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Holdup e velocidade característica em uma extratora pulsada com recheio

Holdup and characteristic velocity in a pulsed packed extraction column

Holdup y velocidad característica en un extractor pulsado con relleno

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Resumo

O presente trabalho tem como objetivo avaliar a hidrodinâmica de um extrator compactado pulsado, com diâmetro interno de 0,026m e comprimento de 1,0m, utilizando o sistema butanol-água líquido. Assim, os parâmetros básicos obtidos para o estudo hidrodinâmico da coluna de extração em questão como fase dispersa, velocidade de escorregamento, velocidade característica e ponto de inundação. A metodologia utilizada no trabalho consistiu em determinar a fração de retenção da fase dispersa, obtida através de testes de interrupções simultâneas na alimentação da coluna. Os efeitos da pulsação de freqüência, vazão de fase dispersa e vazão de fase contínua investigados na análise desses parâmetros. Novas correlações empíricas derivadas das previsões dos parâmetros estudados, obtidas em termos de variáveis operacionais e propriedades físicas do sistema líquido envolvido. O valor absoluto médio do erro relativo (AARE) sempre foi inferior a 5,6%. Boa concordância entre os resultados calculados e experimentais observados para todas as condições operacionais investigadas.

Palavras-chave: Extrator pulsado com recheio; Hidrodinamica; Holdup da fase diispersa; Velocidade característica.

Abstract

The present work aims to evaluate the hydrodynamics of a pulsed packed extractor, with an internal diameter of 0.026m and a length of 1.0m, using the liquid butanol-water system. Thus, the basic parameters obtained for the hydrodynamic study of the extraction column in question as dispersed phase, slip velocity, characteristic velocity and flooding point. The methodology used in the work consisted of determining the holdup fraction of the dispersed phase, obtained through tests of simultaneous interruptions in the column feedings. The effects of frequency pulsation, dispersed phase flow rate and continuous phase flow rate investigated in the analysis of these parameters. New empirical correlations derived from the predictions of the parameters studied obtained in terms of operating variables and physical properties of the liquid system involved. The average absolute value of the relative error (AARE) was always below 5.6%. Good agreement between calculated and experimental results observed for all investigated operating conditions.

keywords: Pulsed packed extractor; Hydrodynamic; Dispersed phase holdup; Characteristic velocity.

Resumen

El presente trabajo tiene como objetivo evaluar la hidrodinámica de um extractor compactado pulsado com um diámetro interno de 0.026m y uma longitude de 1.0m, utilizando el sistema de butanol-agua. Asi, los parámetros básicos obtenidos para el estúdio hidrodinámico de la coluna de extracción, como la fase dispersa, la velocidade de deslizamento, la velocidade característica y el punto de inundación. La metodologia utilizada consistió en determinar la fracción de retención de la fase dispersa, mediante pruebas de interrupciones simultâneas em la alimentación de la columna. Los efectos de la pulsación de frecuencia, el flujo de fase dispersa y fase continua investigados en el análisis de estos parâmetros. Nuevas correlaciones empíricas devivadas fde las predicciones de los parâmetros estudiados, obtenidos em términos de variables operativas y propriedades físicas del sistema líquido involucrado. El valor absoluto médio del error relativo (AARE) siempre há sido inferior al 5,6%. Buen acuerdo entre los resultados calculados y experimentales observados para todas las condiciones operativas investigadas.

Palabras clave: Extractor pulsado com relleno; Hidrodinâmica; Retencion de la fase dispersa; Velocidade característica.

1. Introduction

The extraction processes are suitable for the petrochemical industry to separate aliphatic and aromatic components, for the biochemical industry in the separation of antibiotics and protein recovery, in the separation of spent fuel elements and inorganic chemical (Henley & Seader, 1981). The pulsed packed column came from Van Dijck (1934) ideas as a proposal for an extraction column to increase the mass transfer rate in the process with perforated plates columns. Leading to the development of pulsed packed column that are suitable for applications in the pharmaceutical industry, petrochemical, and nuclear industries (Torab-Mostaedi et al., 2009; Asadollahzadeh et al., 2012). According to Simons (1983); Asadollahzadeh et al. (2012), these columns have a great advantage over the others, since the pulse unit can be removed from the column and the absence of moving mechanical parts makes easier for any future repair. These columns are classified as differential extractors due to the countercurrent flow be established in function of the density difference between the fluids (Kumar et al., 1986).

The purpose of this work is to evaluate the hydrodynamics of a pulsed packed extractor. In the study, holdup data of the dispersed phase, flooding and characteristic velocity of the column are obtained and analyzed. The study of the hydrodynamic extraction columns is important to the construction of the column in terms of geometry, diameter and height. In terms of operational conditions, one can predict the operating limits within a certain operating range. The dispersed phase holdup is related to mass transfer area and the flooding of the column. The dispersed phase holdup is expressed as follows (Treybal, 1963; Napeida et al., 2010):

$$\phi = \frac{v_d}{v_d + v_c} \quad (1)$$

Other parameters to study the hydrodynamics are dependent on the phase dispersed holdup, *e.g.*, slip velocity, characteristic velocity and specific interfacial area. The slip velocity or relative velocity between dispersed and continuous phases in packed columns is given by (Kumar & Hartland, 1994, 1995):

$$V_{slip} = \frac{V_d}{\varepsilon\phi} + \frac{V_c}{\varepsilon(1-\phi)} \quad (2)$$

While the characteristic velocity is the velocity isolated drop in the column under conditions of zero holdups and zero continuous phase flow rate, and hence, it can be identified with the terminal velocity of a single drop (Kumar and Hartland, 1988; Kumar et al., 1985; Coimbra et al., 1998; Napeida et al., 2010).

