

Prediction of Dispersed Phase Holdup in Scheibel Extraction Columns by a New Correlation

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Abstract

In this study, the effect of operating parameters on dispersed phase holdup in liquid-liquid extraction process has been investigated. Three chemical systems (Toluene/Water, Butyl acetate/Water, and n-Butanol/Water) were utilized and holdup was considered in a wide range of interfacial tensions through a Scheibel extraction column. Various rotor speeds were examined on the certain velocities of dispersed and continuous phases. It was found that with increasing rotor speed in a Scheibel extraction column, the drop size was reduced and drops were trapped inside the packed so that an increase in the dispersed phase holdup happened. An obvious increasing trend of dispersed phase holdup was observed as a result of increase in dispersed phase velocity for all systems operating under 2 different rotor speed, namely, 100 and 140rpm. However, the results showed that increase in the velocity of continuous phase would not make significant effect on the holdup. During examining the effect of both rotor speed and dispersed phase velocity, it was found that the holdup would be higher in the chemical system with the lowest interfacial tension compared with two other systems. An empirical correlation was also proposed to predict the dispersed phase holdup with AARE of 8.72%.

Keywords

Scheibel extraction column;
Dispersed phase;
Holdup;
Liquid-liquid extraction;
Rotor speed.

1. Introduction

Liquid-liquid extraction is one of the most important processes in industry. In this separation process, 2 liquid components are separated from each other with the help of a third component, so-called a solvent [1-2]. This

column is a kind of mechanically agitated column, consisting of alternate series of mixing zones and packing sections. Blades in this column are rotated by an engine embedded on the top of the column. The packing section isolates the agitator flow patterns between adjacent stages as back mixing is mitigated [3]. However, back mixing and low operating capacity are disadvantages of this type of column and, accordingly, it has been less used in the industry [4]. On the other hand,

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the mass transfer efficiency of Scheibel extraction column is high and the column is not recommended for systems tending to form an emulsion [5-6]. In recent years, some research has been done on the behavior of drops in a Scheibel extraction column. For example, Bonet and Jeffrey [7] studied the effect of operating parameters on the drop size in the extraction column. Yuan et al. [3] predicted a correlation for the mean drop size in modified Scheibel extraction column. They obtained the dispersed phase holdup in this Scheibel extraction column without providing any model to predict it. Most of these research works have focused on the mean drop size. According to the proposed correlations, by increasing the rotor speed, the dispersed phase holdup will increase [8-9]. In other words, by increasing the rotor speed, the tension in the system would increase and the drops are divided into smaller sizes and, therefore, their ascending speed is reduced. Similar to the rotor speed, dispersed phase velocity has a direct impact on the holdup of dispersed phase. However, different results have been obtained on the effect of continuous phase velocity depending on the type of extraction column [10-12]. To the best of our knowledge, prediction of the dispersed phase holdup in a Scheibel extraction column has not been taken into consideration well.

1.1. Holdup

The dispersed phase holdup is one of the important parameters involved in liquid-liquid extraction devices, which is applied to determine the mass transfer special area, sliding, and average speed. The holdup can be calculated according to the following correlation [9]:

$$\varphi = \frac{V_d}{V_d + V_c} \quad (1)$$

In the present study, the dispersed phase holdup was examined in a Scheibel extraction column with a height of 1.4m. For this purpose, the effects of different factors, such as rotor speed, dispersed phase velocity, continuous phase velocity, interfacial tension, and physical properties of the system on the dispersed phase holdup were examined. Finally, the empirical correlations were suggested to predict the dispersed phase holdup in a Scheibel extraction column.

2. Material and Methods

2.1. Set-up explanation

The experiments were conducted in a glass Scheibel extraction column with an inner diameter of 113 mm. The blades, being moved by a motor embedded in the top of the column, were used for the distribution of drops in the continuous phase. The aqueous and organic phases were stored in the respective supply tanks and pumped into the column when needed. A rotameter was utilized to adjust the flow rates and, in turn, velocities of dispersed and continuous phases. Also, two pairs of input and output were provided on the top and bottom of the column for continuous and dispersed phases. Different parts of the column and its dimensions are illustrated in Fig. 1 and Table 1, respectively.

