



## Drop behavior in a pilot plant Oldshue–Rushton extraction column for three various liquid–liquid systems



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### ABSTRACT

Drop size distribution and Sauter mean drop diameters have been evaluated in an Oldshue–Rushton pilot plant extractor for three various liquid–liquid systems as a function of phase flow rates and intensity of agitation. The Sauter mean drop diameter and drop size distribution were found to depend largely on the speed of agitation, physical properties of liquid systems and direction of mass transfer, but, only partially dependent on the phase flow rates. The experimental data are then fitted by means of a probability density function, namely normal and log-normal function. The mathematical approach is used to determine the constant parameters in these functions and to provide the fit of the experimental data with them. Moreover, an empirical correlation is proposed for estimation of the Sauter mean drop diameter in terms of operating variables, column geometry and physical properties. Good agreement between the prediction and experimental results and the data in the literature was achieved for all investigated operating conditions.

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### 1. Introduction

Solvent extraction is one of the most important processes used in different chemical systems such as food industry, petroleum, and hydrometallurgy. To create a large interfacial area and improve the efficiency of extraction columns, the power required can be supplied by pulsation or stirring. A number of stirred extraction columns such as rotating disc contactor (RDC), Kühni, mixer-settler extraction and Oldshue–Rushton columns were used for the purpose of increasing mass transfer coefficients [1].

The Oldshue–Rushton columns have been widely used in the chemical processes such as solvent extraction. The main industrial application is for low and medium viscosity fluids, generally ranging up to 500 cp and having density differences of at least 0.05. Also, solids may be suspended in one or both phases. In addition, the Oldshue–Rushton column can be used in a co-current mode for any single or multiple-phase continuous co-current contacting operation such as leaching, reacting, bleaching, and many other types of co-current extraction systems [2].

The unit is neither a compartmental or truly continuously differential device, but has some properties of both. The motion of

droplets between compartments in these columns is therefore dependent upon the droplet size, the physical properties of the phases, the phase flow rates and the agitator speed [3,4].

The diameter and height of the extraction columns are very important in the design of these columns to achieve the appropriate mass transfer and flow rates [5]. Maximum throughput and dispersed phase holdup can be dramatically affected by the drop size. In addition, the hold up and drop size are considered to determine mass transfer interfacial area. Indeed, the drop size distribution plays a key role in scaling up of an extraction column. This is because the same mean drop size can be obtained from various drop size distributions which have different interfacial areas [3,6].

A variation in drop size distribution produces a hold-up profile exhibiting a maximum value along the column depending on the operating conditions, which can be used to determine the column flooding [7].

In the absence of agitation, the drop size is determined by the ratio of interfacial tension to buoyancy forces. The effect of external agitation is accounted for in terms of the power dissipation per unit mass [8].

The power input to the agitator is thus transferred to the kinetic, surface, potential and heat energy of the droplets. Design of rotary agitated contactors may be classified in terms of the rate of energy input per unit volume of the continuous phase [8].

Chen and Middleman studied the drop size distribution in agitated liquid–liquid systems [9], and Mlynek et al. analyzed the

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## Nomenclature

AARE	average absolute relative error (-)	$Re_R$	rotor Reynolds number
$C_{II}$	constant parameter in Eq. (11) (-)	$V$	superficial velocity (m/s)
$C_{\psi}$	constant parameter in Eq. (11) (-)	$V_i$	impeller tip velocity (m/s)
$C_{\Omega}$	constant parameter in Eq. (11) (-)	$V_k$	characteristic velocity (m/s)
$d_{32}$	Sauter mean drop diameter (m)		
$D_c$	column diameter (m)	<i>Greek</i>	
$d_e$	equivalent diameter (m)	$\sigma$	interfacial tension (N/m)
$d_R$	rotor diameter (m)	$\alpha$	constant parameter of probability of density function
$e$	cross sectional area (-)	$\beta$	constant parameter of probability of density function
$g$	acceleration due to gravity (m/s <sup>2</sup> )	$\Delta\rho$	density difference between phases (kg/m <sup>3</sup> )
$g(d)$	breakage frequency (1/s)	$\varepsilon$	power dissipated per unit mass (m <sup>2</sup> s <sup>-3</sup> )
$H$	effective height of the column (m)	$\lambda$	coalescence efficiency (1/s)
$h$	collision frequency (1/s)	$\mu$	viscosity (Pa s)
$h_c$	compartment height (m)	$\rho$	density (kg/m <sup>3</sup> )
$k_{1break}$	constant parameter in Eq. (20)	$\rho_m$	density of dispersion (kg/m <sup>3</sup> )
$k_{1coal}$	constant parameter in Eq. (22)	$\varphi$	dispersed phase holdup (-)
$k_{2break}$	constant parameter in Eq. (20)		
$k_{2coal}$	constant parameter in Eq. (23)	<i>Subscripts</i>	
$N$	rotor speed (1/s)	$c$	continuous phase
$n_i$	number of droplets of mean diameter $d_i$ (-)	$d$	dispersed phase
$N_p$	power number	$ln$	log-normal probability density function
$P$	power input per compartment (W)	$n$	normal probability density function
$p$	probability of number density (-)		
$Q$	flow rate of the continuous or dispersed phase (m <sup>3</sup> /s)		
$Re$	Reynolds		

