DISPERSED PHASE HOLDUP IN A LIQUID-LIQUID EXTRACTION COLUMN

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***Abstract*— Dispersed phase holdup was measured in a liquid-liquid extraction column for the butyl alcohol-water system. The column performance has been studied using two columns of the same diameter (0.092m) but different lengths (0.70m, three stages and 0.90m, four stages). The column was operated counter-currently with several continuous and dispersed flows rates. The dispersed phase holdup was measured by the drainage method. Based on holdup data, the results were analyzed with the aim of determining the influence of flows rates and the column number of stages on the behavior of the dispersed phase holdup. An empirical correlation is proposed for estimating the dispersed phase holdup.**

***Keywords*— Hold-up, Extraction.**

I. INTRODUCTION

Liquid-liquid extraction has been recognized as a powerful separation method for many years. Its past application on an industrial scale has been limited, however; it has been considered when separation by other methods such as distillation, evaporation, or crystallization are unsuitable. As a result of escalating energy costs, liquid-liquid extraction can be an economic alternative to other separation processes.

The mass transfer between the flowing liquid phases in an extraction column depends, among other factors, on the contact interfacial area between continuous and dispersed phases. The interfacial area available for mass transfer in a counter-current extraction tower depends upon the volume fraction or holdup, of the dispersed phase, as well as on the mean droplet size. It is therefore important, at the design stage, to be able to predict the dispersed liquid holdup for a given system, column geometry and set of operating conditions.

The performance of an extraction unit operated continuously depends on the amount of solvent present in the extractor. If the amount of solvent is high compared to the feed, the solute mass transfer is favored (Zuniga-Giraldo *et al*., 2006). From an operational point of view, knowledge of the dispersed phase holdup is also essential for inventory purposes (Batey *et al*., 1986).

In a perforated plates extraction column the stages are separated by fixed plates with a high number of small orifices in their surface. Given the great importance of properly estimating the holdup when designing extraction equipments, many authors have published empirical correlations for several types of columns (Baird and Shen, 1984; Kumar and Hartland, 1988; Kumar and Hartland, 1997; Pratt, 1988), that may be used as reference in studies of perforated plates columns with no mechanical shaking

This paper presents a study of the influence of operational and geometrical parameters on the column's dispersed phase holdup. The influences of the dispersed phase and continuous phase flow rates are analyzed. The existence of flooding for the examined conditions is evaluated according to Thornton (1956) model, and an empirical correlation is presented to estimate the dispersed phase holdup.

II. METHODS

The extraction column used in the present work consisted of a glass tube with internal diameter of 0.092 m and height varying from 0.70 to 0.90 m. The stages were separated by perforated plates with 14.32% of free area (65 orifices with 5.5 x 10^{-3}m of diameter).

The experiments consisted in feeding the dispersed phase (n- butyl alcohol) at the bottom of the column to flow upwards countercurrent to the continuous phase (water) fed at the top of the column (Fig. 1). The flow rates of the phases were fixed and the dispersed phase holdup was measured from the relative phase volume, after arresting the inflow and outflow of phases to the column (drainage method). Table 1 lists the physico-chemical data of the systems used and Table 2 presents the range of variables investigated. The experiments were conducted at room temperature, 301 ± 1K.

III. RESULTS AND DISCUSSION

Figures 2 and 3 present the typical variation of the dispersed phase holdup, for columns with three and four stages, with a variation in the dispersed and continuous phase flow rates**.** Both figures evidence that the dispersed phase holdup increases when the dispersed phase flow rate increases.

Table 1: Physico-chemical properties of the system used (n-butyl alcohol – water)

propriety	Phases	
	Dispersed	Continuous
μ x 10^3 (kg/m.s)	2.3	1.0
ρ x 10^{-3} (kg/m^3)	0.81	1.0
σ (N/m^2)	2.46	7.82

Table 2: Range of variables investigated

Variables Studied	Operation Range
Length of column, L (m)	0.70 m (three stages) and 0.90 m (four stages)
U_c x 10^{-2} (m/s)	0.88; 1.95; 3.00 and 4.10
U_d x 10^{-2} (m/s)	0.29; 0.77; 1.25 and 1.74
Number of stages	3 – 4

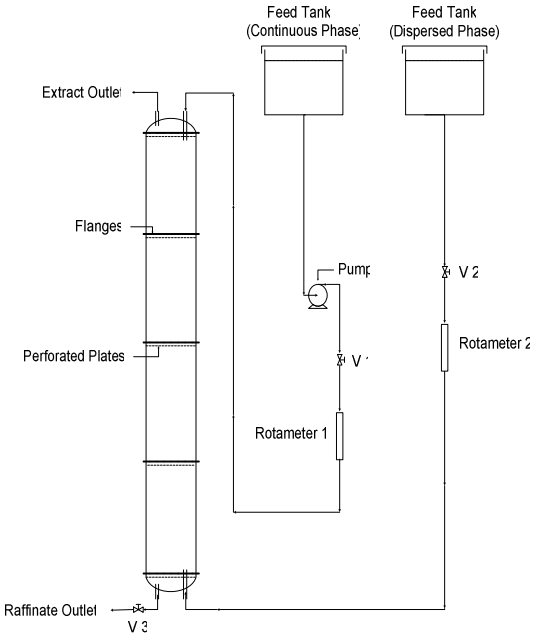

Figure 1 – Schematic diagram of the experimental setup

Distinct effects of the continuous phase flow rate on holdup were found for columns with three or four stages. In columns with three stages (Fig. 2) a strong effect of the continuous phase flow rate on the dispersed phase holdup is evident. In the four stage column (Fig. 3) the continuous phase flow rate has little influences on the holdup. This shows that the column length determines the degree of influence of the continuous phase flow rate on the holdup.

