Prediction of dispersed phase holdup in pulsed disc and doughnut solvent extraction columns under different mass transfer conditions

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ABSTRACT

Using experimental data from a number of pulsed disc and doughnut solvent extraction columns, a unified correlation for the prediction of dispersed phase holdup that considers the effects of mass transfer is presented. Pulsed disc and doughnut solvent extraction columns (PDDC) have been used for a range of important applications such as uranium extraction and nuclear fuel recycling. Although the dispersed phase holdup in a PDDC has been presented by some researchers, there is still the need to develop a robust correlation that can predict the experimental dispersed phase holdup over a range of operating conditions including the effects of mass transfer direction. In this study, dispersed phase holdup data from different literature sources for a PDDC were used to refit constants for the correlation presented by Kumar and Hartland [*Ind. Eng. Chem. Res.*,27 (1988),131–138] which did not consider the effect of column geometry. In order to incorporate the characteristic length of the PDDC (*i.e.* the plate spacing), the unified correlation for holdup proposed by Kumar and Hartland based on data from eight different types of columns [*Ind. Eng. Chem. Res.*,34 (1995) 3925–3940] was refitted to the PDDC data. New constants have been presented for each holdup correlation for a PDDC based on regression analysis using published holdup data from PDDCs that cover a range of operating conditions and physical properties and consider the direction of mass transfer.

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1. Introduction

Various types of solvent extraction contactors, including spray columns, packed columns and Karr columns, have been used for a range of applications in the hydrometallurgical, pharmaceutical and petrochemical industries for many years. The pulsed disc and doughnut solvent extraction column (PDDC) was originally described by Van Dijck [1]. Today, this column has found application in the separation and purification of components that include the spent nuclear fuel and uranium industries [2]. Compared to mixer-settlers, which are frequently used in the mining industry, the PDDC is attractive from both safety and economic stand points, in particular its simplicity of design, less space consumption, higher throughput and no internal moving parts [3].

Introduction of a solvent into any extractor causes the droplets to undergo repeated coalescence and breakage, leading to an equilibrium drop size distribution. The resulting fractional volumetric holdup, x_d, is defined as volume fraction of the active section of the column that is occupied by the dispersed phase:

$$x_d = \frac{v_d}{v_t} \tag{1}$$

where v_d represents the volume of the dispersed phase and v_t represents the total volume of the two phases for the effective length of the column.

The dispersed phase holdup is an important parameter in the design of solvent extraction columns as it is related to the interfacial area for mass transfer and the flood point of the column. Therefore prediction of the dispersed phase holdup is of fundamental importance in the design of solvent extraction columns [4].

Additionally, extraction experiments have revealed that mass transfer direction has a significant effect on the droplet behaviour in liquid dispersions [5]. In the case of solute transfer from the continuous phase to the dispersed phase, smaller drop sizes are observed due to higher breakage rates. The smaller drops have longer residence times and therefore the holdup increases. Conversely for mass transfer from the drop to the continuous phase, the coalescence rate is enhanced creating larger drops and lower holdup [4,6,7]. Therefore, dispersed phase holdup is also affected by the direction of the mass transfer.

In the present work, we analyse the published holdup data for a PDDC both with and without mass transfer and suggest new empirical correlations in terms of physical properties, operating conditions and column geometry.

2. Previous Work

Jeong [8] presented a correlation for the prediction of dispersed phase holdup in a 4.2 cm diameter pulsed disc and doughnut column

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using the kerosene-water system. The doughnut and disc plates were made of 1 mm thick stainless steel. The organic phase (Kerosene) was the dispersed phase. This correlation does not include the physical properties of the phases but is dependent on the operating conditions and plate geometry as follows:

$$x_d = 4.2 \times 10^{-5} h_c{}^{-0.44} A f^{1.28} V_d^{0.93} \tag{2}$$

where h_c is the compartment height (cm); A is the pulsing amplitude (cm); f is the pulsing frequency (min^{-1}); and V_d is the dispersed phase velocity (cm·min^{-1}).

Kumar and Hartland [4] used a large bank of published data including eight different types of solvent extraction columns (but did not include the PDDC) to develop a unified correlation for holdup as a function of various dimensionless groups as follows:

$$x_d = \Pi \cdot \Phi \cdot \psi \cdot \Gamma \tag{3}$$

in which Π allows for the mechanical power input per unit mass, ε; Φ allows for the effect of the phase velocities, V_c and V_d; ψ incorporates the physical properties and Γ represents the geometrical characteristics of the column. The parameters are defined as:

$$\Pi = C_\Pi + \left[\frac{\varepsilon}{g} \cdot \left(\frac{\rho_c}{g \cdot \gamma} \right)^{0.25} \right]^{n_1} \tag{4}$$

$$\Phi = \left[V_d \cdot \left(\frac{\rho_c}{g \cdot \gamma} \right)^{0.25} \right]^{n_2} \cdot \exp \left[n_3 \cdot V_c \cdot \left(\frac{\rho_c}{g \cdot \gamma} \right)^{0.25} \right] \tag{5}$$

$$\psi = C_\psi \left(\frac{\Delta \rho}{\rho_c} \right)^{n_4} \cdot \left(\frac{\mu_d}{\mu_c} \right)^{n_5} \tag{6}$$

$$\Gamma = C_\Gamma \cdot e^{n_6} \left[l \cdot \left(\frac{\rho_c \cdot g}{\gamma} \right)^{0.5} \right]^{n_7} \tag{7}$$

$$\varepsilon = \frac{2\pi^2 (1 - e^2)}{3 h_c C_0^2 e^2} (Af)^3. \tag{8}$$

The constants C_Π, C_Ψ and C_Γ and the coefficients n_1 to n_7 were adjusted to each type of column. For no mass transfer, $C_\Psi = 1$ and different values of C_Ψ were defined for each column type depending on the directions of mass transfer.