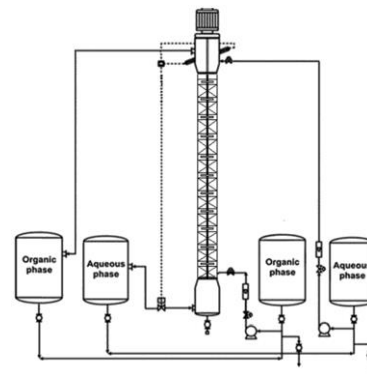


Figure 1. Schematic diagram of the Scheibel extraction column

Table 1. The main dimensions of the Scheibel column

Item	Symbol	Column dimensions (m)
Column internal diameter	D_c	0.113
Compartment height	h_c	0.14
Blade length	D	0.035
Column active height	h	1.43

2.2. Liquid systems

Three chemical systems as toluene-water (high interfacial tension), butyl acetate-water (medium interfacial tension), and normal butanol-water (low interfacial tension) were utilized to make a range of the considerable interfacial tensions. These systems have been suggested as standard

systems by the European Federation of Chemical Engineering [13]. The tests were conducted at different velocities of continuous and dispersed phases for the aforementioned systems with rotor speed ranging from 80 to 160 rpm. The physical properties of these systems are given in Table 2. Properties were obtained using a tensiometer, pycnometer, and Ostwald viscometer at 25°C.

Table 2. Physical properties of the studied systems

Physical property	Toluene-water	n-Butyl acetate-water	n-Butanol-water
ρ_c (kg/m ³)	998.2	997.6	985.6
ρ_d (kg/m ³)	865.2	880.9	846
μ_c (mPa.S)	0.963	1.0274	1.426
μ_d (mPa.S)	0.584	0.734	3.364
σ (mN/m)	36	14.1	1.75

2.3. Experiments procedure

The shut-down method was used to measure the amount of dispersed phase holdup. In this method, after achieving a steady state condition, the position of the intersection of 2 phases on the column was marked. Then, all inputs and outputs of the column were closed simultaneously and the rotor was turned off. When any drops were not present in the column, the change in the interface location was measured again to make a comparison with the initial state. By dividing the measured volume by the active column volume, the quantity of dispersed phase holdup was obtained. After considering the effect of operating parameters and physical properties of the systems on dispersed phase holdup, a correlation was suggested in order to predict holdup in a Scheibel extraction column using Eviews software.

3. Results and Discussion

3.1. Effect of operating parameters on holdup

To study the holdup of dispersed phase in the Scheibel extraction column, the effect of operating parameters, i.e., rotor speed, dispersed and continuous phase velocity, and the interfacial tension are investigated.

Fig. 2 shows the effect of rotor speed. In the mixer, drops form as a result of the break-up of dispersed phase by the shear field and in the other

part, i.e., packed section, drops coalesce with one another. Mixer speed has a significant effect on the drop size and break-up of dispersed phase could occur if dissipative force exceeds the restoring forces. Increase in the rotor speed leads to significant decrease in the mean drop size. This happens because the energy of rotor blades increases as a result of increase in rotor speed and blades with more energy disperse dispersed phase more easily and effectively. The more frequency of collisions between water drops that are well-dispersed and, subsequently, much smaller, because of higher rotor speed, causes a reduction in average velocity of dispersed phase drops towards the top of the column. Under the circumstances, the residence time of dispersed phase would increase, which, in turn, means increase in holdup. On the one hand, it can be seen that at a given low rotor speed, the difference between 3 systems is not noteworthy. But, by increasing the rotor speed, the difference between the responses of systems is more distinguishable when rotor speed of 140rpm is reached. Beyond a critical rotor speed, i.e., rotor speed from 140 to 160rpm, the holdup increases with a much smaller rate. On the other hand, as can be seen in Fig. 2, n-butanol-water system with the smallest interfacial tension displays the furthest holdup. This can be due to the dispersed phase in a system with the lowest interfacial tension, which includes drops with the lowest mean drop size in comparison with systems with higher interfacial tension. Therefore, within the n-butanol-water system having lowest interfacial tension, the rate of coalescence between drops is not intensive enough to produce bigger drops, which have lower frequency of collisions and less residence time inside the column. As a result, the dispersed phase speed will be reduced and, subsequently, the residence time of the drops becomes longer. Therefore, the holdup would be higher in the chemical system with the lowest interfacial tension, compared with two other systems.

