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Fluid Dynamics Study of a Liquid–Liquid Extraction Process in a Column Packed with SMVP

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Abstract: Fluid dynamics of a liquid-liquid extraction process in a 10-cm-diameter column packed with 3.99 m of structured SMVP packing by Sulzer was studied, working with the n-butanol(disperse)/succinic acid (1%/wt.)/water(continuous) with both mass-transfer directions and different phase ratios. The disperse phase holdup and phase velocities during flooding were measured. The fluid dynamic variables experimentally obtained were compared with the various models in the literature, and the Mackowiak and Billet's model for the disperse phase holdup estimation was found to have the best adjustment to the experimental data for both transfer directions. Experimental values of 0.65 and 0.96 were obtained for the d → c and c → d directions, respectively, for the Cp constant in the Streiff and Jancic equation for the calculation of Vslip. The maximum throughput was found between 39.1 and 60.5 m^3/m^2h for the d → c transfer direction and between 46.7 and 65.6 m^3/m^2h for the opposite direction, when the phase ratio varied within 0.35 and 2.0. The Kumar and Hartland's model exhibited the best adjustment under the maximum throughput condition when the model parameters were modified.

Keywords: Liquid extraction, structured packing, throughput limits, holdup models

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INTRODUCTION

Recently, the liquid–liquid extraction has become an attractive option as a separation technique resulting, in some cases, in cost and energy savings. Packed columns are recommended for continuous extraction when few equilibrium stages (5 to 10) are required (1).

In the columns packed with structured fillings without stirring, separation is increased in comparison with random packings. Structured packings are characterized by their high crossed mixing and low axial back mixing, which allows the handling of large loads with small differences of interfacial tension and density, improving the fluid dynamic performance of the equipment and subsequently the separation efficiency (2).

Unfortunately, the available information on the suitable correlations for the design and operation of columns with structured packing is scarce, basically due to the high costs linked to experimental tests.

The SMVP structured packing consists of SMV packing (Sulzer) sections which function as static mixers promoting the disperse phase drops rupture as well as radial mixing, alternated with dual-flow plates which split the column into individual cells remarkably restricting axial mixing. Currently, this type of packing is used in several chemical and petrochemical processes (3).

The purpose of this work is to provide information to assess the calculation models available in the literature and select the most suitable ones to be used in columns with structured packing, specifically SMVP.

The system used in this study was n-butanol(disperse)/succinic acid (solute)/water(continuous), recommended by EFCE. Several parameters affect the fluid dynamics and, consequently, the mass transfer in the packed columns.

The slip velocity or relative velocity between phases is used in mass-transfer coefficient calculations and therefore affects the velocity with which the mass-transfer process is carried out. For a countercurrent process it is defined as (4):

$$V_{slip} = \frac{V_d}{\varepsilon \cdot \phi_d} + \frac{V_c}{\varepsilon \cdot (1 - \phi_d)} \tag{1}$$

The two most common models which set out slip velocity as a function of the characteristic velocity and holdup are the linear and the potential model. The linear model was proposed by Gayler and Pratt (5) and Gayler et al. (6) according to Eq. (2). Slater (7) and Godfrey and Slater (8) questioned the applicability of the linear model and recommended its being replaced with the potential model formulated by Richardson and Zaki (9) for solid particles, according to Eq. (3):

$$V_{slip} = V_k \cdot (1 - \phi_d) \tag{2}$$

$$V_{slip} = V_k \cdot (1 - \phi_d)^m \tag{3}$$

The disperse phase holdup is defined as the fraction of empty volume in the column occupied by the disperse phase. This constitutes, along with the drop diameter, a determinant parameter in mass transfer. The typical behavior established between the surface velocities of the disperse and continuous phases and the disperse phase holdup in liquid–liquid systems is qualitatively presented in Fig. 1. The disperse phase holdup as a function of the continuous phase surface velocity (V_c) is shown in Fig. 1a, with the disperse phase surface velocity (V_d) as a parameter. It can be observed that for V_c up to 70% of the flooding value ($V_{c,f}$), the disperse phase holdup is independent of V_c, and within this velocity range, holdup is only a V_d function. This can be better observed in Fig. 1b, where holdup is represented as a function of the V_d with V_c as a parameter. The following relationship between holdup and V_d (1) is obtained within this range.

$$\phi_d = C_s \cdot V_d \tag{4}$$

where C_s is a constant which depends on the packing as well as the system properties: both phase densities and interfacial tension.

The maximum throughput of the column is determined by the sum of volumetric velocities of both phases at the flooding moment (B_{max}). Hydraulic characteristics in this region significantly differ from those within the normal operation interval. The symptoms indicating column flooding may include the formation of a second interface at a certain column height (different from the main interface) or dragging of one of the phases by the other in the opposite direction to the one it should circulate through. Flooding thus sets the upper limit of the column capacity which is critical for designing the diameter of the packed column (1).

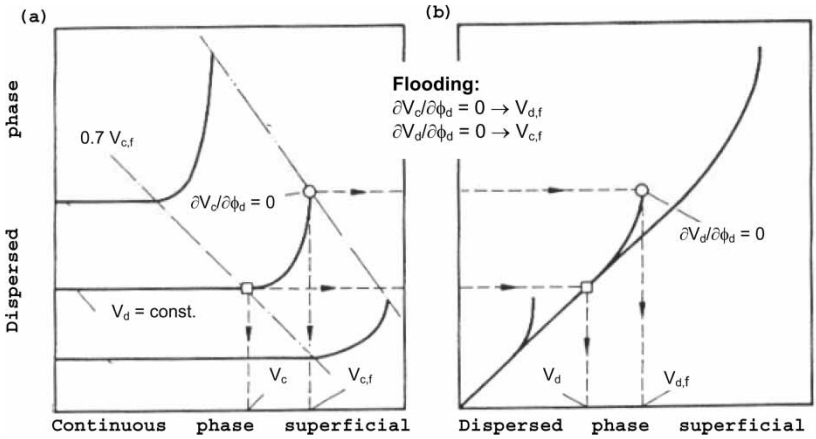

Figure 1. Qualitative description of the hydraulics in packed column extractors (1).

BACKGROUND

Different authors have studied the SMV structured packing performance using the n-butanol/water system with or without succinic acid transfer (4, 10–14). However, the SMVP packing has been little studied. Some of the works published so far are summarized below. The equations proposed by different researchers are shown in Table 1.

