

## THE STUDY OF THE EXTRACTION COLUMNS WITH CYLINDRICAL ROTOR – THE HOLD-UP

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The hydrodynamic behavior of the liquid-liquid extraction columns is characterized by the hold-up of the dispersed phase value, determined in three extraction columns with cylindrical rotor having different constructive characteristics, with the water-carbon tetrachloride system. The values of the hold-up were correlated and expressed under the form of a criterial equation a general correlation, being suggested under the form of:  $\varepsilon_d = a(Ta \cdot F_g)^m Re_{ax}^n V_d^p \delta^r$

### INTRODUCTION

The diversity and the performances of the liquid-liquid extraction columns are characterized by the existence of specific internal constructive elements, which ensure the dissipation of a mechanical energy from the system exterior, achieving a turbulence level necessary for obtaining a corresponding dispersion. A particular solution is represented by the extraction columns with cylindrical rotor, where the flow of the two phases appears between two concentric cylinders, the exterior one being static and the interior one in a rotation movement.<sup>1,2</sup> The phenomena which are characteristic of this type of flow are determined by the report between the centrifugal inertial forces and the viscous forces, expressed by Taylor criterion.<sup>3,4</sup> If it exceeds the value characteristic of the critical threshold of the Ta criterion, the flowing profile evolves to a cell-structured one, characterized by pairs of stable toroid-like whirls, tangent to the exterior cylinder and perpendicular to its axis, and which rotates in opposite directions, the potential flow in countercurrent of the two phases overlapping them.<sup>5,6</sup> In contrast to the special interest shown for this type of flow, called Taylor-Couette-Poiseuille (TCP),<sup>6,7</sup> the columns with a cylindrical rotor have not drawn the researchers' special attention, being indicated for separation processes where a great intensity of the stirring can affect the structural characteristics of the phases, or for the separations in which easily degradable solvents are used, low residence times being indicated.<sup>2</sup>

The paper presents the results of the experimental measurement values of determining the hold-up and the establishment of the operation parameter influence on the hold-up in the extraction columns with a cylindrical rotor.

### EXPERIMENTAL

For this experiment we used a classical extraction installation, with the operation in a stationary regime and the possibility of modifying the operational parameters: the volumetric loading of the column and the rotor speed (Fig. 1). The values of the constructive characteristics for the used columns are given in Table 1, the experiments being performed in the absence of the mass transfer, with the water-carbon tetrachloride binary system. Calculations of the hold-up were performed, using extraction columns with a cylindrical rotor, made of glass and stainless steel. The column stator consists of a glass tube Quick-fit, with a calibrated interior diameter, where a cylindrical rotor made of stainless steel is concentrically installed and which is linked to the training system “engine–rotation speed varying device”.

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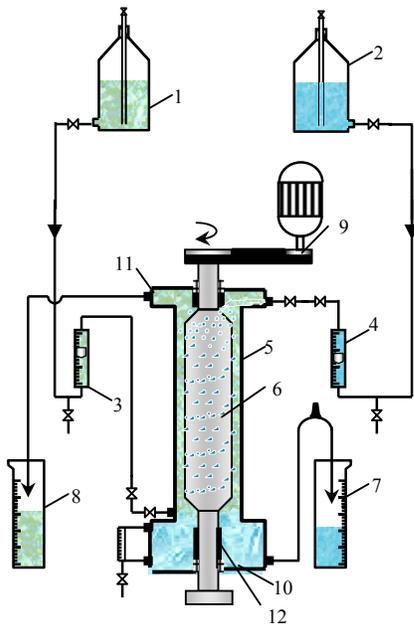


Table 1

The constructive characteristics of the extraction columns

| Characteristic               | I        | II    | III   |
|------------------------------|----------|-------|-------|
| Stator exterior diameter, mm | 46       | 46    | 46    |
| Stator interior diameter, mm | 38       | 38    | 38    |
| Stator height, mm            | 700      | 700   | 700   |
| Rotor diameter, mm           | 36       | 32    | 28    |
| Rotor height, mm             | 1000     | 1000  | 1000  |
| Annular width, $\delta$ , mm | 1        | 3     | 5     |
| Geometric factor, $F_g$      | 0.95     | 0.842 | 0.736 |
| Rotor speed rot/min          | 200-1000 |       |       |

Fig. 1 – The scheme of the experimental installation

1 – container for the continuous phase, 2 – container for the dispersed phase, 3, 4 – rotameters, 5 – stator, 6 rotor, 7, 8 – containers for collecting the extracted and the refined products, 9 – driving group, 10, 11 – phase separators, 12 – muff with longitudinal baffle.

