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Technological design elements for pulsed extraction columns with sieve trays

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Abstract Solvent extraction process is frequently used to obtain chemical compounds used in the nuclear field. In order to make nuclear energy a safe, clean and cheap energy source, one has resorted to the identification of new, innovative methods in the immediate transfer of the laboratory results to an industrial scale. A method is described for the design of liquid extraction columns by scaling up from the results of small scale plant tests. Such a method was applied for scaling up a pulsed column with sieve trays using the dimensions of an experimental model column. The scale-up method is based on the assumption that the height of the column is independent of column diameter, and that increased backmixing accounts entirely for differences in performance on small and large scales. This paper uses the results obtained on an experimental model column for the purpose of designing a high capacity column. The final result consists in determining the diameter and height of a scaled up column by using like parameter the number of transfer units corresponding to a compartment, a value that will stay the same for the laboratory-scale column and for the large-scale (pilot or industrial) column.

Keywords: solvent extraction, pulsed column, scaling up, backmixing, pulsed column design

1. Introduction

For nuclear energy to be considered a safe source of energy, clean and cheap, which would contribute to the fulfillment of the Green Deal is urgently needed to identify the latest solutions, models, methods, procedures for carrying out all the processes related to the operation of a Nuclear Power Plant, [1].

The optimization of the solvent extraction process used for nuclear purposes can be very useful for obtaining chemical compounds of nuclear interest. There are many types of equipment with trays [2; 3] and procedures for mass transfer; the most efficient equipment for the process of solvent extraction proved to be the extraction

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columns with sieve trays. A key element that defines the efficiency of this kind of equipment consists in the selection of a correct design method that will ensure the determination of the optimal dimensions that will allow the correct operation with maximum efficiency of this kind of equipment, [4; 5].

The Romanian school was preoccupied with the intensification of the transfer processes right from the beginning of the researches in the field of chemical engineering, an example in this sense being the works of professor Emilian Bratu, [6 - 9].

In addition to a rigorous design of such equipment, consideration must be given to extending their long and safe operation time by avoiding mechanical or corrosive degradations, [10 - 13].

The design of columns for liquid - liquid extraction involves the determination of two independent factors:

1. the diameter required to ensure the desired capacity;
2. the height required by the degree of separation of the targeted chemical compounds. Ideally, the two dimensions should be determined by theoretical calculations without resorting to other experiments, except for those necessary to obtain phase equilibrium data.

As far as the diameter is concerned, the theoretical calculation is possible because in the literature there are satisfactory correlations of the diameter depending on the limit (eg flooding point) for most types of columns where there is no interfacial turbulence due to surface tension gradients (eg the effect Marangoni), [14].

The calculation of the column height is more difficult as it requires the assessment of the following characteristic quantities:

- the area of the phase contact surface which is determined by droplet dimensions and retention (defined as the average percentage of the total volume of the apparatus occupied by the dispersed phase);
- partial mass transfer coefficients;
- degree of axial dispersion in one or both phases.

There have been developed theoretical methods for calculating the height of columns with sieve trays, in which axial dispersion is absent and for the packed column provided that interfacial oscillations does not occur. Even these simple cases lead to differences between theoretically calculated values and real ones, [14; 15].

For these reasons, in the case of new processes, it is necessary that the design should be based on previous experimentation involving small-scale equipment testing. Experimentation with small equipment, in convenient conditions and with easily accessible materials, is an important advantage of using models as a working tool.

In the past experiments carried out on a model column for solvent extraction we obtained information on: extraction efficiency, the column hydrodynamic behavior, optimal pulsation intensity (amplitude - frequency) that will be used in the calculation of scaling up for the design of a larger size column.

2. Experimental

Scaling up method. Formerly, scaling up was based on the ideal plug-flow model based on the number of transfer units (or stages). These methods, which are satisfactory for mixer-settler devices and for columns with unpulsed sieve trays, are not useful for obtaining good results with other types of contactors due to the undesirable effects of back mixing occurring in them.

Sleicher (1959-1960) proposed two types of of back mixing model, later developed by others (Miyauchi and Vermeulen, 1963; Hartland and Mecklenburg, 1966) called the diffusion model and the backflow models, applicable respectively to differential and to stagewise (compartmental) columns. These models underlie the method of scaling up presented in this paper, [14 - 16].

