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SLIP VELOCITY IN PULSED DISC AND DOUGHNUT EXTRACTION COLUMNS

In the present work, slip velocity has been measured in a 76 mm diameter pulsed disc and doughnut extraction column for four different liquid-liquid systems. The effects of operating variables including pulsation intensity and dispersed and continuous phase flow rates on slip velocity have been investigated. The existence of three different operational regimes, namely mixer-settler, transition, and emulsion regimes, was observed when the energy input was changed. Empirical correlations are derived for prediction of the slip velocity in terms of operating variables, physical properties of the liquid systems, and column geometry for different regimes. Good agreement between prediction and experiments was found for all operating conditions that were investigated.

Key words: dispersed phase hold-up; pulsed disc and doughnut column; slip velocity; characteristic velocity.

Liquid-liquid extraction has long been a key operation in many separation processes found in the chemical, pharmaceutical, environmental, oil, food, nuclear, and hydrometallurgical industries for product purification and/or raw material recovery. Two-phase liquid-liquid countercurrent column extractors have been widely used and have been extensively investigated. The pulsed disc and doughnut column is a development of a pulsed column originally described by Van Dijck [1]. This column has been extensively applied to reprocess the spent nuclear fuel in France and Japan [2,3]. On an industrial scale, the column is employed for uranium separation process by Western Mining Corporation (WMC) at Olympic Dam Operations, South Australia [4]. In comparison to mixer-settlers, this extractor has been stated to have several advantages, *viz*. its lower cost, simplicity of design, less space consumption, and reduction of organic loss [5].

Prediction of dispersed phase hold-up and droplet velocity relative to the continuous phase is of fundamental importance in the design and operation of liquid-liquid extraction. The slip velocity controls the mass transfer coefficient when a solute is transferred between two phases and the holdup together with the drop size determines the interfacial area so that the total mass transfer may be obtained [6-8].

The direct measurement of slip velocity is difficult, so it may be obtained from the flow rates of the dispersed and continuous phase, V_d and V_c, respectively, and dispersed phase hold-up, x_d. For countercurrent flow, it is defined by:

$$V_s = \frac{V_d}{x_d} + \frac{V_c}{1 - x_d} \tag{1}$$

which assumes that the phase velocities are constant across the column cross-section (implying constant drop size) and neglects any circulation effects. The slip velocity can thus be estimated if the hold-up is known.

Slip velocity characteristics of different types of extraction column such as pulsed sieve plate columns [9-11], rotating disc contactors [12,13], Kühni columns [14,15], Karr reciprocating-plate columns [16,17] have been studied experimentally, but few data are available on the prediction of hold-up and slip velocity in pulsed disc and doughnut columns. Many investigations have been concentrated on the development of numerical solutions to describe the operational characteristics of the column [18-20], however no firm design and scale-up criteria have yet been proposed. For the purpose of establishing proper design procedure, there is thus a need for reliable correlations of hold-up and slip velocity in terms of operating variables, liquid physical properties and column geometry.

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In the present work, results are reported on slip velocity determinations carried out in a 76 mm nominal diameter pulsed disc and doughnut extraction column for four liquid-liquid systems. The present study has examined the influence of operating variables including the pulsation intensity, as well as the solvent and aqueous flow rates on the slip velocity. An attempt is also made to develop an empirical correlation for estimation of slip velocity in terms of liquid physical properties, operating conditions, and column geometry.