For determining the hold-up, we used “the volumetric method” based on measuring the dispersed phase volume retained in the column’s active zone. The working algorithm includes the following stages:

- maintaining the column in a functioning stationary regime (the feeding flows, the rotor’s speed, the level of the interface maintained at a constant value, corresponding to an established operation regime) a sufficient time for changing, at least two times, the “liquid dowry” in the column;
- the simultaneous stop of the continuous and dispersed phase input, and of the stirring system, respectively;
- droplets depositing, emptying the column content, separating the obtained biphasic heterogeneous mixture and measuring the entire volume of dispersed phase existing in the active zone of the extraction column while functioning.

On the basis of the constructive characteristics of the columns, we calculated the volume of the active zone of the column, and the hold-up  $\epsilon_d$ , respectively, defined as the volumetric ratio of the solvent in the droplets dispersion formed within the space between the two cylinders.

The experiments followed the establishing of the working parameters influence upon the hold-up, by successive modifications of the three independent variables: the rotor speed, the specific flow of dispersed phase  $V_d$  and the specific flow of continuous phase  $V_c$ .

## RESULTS AND DISCUSSION

The visual analysis of the working of the extraction columns which were used for experiments pointed out the following aspects referring to the hold-up dispersed phase:

- for low rotation velocities, the dispersed phase flows on the rotor surface under the form of a film which moves at high speed, following a helicoidal trajectory toward the lower part of the column;
- by gradually increasing the rotation speed in the active zone of the column, a droplet dispersion is formed, characterized by specific shapes, diameters and distributions, according to the stator-rotor slit size and the rotor rotation.

The performed experiments indicated that the dispersed phase hold-up was influenced by the operational parameters: rotation speed  $n$ , dispersed phase flow  $V_d$ , continuous phase flow  $V_c$ , and the constructive characteristics of the column, represented by the size of the annular width,  $\delta$ , and the geometric factor  $F_g$ , respectively, defined as the rotor/stator diameter ratio (Figs. 2, 3 and 4).

The amplification of the rotation causes the increase of the shearing tensions, induced in the two phases, the intensification of the phenomena of continual breaking of the droplets and the decrease of their average diameter.<sup>8</sup> Therefore, the droplet velocity decreases, the stationary time increases, the droplets performing several rotations round the rotor; the global effect is represented by the increase of the hold-up dispersed phase.

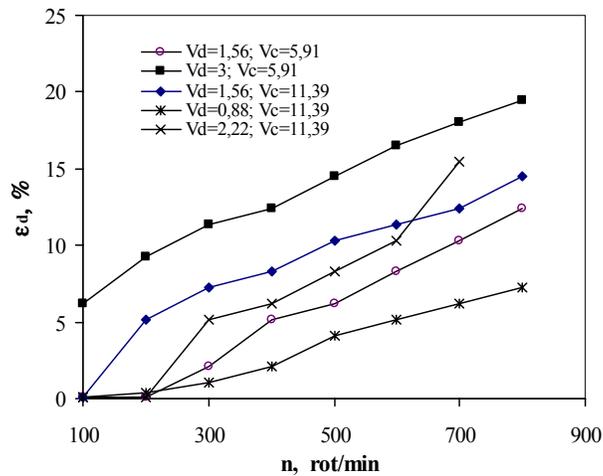


Fig. 2 – The variation of the hold-up according to the column rotation and specific loading for  $\delta = 1$  mm.

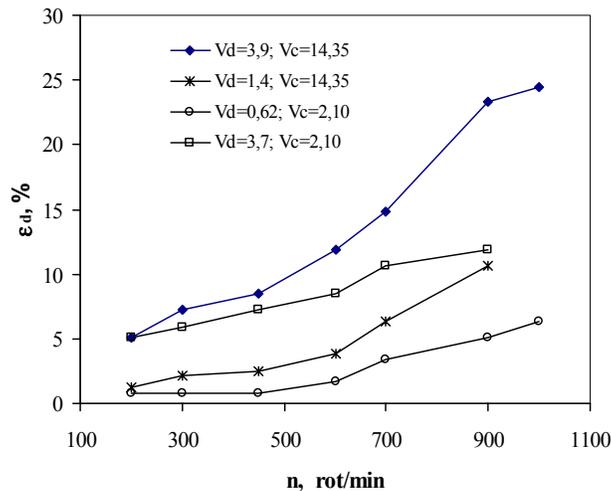


Fig. 3 – The variation of the hold-up according to the column rotation and specific loading for  $\delta = 3$  mm.

