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FLOODING CHARACTERISTICS IN PULSED PACKED EXTRACTION COLUMNS

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Abstract - Flooding behavior of a 76.2 mm diameter pulsed packed column has been determined using four different liquid-liquid systems. The effects of pulsation intensity, flow ratio, interfacial tension, and packing geometry on flood point have been investigated. The results showed that the maximum throughput of the column decreases with an increase in pulsation intensity and flow ratio, while it increases with an increase in interfacial tension. The applicability of the characteristic velocity method to this type of column for the analysis of the flood point was examined and a marked deviation was observed between experimental results and values calculated by this method. Two new empirical correlations for flooding velocity and holdup at flooding are derived in terms of operating variables, packing characteristics, and physical properties of the liquid systems. Good agreement between prediction and experiments has been found for all operating conditions that were investigated.

Keywords: Pulsed packed column; Flooding; Holdup; Characteristic Velocity; Throughput.

INTRODUCTION

Liquid-liquid extraction processes are widely used in different industries, especially in rare earth separation, hydrometallurgical processes, and the nuclear industry. Optimal design of a solvent extraction column involves maximizing the column performance by increasing the rate of mass transfer while achieving high throughputs. With these ideas in mind, Van Dijck (1935) proposed that the volumetric efficiency of a perforated plate could be improved by either pulsing the liquids or reciprocating the plates. Chantry *et al*. (1955) have shown that the height of a packed column required to effect a given degree of extraction is reduced by a factor of three under pulsation. The pulsed columns have a clear advantage over other mechanical contactors when processing corrosive or radioactive solutions since the pulsing unit can be remote from the column. The absence of moving mechanical parts in such columns obviates repair and servicing. These advantages led to development of the pulsed packed column, which has found wide application in a number of industries, including petrochemical, metallurgical, nuclear and chemical industries (Hussain *et al*., 1988).

The design of counter-current flow liquid-liquid extraction columns requires the determination of a suitable cross-sectional area for flow and the height required to achieve a specified degree of mass transfer. Calculation of cross-sectional area is much easier and more reliable than estimation of column height, but at present considerable uncertainty exists unless pilot plant results are used as a basis for design (Godfrey and Slater, 1991). From the industrial point of view, it is desirable that the column should attain high separation efficiency and a large throughput. However, it is not possible to increase the flow rate of the phases indefinitely, since there is a limit to the amount of one phase that can be dispersed in the second phase. When this limit

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*To whom correspondence should be addressed (flooding point) is exceeded, dispersed phase drops can no longer fall or rise against the flow of the continuous phase and stable column operation is unattainable. The maximum volumetric capacity of an extractor, or flooding throughput, is thus the first question that must be dealt with in design of an extraction column (Berger and Walter, 1985). Column diameter is then usually calculated from the knowledge of the column flooding behavior by assuming operating loads as a fraction of flooding throughput or else by considering the operating holdup value as a fraction of the dispersed phase volume fraction at flooding.

When flooding is reached, accumulations of the dispersed phase appear at individual points in the column and these block counter-currents; sometimes a phase inversion can also be observed (Rincón-Rubio *et al*., 1993; Torab-Mostaedi *et al*., 2009). Although pulsed columns can use sieve plate, packing and discs and doughnuts, most investigations reported have dealt with the sieve plate type (Yadav and Patwardlan, 2008; Jahya *et al*., 2009; Jie and Weiyang, 2000) and little information has appeared in the literature regarding the performance of pulsed packed columns. Thus, for the purpose of the establishing proper design procedures for pulsed packed extraction columns, there is a need for sound correlations of flooding velocity and flooding holdup as a function of operating conditions, packing geometry and the physical properties of the liquid phases.

This paper describes an investigation of the flooding behavior of a pulsed packed column as a function of pulsation intensity, phase flow rates and packing characteristics with four different systems. The applicability of the characteristic velocity method to this type of column for analysis of the flood point is examined. New empirical correlations are developed for flooding velocity and holdup at flooding in terms of operating variables, physical properties of the systems and packing characteristics.

EXPERIMENTAL

Description of Equipment

A schematic diagram of the experimental apparatus is shown in Fig. 1. The column, filled with a 2.0m height of packing, had an internal diameter of 76.2 mm and was made of stainless steel. A settler of 127 mm diameter at each end of the column permitted the liquids to coalesce and be decanted separately. The column was pulsed by blowing air at the required amplitude and frequency into the pulse leg. The air pressure was controlled to provide pulses of the required amplitude in the column while the frequency of the pulses was controlled by using two solenoid valves. The inlet and outlet of the column were connected to four tanks, each of 100 liters capacity. The interface location of the two phases was automatically controlled by an optical sensor. An inverted U-tube was used for measuring the pressure drop along the column. The pulsation frequency was measured automatically by using a mini PLC and the pulsation amplitude was obtained by measuring the volume change in the pulse leg and obtaining the volume change within the column from this value. The amplitude used in all experiments was 20 mm and the range of pulsation frequency was 0.3 to 1.6 1/s.