The effect of the dispersed phase velocity on holdup is shown in Fig. 3. As can be seen, in a given rotor speed and by increasing dispersed phase velocity, the coalition between smaller drops would be intensified and, subsequently, by formation of larger drops, mean drop size increases. Therefore, such drops would pass through packed part with more difficulty and, thus, the holdup increases as a result of residence time of dispersed phase. The aforementioned occurrence is

intensified at higher rotor speeds, causing higher value of holdup. Moreover, it is clear that the holdup is sensitive to the interfacial tension as it would be greater for the system with lower interfacial tension, i.e., n-butanol-water, at any rotor speed.

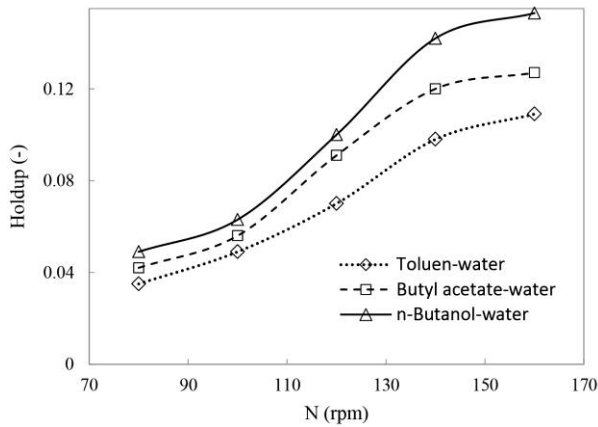


Figure 2. Effect of rotor speed on dispersed phase holdup ($V_d = V_c = 8.8 \times 10^{-4}$ (m/s))

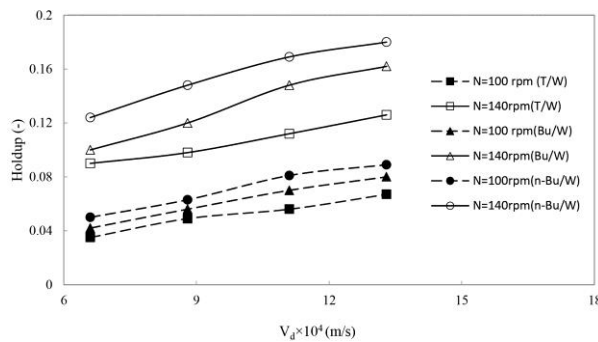


Figure 3. Effect of dispersed phase velocity on dispersed phase holdup ($V_c = 8.8 \times 10^{-4}$ (m/s))

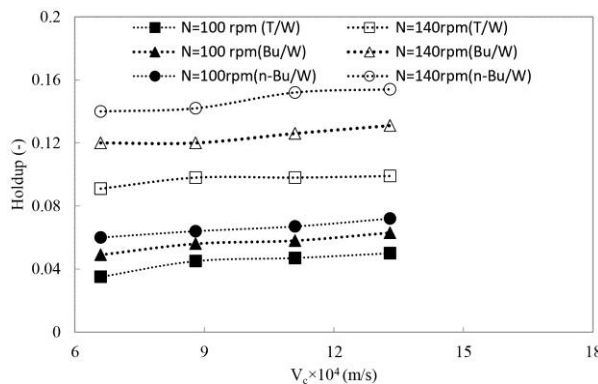


Figure 4. Effect of continuous phase velocity on dispersed phase holdup ($V_d = 8.8 \times 10^{-4}$ (m/s))

Fig. 4 shows the effect of the continuous phase velocity on the dispersed phase holdup. With raising the continuous phase velocity, the drag force increases and it takes the dispersed phase drops entering the bottom of the column more time to go up; thus, the residence time would increase. However, it seems that the range of continuous phase velocity utilized in this work does not make noticeable difference in holdup value. Moreover, three systems with different interfacial tensions exhibit similar response to an increase in the continuous phase velocity at various rotor speeds.