To these aspects we can add the complex character of the biphasic flow between two concentric cylinders, when the vortices can only develop in the continuous phase or in phases, their form and behavior being determined by the flowing regime.<sup>6,7</sup> Thus, the increase of the retention by enhancing the dispersed phase flow may be caused by the whirls behavior in the continuous phase, which amplifies, overloading with dispersed phase.<sup>1</sup>

After a certain shearing speed, determined by the size of the stator-rotor slit and the column loading, when the average dimension of the droplets decreases very much, the small droplets gather under the form of some rings with variable thickness, which are tangent to the exterior cylinder, and which move round the rotor and can move toward the axial direction of the continuous phase flow, this phenomenon representing extreme conditions for column working, before reaching the flooding point.

The statistic processing of the experimental data obtained in various operation conditions of the three extraction columns which were used indicated the possibility of using some regression equations, power types (1) or logarithmic types (2), in order to estimate the hold-up.

$$\varepsilon_d = a \cdot n^b \quad (1)$$

$$\varepsilon_d = c \ln U + d \quad (2)$$

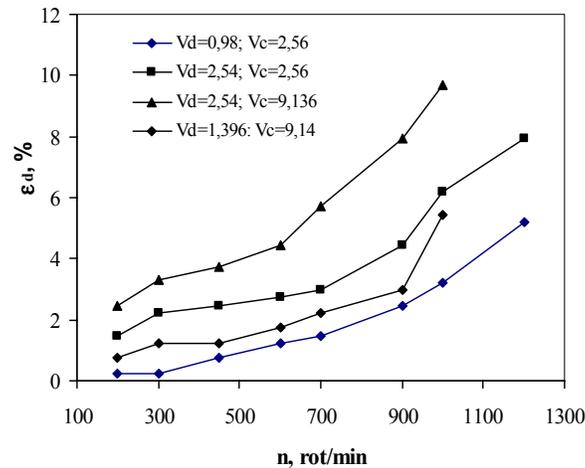


Fig. 4 – The variation of the hold-up according to the column rotation and specific loading for  $\delta = 5\text{mm}$ .

This type of equations, with one variable, has applicability on a restricted field, the values of the coefficients and of the exponents being characteristic to each column and operation field. By dimensional analysis the following adimensional groups were established as significant variables for the analysis of the experimental data:

- Taylor criterion, which correlates the system properties with the rotation and the constructive geometric characteristics of the column;
- $Re_{ax}$  criterion, which correlates the properties and the velocity of the continuous phase;
- the report between the specific flows of the two phases,  $U$ .

A graphical representation of the experimental data which were obtained on the column with  $\delta = 3$ ,  $\epsilon_d$  depending on the product of adimensional groups  $Ta \cdot Re_{ax} \cdot (V_d / V_c)$  is given in Fig. 5.

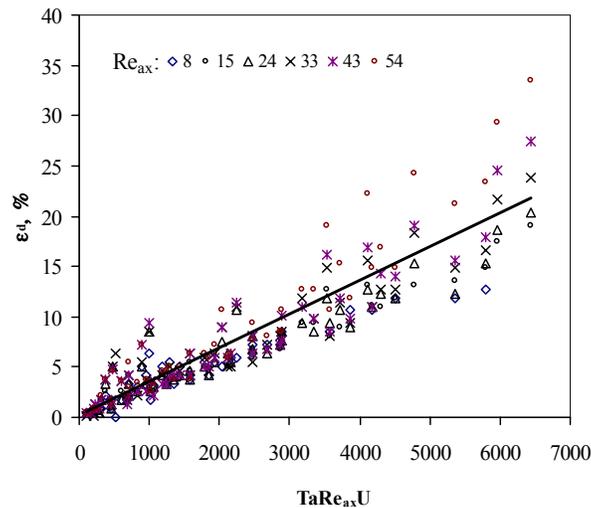


Fig. 5 – The variation of the data regarding the hold-up.