2.1 Difusion model

The mass transfer, in the case of differential contactors, depends on the mass transfer coefficient, the interfacial and the concentration gradient. The first two may be expressed in the form of transfer units (H_{ox} or H_{oy}) which, considering the polydispers nature of the process, is given as follows, assuming the X (for example feed) phase to be dispersed:

$$H_{ox} = \frac{V_x}{(k_{ox} \cdot a)_{av}} \qquad (1)$$

$$H_{ox} = \sum_{i=1}^{n} \frac{V_x \cdot d_i \cdot \phi(\Delta C_x)}{6\, k_{ox,i} \cdot x \cdot f_i} \qquad (2)$$

where: V_x - is the surface velocity of phase x [m/s]; $(k_{ox})_{av}$ - is the average total mass transfer coefficient relative to phase x [m/s]; $(a)_{av}$ - is the average area of the interphase contact surface [m^2]; d_i - droplet diameter of dimension i [m]; $\phi(\Delta C_x)$ - is the correction caused by the polydispersity effect upon the concentration gradient; $\phi(\Delta C_x) \leq 1.0$; x –the fractional retention of the dispersed phase; f_i – the volume fraction of d_i drops that are in retention.

For any system, the $k_{ox,i}$ value will be constant for the same droplet size. Consequently H_{ox} will also be constant in the case of droplet size distribution in a steady state and the concentration gradient will be given by:

$$H_{ox} \frac{dc_x}{dz} = c_x - c_x^* \qquad (3)$$

where: c_x is the solute concentration in phase X [kg/m^3]; c_x^* - the solute concentration in phase X at equilibrium with phase Y [kg/m^3]; z – distance along the column [m].

The concentration gradient will be maximum when the driving force ($c_x - c_x^*$) is maximum. The column operation is more efficient when the phases flow in a counter current way and the height required for a given capacity obtained by integrating equation (3) is therefore minimal. Any deviation from the ideal flow pattern, due to the back mixing of one or both phases, changes the shape of the operating curve, which will be convex to the equilibrium curve, consequently reducing the concentration gradient [17].

The method of scaling up the differential equipment by using the diffusion model is based on the consideration that H_{ox} has the same value in the model column and in the extrapolated column, the differences in performance coming from the different degree of back mixing.

2.2 Backflow model

For compartment type (stagewise) it is recommended to use the backflow model which involves the complete mixing of the phases in contact and the occurrence of back flow caused by the mutual entrainment of the phases leaving the compartment. In this case, the mass transfer is expressed according to the parameter N_{ox}^1 that represents the number of real mass transfer units corresponding to a compartment (the space between two trays), defined as:

$$N_{ox}^1 = \left(k_{ox} \cdot a\right)_{av} \cdot h_c / V_c \tag{4}$$

where: h_c is the height of a compartment [m]; V_c - surface velocity of phase X [m/s]; N_{ox}^1 - is related to Murphree's efficiency through the relation:

$$E_{Mx} = \mathrm{N}_{\mathrm{ox}}^1 /(1 + N_{ox}^1) \tag{5}$$

The value N_{ox}^1 is assumed to remain constant under similar agitation conditions and is used for scaling up, [18 - 20].

3. Results and discussions

Extrapolation of the model column. This paper uses the dimensions of a model (laboratory) column, making the point, at the same time, that the method may be equally well used for scaling up any pulsed column with sieve trays, used for solvent extraction, on which experimental measurements can be made. The purpose of laboratory-scale experiments is to obtain data necessary for the design of equipment on an industrial scale, [21 - 23].

The following requirements must be taken into account for scaling up:

1) the internal column characteristics to be maintained so that we might have the same back mixing in the model and in the equipment on an industrial scale. In the case of pulsed columns with sieve trays, the following remain constant: tray geometry, distance between trays and the product of amplitude x frequency.

2) if the equilibrium relation is nonlinear it can be approximated as linear on smaller areas of concentration to allow the use of analytical methods in order to scale up.

It should be emphasized that interfacial turbulence is the most difficult problem encountered in the design of solvent extraction equipment and it is difficult to establish a satisfactory theoretical model to specify its effects on extractor performance and capacity.