EXPERIMENTAL

Figure 1 shows a schematic arrangement of the experimental apparatus. The main column section consisted of a 74 cm long glass tube of 76 mm internal diameter, enclosing a stack of 30 pairs of disc and doughnut, made of 2 mm stainless steel sheet. The discs and doughnuts were arranged alternately and spaced 10 mm apart, resulting in a 20 mm compartment height, they were held in place by means of three 3.2 mm o.d. SS tie rods with SS spacer sleeves. Two different series of discs and doughnuts were used in the experiments. The discs were 67 and 63 mm in diameter and the doughnut apertures were 36 and 42 mm, giving an open free area of 23.5 and 30.5%, respectively. A settler of 112 mm diameter at each end of the column permitted the liquids to coalesce and decanted separately. The column was pulsed by blowing air at the required amplitude and frequency into pulse leg. The air pressure was controlled by a regulator to provide pulses of the required amplitude in the column, while the frequency of the pulses was controlled using two solenoid valves. The inlet and outlet of the column were connected to four tanks, each of 80 l capacity. The flow rates of both phases were controlled via rotameters. The liquid-liquid interface was maintained approximately 250 mm above the top compartment. This was achieved by using an optical sensor. A solenoid valve (a normally closed type) was provided at the outlet stream of heavy phase. This valve received electronic signals from the sensor. When the interface location was going to change, the optical sensor sent a signal to the solenoid valve and the aqueous phase was allowed to leave the column by opening the diaphragm of the solenoid valve. The organic phase was allowed to leave the column *via* overflow.

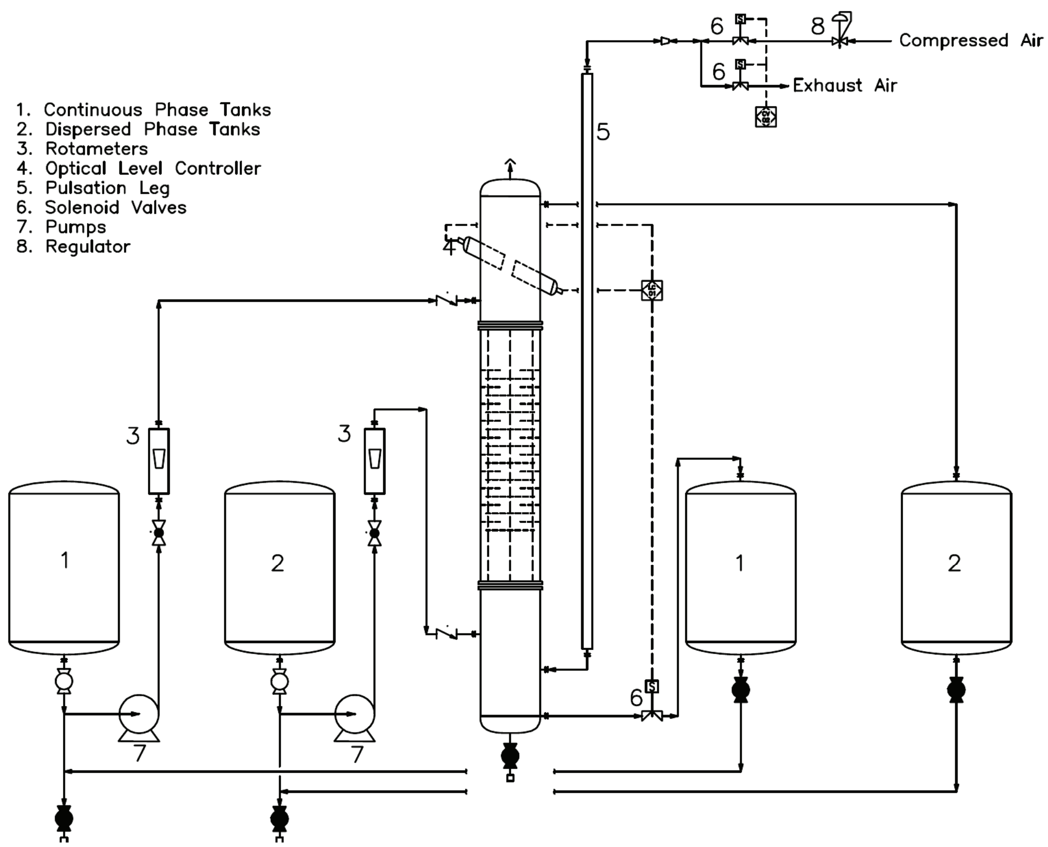

Figure 1. Schematic diagram of the pulsed disc and doughnut column.