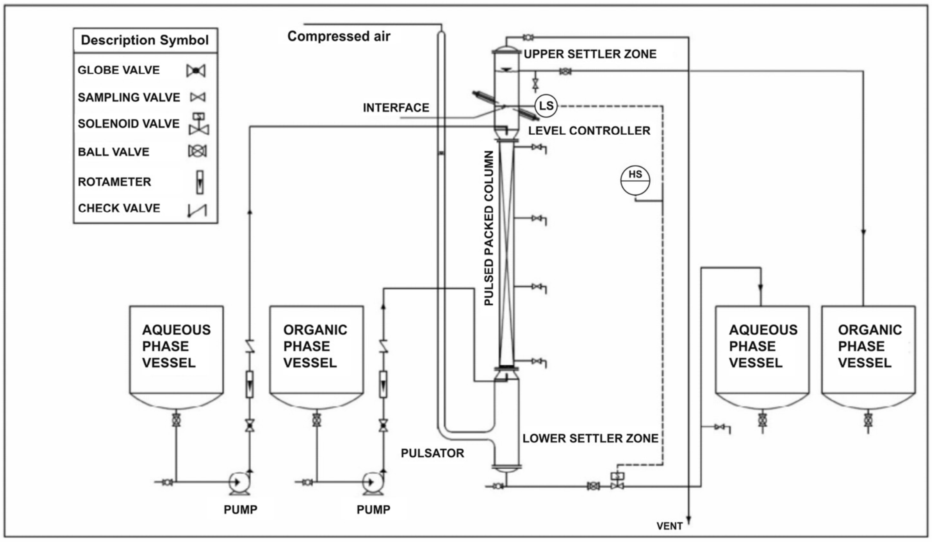

Figure 1: Schematic diagram of the pulsed packed column (LS: Level Switch; HS: Hand Switch)

The properties of the packings used in this work are listed in Table l.

Table 1: Geometrical characteristics of the packings studied.

Packing material	Packing surface (m^2/m^3)	Void fraction (-)
6.3-mm ceramic Raschig ring	595.74	0.46
9.5-mm stainless steel Raschig ring	421.15	0.80

Liquid-Liquid Systems

Four test systems were chosen to cover a range of values of interfacial tension and the recommendations of the European Federation of Chemical Engineering (Míšek *et al.*, 1985) were followed. The systems were kerosene-water, toluene-water, butyl acetate-water and 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 solvents used industrially for extraction is thus covered. Physical properties of these systems are listed in Table 2. Technical grade solvents of at least 99.5 %wt purity were used as the dispersed phase and distilled water was used as the continuous phase.

Procedure

The flood point was determined by indirect measurement of the holdup via the differential pressure. The measurement of the differential pressure proved to be the most rapid and the most exact method for measuring the flood point (Berger and Walter, 1985). In addition, it has the advantage that it can also be used for plant columns, where an observation of drop phase is impossible.

Before carrying out the runs, both phases were mutually saturated with one another. The flood points were then determined by setting the continuous phase flow rate to a desired condition and gradually increasing the dispersed phase flow rate until reaching the desired value. The differential pressure was read and the dispersed phase flow rate increased to its new value until a steady increase in the differential pressure was observed and this clearly signaled flooding. The reproducibility of flooding rates was within ±5%.

When flooding occurred, the dispersed phase and continuous phase inlet and outlet valves were quickly shut. The dispersed phase coalesced at the interface, after which the holdup at flooding was obtained by determining the change of interfacial height.

In each run, after reaching steady state at the desired flow rates, dispersed phase holdup was also measured by a sampling method. Seven samplers were arranged at equal distances along the column. Dispersed phase holdup was measured by rapid withdrawal of 50 to 100 ml samples from the samplers. In this method, the holdup was defined as the ratio of the dispersed phase volume to the total volume of the liquid sample.

Data Analysis

The average absolute value of the relative deviation, AARD, is used to compare the predicted results with the experimental data. This is defined as follows:

$$\mathrm{AARE} = \frac{1}{\mathrm{NDP}} \sum_{i=1}^{\mathrm{NDP}} \frac{|\text{predicted value} - \text{experimental value}|}{\text{experimental value}}$$

in which NDP is the number of data points.

Table 2: Physical properties of systems studies at 20°C (Míšek *et al.*, 1985.; Jahya *et al.*, 1999)

Physical property	Toluene-water	n-Butylacetate-water	n-Butanol-water	Kerosene-water
$\rho_c (kg/m^3)$	998.2	997.6	985.6	998
$\rho_d (kg/m^3)$	865.2	880.9	846	804
μ_c(mPa.s)	0.963	1.0274	1.426	1.00
μ_d(mPa.s)	0.584	0.734	3.364	1.66
σ(mN / m)	36	14.1	1.75	46.5

RESULTS AND DISCUSSION

The effect of pulsation intensity on flooding velocity is shown in Figure 2. As seen in this figure, the higher pulsation intensities, which produced smaller drops, result in lower flooding velocities. The rise of pulsation intensity results in higher shear stress and intense drop breaking. It appears that the number of drops in the column increases due to the reduction of relative velocity between the dispersed phase and continuous phase, which results in an increasing hold-up, such that the critical hold-up is reached at a lower total throughput. It is also found that flooding velocities decrease with an increase in the flow ratio of the dispersed phase to continuous phase. Dispersed phase holdup increases with an increase in flow ratio and, consequently, the column becomes unstable at lower dispersed phase flow rate.