According to Gayler & Pratt (1951), the characteristic velocity as follows:

$$V_{slip} = V_K(1-\phi) \quad (3)$$

Applies for a condition dispersed phase holdup: $\phi < 0.20$.

Another equation for the characteristic velocity used in sedimentation and fluidization systems, applied to liquid-liquid system to values dispersed phase holdup between 0 to 0.30, is given by (Richardson & Zaki, 1954):

$$V_{slip} = V_K(1-\phi)^m \quad (4)$$

Where m is a function of the Reynolds number

Another important factor in the study of the hydrodynamic behavior when associated with mass transfer is the specific interfacial area, which depends on the dispersed phase holdup. To packed column it's defined as (Simons, 1983):

$$a = \frac{6\varepsilon\phi}{d_{32}} \quad (5)$$

Drop size has an important parameter on the dispersed phase holdup and efficiency mass transfer (Gholam Samani et al., 2012). Drop size distribution is described by the relation of Sauter mean diameter (d_{32}):

$$d_{32} = \frac{\sum_{i=1}^{n} n_i d_i^3}{\sum_{i=1}^{n} n_i d_i^2} \quad (6)$$

The operation efficiency of the columns is considered in terms of maximum volumetric capacity, also called flood points. Values above the maximum makes the operation unstable. Thus, the investigation of the flooding point must be verified. The column flood point characteristics to be directly affected by the operating variables and physical properties of liquid systems (Torab-Mostaedi et al., 2011). The flooding phenomenon generally occurs when one of the fed phases is prevented from following its original route. Therefore, a column must operate with the phases' velocities below the flooding velocity, once it is not possible to increase the flow rates of the phases indefinitely, there is a limit of the quantity of a phase that can be dispersed in the second phase (McCabe et al., 1998; Asadollahzadeh et al, 2011). Although it depend on the feed, other factors can influence it, as agitation intensity, interfacial tension, viscosity and the direction of mass transfer.

Thornton (1956); Seader & Henley (2006) describes the flooding point as the maximum holdup obtained from the graphical analysis of the ratio between the dispersed phase velocity and the characteristic velocity ($\frac{V_d}{V_K}$) *versus* the holdup (ϕ_d).

Applying partial derivatives in Eq. (3) considering the slip velocity relationship for columns packed, it is obtained:

$$\left(\frac{\partial V_d}{\partial \phi}\right)_{V_c} = 0 \tag{7}$$

and

$$\left(\frac{\partial V_c}{\partial \phi}\right)_{V_d} = 0 \tag{8}$$

Thus,

$$V_c = \varepsilon V_K\left[1 - 2\left(\phi_f\right)\right]\left[1 - \left(\phi_f\right)\right]^2 \tag{9}$$

$$V_d = 2\varepsilon V_K\left[1 - \left(\phi_f\right)\right]\left(\phi_f\right)^2 \tag{10}$$

Combining equations 9 and 10 is obtained an expression for the dispersed phase holdup in the flooding, as follows:

$$\phi_f = \frac{[R^2+8R]^{0.5}-3R}{4[1-R]} \tag{11}$$

where $R = Q_d/Q_c$

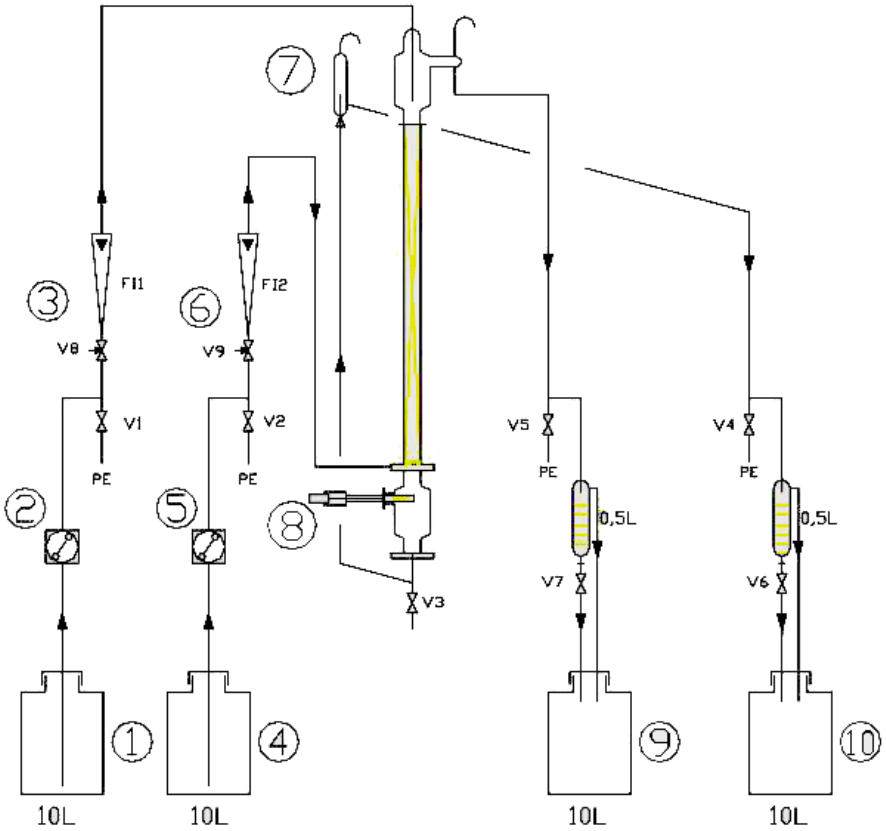
This paper aims to investigate the dispersed phase holdup, slip and characteristics velocity and evaluate the maximum capacity in terms of point flood in a pulsed packed column as a function of frequency pulsation and phases ratio to n-butanol/water system. Finally, new empirical correlations are developed for dispersed phase holdup, slip velocity, and characteristic velocity in terms of operating variables and physical properties of the system liquid studied.