The liquid-liquid systems that studied were kerosene (Shellsol 2046)-water, toluene-water, butyl acetate-water, *n*-butanol-water. With respect to the interfacial tension, they cover a range from 1.75 to 46.5×10^{-3} N/m. The major part of industrially used solvents for extraction is thus covered. Distilled water was used as the continuous phase and technical grades solvents of at least 99.9 mass% purity were used as the dispersed phase. The physical properties of the liquid-liquid systems used in the experiments are given in Table 1. The physical properties were obtained under equilibrium conditions.

Before starting a series of experiments, the aqueous and organic phases were recirculated through the column for several times to ensure mutual saturation. After filling the column with the aqueous phase, the amplitude and frequency of pulsation were then adjusted to the desired values. The organic dispersed phase introduced at the bottom of the column. The system was allowed to reach steady state, which usually necessitated three to four changes of the column volume. Holdup measurements were obtained using the shut down (displacement) method. In order to use this method, at the end of a run, the inlet and outlet valves were shut simultaneously and the dispersed phase was allowed to disengage to the interface at the top of the column. A period of 10-15 min was allowed for the dispersed phase to settle. The change in interface height between operation and after settling was measured and then converted into the corresponding volume to determine hold-up. In the settler of an extraction column, there are no internals and agitation and the droplets are very large compared to the active part of the column. Consequently, the value of the holdup is very low in the settler compared to the active part of the column. So, the method has enough accuracy for measuring of holdup in the column.

All the experiments were carried out far from flooding conditions. The values of slip velocity were calculated using Eq. (1). In order to consider the effect of column geometry on slip velocity characteristics and also to increase the total number of data points, the experimental data are taken from van Delden *et al.* [23] obtained for the extraction of caprolactam with toluene.

RESULTS AND DISCUSSION

Visual observation of the column in operation with these three liquid systems indicated that three regimes, mixer-settler, dispersion (transition), and emulsion, occurred depending upon the pulsation intensity. A stepwise movement of the droplets within the column indicated the occurrence of mixer-settler regime (Figure 2a). The existence of dispersion regime revealed by non-uniform drop size distribution and no coalescence of dispersed phase droplets (Figure 2b). The emulsion regime followed the dispersion regime and was characterized by uniform distribution of dispersed phase drops in the continuous phase in each compartment of the column (Figure 2c).

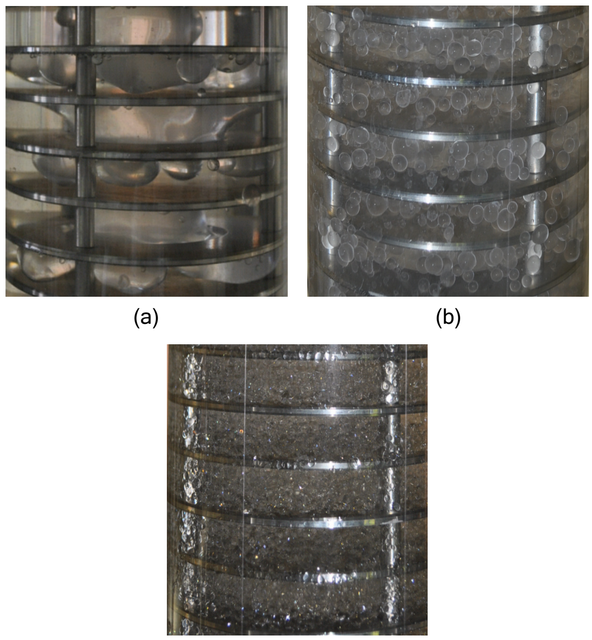

Figure 2. Existence regimes by the pulsed disc and doughnut column: a) mixer-settler, b) transition and c) emulsion regimes.