Moreover, when the continuous phase is an aqueous solution of low viscosity, the rising velocity of the dispersed drops depends on the drop diameter, while the drop diameter in the column changes with the interfacial tension. Therefore, the holdup of the dispersed phase and, consequently, the flooding point are expected to vary with interfacial tension. It is known that, upon increasing interfacial tension, the size of drops is increased and the residence time of them in the column is decreased. Hence, the relative velocity between two phases increases and, consequently, the value of the dispersed phase holdup is decreased and the column operation is more stable. Therefore, lower interfacial tension results in lower total allowable throughput than in the case of higher interfacial tension, due to the smaller drops.

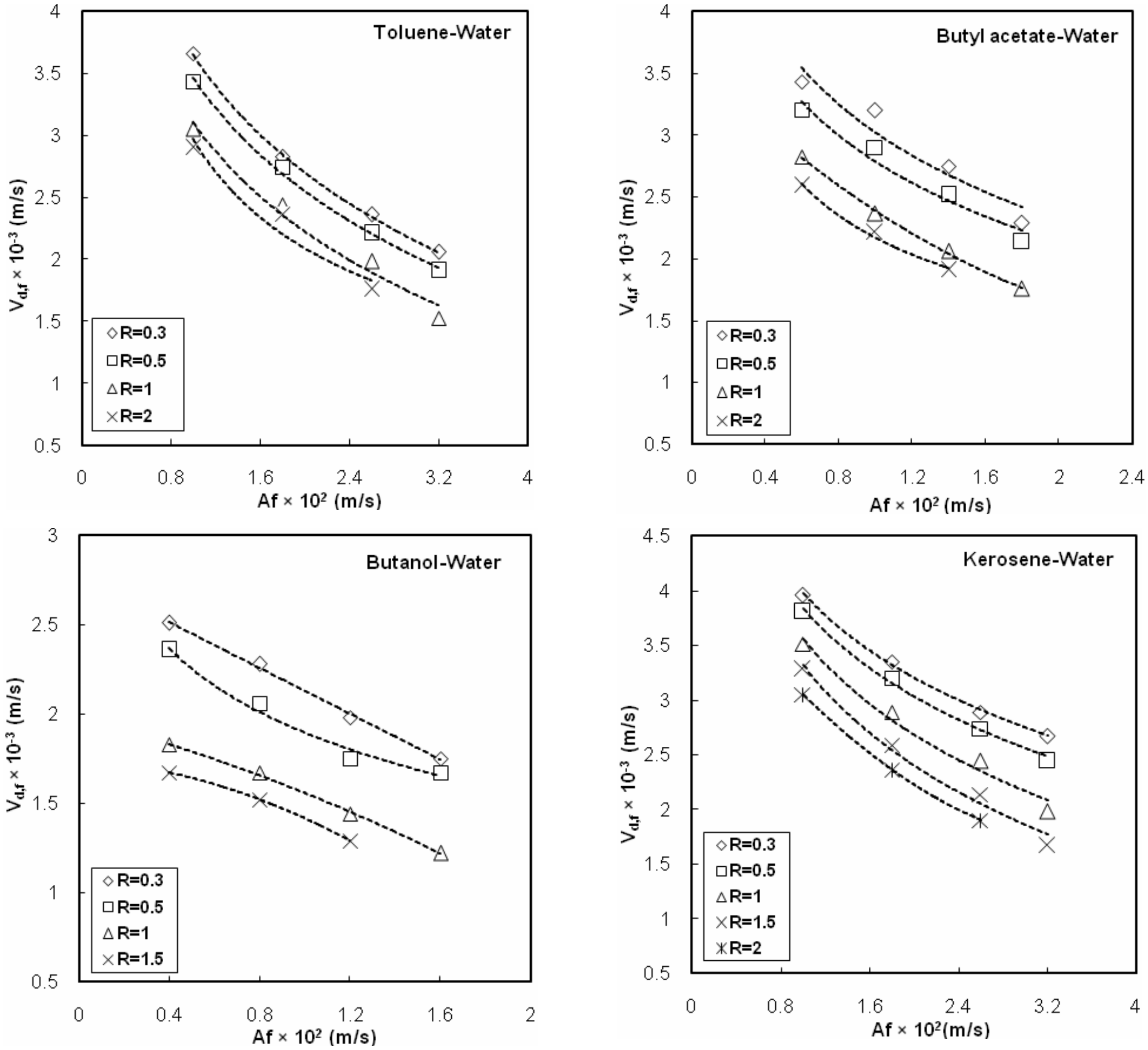

Figure 2: Effect of pulsation intensity and dispersed/continuous phase ratio (R) on the velocity of the dispersed phase at flooding

The effect of pulsation intensity on holdup at flooding is given in Figure 3. As shown in this figure, holdup at flooding increases with an increase in pulsation intensity due to the smaller drops. This figure also shows that the maximum dispersed phase holdup increases with an increase in solvent flow ratio. However, the value of holdup at flooding increases upon decreasing interfacial tension due to the formation of smaller drops.