2. Methodology

2.1 The Equipament

Hydrodynamic tests were conducted in a column with height of 1.0 m and 0.026m of internal diameter, filled with a 0.9 m height packing of glass Raschig rings with internal diameter of $0.00324m \pm 0.00016$, and the fractional packing voidage was 0.65. Compressed air was used as pulsator and amplitude of pulsation was 0.05 m. A schematic diagram of this column is showed in the Figure 1.

Figure 1 - Schematic flow diagram of the pulsed packed column.



Notes: (1) heavy phase tank; (2) heavy phase pump; (3) F11 rotameter; (4) light phase tank; (5) light phase pump; (6) F12 rotameter; (7) interface level control through lateral adjustment; (8) pneumatic pulser; (9) extract tank; (10) raffinate tank. V1, V2, V3, V4, V5, V6 and V7 sampling valves; V8 and V9 globe valves.
Source: The author.

According to the diagram shown in Figure 1 the two phases, light and heavy, were fed into the column in the reservoirs 1 and 4. The light phase (4) was introduced from the bottom and the heavy phase (1) from the top. In the reservoirs indicated in 9 and 10, the column outlet products were stored. The column was fed through pumps 2 and 5, with flow rates measured through rotameters 3 and 6. In 7 the level controller of the formed liquid interface is shown and in 8 the extraction pulsation system.

2.2 Experimental

The physical properties of the liquid system are listed in Table 1

Table 1 - Physical properties of system studied at 25°C.

Physical properties	n-Butanol/water
ρ_d (kg/m^3)	806.99
ρ_c (kg/m^3)	997.06
μ_d (mPa.s)	2.59
μ_c (mPa.s)	0.91
σ * (mN/m)	1.75

*Misek et al., (1978); Napeida et al., (2010).

N-butanol at 99.77 wt% purity was used as the dispersed phase and water was used as the continuous phase. Densities and viscosities of the components under study were determined with an Anton Paar DSA 5000 densimeter and an Anton Paar SVM 3000 viscometer, respectively.

The method of simultaneous interruptions was used for the calculation of the dispersed phase holdup. This method consists on interrupting the fluid inputs into the column, and subsequent measurement of the dispersed phase volume present within the column. First, the column was filled with the continuous phase, and then introduced into the dispersed phase. Pulsation frequency was adjusted, and after steady state has been reached, the flow rates of both phases were interrupted simultaneously. The volume of the dispersed phase was then measured after all present liquid had been drained from the bottom of the column and the dispersed phase holdup calculated using Eq. (1).

Reproducibility of the holdup values was within ±1%. In order to obtain flooding points, adjustment of the continuous phase flow rate and gradual increasing of dispersed phase flow rate were used. When an interface was formed at the base of the column, feeds were interrupted and the dispersed phase holdup measured as previously mentioned. The reproducibility of holdup values was within ±3.8%.

2.3. Drop size distribution and specific interfacial area

To determine the size of the drops was possible by using the photographic method (Cannon Eos Rebel T4i 18 megapixels camera) and quantified with the aid of image

processing software, according to the methodology proposed by Stella et al. (2008) adapted for his experimental condition.

Drop size was measured at different frequencies in various parts of the column. The mean drop size was calculated by Eq. (6) and specific interfacial areas were calculated using Eq. (5).

2.4. Data analysis

To compare the calculated results with experimental data, is used the average absolute value of relative error (AARE), defined as:

$$AARE = \frac{1}{NDP} \sum_{i=1}^{NDP} \frac{|calculated\ value - experimental\ value|}{experimental\ value} \quad (12)$$