Typical variation of slip velocity with pulsation intensity is given in Figure 3. In the mixer-settler region of operation, occurring at low agitation levels, holdup is high due to the presence of thick layers of the dispersed phase under the discs and doughnuts. As pulsation rate increases, large drops are generated due

Table 1. Physical properties of the chemicals used at 20 °C [21,22]

Physical property	Kerosene-water	*n*-Butanol-water	*n*-Butylacetate-water	Toluene-water
ρ_c / kg m^{-3}	998	985.6	997.6	998.2
ρ_d / kg m^{-3}	804	846	880.9	865.2
μ_c / mPa s	1.00	1.426	1.0274	0.963
μ_d / mPa s	1.66	3.364	0.734	0.584
σ / mN m^{-1}	46.5	1.75	14.1	36

to pulsation do not have enough time to collect under internals. These large drops have low residence time and they have high rising velocity. Therefore, in the mixer-settler regime, the slip velocity increases with increasing pulsation intensity until a maximum is reached, corresponding to beginning of dispersion region. In transition regime, with increasing pulsation intensity the drop size and, as a consequence, the drop velocity are reduced. For still higher pulsation rates corresponding to the emulsion region, the holdup increases rapidly due to formation of very small drops. Consequently, slip velocity decreases with an increase in pulsation intensity.

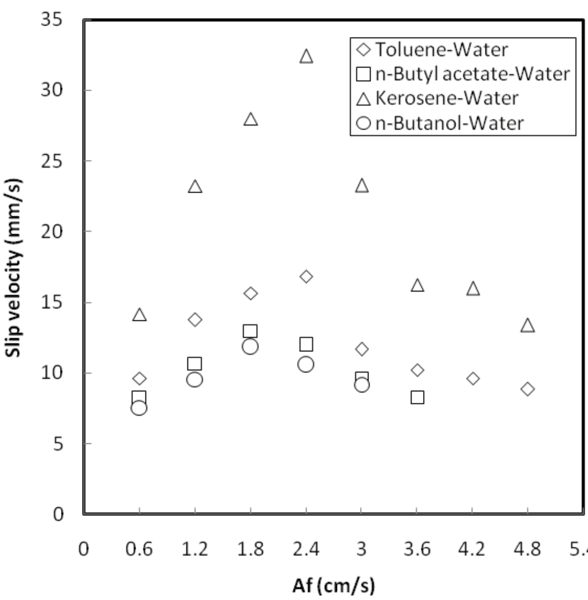

Figure 3. Effect of pulsation intensity on slip velocity ($V_c = V_d = 1.132$ mm/s).

Figure 3 also shows the effect of interfacial tension on slip velocity. It is well known that larger drops are produced from high interfacial tension system than are generated from lower interfacial tension system. On this basis, slip velocity increases with an increase in interfacial tension.

The effect of dispersed phase velocity on slip velocity is shown in Figure 4. As can be seen in this figure, slip velocity increases with increasing of dispersed phase velocity. The number of dispersed drops increases with increase in dispersed phase velocity and consequently hold-up of dispersed phase increases according to its definition. However, an increase in dispersed phase flow rate tends to increase the drop size. A higher dispersed phase flow rate leads not only to a larger drop formation diameter, but also to higher coalescence frequencies. In the present case, the effect of drop size velocity is larger than that