Calculation of the column diameter. The diameter of the extrapolated column is obtained by imposing the condition that the sum of the surface flow velocities of the two phases (organic and aqueous) be the same in the model column and in the extrapolated column, [21], namely,

$$V_{c,m} + V_{d,m} = V_{c,ex} + V_{d,ex} \qquad (6)$$

where $V_{c,m}$, $V_{c,ex}$ is the surface flow velocity of continous phase in the model column, respectively, in the extrapolated column; $V_{d,m}$, $V_{d,ex}$ is the surface flow velocity of dispersed phase in the model column, respectively, in the extrapolated column.

Tacking into account the following notations

$$V_{c,m} = \frac{G_{c,m}}{\frac{\pi d_{c,m}^2}{4}}; V_{d,m} = \frac{G_{d,m}}{\frac{\pi d_{c,m}^2}{4}} \quad ;$$

$$V_{c,ex} = \frac{G_{c,ex}}{\frac{\pi d_{c,ex}^2}{4}} \quad ; V_{d,ex} = \frac{G_{d,ex}}{\frac{\pi d_{c,ex}^2}{4}}$$

$$V_{c,m} + V_{d,m} = \frac{G_{c,m} + G_{d,m}}{\frac{\pi d_{c,m}^2}{4}} \quad ;$$

$$V_{c,ex} + V_{d,ex} = \frac{G_{c,ex} + G_{d,ex}}{\frac{\pi d_{c,ex}^2}{4}}$$

relation (6) becomes,

$$\frac{G_{c,m} + G_{d,m}}{\frac{\pi d_{c,m}^2}{4}} = \frac{G_{c,ex} + G_{d,ex}}{\frac{\pi d_{c,ex}^2}{4}},$$

where from:

$$d_{c,ex} = d_{c,m}\sqrt{\frac{G_{c,ex} + G_{d,ex}}{G_{c,m} + G_{d,m}}} \qquad (7)$$

with $d_{c,m}$= 0.027m; $G_{c,m}$= 0.6 l/h; $G_{d,m}$= 0.2 l/h; $G_{c,ex}$= 60 l/h; $G_{d,ex}$= 20 l/h one obtains $d_{c,ex}$= 0.27m.

Calculation of column height. The height of the extrapolated column is determined by calculating the number of compartments necessary to obtain the required degree of separation and being aware of the fact that the height of a compartment is the same in the model column and in the column whose dimensions we want to determine.

The recommended procedure consists in substituting the experimental value of the exit extract phase composition, together with the estimated backmixing parameters, the column length or number of compartments and the extraction factor E into the appropriate model solution and solving for height column. This value is then assumed to apply to the fullscale equipment and is substituted together with the corresponding backmixing parameters and desired exit composition into the same model to obtain the height or number of stages required.

Analytical solutions are used when the equilibrium can be described by a linear relation, such as,

$$C_x^* = m \cdot c_y + q \tag{8}$$

where: m – slope of equilibrium straight line; q – ordinate at the origin of the equilibrium line.

With this purpose in view, the inlet stream and the extract concentrations are expressed in dimensional form featuring the following expressions for the back flow model:

$$X = \frac{m\,C_y^{N+1} + q}{C_x^o - (mC_y^{N+1} + q)} \cdot C_x^o - (mC_y^{N+1} + q) \tag{9}$$

$$Y = \frac{m(C_y^1 - C_y^{N+1})}{C_x^o - (mC_y^{N+1} + q)} \tag{10}$$

where: C_x^o ; C_x^N - concentration of the solute in the aqueous phase, at the inlet and, respectively, at the outlet [g/l]; C_y^{N+1}; C_y^1 - concentration of the solute in the organic phase, at the inlet, and, respectively, at the outlet [g/l].

The appropriate solutions for the back flow model are the following:

$$X_n = A_1 + A_2 \cdot \mu_2^n + A_3 \cdot \mu_3^n + A_4 \cdot \mu_4^n \tag{11}$$

$$Y_n = A_1 + A_2 \cdot a_2 \cdot \mu_2^n + A_3 \cdot a_3 \cdot \mu_3^n + A_4 \cdot a_4 \cdot \mu_4^n \tag{12}$$

where *n* is the number of compartments while μ_i are the roots of the characteristic equation in (μ – 1); the back mixing is expressed as the degree of back mixing δ_j defined as the ratio between the amount of material returning and the amount of material moving forward (in the desired direction), [14].