3.3 Prediction of correlation for holdup

As far as it is known, mean drop size along with dispersed phase holdup establishes the mass transfer coefficients and determines the interfacial area of mass transfer. Therefore, suggestion of correlations consisting mainly of operation conditions, the physical properties of the phases, and the column geometry would be beneficial to design and analyze Scheibel extraction columns. Table 3 shows some empirical predictive correlations for the holdup.

Average absolute relative error (AARE%) values are calculated as follows:

$$AARE\% = \frac{1}{n} \sum_{k=1}^n \left| \frac{\varphi(\text{exp}) - \varphi(\text{cal})}{\varphi(\text{exp})} \right| \times 100 \tag{2}$$

Where, n is total amount of data, $\varphi(\text{exp})$ is dispersed phase holdup obtained from experiments and, $\varphi(\text{cal})$ is dispersed phase holdup according to correlation.

Analysis of the variables affecting the holdup leads to suggestion of a functionality between following variables and the holdup. Therefore, the following correlation can be suggested:

$$\varphi = f(N, V_d, V_c, \sigma, \rho_d, \rho_c, \mu_d, \mu_c, g, D) \tag{3}$$

Using the Buckingham theory, the variables involved can be stated as some dimensionless groups. Thus, the following equation is suggested to elucidate the effect of operational parameters and physical properties on the dispersed phase holdup.

$$\varphi = C_1 \left(\frac{V_d^2 \rho_d}{g \mu_c} \right)^{C_2} \times \left(\frac{\mu_d}{\mu_c} \right)^{C_3} \times \left(\frac{N^4 D^4 \rho_c}{\sigma g} \right)^{C_4} \times \left(1 + \frac{V_d}{V_c} \right)^{C_5} \tag{4}$$

The equation coefficients are determined by Eviews software as $C_1 = 1.54$, $C_2 = 0.23$, $C_3 = -0.73$, $C_4 = 0.47$ and $C_5 = -0.01$.

In the above correlation, experimental data of Yuan et al. [3] are also used to get more accurate coefficients. According to Eq. 4, the dispersed phase holdup is highly dependent on rotor speed. To make a comparison between calculated values of holdup calculated by Eq. 4 and experimental data, Fig. 5 is plotted with AARE about 8.72%. As it can be seen, there is a good agreement between experimental data and correlation results. Therefore, the derived correlation can be used as a suitable basis for approximation of holdup in a Scheibel extraction column.

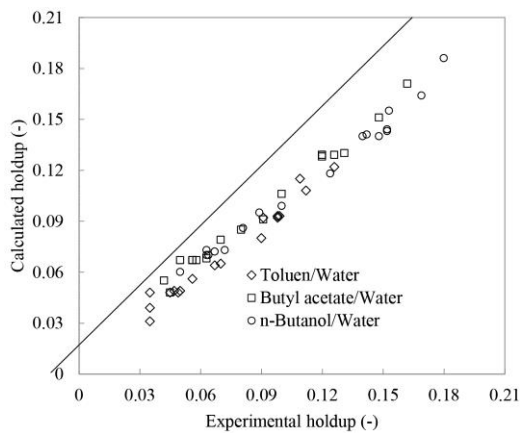


Figure 5. Comparison between experimental data and Eq. 4

4. Conclusion

In this paper, the effects of operating parameters and physical properties on holdup were considered in a Scheibel extraction column. The systems utilized included high, medium, and low interfacial tensions so that they could cover a wide range. The increase in rotor speed had a strong effect on holdup as the holdup would increase with increase in rotor speed. The velocities of dispersed and continuous phases were effective on holdup as well. However, according to the experimental observations, it was obvious that the effect of continuous phase velocity was not very important. Also, with increasing the interfacial tension, the holdup value decreased. An equation was suggested to predict the dispersed phase holdup by examining the impact of operating parameters and physical properties of the system with an average error rate of around 6.5%, showing a good agreement with the experimental data.