Jancic et al. (10) tested the performance of the stainless steel SMV static mixers in a 10-cm-diameter and 6-m packed section column, in countercurrent liquid-liquid extraction processes at 20°C (constant temperature). Four testing systems were used, including the n-butanol/succinic acid(solute)/water. The experiments were conducted without the presence of solute and with mass transfer in the d → c and c → d directions, varying the phase ratio as well as the extraction factor. The maximum throughput for the n-butanol-water (without solute) system showed the smallest value among all the studied systems ($40\,m^3/m^2h$), due to the low interfacial tension and to the consequently lower drop diameter. When using succinic acid as a solute, a 40% increase in the column total capacity for the mass transfer from the disperse to the continuous phase regarding the extraction in the opposite direction was reported, which is attributed to a disperse phase holdup decrease due to the presence of drops with larger diameter.

Streiff and Jancic (11) carried out studies on the maximum throughput and disperse phase holdup, with and without mass transfer, using a 10-cm-diameter and 5 m SMV structured packing, with high and low interfacial tension systems (including the n-butanol–succinic acid-water system). These researchers supposed that the slip velocity is independent of holdup, and an average [Eq. (1.2)] can be calculated which would be based on the characteristic slip velocity and a parameter C_p (not reported) which takes into account the drop-drop and drop-packing interactions. They propose two equations for the flooding velocities calculation [Eqs. (1.3a) and (1.3b)]

Mackowiak and Billet (12) worked with random and structured packings such as the metallic Montz B1-300. They do not include the SMV packing nor the n-butanol–succinic acid-water system. They determined the disperse phase holdup, drop diameter, and flooding velocities using eight systems with the d → c and c → d directions. In relation to the disperse phase holdup, they present two models [Eqs. (1.7a) and (1.7b)], which are both valid when $V_c < 0.7\ V_{c,f.}$ The first one introduces the C_s constant, which is characteristic of the packing and the system; the second one is an approximate expression, valid for $\phi < 0.25$. This approximate expression includes the $C_{h,MB}$ constant, which depends on the packing geometry and is independent of the system. This can be experimentally determined if the holdup values as a function of the disperse phase velocity are known for a given system and packing. The maximum throughput equations proposed by the authors introduce a constant characteristic of the packing into flooding condition [$C_{f,Mb,}$ see Eq. (1.8)], which must be experimentally determined as the

Table 1. Published correlations for fluid-dynamics study in packed columns

Correlation	Comments
Streiff and Jancic (1)	
$\frac{d_{vs}}{d_h} = C_F \cdot We_c^{-0.5} \cdot Re_c^{0.15}$ (1.1)	$C_F = 0.12$ (no mass transfer)
$\overline{V}_{slip} = C_p \cdot V_{so}$ (1.2)	$C_p = f$ (packing type)
$V_{so} = \left[\frac{4}{3} \cdot \frac{\Delta\rho \cdot g \cdot d_{vs}}{\rho_c \cdot C_D}\right]^{0.5}$ (1.2a)	
$V_{c,f} = C_p \cdot V_{so} \cdot \epsilon \cdot (1 - \phi_{d,f})^2$ $V_{d,f} = C_p \cdot V_{so} \cdot \epsilon \cdot \phi_{d,f}^2$ (1.3a and 1.3b)	
$\phi_{d,f} = \frac{\lambda^{0.5}}{1 + \lambda^{0.5}}$ (1.4)	
Maćkowiak and Billet (12)	
$d_{vs} = C_{2MB} \cdot \sqrt{\frac{\sigma}{\Delta\rho \cdot g}}$ (1.5)	$C_{2MB} = 0.8$ (if $\rho_d > \rho_c$) 1.0 (if $\rho_c > \rho_d$)
$V'_{so} = \left[\frac{4 \cdot g \cdot \Delta\rho \cdot \sigma}{\rho_c^2 \cdot C_D}\right]^{1/4}$ (1.6)	
$\phi_d = C_s \cdot V_d$ If $V_c < 0.7V_{c,f}$ (1.7a)	
$\phi_d \approx C_{h,MB} \cdot \frac{V_d}{\varepsilon} \cdot \left(\frac{\rho_c^2}{4 \cdot g \cdot \Delta\rho \cdot \sigma}\right)_{C_D=1}^{1/4}$ (1.7b)	
$V'_k = C_{f,MB} \cdot V_{so}$ (at flooding) (1.8)	
Seibert and Fair (14)	
$d_{vs} = 1.15 \cdot \eta \cdot \sqrt{\frac{\sigma}{\Delta\rho \cdot g}}$ (1.9)	$\eta = 1$ c → d (or no mass transfer)

(*continued*)

Table 1. Continued

Correlation	Comments
$V_{slip} = V_{so} \cdot \exp\left(\frac{-6 \cdot \phi_d}{\pi}\right) \cdot \mathrm{Cos}\left(\frac{\pi \cdot \zeta}{4}\right) + \left[1 - \mathrm{Cos}\left(\frac{\pi \cdot \zeta}{4}\right)\right] \cdot \frac{V_c}{\varepsilon \cdot (1 - \phi_d)}$ (1.10)	$\eta = 1 - 1.8$ d → c AAPE = φ_d (12%) B_{max} (12%)
$\zeta = \frac{a_p \cdot d_{vs}}{2}$ (1.11)	
$\phi_d = \frac{V_d \cdot [\mathrm{Cos}(\pi \cdot \zeta/4)]^{-2}}{\varepsilon \cdot [V_{so} \cdot \exp(-6 \cdot \phi_d/\pi) - V_c/\varepsilon \cdot (1 - \phi_d)]}$ (1.12)	
$\phi_{d,max} = \frac{\pi}{6} = 0.52$ (1.13)	
$1.08 \cdot V_{c,f} + \left[\cos\left(\frac{\pi \cdot \zeta}{4}\right)\right]^{-2} \cdot V_{d,f} = 0.192 \cdot \varepsilon \cdot V_{so}$ (1.14)	
Kumar and Hartland (4)	
$\frac{d_{vs}}{\sqrt{\sigma/\Delta\rho \cdot g}} = C_{1KH} \cdot \left(\frac{\mu_W \cdot g^{1/4} \cdot \rho_W}{\Delta\rho^{1/4} \cdot \sigma^{3/4} \cdot \rho_d}\right)^{0.19}$ (1.15)	$C_{1KH} = 2.54$ (no mass transfer) = 2.24 c → d = 3.13 d → c
$V_{slip} \cdot \left(\frac{\rho_c}{g \cdot \mu_c}\right)^{1/3} = C_{3KH} \cdot \varepsilon^{-0.17} \cdot \left(\frac{\Delta\rho}{\rho_c}\right)^{0.41} \times \cdot \left[\frac{1}{a_p} \cdot \left(\frac{\rho_c^2 \cdot g}{\mu_c^2}\right)^{1/3}\right]^{0.59} \times \cdot \left(\frac{\mu_d}{\mu_c}\right)^{-0.10} \cdot (1 - \phi_d)$ (1.16)	$C_{2KH} = 5.34$ (no mass transfer) = 6.16 c → d = 3.76 d → c