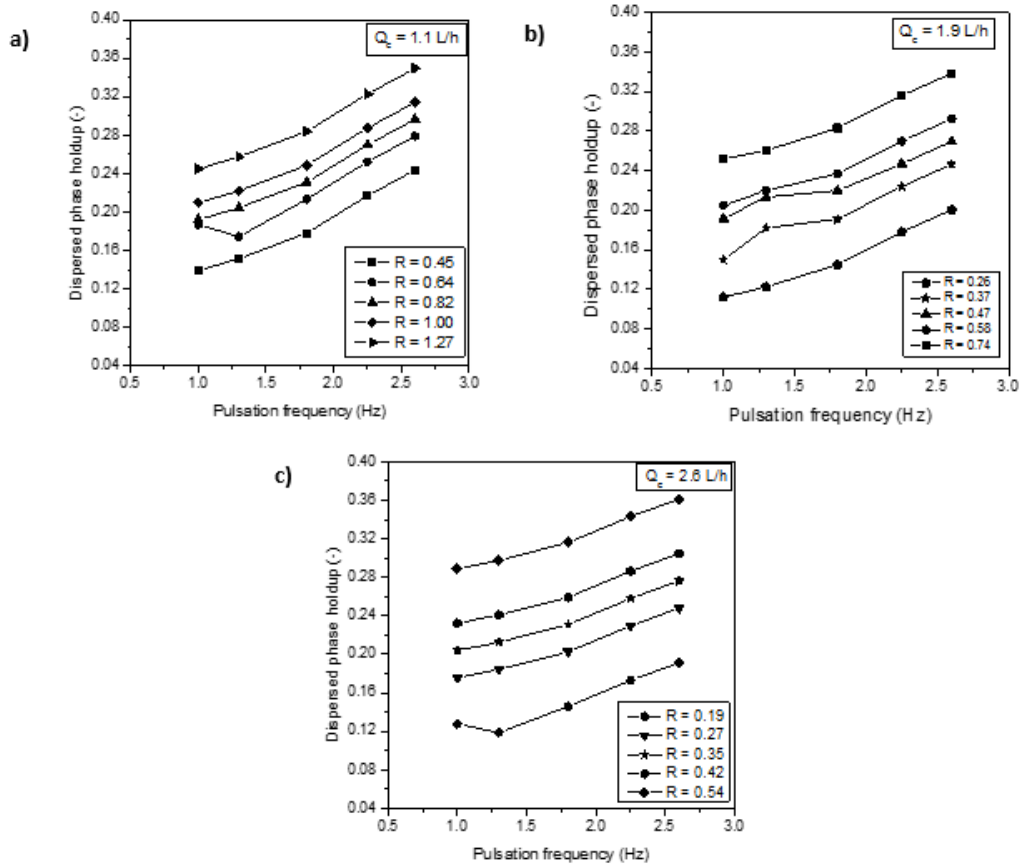
where NDP represents the number of data points.

3. Results and discussion

3.1. Dispersed phase holdup, drop size distribution, slip velocity and characteristic velocity

The holdup of the dispersed phase is influenced by various variables of the process. To study the column in this work, the frequency pulsation and the phases flow rate directly influence these values. The effect of the pulsation frequency on the dispersed phase holdup is given in Figure 2.

Figure 2 – Effect of pulsation frequency on dispersed phase holdup.

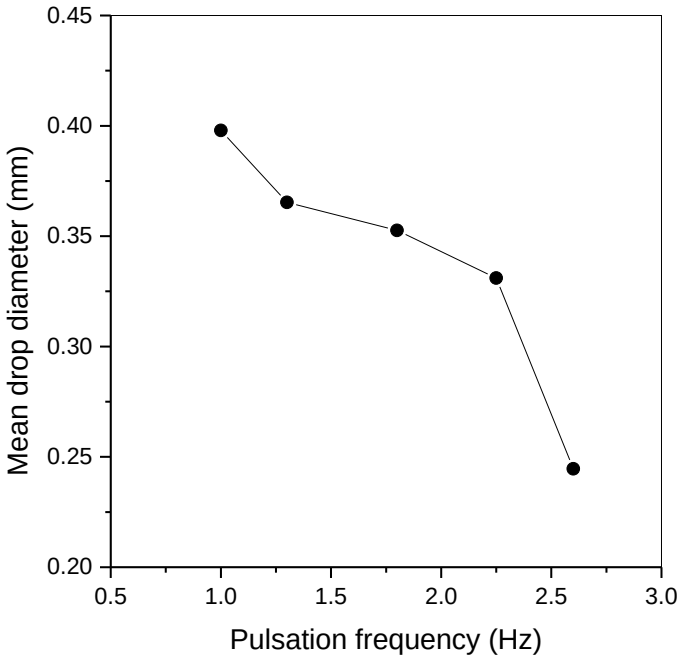


a) Qc = 1.1 L/h; b) Qc = 1.9 L/h; c) Qc = 2.6 L/h. Source:: the authors
Source: The authors.

According to this figure, the dispersed phase holdup increases with increasing pulsation frequency. The increase in the energy system makes it occurs higher dispersion in the middle due to breakage of the drops. The more dispersed in the environment smaller drops the dispersed phase holdup get higher. The pulsation frequency assistance to increase shear forces, leading to breakage of the drops favoring the increase of the dispersed phase holdup. It is also observed an increase in the holdup with the increase of the phase ratios. Maximum values of the dispersed phase holdup were obtained for high of dispersed flow rates and high values of pulsation frequency. However, excessive increases can cause the droplet coalescence and impair the stability of the operation.