Table 3. Proposed correlations for dispersed phase holdup

Reference	Equation	Column
Napeida et al. [9]	$\phi = 13.38 \left(\frac{g}{N^2 D} \right)^{-1.43} \left(\frac{\mu_d}{\mu_c} \right)^{1.24} \left(\frac{V_d \rho_c^{0.25}}{g^{0.25} \sigma^{0.25}} \right)^{0.34}$	Hanson mixer-settler
Kasatkin et al. [14]	$\phi = 1.58 \left(\frac{N d_r}{V_c} \right) \left(\frac{V_d}{V_c} \right)^{0.96} \left(\frac{V_c^2}{g D_c^2} \right)^{0.96} \left(\frac{\Delta \rho}{\rho_c} \right)^{-1.31} \left(\frac{d_s^2 - d_r^2}{D_c^2} \right)^{-0.7} \left(\frac{h_c}{H} \right)^{-0.426} \left(\frac{\rho_c V_c D_c}{\mu_c} \right)^{-0.13} \left(\frac{\rho_c V_c^2 D_c}{\sigma} \right)^{0.245}$	RDC column
Sarkar et al. [15]	$\phi = 0.1728 + 0.4406 V_d + 0.1185 H + 0.0247 N$	Multi-impeller column
Tsouris et al. [16]	$\phi = 0.026 N^{4.58} V_d^{0.47} V_c^{0.27}$	Multi-impeller column
Kumar and Hartland [17]	$\phi = 8.28 \left[0.27 + \left(\frac{\epsilon}{d} \left(\frac{\rho_c}{g \sigma} \right)^{0.78} \right) \right] \left[V_d \left(\frac{\rho_c}{g \sigma} \right)^{0.25} \right]^{0.87} \left(\frac{\Delta \rho}{\rho_c} \right)^{-0.58} \left(\frac{\mu_d}{\mu_c} \right)^{0.18} \left[h_c \left(\frac{\rho_c g}{\sigma} \right)^{0.5} \right]^{-0.39} \exp \left[3.34 V_c \left(\frac{\rho_c}{g \sigma} \right)^{0.25} \right]$	RDC column
Oliveira et al. [18]	$\phi = (0.6 \pm 0.2) Q_d^{0.8 \pm 0.2} N^{1.6 \pm 0.3}$	Kuhni column
Kadam et al. [12]	$\phi = 458 \left(\frac{P}{V} \right)^{0.41} \left(\frac{\sigma^3 \Delta \rho}{\mu_c^2 g} \right)^{-0.071} (V_d)^{0.96} (V_c)^{0.24}$	ARDC column
Hemmati et al. [19]	$\phi = 0.089 \left(\frac{N^2 d_R}{g} \right)^{0.254} \left(\frac{\mu_d}{\mu_c} \right)^{0.272} \left(\frac{d_R}{h} \right)^{0.256} \left(1 + \frac{V_c}{V_d} \right)^{-1.04}$	PRDC column
Asdollahzade et al. [20]	$\phi = 15.86 \left(\frac{N^2 d_R^2}{\epsilon g h} \right)^{0.35} \left(\frac{\mu_d}{\mu_c} \right)^{-0.21} \left(1 + \frac{V_c}{V_d} \right)^{1.14} \left(\frac{\sigma g}{\rho_c V_d^4} \right)^{-0.98}$	Multistage column

Nomenclature

D	Blade length (m)
D_c	Internal diameter of column
d_r	Rotor diameter (m)
d_s	Stator diameter (m)
g	Gravitational constant (=9.81 m/s ²)
H	Effective height of the column (m)
h_c	Compartment height (m)
N	Rotor speed (1/s)
n	Number of droplets of mean diameter d_i
P/V	Power per unit volume (W/m ³)
Q	Flow rate (m ³ /s)
V	Superficial velocity (m/s)
AARE	Average absolute relative error

Greek letters

ϵ	Power dissipated per unit mass (m ² s ⁻³)
ρ	Density (kg/m ³)
$\Delta\rho$	Density difference between two phases (kg/m ³)
μ	Viscosity (Pa s)
σ	Interfacial tension between two phases (N/m)
ϕ	Hold – up

Subscripts

c	Continuous phase
d	Dispersed phase

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