(*continued*)

Table 1. Continued

Correlation	Comments
$\phi_d = C_{2KH} \cdot \varepsilon^{-1.11} \cdot \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.5} \cdot \left[\frac{1}{a_p} \cdot \left(\frac{\rho_c^2 \cdot g}{\mu_c^2}\right)^{1/3}\right]^{-0.72} \times \left(\frac{\mu_d}{\mu_c}\right)^{0.1} \times \left[V_d \cdot \left(\frac{\rho_c}{g \cdot \mu_c}\right)^{1/3}\right]^{1.03} \times \exp\left[0.95 \cdot V_c \cdot \left(\frac{\rho_c}{g \cdot \mu_c}\right)^{1/3}\right]$ (1.17)	$C'_{3KH} = 0.30$ (no mass transfer) $= 0.27$ $c \rightarrow d = 0.38$ $d \rightarrow c$.
$V_{c,f} \cdot \left[1 + 0.36\left\{\frac{1}{a_p}\left(\frac{\Delta\rho^2 \cdot g}{\mu_c^2}\right)^{1/3}\right\}^{0.41} \cdot \lambda^{1/2}\right]^2 \cdot \sqrt{\frac{a_p}{g}} = \alpha_{KH} C'_{4KH} \cdot \varepsilon^{1.61} \cdot \left(\frac{\Delta\rho}{\rho_d}\right)^{0.38} \times \left[\frac{1}{a_p} \cdot \left(\frac{\Delta\rho^2 \cdot g}{\mu_c^2}\right)^{1/3}\right]^{0.67} \times \left(\frac{\mu_c}{\sqrt{\Delta\rho \cdot \sigma / a_p}}\right)^{0.12}$ (1.18)	$\alpha_{KH} = 1$ (continuous phase wetting) $\alpha_{KH} = 1.25$ (dispersed phase wetting) AAPE = φ_d: 18.7%. V_{slip}: 15.6%. $B_{máx}$: 19.5%.

slope of the graphic representation of V_{slip}/V_{so} as a function of $(1\text{-}\phi)$ within the flooding region. They report larger holdup values in the $d \rightarrow c$ direction due to presence of smaller drops.

Seibert et al. (13) used the n-butanol–succinic acid (0.8%wt./wt.)-water system, and the SMV packing, only in the $d \rightarrow c$ transfer direction. Their results showed that the disperse phase holdup varied within 0.10 and 0.48(m^3/m^3), when using phase ratios between 0.4 and 5. Seibert and Fair (14) used the SMV packing, with the n-butanol–succinic acid (0.8%wt./wt.)/water(c) system, among others, in the $d \rightarrow c$ and $c \rightarrow d$ directions. In the drop diameter calculation model [Eq. (1.9)] they introduced a correction factor dependent on the mass-transfer direction. The slip velocity [Eq. (1.10)] model is inferred based on the characteristic slip velocity or terminal velocity (V_{so}) first, and then a drop-drop interaction function and tortuousness [Eq. (1.11)] are introduced to determine the effect of the packing on the drop movement.

The disperse phase holdup model [Eq. (1.12)] is directly proportional to its surface velocity, and depends on the packing characteristics as well as the physical properties of the system. The model proposed for the

maximum throughput is given by Eq. (1.14), which contains porosity, tortuousness, and drop velocity parameters.

Kumar and Hartland (4) present empirical correlations to determine drop diameter, disperse phase holdup, slip velocity, and maximum loads in packed columns. They used results published in the literature comprising random and structured packing with different characteristics (including SMV) as well as a wide range of physical properties, column diameters and transfer directions. The correlation parameters were determined by adjusting a huge experimental database from the literature to equations resulting from the dimensional analysis and using the Marquardt's algorithm. In the models employed to determine the slip velocity [Eq. (1.16)] and the disperse phase holdup [Eq. (1.17)], they concluded that the term including the interfacial tension was negligible. They used 2023 pieces of data, from which 67% had no mass transfer, 15% was in the $c \rightarrow d$ direction, and 18% in the $d \rightarrow c$ direction.

The model to estimate limiting loads [Eq. (1.18)] was developed based on 845 measurements under maximum throughput conditions, from which 90% had no mass transfer, 6% were in the $c \rightarrow d$ direction, and 4% in the $d \rightarrow c$ direction. The values of the C_{4KH} adjustable parameter were a function of the packing type [0.07 for the SMV packing (Sulzer)].

Manuale (15) worked with several structured packing similar to the SMV, including the standard aluminum one (STD 90°) with 0.93 empty fraction and $a_p = 600$ (m^2/m^3), the specific one for liquid–liquid applications (ESP 90°) (similar ε and a_p) and the specific one for liquid-liquid operations oriented to 45° (ESP 45°) identical to the former one but varying the rotation angle. He used the n-butanol–succinic acid/water system in both transfer directions, dispersing always the organic phase and reports values of $C_{f,MB}$ [Eq. (1.8)] within 0.65 and 0.45, these being within the order of magnitude reported by Billet (1) for structured packings.

Table 2 summarizes the little specific-information published concerning SMV and SMVP packing. A maximum throughput decrease for the toluene/acetone/water system is reported due to the presence of the dual-flow plates and an capacity decrease for the $d \rightarrow c$ direction when using the SMV packing compared with the SMVP. For the n-butanol–succinic acid/water system, only the information for the $c \rightarrow d$ transfer direction without dual-flow plates is available. However, it is important to remark that the capacity is of the order of 50 m^3/m^2h.

EXPERIMENTAL EQUIPMENT AND METHODOLOGY

The extraction plant flow diagram is shown in Fig. 2. The packed column (C-1) has 19 sections of stainless steel Sulzer SMV 350Y structured filling, 0.21 m in height, and 0.10 m in diameter each, alternated with the liquid redistribution plates, reaching a total packing height of 3.99 m. The packing

Table 2. Capacities at flooding

Packing	System	Capacity ($m^3/(m^2 \cdot h)$)	Mass transfer direction	Reference
Sulzer SMV	Toluene(d)/ acetone/ water (c)	90	c → d	(10, 13)
Sulzer SMVP	Toluene(d)/ acetone/ water (c)	60	c → d	(11, 3)
Sulzer SMV	Butanol(d)/ Succinic acid/water (c)	50	c → d	(10, 13)
Sulzer SMV	Toluene(d)/ acetone/ water (c)	150	d → c	(10, 13)

specific surface area is $350\,m^2/m^3$, and the empty fraction is 0.98. Figure 3 shows a picture of the packing and the dual-flow plates.