drop size distributions of the liquid–liquid dispersion in a stirred vessel at high phase ratio [10].

The hydrodynamic mechanism of splitting in the dispersion process investigated by Hinze [11], it was observed that the difference in density between the dispersed and the continuous phase has an important effect on the way in which break up occurs.

Tsouris et al. [12] studied the drop size distributions in the Oldshue–Rushton column and it was observed that the ratio of the characteristic flow to the impeller tip velocity had the strong effect on the Sauter mean drop diameters.

The effects of the dispersed phase distributor, column geometry, disc speed, physical properties, and disperse, continuous and total flow rates on drop sizes in an extractor RDC column were studied by Al-Rahawi. Two equations were derived to predict the average mean drop diameter in the RDC column. The effects of the dispersed phase distributor, disc speed and physical properties were found to be significant and dominated the drop sizes. Meanwhile, the effect of column size was insignificant [13].

The effect of the dispersed phase holdup, rotor speed, and column height on the dispersed phase mean drop size  $d_{32}$  was quantified by Kirou et al. [14] who suggested that  $d_{32} \sim N^{-1.26}$ . The Sauter mean diameter decreased everywhere in the contactor with increasing agitation speed. This behavior was observed for holdup values below 20%.

Al-Aswad et al., investigated the effect of acetone transfer between Clairsol350 (organic phase) and water (aqueous phase) on the Sauter mean drop diameter with experimental work in rotating disc contactor, the smaller drop size was observed from the continuous phase to the dispersed phase [15].

Komasawa and Ingham [16] studied the effect of agitation speed and mass transfer on the Sauter mean diameter in the Oldshue–Rushton contactor. They observed that  $d_{32}$  decreases as the rotor speed increases up to a certain agitation level, above which drop breakage and coalescence become equally important and  $d_{32}$  remains unchanged.

Experimental studies in agitated columns have shown that drop size distributions are broad in the first stages, becoming narrower

and shifting toward smaller drop sizes along the column until a steady-state distribution is obtained [12,17,18].

Although the Oldshue–Rushton column has been used effectively for a number of separation processes, there is scanty information in the literature on the performance of this type of extractor. So, the development of a new correlation for drop size with reliable experimental data in the Oldshue–Rushton column for different systems can be helpful for determination of system performance and scale-up of extractor.

The aim of this research work is to acquire a sufficient understanding of drop behavior in an Oldshue–Rushton pilot plant column in order to provide basic data required for column design. Drop size distribution has been studied by changing the operating parameters such as disperse and continuous phase flow rates and agitation speed. The effect of solute transfer on the drop size in the pilot plant column was investigated. Three chemical systems, including toluene–water, n-butyl acetate–water, n-butanol–water were chosen to study the effect of the interfacial tension on the droplet size distributions. Furthermore, empirical correlations were derived for the determination of drop size distribution and Sauter mean drop size. The predicted correlation is very useful for estimation of the mass transfer interfacial area.

## 2. Experimental procedure

### 2.1. Description of pilot plant extractor

A pilot plant Oldshue–Rushton column contactor, 113 mm in diameter, a working height of 700 mm with nine compartments, was used in the experiments. The stirrer is located in the center of each compartment with 6-blade impellers of 50 mm diameter. All internal parts of the column and the blade impellers were made of AISI 316 stainless steel. The blade impellers are mounted on a shaft, which is driven by an electric motor via a variable speed gear box. The speed of rotation could be measured and indicated by an electronic digital rotation speed meter adjusted by regulating the DC voltage of the source. The specific column geometries studied

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