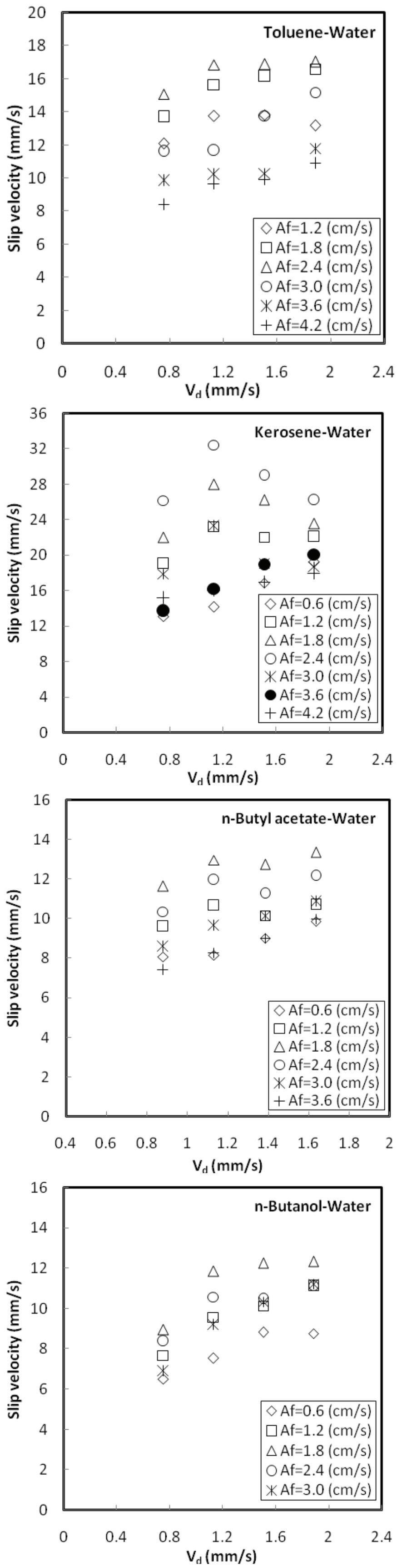

Figure 4. Effect of dispersed phase velocity on slip velocity ($V_c = 1.132$ mm/s).

of hold-up, and consequently slip velocity increases with increase in dispersed phase velocity in most experiments. Similar results were obtained in some other types of extraction columns [9].

The effect of continuous phase velocity on slip velocity is given in Figure 5. By increasing the continuous phase velocity, the drag force between the dispersed drops and continuous phase increases, so the drop movement will be limited and the residence time will increase. Consequently, the value of slip velocity decreases with increasing of continuous phase velocity.

The effect of fractional free area on slip velocity is shown in Figure 6. Similar to other types of extraction columns, slip velocity increases with an increase in fractional free area. An increase in the fractional free area (or doughnut aperture diameter) reduces the shear forces on drops resulting in an increase in drop size, which leads to increment in the slip velocity.

One of the main objectives of this study is to derive empirical correlation to represent the slip velocity in pulsed disc and doughnut columns. For this reason, empirical correlations are derived for dispersed phase holdup for different operating regimes in the present work:

$$V_s = 47.32\left(\frac{\mu_c^4 g}{\Delta\rho\sigma^3}\right)^{-0.34}\left(1+\frac{V_d}{V_c}\right)^{0.39}\left(\frac{\Delta\rho}{\rho_c}\right)^{0.79}\left(\frac{\rho_c\sigma^4}{\psi\mu_c^5}\right)^{-0.14}\left(\frac{d_a\rho_c\sigma}{\mu_c^2}\right)^{-0.23} \quad \text{(mixer-settler regime)} \qquad (2)$$

$$V_s = 159.81\left(\frac{\mu_c^4 g}{\Delta\rho\sigma^3}\right)^{0.08}\left(1+\frac{V_d}{V_c}\right)^{0.42}\left(\frac{\Delta\rho}{\rho_c}\right)^{1.58}\left(\frac{\rho_c\sigma^4}{\psi\mu_c^5}\right)^{0.18}\left(\frac{d_a\rho_c\sigma}{\mu_c^2}\right)^{-0.34} \quad \text{(dispersion and emulsion regimes)} \qquad (3)$$

where ψ is power input per unit mass. Jealous and Johnson [24] were the first to consider power dissipation in pulsed sieve-plate extraction columns. They derived an equation for instantaneous power dissipation by assuming that the friction effects in a perforated plate were similar to those in an orifice. The in-