Different packings have different interactions with the drops, because of their differences in void fraction, surface area, and geometry. Therefore, the packing type has some influence on the flood point. Figure 4 shows that the flooding velocity of the column with stainless steel Raschig rings is about 30% higher than that of the same column with ceramic Raschig rings, while holdup at flooding of the column with stainless steel Raschig rings is lower than that with ceramic Raschig rings. This is mainly because the stainless steel Raschig rings have a higher void fraction than the ceramic Raschig rings, which causes the dissipated energy per unit mass in the pulsed packed column with the S.S. Raschig rings to be much lower than with ceramic Raschig rings.

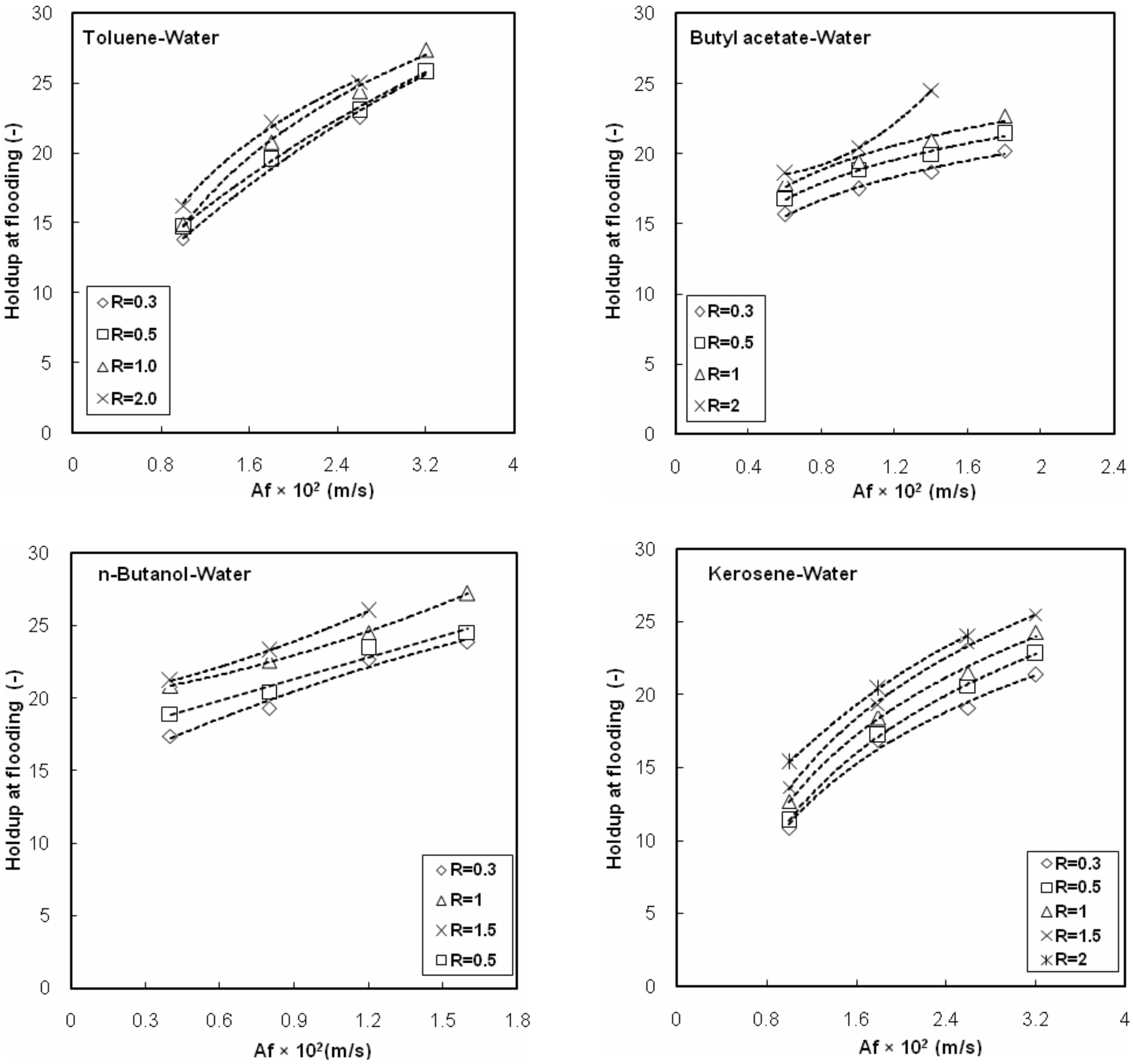

Figure 3: Effect of pulsation intensity on holdup at flooding

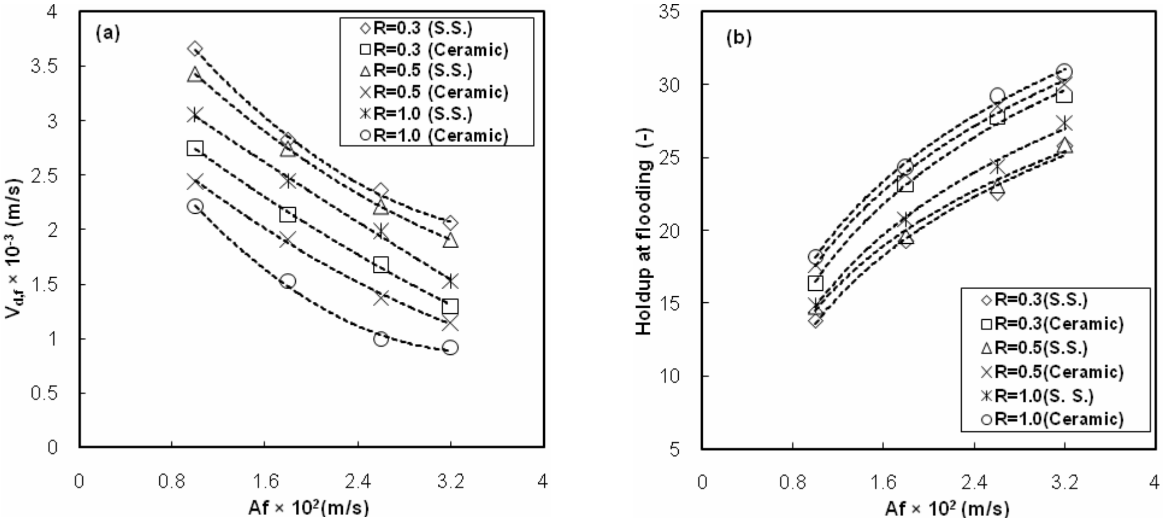

Figure 4: (a) Experimental variation in V_{df} with Af; (b) Variation in ϕ_f with Af (Toluene-Water).

Prediction of the Flood Point in Pulsed Packed Columns

One of the main objectives of this study is to define a suitable method for the prediction of flooding conditions in pulsed packed columns. Previous empirical correlations and the characteristic velocity concept are compared to the experimental data obtained in the present work.

The Potnis, Bijawat, and Doraiswamy Equation

Among the equations reported in the literature for the flood point in pulsed packed columns that of Potnis *et al.* (1955) is important.