Combining Eqs. (3) to (8) resulted in a unified correlation [4] for eight different types of solvent extraction columns which did not include the PDDC. The resulting correlation is:

$$
\begin{aligned}
x_d = & \left[C_\Pi + \left\{ \frac{\varepsilon}{g} \left(\frac{\rho_c}{g \cdot \gamma} \right)^{0.25} \right\}^{n_1} \right] \cdot \left[V_d \cdot \left(\frac{\rho_c}{g \cdot \gamma} \right)^{0.25} \right]^{n_2} \\
& \times \exp \left[n_3 \cdot V_c \cdot \left(\frac{\rho_c}{g \cdot \gamma} \right)^{0.25} \right] \left(\frac{\Delta \rho}{\rho_c} \right)^{n_4} \cdot \left(\frac{\mu_d}{\mu_c} \right)^{n_5} \cdot C_\Gamma \\
& \times e^{n_6} \left[l \cdot \left(\frac{\rho_c \cdot g}{\gamma} \right)^{0.5} \right]^{n_7}.
\end{aligned}
\tag{9}
$$

Delden [9] used Eq. (9) to predict holdup in a PDDC based on his data for the forward and back-extraction of caprolactam using toluene. The holdup data was obtained under different mass transfer conditions but this was not taken into account when developing a holdup correlation. Very large deviations (Average absolute value of the relative error, AARE = 68.8%) between experimental and predicted values were found when using the original fit parameters provided with the unified correlation (Eq. (9)) presented by Kumar and Hartland [4] (the correlation fit parameters can be found in Table 1). Delden [9] refitted the constants for Eq. (9), as shown in Table 1, and the AARE was reduced to 12.2%. This study did not consider the effect of mass transfer direction on holdup. More recently, Rajnish [10] refitted the holdup data from a PDDC using Eq. (9) for a system of nitric acid 0.5 mol·L^{-1} (continuous aqueous phase) and 30% (by volume) TBP in normal paraffinic hydrocarbon (an industrial substitute of n-dodecane; dispersed organic phase). The refitted constants by Rajnish ($C_\Pi = 3.11$ and $n_1 = 0.68$) were able to predict the experimental data to within $\pm 20\%$ but again mass transfer direction still was not included in this correlation.

The most extensive study on holdup using a PDDC has been done by Jahya [11] and included the operation of non-mass transfer conditions as well as dispersed to continuous phase and continuous to dispersed phase mass transfer. He made use of an existing correlation for holdup (Eq. (10)) which is for a pulsed sieve-plate liquid–liquid extraction column (PSPC) [12] and refitted his experimental data.

$$x_d = k \left(\frac{(Af)^3 \rho_c{}^{0.25}}{\beta h g^{1.25} \gamma^{0.25}} \right)^{0.31} \left(\frac{V_d^4 \rho_c}{g \gamma} \right)^{0.3} \left(1 + \frac{V_d}{V_c} \right)^{0.14} \left(\frac{\Delta \rho}{\rho_c} \right)^{-0.79} \left(\frac{\mu_d^4 g}{\rho_c \gamma^3} \right)^{-0.01} \tag{10}$$

where the parameter k is fitted under different extraction system without considering the mass transfer direction. At the same time, another existing correlation [12] for a pulsed perforated-plate extraction column was used to predict the holdup data from a PDDC by Jahya [11]:

$$x_d = k_1 e^{[k_2 |Af - (Af)_m|]} V_d^{0.86} (V_c + V_d)^{0.28} \Delta \rho^{-0.30} \rho_d^{-0.93} \mu_d^{0.77} \varepsilon^{-0.56} h^{-0.56} \tag{11}$$

in which

$$(Af)_m = 9.69 \times 10^{-3} \left(\frac{\gamma \varepsilon \Delta \rho^{0.25}}{\mu_d^{0.75}} \right)^{0.33}. \tag{12}$$

k_1 and k_2 were constant parameters (refer to Table 2 for values of constants k_1 and k_2 and corresponding AARE). It can be seen from Table 2 that the parameters in Eq. (11) were different depending on the pulsing conditions and the extraction systems being studied.

Table 2
Fitted parameters and AARE for predicting holdup using Eq. (11) and Jahya experimental data [11]

System	Pulsing conditions	k_1	k_2	AARE/%
Shellsol–H$_2$O		2.10×10^6	44.53	14.2
Toluence–Acetone–H$_2$O	Low pulsation	4.192×10^7	-586.12	5.9
	High pulsation	6.929×10^6	110.03	16.9
Kerosene–Alamine–H$_2$SO$_4$	Low pulsation	3.959×10^6	12.17	14.3
	High pulsation	9.544×10^6	18.10	9.5

Table 1
Original and refitted constants for predicting holdup in a PDDC using Eq. (9) and Delden [7] experimental data

	C_Π	C_Γ①	n_1	n_2	n_3	n_4	n_5	$n_6$②	n_7	AARE/%
Kumar& Hartland constants	0.27	6.87	0.78	0.87	3.34	-0.58	0.18	0	-0.39	68.8
Delden (refitted constants)	2.39	0.45	0.34	0.87	3.34	-0.58	-0.08	0	-0.12	12.2

① Best values of C_Γ for different types columns.
② n_6 was fixed at -1 for both Kühni and packed columns, and zero for other agitated columns.