The column case is made of stainless steel and has disperse (organic) and continuous (aqueous) phase distributors at the bottom and top, respectively.

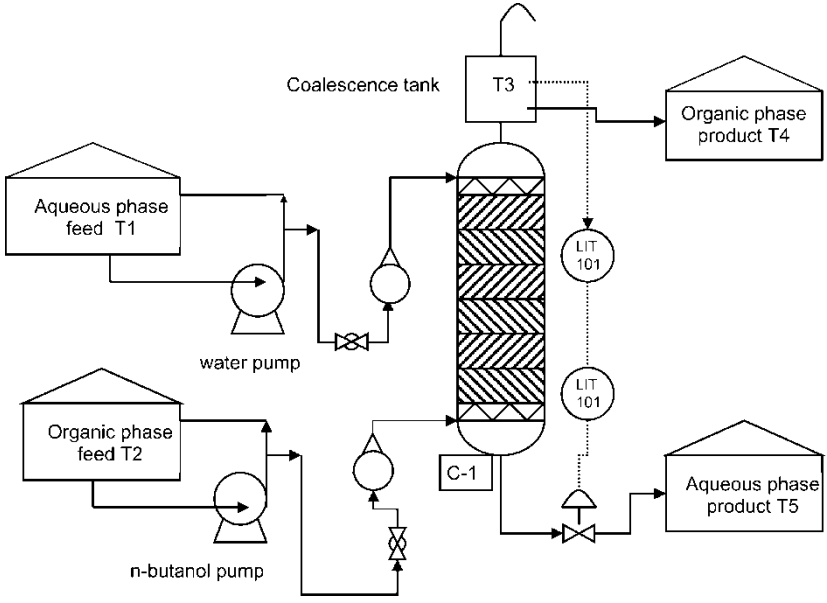

Figure 2. Flow diagram of experimental equipment.

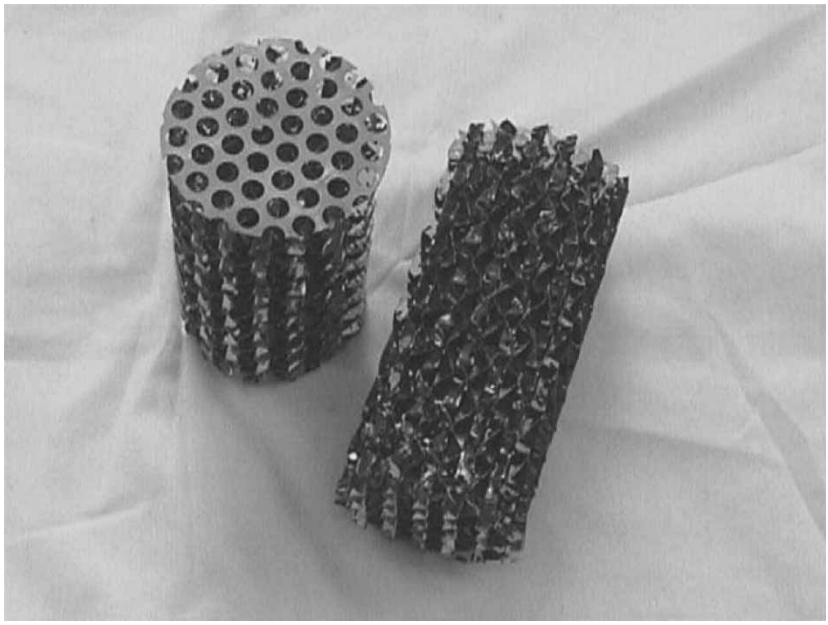

Figure 3. SMVP Packing.

A 0.15 m glass section was installed at the bottom of the column to observe the possible drop dragging in the continuous phase. Two glass viewers were installed at the distributor to view the drops sight and detect the presence of a second interface in the flooding tests. A coalescence tank (T3), with an interface level sensor-transmitter associated to the PID level control (LIC-101), is located at the column top, whose final control element is a needle valve that regulates the continuous phase outlet. Tests in both mass-transfer directions were carried out as well as seven different phase ratios (Vd/Vc) for each one.

The organic phase was fed with 1%/wt. succinic acid for the $d \rightarrow c$ direction, whereas for the opposite direction, the same was made for the aqueous phase.

The water used in the tests was previously demineralized and its conductivity was kept constant at 10 μmhos for all the experiments. The tests were conducted at a temperature ranging within 26° and 28°C. The viscosity, density, and interfacial tension were measured on the extraction column ends in both phases in all tests. The incoming flows were adjusted through a set of valves and rotameters whereas the outgoing flows were measured with a digital electronic turbine-type flow meter which can be used as a flow meter and a counter, as desired. Rotameters have an appreciation of 0.07 lt/min and the digital flow meters of 0.01 lt/min.

In order to determine the disperse phase holdup, all outlet and inlet valves of the column phases were closed simultaneously and the disperse phase was decanted into the upper tank, and emptied and weighed later.

Once the disperse phase holdup in the steady state was determined, the velocities of both fluids were increased, keeping the phase ratio constant up to observing the flooding signals. We considered that flooding condition was reached when one of this conditions was observed: the disperse phase drops were dragged by the continuous phase; a second interface was formed at the distributor of the disperse phase; and/or instability on the main interface occured. The reported values result from averaging two experimental tests carried out under equal conditions.

EXPERIMENTAL RESULTS

Figure 4 shows the slip velocity as a function of holdup for both mass-transfer directions. When applying the linear model [Eq. (2)], no significant differences were observed between both mass-transfer directions; all data adjusted to a straight line were found to have a 0.95 correlation index; and the characteristic velocity (V_K) calculated as the straight line slope was found to have a value of 0.042 m/s.

Slip velocity experimental values are shown in Table 3, along with those obtained when applying the different researchers' models. The absolute average percentage error (AAPE) is indicated for each model.