Drop size distribution is essential for the study of specific interfacial area. Smaller size contributes to higher specific interfacial areas. Figure 3 shown drop size behavior with the pulsation frequency

Figure 3 - The effect of pulsation frequency on the drop size distribution

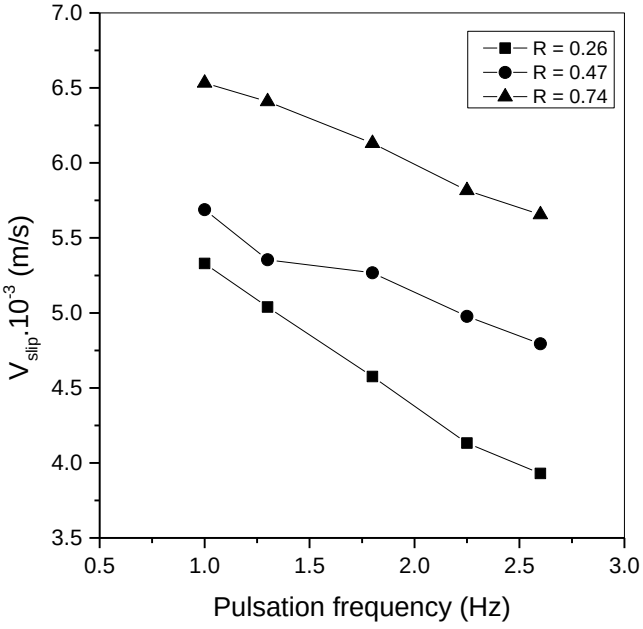


Source: The authors.

The increase of the intensity of the pulsation frequency contributes more strongly for the breakage of drops as mentioned above. So higher frequencies cause a higher collision occurs between the drops and the wall of the column and drops to the packed thus favouring shear forces to get higher (Ousmane et al., 2011; Gholam Samani et al., 2012).

The slip velocity as a function of the pulsation frequency is shown in Figure 4

Figure 4 - The effect of pulsation frequency on slip velocity.

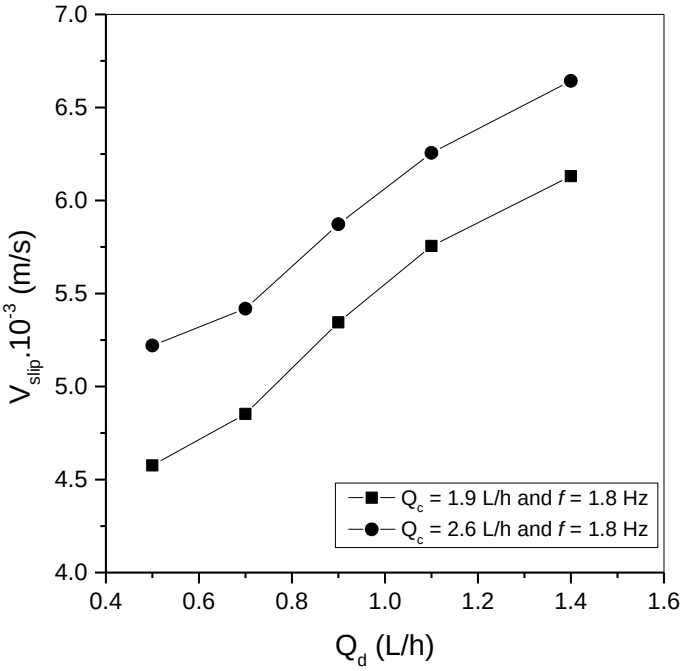


Source: The authors.

The analysis of the figure shows that the slip velocity decreases with increasing pulsation frequency due to decrease the size of the drops. This effect causes drops to remain more time at column bed thus reducing the velocity between the dispersed phase in drops and the continuous phase. Small drops sizes in the column flow slowly, i.e., the residence time is longer compared with larger drops sizes. It is also observed that slip velocity is higher for superior to phases ratios when compared to lower ratios values.

The behavior of the slip velocity according to the dispersed phase flow is presented in Figure 5.

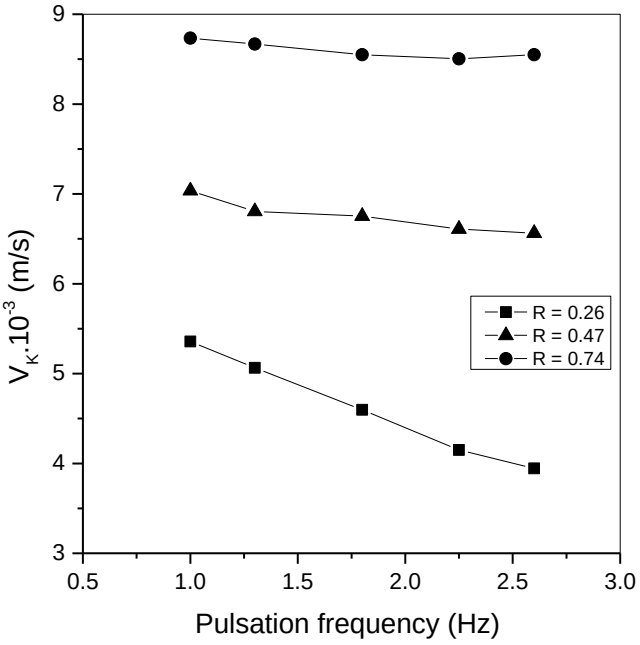
Figure 5 - The effect of dispersed phase flow rate on slip velocity.



Source: The authors.

The figure indicates that the slip velocity increases when the continuous phase flow rate is increased. This occurs due to the reduction in the contact time between the two phases that is verified in the column when the flow of the continuous phase is increased. The effect of pulsation frequency on the characteristic velocity is shown in Figure 6.