A. Flooding Study

The most important concept in understanding the principle involved in the flooding in liquid-liquid extraction column is about slip velocity and characteristic velocity. According to Godfrey and Slater (1991) the slip velocity (or relative velocity) can be related to dispersed phase holdup fraction through the following equation

$$u_{slip} = \frac{u_c}{1-\phi} + \frac{u_d}{\phi} \qquad (1)$$

where u_c and u_d are the superficial velocities of the continuous and dispersed phases, respectively.

The validity and usefulness of this definition is determined by varying u_c and u_d so that holdup ϕ varies, and finding an empirical function of ϕ which uniquely correlates the results in terms of u_{slip}. Gayler (1953) proposed that for many different types of columns a satisfactory correlating equation was

$$\frac{u_d}{\phi} + \frac{u_c}{1-\phi} = u_k(1-\phi) \qquad (2)$$

where u_k , the characteristic velocity, is the mean relative velocity of droplets extrapolated to zero flow rates (and hold-up) and can be identified with the terminal velocity of a single drop in the equipment concerned.

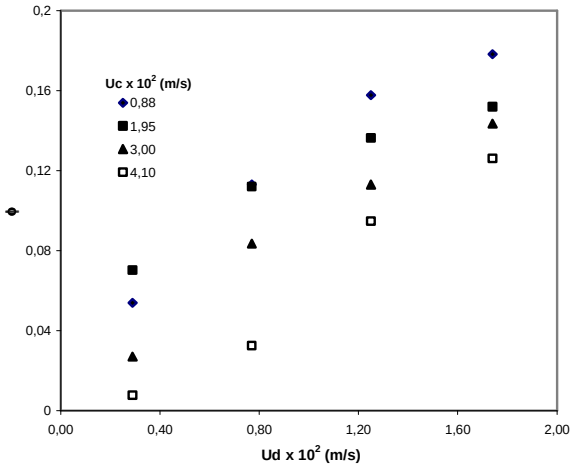

Figure 2 – Variation in dispersed phase holdup with the continuous phase flow rate (N=3 stages)

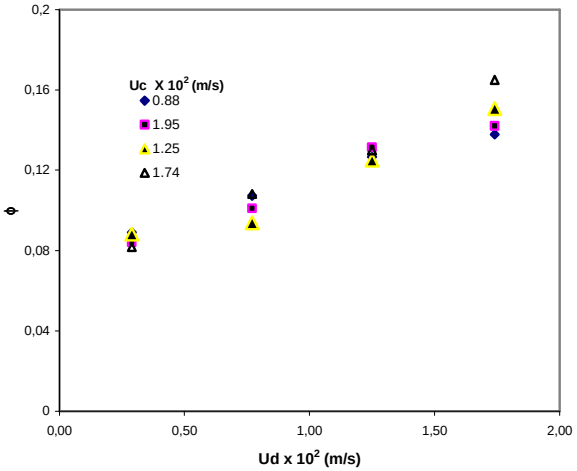

Figure 3 – Variation in dispersed phase holdup with the continuous phase flow rate (N=4 stages)

Dell and Pratt (1951) introduced the idea of differentiating a velocity/holdup relationship to obtain limiting values of superficial velocity, supposing that near the maximum flow rates, small changes of one flow rate with the other fixed will give a large increase in ϕ. The equations describing this condition are:

$$\left(\frac{\partial u_d}{\partial \phi}\right)_{u_c} = 0 \quad \text{and} \quad \left(\frac{\partial u_c}{\partial \phi}\right)_{u_d} = 0 \qquad (3)$$

Thornton (1956) argues that a fixed u_c and a progressive increase of u_d will result in a progressive increase of the holdup ϕ. This may be clearly seen in Figure 4 in which the u_d/u_k velocity ratio is related to the dispersed liquid holdup (ϕ). According to Thornton (1956), the flooding point is obtained when the curve formed reaches a maximum, that is $(\partial u_d/\partial\phi)_{u_c} = 0$ (Fig. 4).

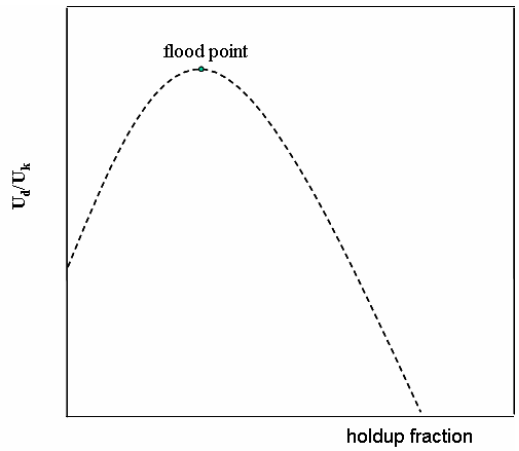

Figure 4 – Typical curve for estimating the flooding holdup in extraction columns.