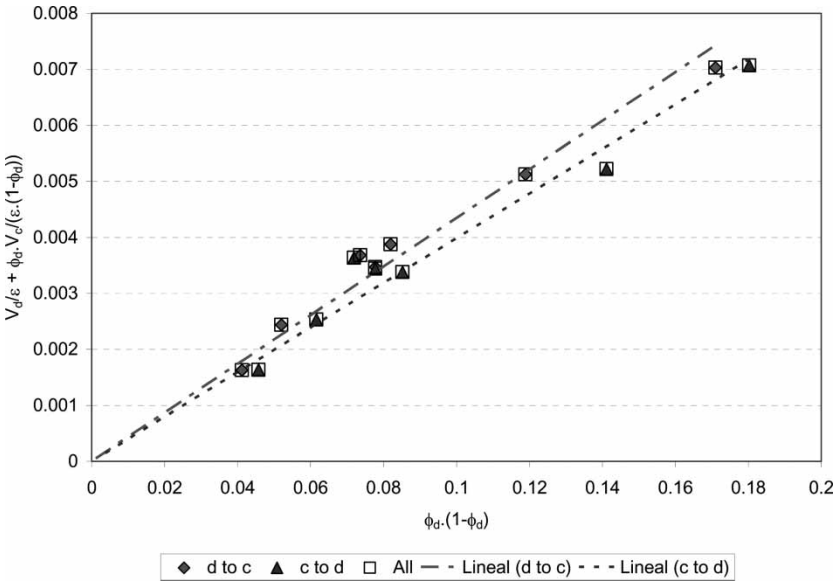

Figure 4. Lineal model of Vslip.

Table 3. Comparison between slip velocity models

Transfer direction	Phase ratio ($\lambda = V_d/V_c$), adim.	Slip velocity, $V_{slip} \times 10^2$ (m/s)			
		Experimental	Streiff and Jancic (V_{slip} average)	Seibert and Fair	Kumar and Hartland (C_{3KH} modified)
d → c.	0.36	3.79	4.02	4.57 (1)	4.37
	0.46	4.60		4.09	4.09
	0.55	4.43		4.39	4.26
	0.77	4.08		4.16	4.14
	1.20	4.31		4.04	4.09
	1.48	3.70		3.73	3.95
	1.96	3.21		3.13	3.75
	AAPE		10	6	9
c → d.	0.35	3.40	3.66	3.57	4.26
	0.45	4.66		2.97	3.81
	0.55	3.84		3.35	4.07
	0.75	4.05		3.11	3.89
	1.00	3.59		3.15	3.92
	1.50	3.07		2.60	3.58
	2.00	2.99		2.03	3.25
	AAPE		12	20	13

To analyze the Streiff and Jancic's proposal, the average slip velocity for every transfer direction was calculated (being reported as the result in the corresponding box in this model); then the error of each experimental value was calculated in respect to the average value. For this reason, in these authors' case, the AAPE represents the error of the experimental values in relation to the average value, resulting in a maximum of 12%.

Values for the dimensionless C_p constant [Eq. (1.2)] of 0.65 and 0.96 were obtained for d → c and c → d directions, respectively, thus proving what the authors indicated about the effect of the drop-drop and drop-packing interactions on this constant.

The average slip velocity experimental values show a smaller value for the c → d transfer direction, which can be due to the fact that drops smaller than those for the opposite direction are formed, which result in a greater holdup and lower slip velocity.

When using Seibert and Fair's model (14) [Eq. (1.10)], an important difference is established between the error obtained for both transfer directions, being the percentage deviation for the d → c direction 14 points lower. This can be due to the fact that when depending explicitly on the disperse phase holdup, the slip velocity is affected by the error associated to

the Seibert and Fair's model for the disperse phase holdup calculation, which is occasionally of difficult convergence.

When the Kumar and Hartland's model is applied (4) [Eq. (1.16)] with the C_{3KH} constant recommended by the authors, a 39% error was obtained for the c → d transfer direction. With a view to improve the adjustment, the C_{3KH} parameter was modified, minimizing the sum of the differences among the values provided by the model and the experimental value by varying C_{3KH}; in this case a value of $C_{3KH} = 0.45$ was obtained for both transfer directions. Thus the error could be reduced from 39% to 13% for the c → d transfer direction (Table 3), this value being lower than that reported by the authors (15.6%).

Similar deviations are observed when the models of Kumar and Hartland and Streiff and Jancic are used.

Holdup

The holdup experimental values in the steady state are shown in Fig. 5 as a function of V_d for all V_c values used in this study as well as both mass-transfer directions. The experimental values follow the expected linear trend with correlation indexes slightly above 0.9. This indicates that the continuous phase velocities used in these tests were 70% below the column flooding value, range within which the holdup solely depends on the disperse phase velocity.

In general, greater holdup values were observed when the transfer direction was c → d. This fact indirectly proves that drop diameter in the

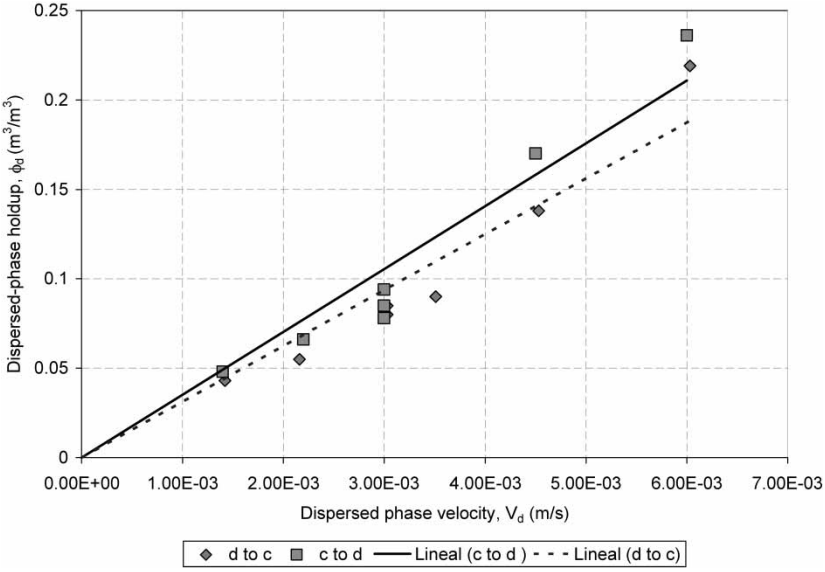

Figure 5. Dispersed-phase holdup as a function of the dispersed-phase load.

c → d transfer direction is smaller than that in the opposite direction, which results in a higher holdup. This is in agreement with the results obtained in the studies mentioned before.

The disperse phase holdup parity graphics for the different models assessed are shown in Figs. 6 and 7.