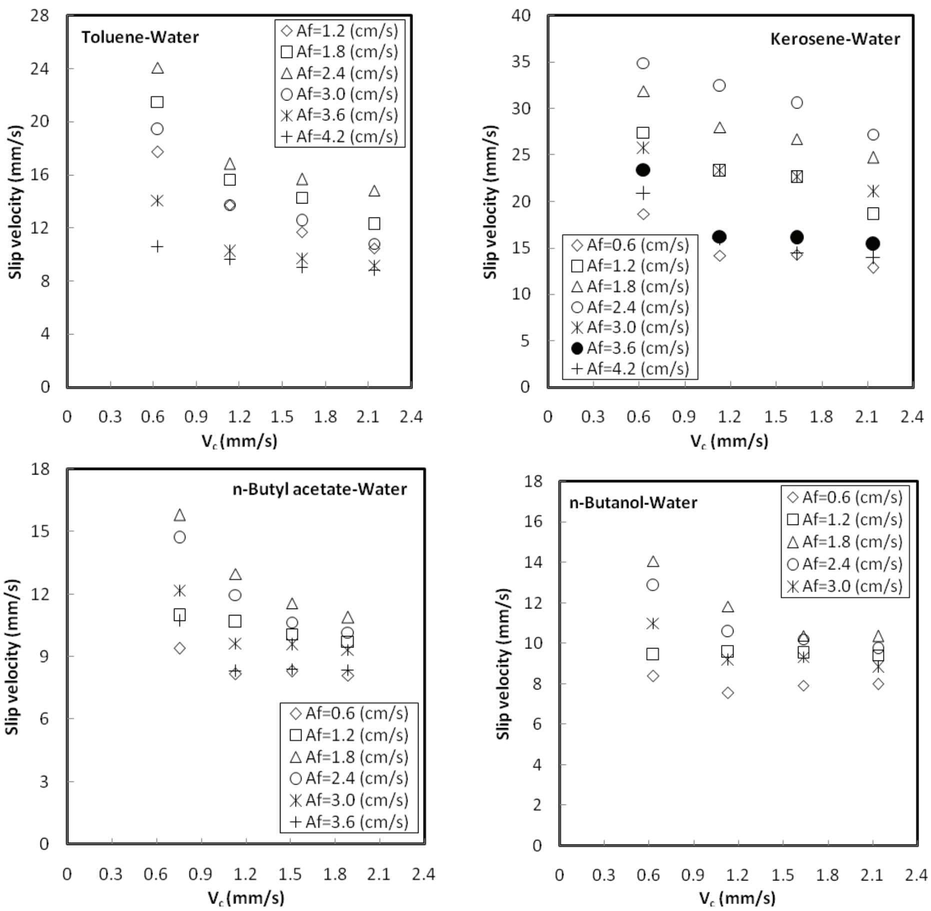

Figure 5. Effect of continuous phase velocity on slip velocity (V_d = 1.132 mm/s).

tegrated form of the Jealous and Johnson (1955) for ψ, as obtained by Hafez and Baird [25] for a sinusoidal wave, is:

$$\psi = \frac{2\pi^2\left(1-e^2\right)\left(Af\right)^3}{3h_c C_O e^2} \quad (4)$$

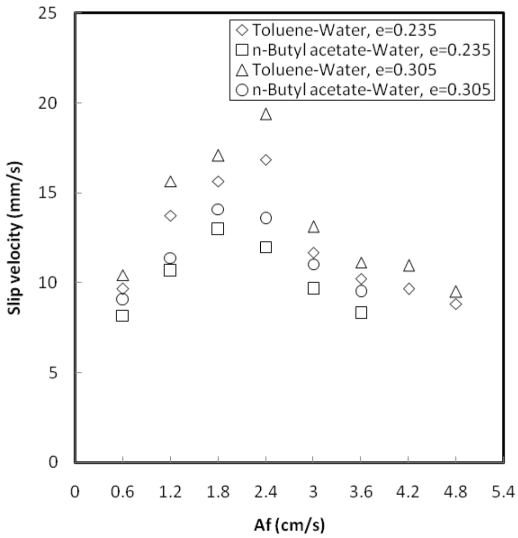

Figure 6. Effect of fractional free area on slip velocity (V_c = V_d = 1.132 mm/s).