$$\frac{\left(V_{cf}^{0.5}+V_{df}^{0.5}\right)\rho_c}{a\,\mu_c} = 0.5303(e)^{0.05} \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.256}\left(\frac{\mu_d^2\, d_T\, g}{\sigma^2}\right)^{-0.0051}\left(\frac{a^3\,\mu_c^2}{g\,\Delta\rho\,\rho_c}\right)^{-0.252}\left(\frac{\sigma\,\rho_c}{\mu_c^2\, a}\right)^{0.283}\left(\frac{Af\,\rho_c}{a\mu_c}\right)^{-0.11} \quad (1)$$

This correlation contains the physical properties of the system, the pulsation data and the packing characteristics. However, the effect of the phase ratio on maximum throughput of the column was not considered in Equation (1), while it is widely accepted that flooding velocity varies significantly with phase ratio. Flooding velocities calculated according to this equation are compared in Fig. 5 with the data obtained in the present work. It is clear that the equation does not provide good agreement between the experimental data and calculated values.

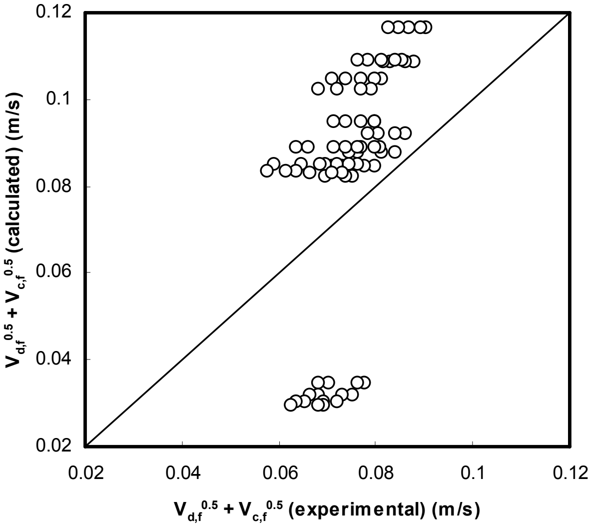

Figure 5: Comparison of experimental results with those predicted by Equation (1)

The Characteristic Velocity Concept

Semi-theoretical relationships for predicting flooding velocities are generally derived from holdup models and many of such relationships are based on the slip velocity approach. The concept of characteristic velocity (Gayler *et al.*, 1953) is very useful for relating the dispersed phase holdup and flow rate as follows:

$$V_s = \frac{V_d}{e\phi} + \frac{V_c}{e(1-\phi)} = V_k\left(1-\phi\right) \qquad (2)$$

It was found that this equation could only be used in the condition of low dispersed phase holdup and no obvious coalescence. Holdup has been correlated using the characteristic velocity approach by Spaay *et al*. (1971). They have identified two regions as follows:

(1) Small drop size where V_k is independent of packing size.