Concerning the Mackowiak and Billet's model (12), the results using Eqs. 1.7a and 1.7b are presented. Equation 1.7a requires the constant C_s obtained in Fig. 5, which showed a value of 31.2 and 35.1 s/m for the d → c and c → d directions, respectively. The C_s constant appears to be slightly influenced by the transfer direction since the average physical properties for both transfer directions are very similar and the packing is the same. According to Mackowiak and Billet, the value of C_s obtained should be the same, but this is not the case. More experiments should be conducted in order to discard the effect of experimental errors on this discrepancy.

The average value of the $C_{h,MB}$ dimensionless constant is needed for the disperse phase holdup calculation according to Eq. 1.7b. This value was 1.7 and 1.9 for the d → c and c → d directions, respectively. This result is in agreement with the authors' proposal since it turned to be practically independent of the transfer direction for $\phi_d \leq 0.25$. For both equations the average percentage absolute error was under or equal to 15%.

Regarding the Seibert and Fair's model, it is worth mentioning that it presented convergence problems, specifically for the c → d transfer direction, where the greatest AAPEs occurred (22%), although it should be

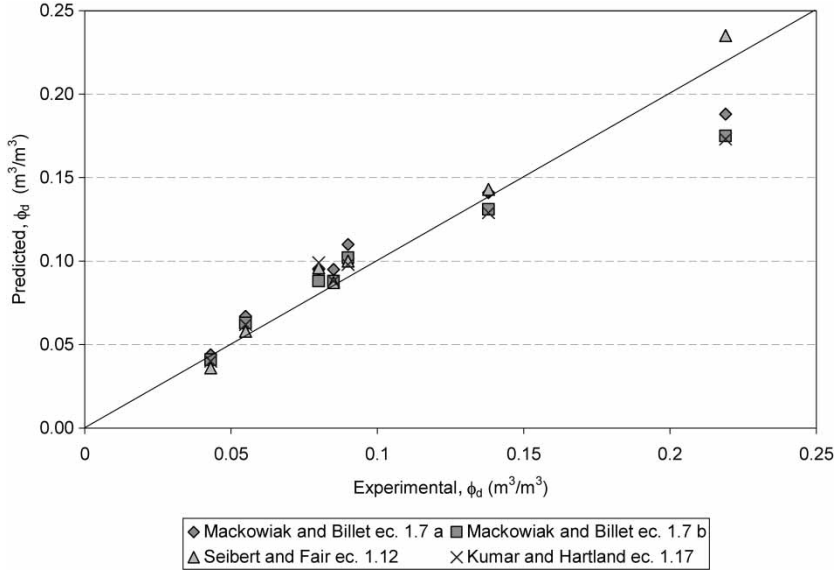

Figure 6. Dispersed phase holdup parity plot (d → c).

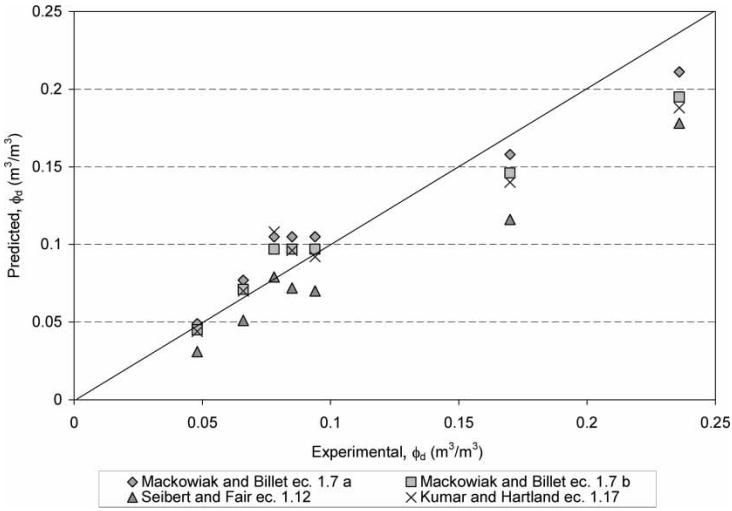

Figure 7. Dispersed phase holdup parity plot (c → d).

remarked that the model presented the lowest average percentage absolute error (9%) for the d → c direction.

When using the Kumar and Hartland's model (Equation 1.17) (4) with the C_{2KH} constant suggested by the authors, average percentage absolute errors as high as 64% were found for the c → d direction. This may be attributed to the fact that more than 70% of the data used by the authors did not included mass transfer.

These results suggested that the model error could be minimized through the adjustment of the value of the C_{2KH} constant, which represented a substantial improvement of the model adjustment to the experimental results (AAPE = 12% for d → c, 15% for c → d), both below the values reported by the authors (18.7%).

The analysis of Figs. 6 and 7 leads us to infer that, in general terms, all studied models fit the experimental results generating average percentage deviations under or equal to 22%. To differentiate among every model, a statistical analysis, similar to the proposed by Ansari et al. (16), was performed, consisting of calculating six statistical parameters: the average percentage error (E1), the absolute average percentage error (AAPE)(E2), the percentage standard deviation (E3), the average error (E4), the absolute average error (E5), and the standard deviation (E6), in order to determine eventually the "relative performance factor (RPF)." The minimum and maximum possible values for RPF are 0 and 6, which helps in determining a criterion to discriminate among models. The Mackowiak and Billet's Eq. (1.7b) presents the best adjustment for both transfer directions (RPF = 1.07 d → c, RPF = 0.03 c → d).

Table 4. Relative performance factors (holdup)

Transfer direction	Model	Number of data points	E1	E2	E3	E4	E5	E6	RPF
d → c	Maćkowiak and Billet, Ec. 35	7	1.66	10.2	4.68	−0.31	1.20	0.89	1.07
	Seibert and Fair	7	4.79	9.45	13.6	0.64	0.84	1.82	4
	Kumar and Hartland	7	2.06	11.9	5.82	−0.30	1.36	0.85	2.25
c → d	Maćkowiak and Billet, Ec. 35	**7**	1.64	12.4	4.65	−0.41	1.52	1.17	0.03
	Seibert and Fair	7	−22.0	22.4	62.2	−2.57	2.60	7.27	6
	Kumar and Hartland	7	1.29	15.1	3.64	−0.56	1.84	1.58	0.69