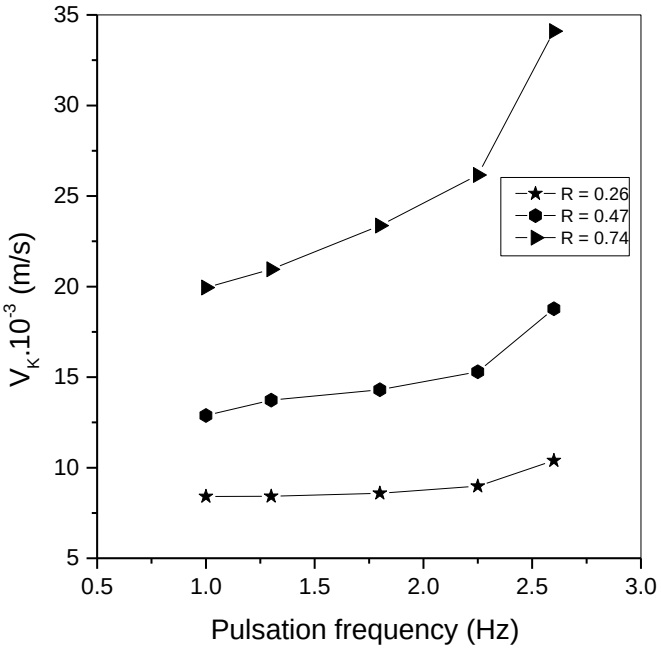
Figure 6 - Effect of pulsation frequency on characteristic velocity using Eq. (3).



Source: The authors.

The analysis of figure indicates that characteristic velocity decreases with increasing pulsation frequency. This effect is explained by the increase of the breaking of the drops due to increasing pulsation frequency, the drops ascend more slowly, decreasing the slip velocity that relates the velocity between the drops and the continuous phase. Figure 7 shows the characteristic velocity.

Figure 7 - Effect of pulsation frequency on characteristic velocity using-Eq. (4).

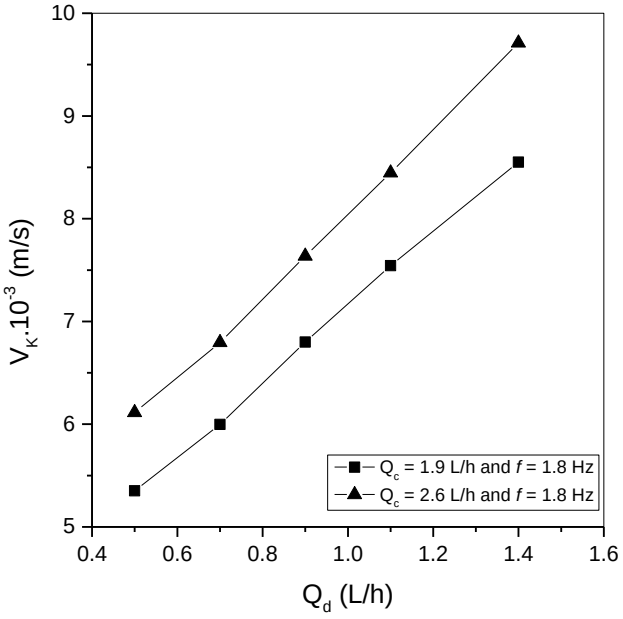


Source: The authors.

Figure 7 shows the characteristic velocity calculated by Eq. (4) is higher compared with the calculated using Eq. (3) (Figure 6) influencing the flooding study obtaining lower values due to the characteristics velocity are higher .

The effect of the dispersed phase flow in the characteristic velocity is showed in Figure 8.

Figure 8 - The effect of dispersed phase flow rate on characteristic velocity.

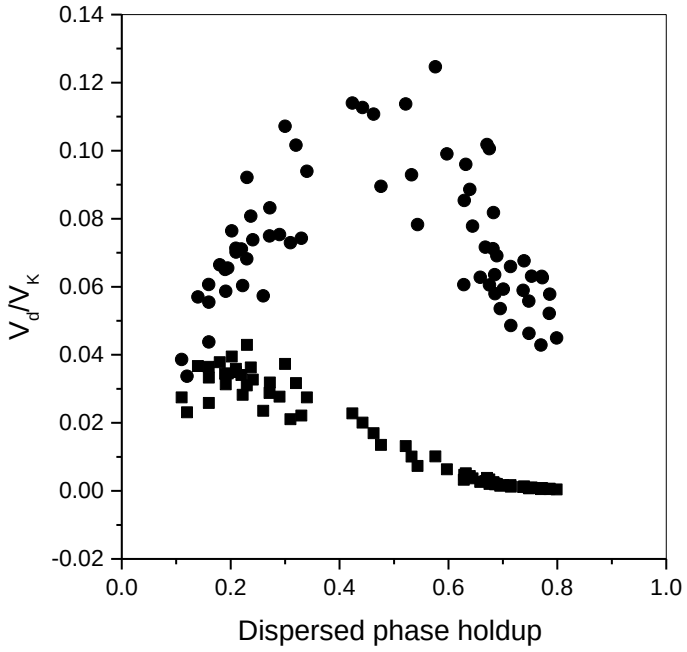


Source: The authors.

According to figure the characteristic velocity increases with the increase of the dispersed phase flow, but the increase is accentuated when comparing the increase of the continuous phase in the middle. The drops flow more rapidly because there are less amount of dispersed phase, i.e., the smaller the rise time of the drops, then directly affect the increasing characteristic velocity values.