Equations (2) and (3) are derived based on the experimental results of the present work and the data taken from van Delden *et al.* [23]. The comparison of experimental data with those calculated by Eqs. (2) and (3) is shown in Figure 7. This figure shows that the experimental results are in good agreement with calculated values obtained by applying the presented correlations. These correlations reproduce the experimental results with an average relative deviation of 10.96%.

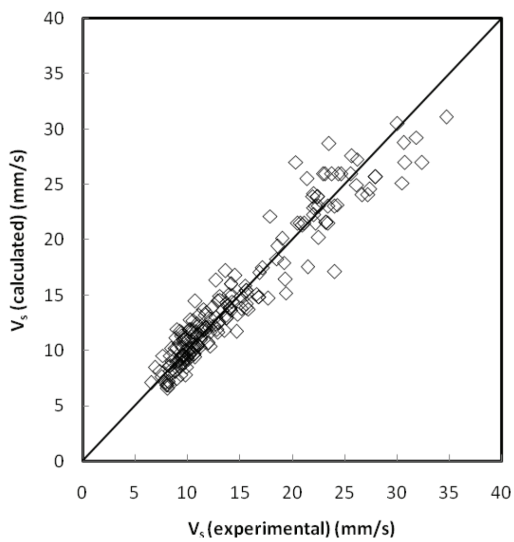

Figure 7. Comparison of experimental data of slip velocity with calculated values.

CONCLUSIONS

This paper presented an experimental study on slip velocity of a pulsed disc and doughnut extraction column. The results showed that depending on pulsation intensity and throughput the column can be operated in different flow regimes, the so-called mixer-settler or dispersion or emulsion regimes. The results showed that slip velocity decreased with increasing pulsation intensity towards mixer-settler regime, until finally a maximum was reached corresponding to the transition regime. Following this, the slip velocity increased with further increase in pulsation intensity, corresponding to emulsion regime. Slip velocity was also found to increase with increases in dispersed phase velocity and interfacial tension, while it decreased with an increase in continuous phase velocity. Empirical correlations are derived for prediction of slip velocity for design purposes. Since there is little experimental data on this type of extraction column, the present work is of use to those looking to use this type of contactor.

Nomenclature

A	Amplitude of pulsation (m)
C_O	Orifice coefficient
d_a	Doughnut aperture diameter (m)
e	Fractional free area
f	Frequency of pulsation (s^{-1})
g	Acceleration due to gravity (m/s^2)
h_c	Compartment height (m)
V	Superficial velocity (m/s)
V_s	Slip velocity (m/s)
x_d	Dispersed phase hold-up

Greek symbols

σ	Interfacial tension (N/m)
$\Delta\rho$	Density difference between phases (kg/m^3)
μ	Viscosity (Pa s)
ρ	Density (kg/m^3)
ψ	Power dissipated per unit mass (m^2/s^3)

Subscripts

c	Continuous phase
d	Dispersed phase

REFERENCES

[1] W.J.D. Van Dijck, US Patent 2,011,186 (1935)

[2] G. Angelov, E. Journe, A. Line, C. Gourdon, Chem. Eng. J. **45** (1990) 87-97

[3] G. Angelov, C. Gourdon, A. Line, Chem. Eng. J. **71** (1998) 1-9

[4] A. Jahya, G.W. Stevens, H.R.C. Pratt, Solvent Extr. Ion Exc. **27** (2009) 63-82

[5] R.L. Movsowitz, R. Kleinberger, E.M. Buchalter, Bateman Projects Ltd., Halfa, Israel, 1997