(2) Larger drop size where the drops interact with the packing; thus, V_k is dependent on packing size.

This leads to the following correlations for V_k.

$$V_k = 6.32\times10^{-3} d_{32}^{0.727} \Delta\rho^{0.815} \left(2Af^2\right)^{-0.254} \left[a\left(1-e\right)\right]^{-0.184} \qquad \left(\text{region } 1\right) \qquad (3)$$

for

$$C_5 = d_{32}^{0.787} \Delta\rho^{0.255} \left(2Af^2\right)^{-0.144} \left[a\left(1-e\right)\right]^{-0.426} \le 0.406$$

$$V_k = 2.57\times10^{-3} d_{32}^{-0.06} \Delta\rho^{0.56} \left(2Af^2\right)^{-0.11} \left[a\left(1-e\right)\right]^{-0.61} \mu_c^{-0.35} \qquad \left(\text{region } 2\right) \qquad (4)$$

for

$$C_5 \ge 0.406$$

These are only for Raschig rings with 0.55<e<0.88. The values of d_{32} were calculated by the semi-empirical correlation proposed by Spaay *et al*. (1971).

At the flood point, the phase flow rates can be obtained by differentiating Equation (2) with respect to ϕ_f, treating V_d and V_c as dependent variables, and setting the differentials equal to zero. Thus:

$$\left(\frac{dV_d}{d\phi}\right)_{V_c} = 0 \Rightarrow V_{d,f} = 2eV_k(1-\phi_f)\phi_f^2 \qquad (5)$$

$$\left(\frac{dV_d}{d\phi}\right)_{V_c} = 0 \Rightarrow V_{c,f} = eV_k(1-2\phi_f)(1-\phi_f)^2 \qquad (6)$$

where ϕ_f, the limiting value of the holdup at the flood point, is obtained by solving Equations (5) and (6) for ϕ_f and putting $R=V_d/V_c$,; thus:

$$\phi_f = \frac{\left(R^2+8R\right)^{0.5} - 3R}{4\left(1-R\right)} \qquad (7)$$

In the present work, the values of V_k were obtained from Equations (3) and (4) and the calculated values of V_k were used in Equation (5) for calculating the flooding velocity. Equation (7) was used for calculating the holdup at flooding. Figure 6 shows the comparison of experimental data and calculated values obtained by using Equations (3), (4), (5) and (7). As can be seen in this figure, there is a large deviation between experimental results and calculated values.

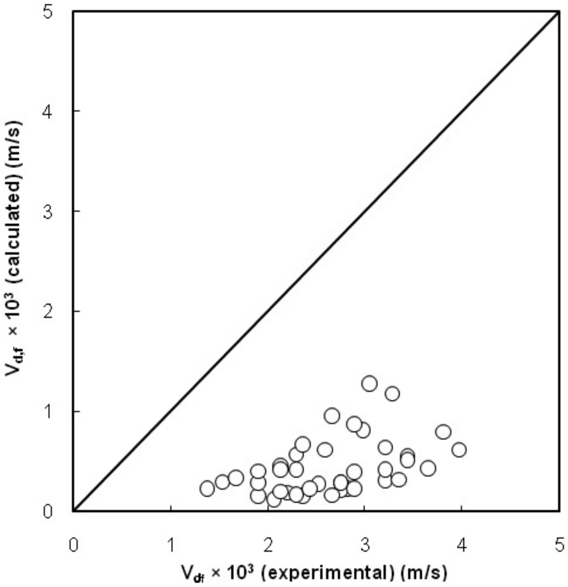

Figure 6: Comparison of experimental results with those predicted using the equations of Spaay *et al*. (1971).

Several modified equations that take into account the drop coalescence under high dispersed phase holdup have been developed, but it is considered that the equation:

$$V_s = \frac{V_d}{e\phi} + \frac{V_c}{e(1-\phi)} = V_k\left(1-\phi\right)^m \qquad (8)$$

has very wide applicability (Godfrey and Slater; 1991). Since the pulsed packed column is usually operated within the high dispersed phase holdup region (Jie and Weiyang, 2000), Equation (8) was used for prediction of the flood point in this work. At the flood point, the phase flow rates could be obtained by differentiating Equation (8) with respect to holdup, treating V_d and V_c as dependent variables and setting the differential equal to zero. Thus:

$$\left(\frac{dV_d}{d\phi}\right)_{V_c} = 0 \Rightarrow V_{d,f} = e\left(1+m\right)V_k(1-\phi_f)^m\phi_f^2 \qquad (9)$$

$$\left(\frac{dV_c}{d\phi}\right)_{V_d} = 0 \Rightarrow V_{c,f} = eV_k\left(1-\phi_f\right)^{m+1} \left(1-\left(m+1\right)\phi_f\right) \qquad (10)$$