The characteristic velocity values can also be used to determine the typical flooding curve of the column and the flooding holdup, ϕ_f. Figure 9 shows the two curves obtained using the two models (Eqs. 3 e 4)

Figure 9 - Typical flooding curve. (●: Values using Eq. 3; ■: Values using Eq. 4).



Source: The authors.

Analyzing the behavior of the two curves shown in Figure9, it is observed that the curve obtained by Eq. (3) flooding holdup (ϕ_f) is found in the range of approximately 0.40, while using the Eq. (4) (ϕ_f) is in the range of 0.2. Comparing the flooding ranges with those obtained experimentally it is realized that the data, it's using better Eq. (3) than Eq. (4). Although, the experimental values obtained dispersed phase holdup is within the validity range of Eq. (4). Many authors indicate Eq. (4) as superior. Thornton (1959) used Eq. (3) in his study.

Therefore, as observed in the data shown in Figure 9, it can be seen that in both curves formed, the values of the holdup can be obtained and, consequently, the characteristic velocity

of flooding of the column. However, it is clear that the curve obtained with the equation 3 presented a clearer indication of the maximum point formed when the column reaches the flooding holdup. This can be proven even with the visual observation of the phenomenon that was perceived in the experiments. Hence the conclusion is that the use of equation 4 should only be done with smaller Re ranges than the one used in the present study.

3.2. News empirical correlations

Due the lack of correlations that explain the hydrodynamic behavior of such columns under different experimental conditions, propose empirical equations, is fundamental importance for better data analysis. Therefore, new correlations for prediction of dispersed phase holdup, slip velocity and characteristic velocity are developed in terms of the operating parameters and physical properties in the present extraction column.

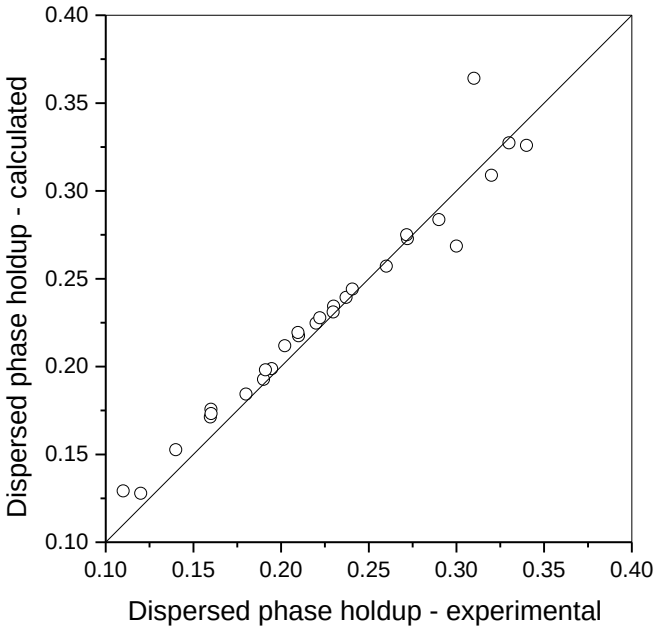
3.2.1. Phase dispersed holdup

The new correlation derived for prediction of dispersed phase holdup is defined as follows:

$$\phi = 1.229 \left(\frac{V_d}{V_c}\right)^{0.034} \left(\frac{V_d}{Af}\right)^{0.045} \left(\frac{\rho_c V_d^3}{\mu_d g}\right)^{0.265} \left(\frac{\mu_d a}{\rho_c V_d}\right)^{0.701} \quad (13)$$

The comparison of experimental results with those calculated by Eq. (13) is shown in Figure 10.

Figure 10 - Comparison between experimental data and calculated values using Eq. 13.



Source: The authors.

This correlation is obtained from 28 experimental data. This Eq. (13) reproduces the experimental data with an average absolute value of the relative error (AARE) of 4,57%. Thus good agreement between calculated and experimental was observed for all investigated operating conditions.

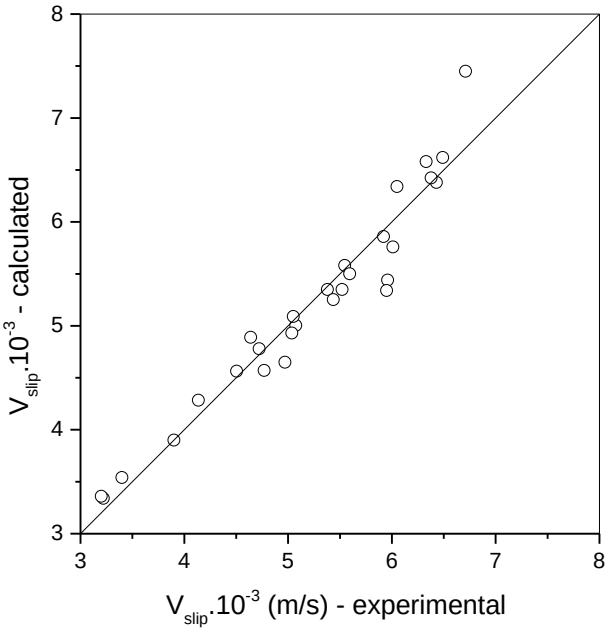
3.2.2. The slip velocity

The new correlation derived for prediction of slip velocity is defined as follows:

$$\frac{V_{slip}}{V_d} = 3.482 \left(\frac{V_c}{V_d}\right)^{0.402} \left(\frac{V_d}{Af}\right)^{0.283} \left(\frac{\mu_d g}{\rho_c V_d^3}\right)^{0.190} \quad (14)$$

The comparison of experimental results with those calculated by Eq. (14) is shown in Figure 11.