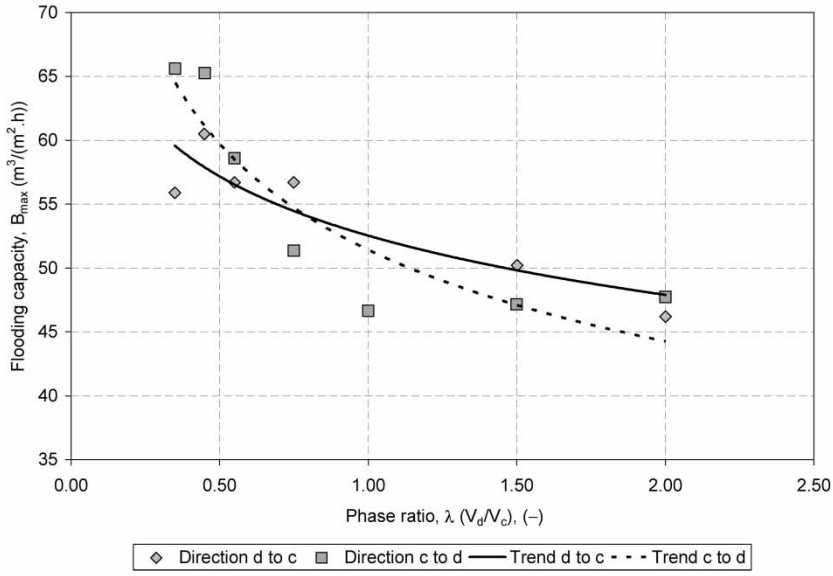

Figure 8. Capacities at flooding point for n-butanol (dispersed)—water (continuous)—succinic acid with SMVP packing.

An outstanding aspect in Figs. 6 and 7 is that for both transfer directions and all models, the adjustment gets substantially worse from ϕ_d values higher than approximately 0.15. The only authors who establish an application interval for their ϕ_d model are Mackowiak and Billet (12), however, the limit they set is $\phi_d < 0.25$.

According to Table 4 the statistical analysis indicates that the Mackowiak and Billet's model (1986) presents the least RPF for both transfer directions.

Maximum Throughput

The maximum experimental capacity obtained for both transfer directions under study is shown in Fig. 8. In general, the maximum throughput was found between 39.1 and 60.5 m^3/m^2h for $d \rightarrow c$, and between 46.7 and 65.6 m^3/m^2h for the opposite one, when the phase ratio varied between 0.35 and 2. These values are of the same order of magnitude as that previously reported for this system and SMV packing (see Table 2).

Table 5. Flooding capacities

Mass-transfer direction	Phase ratio, λ (V_d/V_c), adim	Capacity at flooding, B_{max} ($m^3/(m^2 \cdot h)$)				
		Experimental	Streiff and Jancic	Maćkowiak and Billet	Seibert and Fair	Kumar and Hartland ($C_{4\ KH}$ modified)
$d \rightarrow c$	0.35	55.9	76.3	68.2	43.6	56.6
	0.45	60.5	74.5	64.2	43.6	54.8
	0.54	56.7	73.4	61.4	43.6	53.7
	0.75	56.7	72.1	57.5	43.6	52.3
	0.99	39.1	72.0	54.6	43.6	51.6
	1.50	50.2	72.5	51.4	43.6	51.6
	2.00	46.2	73.8	49.8	43.6	52.2
	AAPE		44	13	18	10
$c \rightarrow d$	0.35	65.6	71.4	67.2	35.2	57.7
	0.45	65.3	69.6	63.2	35.2	55.9
	0.55	58.6	68.5	60.4	35.3	54.7
	0.75	51.3	67.4	56.6	35.4	53.4
	1.00	46.7	67.0	53.8	35.5	52.7
	1.50	47.2	67.7	50.7	35.6	52.7
	2.00	47.7	69.0	49.1	35.7	53.3
	AAPE		28	6	34	10

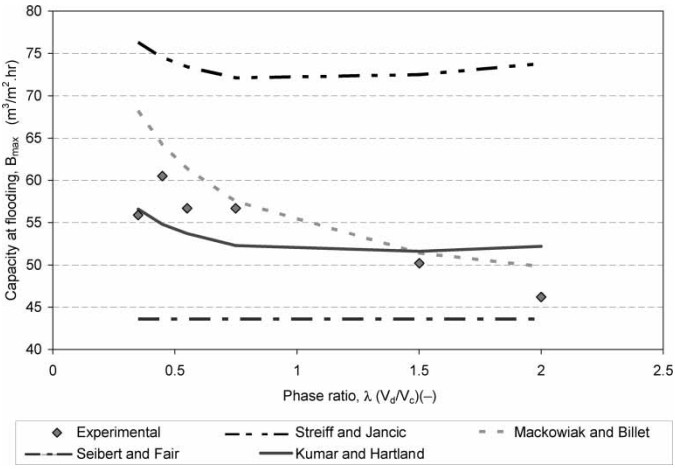

Figure 9. Capacity at flooding obtained with the different models (d → c).

Unfortunately, due to the little availability of published experimental data on this packing and system, it was not possible to assess the dual-flow plates effect on the maximum throughput.

A higher point dispersion for the d → c direction is clearly evidenced in Fig. 8, which makes it impossible to specify accurately the zone from which the maximum load remains independent of the phase ratio. For the c → d direction this value is approximately $47\,m^3/m^2h$.

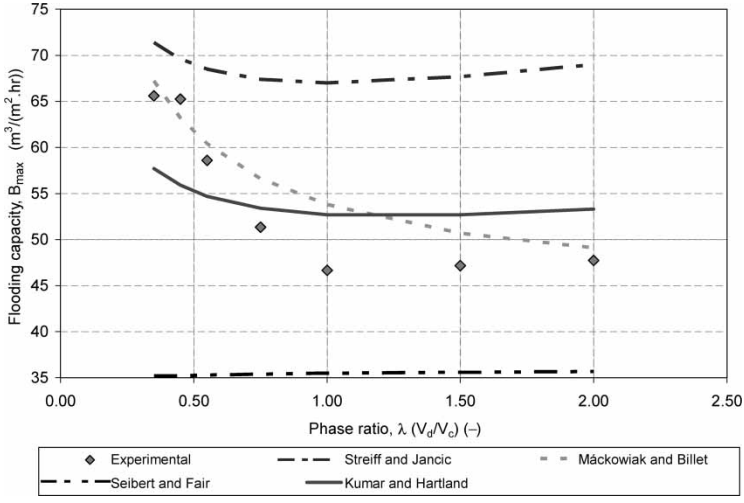

Figure 10. Capacity at flooding obtained with the different models (c → d).