Combining Equations (9) and (10), the limiting value of holdup at flooding is obtained as follows:

$$R = (m+1)\phi_f^2 / (1-\phi_f).(1-\phi_f - m\phi_f) \qquad (11)$$

The values of V_k and m obtained from Equation (8) are listed in Table 3. According to this table, the values of m are between -0.22 and 3.64, while this range was reported to be between 0 and 4 for a rotating disc contactor (RDC) (Godfrey and Slater, 1991), from 0.3 to 1.5 for a packed column (Godfrey and Slater, 1991), from -3 to 1 for a sieve plate column (Hamidi *et al*., 1999) and -0.9 to 3.6 for a Grasser raining bucket contactor (Giraldo-Zuniga *et al*., 2006). The difference in m values is probably due to the difference in the extractor construction and the physical properties of the liquid systems used. Flooding velocities and holdup at flooding are calculated using Equations (4) and (6). The comparison between experimental results with those calculated by the characteristic velocity method is presented in Figure 7. The results confirm that this method is not applicable to the present column, since a marked deviation is observed between the experimental data and calculated values.

Table 3: The values of characteristic velocity and m.

n-Butyl acetate-Water			Toluene-Water			Kerosene-Water		
Af (cm/s)	**m**	**V_k (mm/s)**	**Af (cm/s)**	**m**	**V_k (mm/s)**	**Af (cm/s)**	**m**	**V_k (mm/s)**
0.6	1.59	17.89	1	2.50	23.92	1	3.64	32.09
1	0.636	13.94	1.8	2.0	17.46	1.8	2.89	23.31
1.4	0.011	11.615	2.6	0.47	11.38	2.6	1.834	18.35
1.8	-0.22	10.20	3.2	0.108	9.17	3.2	0.733	12.39

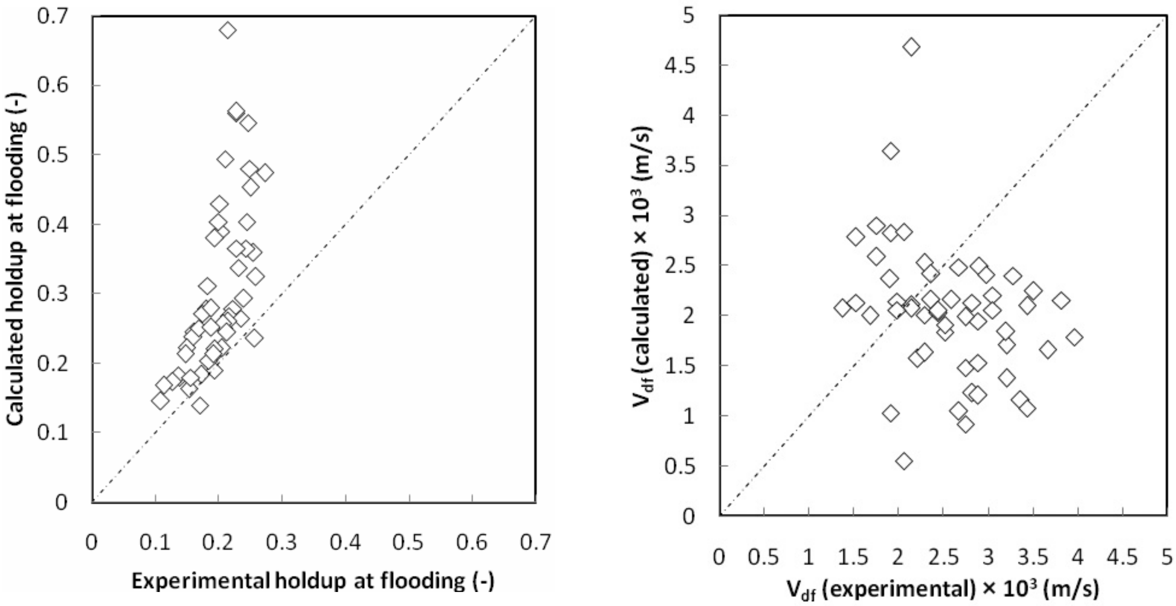

Figure 7: Comparison of experimental data with calculated values using the characteristic velocity approach

New Predictive Correlations for Flooding Velocity and Holdup at Flooding

Since there is no accurate correlation for the prediction of flooding conditions in pulsed packed columns, new empirical correlations were developed in terms of the physical properties of the systems and the operating and packing characteristics for the prediction of the flooding velocity and holdup at flooding. Based on 81 flooding velocity data points, the following expression is obtained:

$$V_{df} = 2.31\left(\frac{Af.\mu_c}{\sigma}\right)^{-0.41}\left(\frac{\sigma^3\rho_c}{g\mu_c^4}\right)^{-0.50}\left(\frac{\mu_c}{\mu_d}\right)^{-0.08}\left(\frac{e^3.g}{a}\right)^{0.127}\left(\frac{\sigma.\rho_c}{\mu_c^2 a}\right)^{0.35}\left(1+R\right)^{-0.43} \tag{12}$$

The average absolute value of the relative deviation (AARD) between the predicted values of V_{df} and the experimental points is 6.454%, with a maximum absolute error of 22.7%. The comparison of experimental data with those calculated by Equation (12) is illustrated in Figure 8. According to this figure, there is a good agreement between experimental and calculated data.