[6] J.C. Godfrey, M.J. Slater, Chem. Eng. Res. Des. **69** (1986) 130-141

[7] A. Kumar, S. Hartland, Ind. Eng. Chem. Res. **34** (1995) 3925-3940

[8] E. Hashtochahar, S.J. Safdari, A. Haghighiasl, M. Torab-Mostaedi, Can. J. Chem. Eng. **88** (2010) 807-818

[9] A. Kumar, S. Hartland, In Liquid-Liquid Extraction Equipment, J.C. Godfrey, M.J. Slater, Eds., John Wiley and Sons, New York, USA, 1994, pp. 625-735

[10] A. Kumar, S. Hartland, Chem. Eng. Res. Des. **61** (1983) 248-252

[11] A. Kumar, S. Hartland, Chem. Eng. Process **23** (1988) 41-59

[12] P. Kalaichelvi, T. Murugesan, J. Chem. Tech. Biotechnol. **69** (1997) 130-136

[13] A. Kumar, S. Hartland, Chem. Eng. Commun. **56** (1987) 87-106

[14] A. Kumar, Ph.D. Thesis No. 7806, Swiss Federal Institute of Technology, Zürich, Switzerland, 1985

[15] P.J. Bailes, J. Gledhill, J.C. Godrey, M.J. Slater, Chem. Eng. Res. Des. **64** (1986) 43-55

[16] A. Kumar, S. Hartland, Can. J. Chem. Eng. **67** (1989) 17-25

[17] M.C. Camurdan, M.H.I. Baird, P.A. Taylor, Can. J. Chem. Eng. **67** (1989) 554-559

[18] M.S. Aoun Nabli, P. Guiraud, C. Gourdon, Chem. Eng. Res. Des., A **76** (1998) 951-960

[19] M.S. Aoun Nabli, P. Guiraud, C. Gourdon, Chem. Eng. Sci. **52** (1997) 2353-2368

[20] N. Bardin-Monnier, P. Guiraud, C. Gourdon, Chem. Eng. Process **42** (2003) 503-516

[21] T. Míšek, R. Berger, J. Schröter, EFCE Publ. Ser. **46** (1985) 1-20

[22] A. Jahya, G.W. Stevens, H.R.C. Pratt, in Proceedings Chemeca '99, Newcastle, Australia, 1999

[23] M.L. van Delden, G.S. Vos, N.J.M. Kuipers, A.B. de Haan, Solvent Extr. Ion Exc. **24** (2006) 519-538

[24] A. C. Jealous, H.F. Johnson, Ind. Eng. Chem. **47** (1955) 1159-1166

[25] M.M. Hafez, M.H.I. Baird, Chem. Eng. Res. Des. **56** (1978) 229-238.

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NAUČNI RAD

RELATIVNA BRZINA IZMEĐU FAZA U PULZIRAJUĆIM EKSTRAKCIONIM KOLONAMA SA DISKOVIMA I PRSTENOVIMA

U ovom radu merena je relativna brzina između faza u pulzirajućoj ekstrakcionoj koloni sa diskovima i prstenovima, prečnika 76 mm, za četiri različita sistema tečno-tečno. Ispitivan je uticaj operativnih promenljivih, uključujući intenzitet pulzacije i protoke dispergovane i kontinualne faze, na relativnu brzinu između faza. Konstatovana je pojava tri različita režima proticanja, pri promeni energije unete u sistem, i to: mešanje-odvajanje, prelazni i emulzioni. Izvedene su empirijske korelacije za određivanje relativne brzine između faza u funkciji operativnih promenljivih, fizičkih osobina tečnih sistema i geometrijskih karakteristika kolone, za različite režime proticanja. Zabeleženo je dobro slaganje predskazanih i eksperimentalnih vrednosti pri svim ispitivanim eksperimentalnim uslovima.

Ključne reči: sadržaj dispergovane faze, pulzirajuća kolona sa diskovima i prstenovima, relativna brzina između faza, karakteristična brzina.