In short, the scaling up procedure is as follows:

1) the values of the back mixing parameter δ_j are obtained for the model column and for the large-scale column, from the appropriate correlations;
2) one replaces values Y^1, δ_j, F (extraction factor) obtained for the model column in the expressions for Y^1 and Γ^1 and we associate them with the characteristic equation to be solved for N_{ox}^1 (Hartland and Mecklenburg), [16];
3) one replaces the N_{ox}^1 value together with Y^1 and the back mixing parameter δ_y, estimated for the scaled up column in the simplified solution of the back flow model, [14]. By solving this equation one obtains the number of column compartments.

The calculation of extrapolated column height. One writes X for the aqueous dispersed phase $(M_xO_y(AO_z)_w)$=M and Y for the continuous organic phase (Tri Butyl Phosphate TBP - Kerosene). The concentrations in the two phases are as follows:

$$c_x^0 = 434\ g\ M\ /l\ ;\ c_x^N = 1.66\ g\ M\ /l; c_y^1 = 123.17\ g\ M\ /l; c_y^{N+1} = 0$$

The equilibrium relation is:

$$c_x^* = c_y \tag{13}$$

The flow rates in the model column are: G_a=200ml/h – flow rate of aqueous phase and G_o=600ml/h – flow rate of organic phase.

Non - dimensional concentration Y^1:

$$Y^1 = \frac{c_y^1}{c_x^0} = 0.254 \tag{14}$$

Extraction factor F:

$$F^0 = \frac{mG_{n,in}}{G_{0,ies}} \tag{15}$$

$$F^1 = \frac{mG_{n,ies}}{G_{0,in}}, \text{ with m = 1} \tag{16}$$

The densities of the two phases are $\rho_1[M]$ - continous, and $\rho_2[TBP - Kerosen]$-dispersed.

$G_{A,\,ies} = G_{A,\,in} - G_{A,\,in} \times c^o{}_x$, $\rho_1[M] = 1450\ kg/m^3$, yields $G_{A,\,ies}$ = 0.133 l/h

$C_{0,\,ies} = G_{0,\,in} - G_{A,\,in} \times c^o{}_x$, $\rho_2[TBP - Kerosen] = 973\ kg/m^3$, yields $C_{0,\,ies}$ = 0.667 l/h

Relations (15) and (16) yield:

$F^o = \dfrac{0.200}{0.667} = 0.29285$ and $F^1 = 0.2217$, based on which one calculates:

$F_{med}=(F^0 + F^1)/2$ resulting F_{med}=0.2608

We consider the aqueous phase (M), X, as dispersed and the organic phase (TBP-Kerosene), Y, as continuous. One calculates the value of Γ^1 with the relation:

$$\Gamma^1 = \frac{Y^1}{F} \tag{17}$$

resulting in Γ^1=0.974.

Because the back mixing takes place only in the continuous phase, $\alpha_x = 0$.

For the continuous organic phase, α_y is calculated with the relation:

$$\alpha_y = \left(d_{c,m}\right)^{0,802}\left(f \cdot A\right)^{0,101}\left(0.1703 + 0.3017\frac{V_d}{V_c}\right) \tag{18}$$

where: d_{cm} - diameter of the model column [cm]; (f.A) - frequency - amplitude product [cm/s], results for the model column α_y=2.691, this one featuring frequency f=1.5 cycles/s and amplitude A=2.2 cm, [15].

Having calculated these values, one uses the Harland and Mecklenburg method [15] to obtain N_{ox}^1 (the number of transfer units corresponding to a compartment).

The Harland and Mecklenburg equations are as follows:

$$A_i^2\left(1 = \alpha_y\right)\left(1 + N_{0x}^1\right) - \left[1 + 2\,\alpha_y + N_{0x}^1\left(F + \alpha_y\right)\right]A_i + \alpha_y = 0 \tag{19}$$

$$g_i = \frac{1 - \alpha_x\left(A_i - 1\right)}{1 + \left(1 + \alpha_y\right)\left(A_i - 1\right)} \tag{20}$$

$$i=\frac{\dfrac{g_1A_1-A_1^N}{(A_1-1)g_1}-\dfrac{g_3A_3-A_3^N}{(A_3-1)g_3}}{\dfrac{1-F\cdot A_1^N}{(A_1-1)g_1}-\dfrac{1-F\cdot A_3^N}{(A_3-1)g_3}} \qquad (21)$$

We obtain the value Γ^1 calculated by using the relation (17).