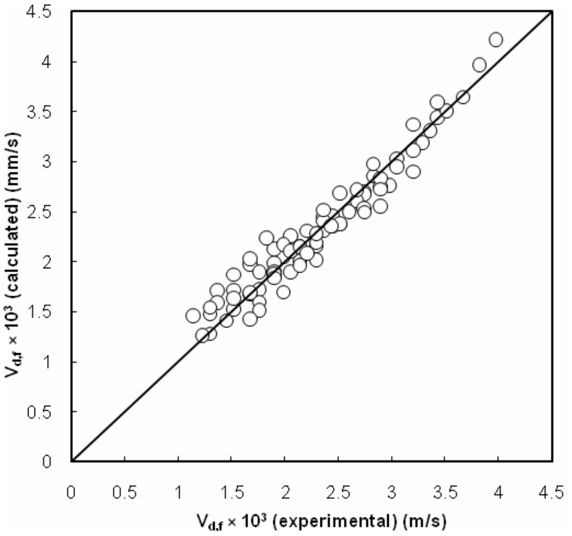

Figure 8: Comparison of experimental data with calculated ones from Eq. (12).

The experimental values of holdup at flooding are also correlated in terms of the physical properties of systems and the operating conditions and packing characteristics as follows:

$$\phi_f = 0.189\left(e\right)^{-0.486}\left(\frac{Af.\mu_c}{\sigma}\right)^{0.385}\left(\frac{\sigma^3\rho_c}{g\mu_c^4}\right)^{-0.013}\left(\frac{\mu_c}{\mu_d}\right)^{-0.075}\left(\frac{\sigma.\rho_c}{\mu_c^2 a}\right)^{0.26}\left(1+R\right)^{0.20} \tag{13}$$

The correlations were derived by the least squares method using "Eviews software", version 3.1. The comparison between the experimental data and those predicted by using Equation (13) is given in Figure 9. This figure reveals good agreement between the calculated values and experimental results. The value of the AARD for the predicted values of holdup at flooding obtained by applying Equation (13) to the experimental results is 5.45%, with a maximal error of 22.37%.

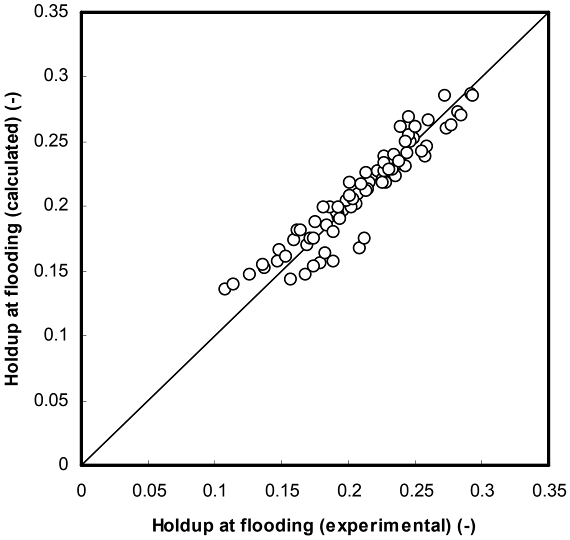

Figure 9: Comparison of experimental results with calculated values from Eq. (13).

CONCLUSIONS

This paper presents an experimental investigation of the flooding behavior of a pulsed packed column. The results showed that the maximum throughput decreases with an increase in pulsation intensity and flow ratio, while it increases with an increase in interfacial tension. It was found that the characteristic velocity approach is not applicable to this type of extraction column for prediction of the flood point. New empirical correlations for prediction of flooding velocity and holdup at flooding are proposed. These equations are shown to describe the experimental data satisfactorily. Since there is little experimental data on this type of column, the present work is of use to those desiring to employ this type of contactor.

NOMENCLATURE

a	specific interfacial area	m^2/m^3
A	amplitude of pulsation	m
d_{32}	Sauter mean drop diameter	m
d_T	column diameter	m
f	frequency of pulsation	s^{-1}
e	void fraction of packing	(-)
g	acceleration due to gravity	m/s^2
m	exponent	(-)
R	flow ratio (V_d/V_c)	(-)
V	superficial velocity	m/s
V_s	slip velocity	m/s
V_k	characteristic velocity	m/s

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
ϕ	dispersed phase holdup	(-)

Subscripts

c	continuous phase
d	dispersed phase
f	flooding

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