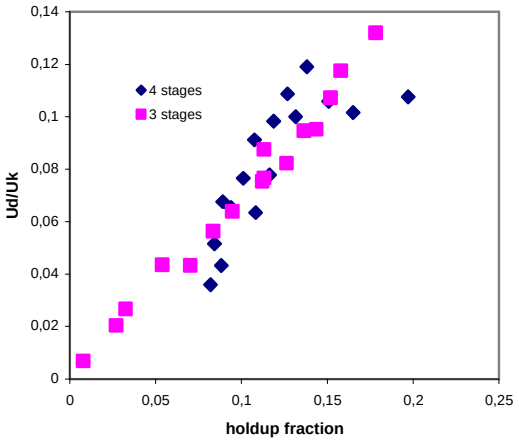

Figure 5 – Experimental results represented as proposed by Thornton (1956) for diagnosing the flooding conditions.

Figure 5 illustrates the values experimentally obtained in the present study represented as proposed by Thornton (1956), for diagnosing the flooding conditions.

One observes in Fig. 5 that the holdup characteristic of the flooding point was not reached for the flow conditions used. Dispersed and continuous phases flow rates examined in the present work define an operation range in the extraction column where the flooding phenomenon does not occur, with a dispersed phase retention of less than 0.20.

B. Empirical correlation for dispersed phase holdup fraction

From the experimental data obtained for the investigated liquid-liquid system by dimensionality theory methods it was obtained a generalized relation between the dispersed phase holdup fraction, ϕ, and the kinematic and geometric factors which govern it: Reynolds Number for the dispersed phase, $Re_d = u_d D \rho_d / \mu_d$, Reynolds Number for the continuous phase, $Re_c = u_c D \rho_c / \mu_c$, Froude Number for the dispersed phase $\mathrm{Fr_d} = u_d^2 / D g$ and ratio of the column height to column diameter. Following, with the use of the multivariable linear interaction techinque, the dimensional groups were grouped through the following expression:

$$\phi = 0{,}066 \left(\frac{L}{D}\right)^{-0{,}52} \left(\frac{u_d D \rho_d}{\mu_d}\right)^{0{,}73} \left(\frac{u_c D \rho_c}{\mu_c}\right)^{-0{,}20} \left(\frac{u_d^2}{D g}\right)^{-0{,}02} \quad (4)$$

The correlation is able to estimate the experimental dispersed phase holdups with an average deviation of 19,41% (Fig. 6).

IV. CONCLUSIONS

This work has shown that the dispersed phase holdup tends to increase when the dispersed flow rate is increased in a staged liquid-liquid extraction column. The effect of the continuous phase flow rate depends on the column length; for the 3 stage column it becomes negligible.

The range of experimental flow rates examined, leading to dispersed holdups below 0.20, did not reach flooding conditions.

Besides analyzing the influences of several parameters, an empirical correlation for estimating the dispersed phase holdup in staged liquid-liquid extraction columns is proposed.

NOMENCLATURE

L – Height of the column (m)
D – Inner diameter of the column (m)
g – Gravity acceleration (m^2/s)
u_c – Continuous phase superficial velocity (m/s)
u_d – Dispersed phase superficial velocity (m/s)
u_k – Characteristic velocity (m/s)
N – Stages Number

Greek letters

ϕ – Dispersed phase holdup fraction (%)
ρ_c – Density of the continuous phase (kg/m^3)

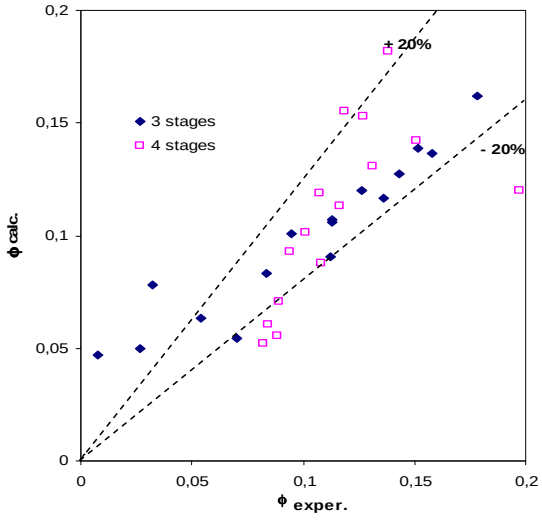

Figure 6 – Comparison between calculated and experimental dispersed liquid holdups

ρ_d – Density of the dispersed phase (kg/m^3)

$\Delta\rho$ – Difference between densities of the dispersed phase (k/cm^3)

μ_c – Viscosity of the continuous phase (kg/m.s)

μ_d – Viscosity of the dispersed phase (kg/m.s)

σ - Interfacial tension (N/m^2)

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