Table 6. Relative performance factors (flooding)

Transfer direction	Model	Number of data points	E1	E2	E3	E4	E5	E6	FRP
d → c	Streiff and Jancic	7	43.5	43.5	123.00	2132	2132	6034	6
	Maćkowiak and Billet	7	12.5	12.5	35.4	597	597	1689	1.06
	Seibert and Fair	7	−14.8	18.1	41.8	−859	987	2429	1.83
	Kumar and Hartland	7	3.79	10.2	10.7	107.1	481	303	0
c → d	Streiff and Jancic	7	27.9	27.9	78.9	1403	1403	3971	4.55
	Maćkowiak and Billet	7	5.46	6.37	15.5	266.5	325	754	0.52
	Seibert and Fair	7	−33.8	33.8	95.7	−1920	1920	5433	6
	Kumar and Hartland	7	1.04	10.5	2.96	− 27.8	576	78.7	0.31

A higher maximum throughput should be expected for the d → c direction; however, no significant differences were observed between both transfer directions. This might be due, on the one hand, to the experimental data dispersion resulting from the uncertainty in the determination of the flooding exact moment, and, on the other, to the low acid concentration used. However, it is important to underscore that the experimental results follow the decreasing trend for phase ratios lower than the unit, as reported by the packing manufacturer (3).

The resulting values from the different models along with their deviations are presented in Table 5. Figures 9 and 10 present the maximum throughput values obtained with the different models for both mass-transfer directions. It can be observed that only the models of Mackowiak and Billet and Kumar and Hartland follow the trend of the experimental data.

It should be highlighted that the values given by the Kumar and Hartland's model (4) are those obtained when performing the adjustment procedure of the respective constant for every transfer direction, analogous to the one described before for holdup. Certainly, the adjustable C_{4KH} parameter [Eq. (1.18)] seems to be a function only of the packing type since its value (0.09) did not vary for any transfer direction, but was quite different to that reported by the authors (0.22). The adjustment improved with the new value of the C_{4KH} parameter, compared to the one reported by the authors (AAPE = 19.5%).

Since the models of Mackowiak and Billet (1986) and Kumar and Hartland (1994) present very similar errors which are considerably smaller than the other models, the statistical analysis proposed by Ansari et al. (16) was applied. Table 6 reflects the statistical analysis which indicates that Kumar and Hartland's model exhibits the smallest RPF for both transfer directions (RPF = 0,31 for c → d and 0 for d → c).

CONCLUSIONS

The conclusions listed below are restricted to the system and packing used in this study. Experimental values of the Streiff and Jancic's constant (C_p), of 0.65 (d → c) and 0.96 (c → d), were obtained. However, more experiments have to be conducted to corroborate these values and their dependence on the mass transfer direction.

Slip velocity and holdup predictions from different existing models are in agreement with the experimental data collected here, but for flooding capacity some models are not able to predict the descending trend for phase ratio lower than one. The Mackowiak and Billet's model for the holdup calculation provided the best adjustment to experimental data, according to the statistical analysis carried out, with a relative performance factor of 1.07 and 0.03 for d → c and c → d, respectively.

The maximum throughput was found between 39.1 and 60.5 m^3/m^2h for the $d \rightarrow c$ transfer direction and between 46.7 and 65.6 m^3/m^2h for the opposite direction, when the phase ratio varied between 0.35 and 2.0. No significant differences were observed between both transfer directions, probably due to the uncertainty regarding the exact flooding moment and the low concentration of acid employed.

The Kumar and Hartland's correlation for the flooding velocities calculation improved significantly its adjustment to the experimental data when the model constant was modified. Additionally, the statistic analysis indicated that this model is the most suitable one to predict the limiting throughput for this system and packing under the experimental conditions studied, with a relative performance factor of 0 and 0.31 for $d \rightarrow c$ and $c \rightarrow d$, respectively.

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NOMENCLATURE

Symbol	Variable	Units
a_p	Parking surface area per unit volume of column	(m^2/m^3)
AAPE	Absolute average percentage error	
B, $B_{máx}$	Sum of the superficial velocities, at any condition and at flooding	$(m^3/(hr \cdot m^2))$
C_{1KH}	Parameter of ec. (1.15)	(adim)
C_{2KH}	Parameter of ec. (1.17)	(adim)
C_{2MB}	Parameter of ec. (1.5)	(adim)
C'_{3KH}	Parameter of ec. (1.16)	(adim)
C'_{4KH}	Parameter of ec. (1.18)	(adim)
C_D	Drag coefficient	(adim)
C_F	Parameter of ec. (1.1)	(adim)
$C_{f,\,MB}$	Parameter of the packing at flooding condition, ec. (1.8)	(adim)
$C_{h,\,MB}$	Parameter of the packing geometry, ec. (1.7b)	(adim)
C_p	Parameter of ec. (1.2)	(adim)
C_s	Parameter of the packing and the system properties, ec. (1.7a)	(s/m)
d_h	Hydraulic diameter of packing, $d_h = \frac{4 \cdot \varepsilon}{a_p}$	(m)
d_{vs} o d_{32}	Sauter mean drop diameter	(m)

g	Acceleration due to gravity	(m/s^2)
m	Exponent on ec. (3)	(adim)
Re,i	Reynolds number based on phase "i"	(adim)
V_i	Superficial velocity of "i" phase $(m^3/(s.\ m^2))$	(m/s)
$V_{i,f}$	Superficial velocity of "i" phase at flooding $(m^3/(s \cdot m^2))$	(m/s)
V_k	Characteristic velocity, ec. (2,3)	(m/s)
V_{slip}	Slip velocity	(m/s)
$\overline{V}_{slip}$	Average slip velocity, ec. (1.2)	(m/s)
V_{so}	Terminal velocity	(m/s)
We_i	Weber number of "i" phase $= \frac{\rho_i \cdot V_i^2 \cdot d_{vs}}{\sigma}$	(adim)

Greek letters

Symbols	Variable	Units
α_{KH}	Parameter of ec. (1.18)	(adim)
$\Delta\rho$	Density difference between phases	(kg/m^3)
ε	Void fraction of packing	(adim)
ζ	Dimensionless tortuosity factor, ec. (1.10)	(adim)
η	Drop size correction factor ec. (1.9)	(adim)
λ	Phase ratio dispersed/continuous	(adim)
μ_i	Dynamic viscosity of "i" phase	(Pa.s)
μ_w	Dynamic viscosity of water at $20^\circ C = 0.9$ cP. ec. (1.15)	(Pa.s)
ρ_i	Density of "i" phase	(kg/m^3)
ρ_w	Density of water	(kg/m^3)
σ	Interfacial tension	(N/m)
ϕ_d	Fraction of dispersed phase holdup	(m^3/m^3)
$\phi_{d,f}$	Fraction of dispersed phase holdup at flooding	(m^3/m^3)
$\phi_{d,máx}$	Maximum fraction of dispersed phase, ec. (1.13)	(m^3/m^3)

Subscripts

c	Continuous phase
d	Dispersed phase
w	Water phase

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