop size distribution curve becomes wider when *af* is lower. An increase of *af*, on the contrary, makes the curve trends to represent a uniform size (Jones, 1962). Some investigations have been carried out on the effect of the column geometry in pulsed extraction columns (Spaay et al., 1971; Lorenz et al., 1990; Yadav and Patwardhan, 2008). The column diameter has almost no effect on the drop size, but the first 2–3 sieve plates affect the drop size: thay bring about a breakage of the drops while in the rest of the column the drop size varies only slightly (Lorenz et al., 1990).

The volumetric flow rates of the continuous and dispersed phases also slightly change the drop size (Jones, 1962) especially when the phase velocities are too smaller than *af* (Boyadzhiev and Spassov, 1982). Yadav and Patwardhan (2008) presented a review on the drop size in the pulsed sieve plate columns. Usman et al. (2009) investigated the effect of the pulse intensity, and the dispersed phase and continuous phase velocities on the Sauter mean diameter. Drop size

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Nomenclature

af	pulse intensity (m/s)
a_p	specific surface area (m^2/m^3)
d_{32}	Sauter mean diameter (m)
d_{10}	arithmetic or number mean diameter (m)
d_i	drop diameter (mm)
g	acceleration due to gravity (=9.81 m/s^2)
h	height at each point of active column (m)
H_0	the overall height of active column (m)
n_i	number of droplets of mean diameter d_i
P	probability of number density
Q	volumetric flow rate (m^3/s)

Subscript

c	continuous phase
d	dispersed phase
n	normal probability density function
ln	log-normal probability density function

Greek letters

α	constant parameter of probability of density function
β	constant parameter of probability of density function
ε	void fraction
μ	viscosity (N s/m^2)
ρ	density (kg/m^3)
$\Delta\rho$	density difference between two phases (kg/m^3)
σ	interfacial tension between two phases (N/m)

was a function of the flow regime and, at the end of mixer-settler regime, superficial velocity strongly affected the drop size. Pietzsch and Pilhofer (1984) modeled the drop size with the balance between forces acted on the drop where the stable drop size was calculated with buoyancy, inertial, and drag forces as destroyers and the interfacial tension force as a stabilizer of the drop.

There are few works implemented on the pulsed packed columns, more specifically the drop size. Spaay et al. (1971) presented a semi-empirical correlation on the Sauter drop diameter. They considered the effects of liquid properties, *af*, bed porosity, and specific surface area of the packing. According to their correlation, the latter parameter has a little influence on the drop size while the bed porosity has a strong effect in a manner that a decrease in the bed porosity results in a smaller drop size. The flow rates of the continuous and dispersed phases were considered to be negligible in this correlation; alternately, *af* had an important role in the magnitude of the drop diameter. In order to develop appropriate procedures for design of pulsed packed columns, it is vital to acquire the knowledge of average drop size and its distribution.

In this work, the Sauter drop size and drop size distribution behavior have been explored in a pulsed packed extraction column. The effects of operating variables (*af* and flow rates of the continuous and dispersed phases) and the height of active column on the drop size distribution were examined. In addition, empirical correlations have been proposed for the Sauter mean drop diameter and drop size distribution as a function of operating conditions, physical properties of the liquid systems and active column height. The drop size distribution and the mean drop size thus estimated are helpful in calculation of the interfacial area for mass transfer in this system.

2. Experimental

2.1. *Description of equipment*

A glass pilot plant pulsed packed column with 1.4 m height of packing and 0.055 m internal diameter was used in these experiments. A settler of 0.073 m diameter at both ends of the column was employed to separate the two liquid phases. The pulse was introduced to the liquid level with a reversible mechanical piston. Amplitude and frequency were adjusted using setting movement amplitude of the piston and a digital controller, respectively. The interface location of the two phases at the top of the column was automatically controlled by an optical sensor. A schematic flow diagram of the experimental equipment is shown in Fig. 1 that this type of column works with the countercurrent flow. The column was filled with Raschig rings of 0.625 cm size and 60% void fraction.

2.2. *Liquid systems*

Three liquid–liquid systems were selected to cover a wide range of interfacial tension values according to the recommendations of the European Federation of Chemical Engineering (Míšek et al., 1985). The systems were kerosene–water, toluene–water, and butyl acetate–water. The physical properties of these systems are listed in Table 1. The technical grade solvents of at least 99.5 wt% purity were used as the dispersed phase. All experiments were carried out at the $20 \pm 1\,^{\circ}C$.

2.3. *Droplet size measurements*

Drop sizes were determined by taking a digital photo of the column contents using a Nikon D5000 digital camera and comparing the drop dimensions with the known size of the packing as a reference. The drop size was measured at four hights of the active column ($h = 10 \pm 10$, 50 ± 10, 90 ± 10, and 130 ± 10 cm that the zero point is considered to be the bottom of the active column).

To determine the size of the drops, the recorded photos were analyzed by AutoCAD software. More than 1500 drops were analyzed for each experimental condition at four points of the active column to guarantee the statistical significance of the determined Sauter mean drop diameter. The Sauter mean drop diameter, d_{32}, was calculated at the exerted experimental conditions as follows:

$$d_{32} = \frac{\sum_{i=1}^{n} n_i d_i^3}{\sum_{i=1}^{n} n_i d_i^2} \tag{1}$$

where n_i is the number of droplets of mean diameter d_i within a narrow size range i.

3. Results and discussion

In an apparatus with an agitator, drops frequently run into eddies. The resulting collisions are responsible for the drops breakup into two or several smaller ones (Schlauch, 2007). In contrast, the drop breakage in pulsed packed columns should be related to the impingement of the drop on the packing wall. Indeed, the exerted force from the packing wall against