By using a statistic calculation application, based on multiple linear regressions, we have established criterial relations (3) for each column, the value of the coefficients and of the exponents being given for the three columns in Table 2.

$$\epsilon_d = aTa^b Re_{ax}^c (V_d / V_c)^d \quad (3)$$

Table 2

The parameters of the equations for the calculation of the hold-up

| $\delta$ , [mm] | a        | b     | c     | d      |
|-----------------|----------|-------|-------|--------|
| 1               | 0.547    | 0.68  | 0.931 | 0.998  |
| 3               | 0.000111 | 1.194 | 0.94  | 0.8004 |
| 5               | 0.00145  | 0.829 | 0.814 | 0.712  |

The correlations, valid for each given system and every type of column, allow the estimation of the hold-up for different loadings of the column and the whole rotation field of the rotor. A comparison between the experimental data and the calculated ones with the suggested equations is shown in Fig. 6, with a  $\pm 27\%$  variation.

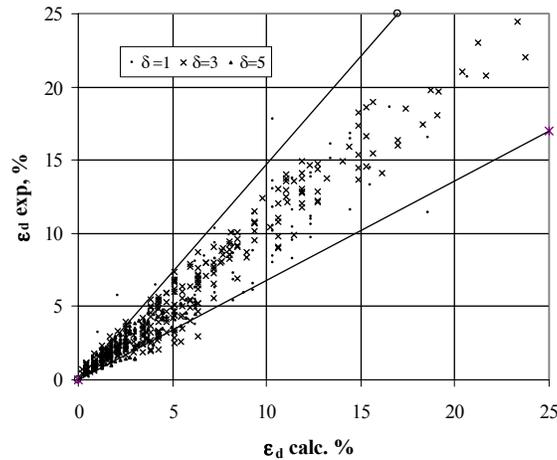


Fig. 6 – The comparison between the experimental data and the ones computed.

The communicated studies referring to the hold-up in columns with cylindrical rotor are few, Davis<sup>1</sup> indicating a general correlation of the type:

$$\varepsilon_d \propto (Ta \cdot F_g)^2 \cdot \delta^{-3} \cdot V_d^{1/2} \cdot 10^{-6} \quad (4)$$

The equation verifies, with an acceptable precision, only the data obtained in the column with  $\delta = 3$  mm. A relation with a general applicability for any column and system was obtained by processing the experimental data using the correlation suggested by Davis<sup>1</sup>, in which the  $Re_{ax}$  criterion was introduced, taking into account the influence of the continuous phases speed:

$$\varepsilon_d = 10^{-6,113} (Ta \cdot F_g)^{0,9582} Re_{ax}^{0,1742} V_d^{0,817} \delta^{-2,6} \quad (5)$$

The equation presents a general applicability, ensuring an estimation of the hold-up with a variation of  $\pm 15\%$ , as opposed to the experimentally determined values.

## CONCLUSIONS

Following the performed experiments, there resulted a great functioning flexibility and stability for the column characterized by an increase of the stator-rotor slit size,  $\delta = 3$  mm. In this column, the operation variables modified within large fields, with a good reproducibility of the experimental data.

The droplets are evenly distributed on the whole height of the rotor, without zones with uneven distribution, droplets overcrowding, or stagnant zones on the rotor surface, these phenomena being noticed when the other two columns were functioning.

For the assessment of the hold-up we established a general calculation equation which leads to corresponding values for the whole operation field.

The value of the maximum hold-up in the three types of columns varies from 9% to 25% for the water-carbon tetrachloride system; given the used experimenting conditions, the minimum values are obtained in the column with  $\delta = 5$  mm, where the flowing section is much greater, the reduced value of the shearing tensions ensuring a free movement of the droplets.

#### Nomenclature

|           |  |
|-----------|--|
| $D_s$     | stator diameter, m   |
| $D_r$     | rotor diameter, m  |
| $F_g$     | geometric factor, $F_g = D_r/D_s$                              |
| $n$       | rotor speed rot/min  |
| $R_r$     | rotor radius, m;   |
| $Re_{ax}$ | axial Reynolds number, $Re_{ax} = (\rho_c u_c \delta)/\eta_c$  |
| $Ta$      | Taylor number, $Ta = (\rho_c n \delta^{3/2} R_r^{1/2})/\eta_c$ |
| $U$       | report flow faze, $U = V_d/V_c$                                |
| $V_c$     | specific flow of continuous phase, $m^3/m^2h$                  |
| $V_d$     | specific flow of disperse phase, $m^3/m^2h$                    |
| $\delta$  | annular width, m;  |
| $\rho_c$  | density of continuous phase, $kg/m^3$                          |
| $\eta_c$  | viscosity of continuous phase, $kg/(ms)$                       |

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