Next, the calculation algorithm consists of the following steps:

1. a value is proposed for N_{ox}^1;
2. one replaces the known values and one solves the equation of degree II (19) in A; one obtains A_1 and A_3;
3. one calculates g_1 and g_3 using equation (20);
4. one calculates Γ^1 (entering the values A_1, A_3, g_1, g_3 obtained) in equation (21) and one compares them with the value of Γ^1 obtained from equation (17);
5. if the two Γ^1 values coincide or are very close then the value assumed for N_{ox}^1 is good;
6. if the two values of Γ^1 do not coincide, another value is assumed for N_{ox}^1 until the condition listed under point (5) is met.

The iterative calculation performed to obtain the value N_{ox}^1 according to the algorithm described above is presented in Table 1.

Table 1 Parameters used for the calculation of the N_{ox}^1 value

No.	N_{ox}^1	A_1	A_3	g_1	g_3	Γ^1
1	0.1	0.938	0.714	1.2973	-17.512	0.865
2	0.2	0.894	0.679	1.64	-5.41	0.965
3	0.25	0.882	0.662	1.772	-4.04	0.978
4	0.23	0.887	0.668	1.715	-4.443	0.974

We obtained N_{ox}^1=0.23 for the model column, a value that remains constant for the up-scaled column whose diameter was calculated as $d_{c,ex}$=27cm.

One calculates the degree of back mixing for the extrapolated column with formula (18) and one obtains the value $\alpha_{y,ex}$=4.2961.

The simplified solution of the back flow model given by Pratt [17] is:

$$N=\frac{\log\dfrac{a_4(\mu_4-\mu_3)(F-Y^1)}{F^2(1-\mu_3)\mu_4(1-Y^1)}}{\log\mu_4} \qquad (22)$$

valuable for $F\neq 1$, x=0 and $y\neq 0$, where F=0.2608 and Y^1=0.254, μ_3 and μ_4 are the roots of the characteristic equation of the second order in (μ-1) given below:

$$(1+\alpha_y)(1+N_{ox}^1)(\mu-1)^2+[1+N_{ox}^1(2-F+\alpha_y)](\mu-1)+N_{ox}^1(1-F)=0 \qquad (23)$$

μ_3 is the lesser root and a_4 is calculated with relation:

$$a_4 = F\ \frac{1-\alpha_x(\mu_4 - 1)}{\mu_4 + \alpha_y(\mu_4 - 1)} \qquad (24)$$

By substituting the known values in equations (22), (23) and (24), we have obtained: $\mu_3 = 0.73$; $\mu_4 = 0.9033$; $a_4=0.53457$ and N=29.3. The value obtained from the N=29.3 calculation is rounded to N=30.

The height of the extrapolated column is calculated with the relation:

$$H_{c,ex} = N^* h_c$$

where: h_c is the height of a compartment and N is the number of compartments.

With $h_c=0.05$ m one obtains $H_{c,ex} = 1.5$ m.

The dimensions of the extrapolated column are:

- column diameter: $d_{c,ex}=27$ cm
- column height: $H_{c,ex}=150$ cm
- height of a compartment: $h_{comp,\ ex}=5$ cm
- diameter of tray: $d_{tray,\ ex}=26.22$ cm
- free tray area: a=23%
- diameter of tray holes: d=2 mm
- pulsation amplitude: A=2.2 cm
- pulsation frequency: f=1.5 Hz

4. Conclusions

A scaling method was developed for scaling up a pulsed column with sieve trays using the results obtained on an experimental model column.

The method can be used to up-scale or down-scale any pulsed column for which experimental measurements can be made.

The up-scaling method proposed in this paper has a more rigorous theoretical foundation than the methods based on plug-flow and therefore will be used predominantly whenever we have experimental results

The method is based on the hypothesis that N_{ox}^1 (the number of transfer units corresponding to a compartment) remains constant being equal for the model column and for the column whose dimensions we want to determine

For pulsed columns with sieve trays, this method implies that while the up-scaled column diameter increases, by maintaining fluid velocities constant, the height of the compartment remains unchanged.

The method is based on the assumption that the equilibrium relation for the contact phases is a linear relation.

The application of the above-mentioned calculation yielded values for the dimensions of an up-scaled pulsed column with sieve trays.

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