Figure 11 - Comparison between experimental data and calculated values using Eq. (14).



Source: The authors.

In Figure 11 it is important to note that the behavior identical to that in Figure 10 was obtained. That is, in Figure 11 a good approximation between the slip velocity experimental values and those found using equation 14 was also verified (AARE = 5,59%). This also proves the consistency of equation 14 for determining the slip velocity in the operating range used in the experiments.

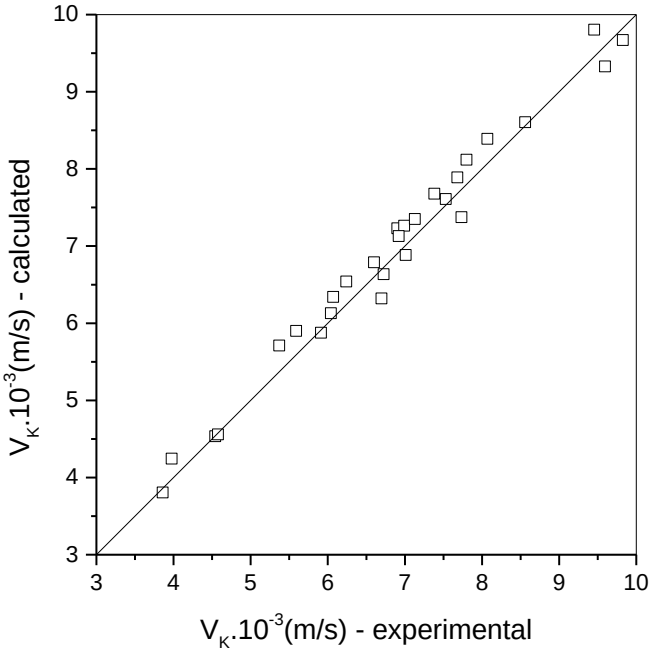
3.2.3. Characteristic velocity

The new correlation derived for prediction of characteristic velocity is defined as follows:

$$\frac{V_K}{V_d} = 6.851 \left(\frac{V_c}{V_d}\right)^{0.372} \left(\frac{V_d}{Af}\right)^{0.047} \left(\frac{\mu_d g}{\rho_c V_d^3}\right)^{0.082} \left(\frac{\mu_d a}{\rho_c V_d}\right)^{-0.100} \quad (15)$$

The comparison of experimental results with those calculated by Eq. (15) is shown in Figure 12.

Figure12 - Comparison between experimental and calculated values using Eq. (15).



Source: The authors.

The average absolute value of the relative error (AARE) between the calculated values of V_K and the experimental points is 5,28%. The figure reveals very good agreement between the calculated values and the experimental data pertaining to the characteristic velocity.

4. Conclusions

This paper presents an experimental study of dispersed phase holdup, slip velocity, characteristic velocity and flooding point.

Results showed That the dispersed phase holdup increases by raising pulsation frequency and phase ratio. Slip velocity decrease with an increase in frequency pulsation and continuous phase flow rate. The same effect was observed in the characteristic velocity results, both velocities analyzed can be used to describe the hydrodynamic behavior a function of frequency pulsation and phases ratio.

The experimental results investigation of the flooding behavior in the column has shown that the maximum is obtained was 0.40 using Equation 3. New empirical correlations for prediction of dispersed phase holdup, slip velocity e characteristic velocity are proposed. These equations are shown to describe the experimental data satisfactorily.

Parameters investigated contributes significantly to the study of the hydrodynamics of extraction columns, as from these data other parameters could be investigated. The results also contribute to the expansion of data for pulsed packed column extraction columns.

5. Considerações Finais

In the present work, the hydrodynamics of a pulsed and packed extractor was evaluated through experimental data of the holdup fraction of the dispersed phase. Influences of the flow rates of the two feed currents, light and heavy, on the holdup fraction, column flooding and speeds, characteristic and slip were analyzed. Empirical equations were obtained with a good approximation range, both for holdup and speeds. Future work in this line of research may be carried out by expanding the column operating ranges, or by analyzing other liquid systems. Another suggestion may be the study of the mass transfer of a solute between the two liquid phases, since research in pulsed and filled extractors is still rare in the current literature.

Nomenclature

a specific interfacial area (m^2/m^3)

A amplitude of pulsation (m)

d_{32} Sauter mean diameter (m)

f frequency of pulsation (s^{-1})

g gravity acceleration (m/s^2)

m parameter

Q flow rate

R flow ratio $\left(\frac{Q_d}{Q_c}\right)$ (-)

R flow ratio $\left(\frac{V_d}{V_c}\right)$ (-) Eq. (11)

v volume (m^3)

V superficial velocity (m/s)

V_{slip} slip velocity (m/s)

V_K characteristic velocity (m/s)

Greek symbols

σ interfacial tension (N/m)

ε void fraction (-)

μ viscosity (Pa.s)

ρ density (Kg/m^3)

ϕ dispersed phase holdup (-)

Subscripts

c continuous phase

d dispersed phase

f flooding

Acknowledgements.

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