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Column Design Parameters for Foam Fractionating Human Placental Extract*

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ABSTRACT

In the present investigation, column design parameters for foam fractionation of human placental extract have been studied. A design procedure using the transfer unit concept has been presented. The number of transfer units has been computed with the help of a quasi-equilibrium diagram especially constructed for the present system. In order to determine the height of a transfer unit, an empirical correlation for the present system has been proposed.

INTRODUCTION

In the isolation and purification of enzymes and proteins from biological sources, the foam fractionation technique has been used successfully by Ahmad (1), Lalchev and Exerowa (2), Lalchev et al. (3), Sarkar et al. (4), and Bhattacharya et al. (5). This separation technique has recently drawn a lot of attention as it is very effective in isolating and purifying environmentally sensitive (pH, temperature, chemicals, etc.) surface-active materials. It is therefore important to evaluate design parameters for such foam fractionating columns. In the present work a design procedure based on the concept of transfer units has been used and the column design parameters evaluated.

* The authors dedicate this paper to Prof. R. N. Mukherjea on his sixty-fifth birthday.
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THEORY

The concept of transfer units is useful in designing a foam fractionation column. The number of transfer units (NTU) for such a foam fractionation column may be computed from the equation

$$\text{NTU} = \int_{X_B}^{X_F} \frac{d\overline{X}}{\overline{X}^* - \overline{X}} \tag{1}$$

where $\overline{X}_B^*$ = effective concentration of solute protein, $\overline{X}$, in equilibrium with X_B (kg/m^3)
X_F = concentration of protein in the overflowing foam on a gas-free basis (kg/m^3)
X_B = concentration of protein in the effluent (kg/m^3)
$\overline{X}^*$ = effective upflow protein concentration in equilibrium with X on a gas-free basis (kg/m^3)
$\overline{X}$ = effective upflow protein concentration on a gas-free basis (kg/m^3)
X = protein concentration in liquid (kg/m^3)

A few investigators [Lemlich (6), Goldberg and Rubin (7)] have suggested analytical equations for calculating NTU, assuming a linear equilibrium isotherm. However, this procedure cannot be used for the present system since the equilibrium relationship between solute concentration in the foamate and in the residue is not known. This necessitates computation of the equilibrium data from experiment and then plotting these values in the form of an equilibrium diagram. In the present investigation, as the solute protein is not a unique component, it is not possible to obtain true equilibrium data. However, a quasi-equilibrium relationship between the solute concentration in the foamate and that in the effluent may be obtained experimentally.

The calculation of NTU again requires knowledge of the material balance of the solute to give the equation for the operating line. An overall material balance around the entire column may be written as

$$L = F + B \tag{2}$$

While the solute balance gives

$$LX_L = FX_F + BX_B \tag{3}$$

or

$$X_F = (L/F)X_L - (B/F)X_B \tag{4}$$

where L = feed solution flow rate (m^3/s)
F = flow rate of the foamate (m^3/s)
B = flow rate of the effluent (m^3/s)
X_L = concentration of protein in the feed solution (kg/m^3)

Equation (4) presents a straight line having a slop (L/F) and a negative intercept on the ordinate [$(B/F)X_B$]. Thus, by knowing the flow rates of the individual streams, the operating line can be constructed. Since only the terminal conditions have been used to construct the operating line, some error is expected. However, for dilute feed concentrations the possibility of departure from linearity is small.

Arbitrary equations for evaluating the height of a transfer unit (HTU) has been given by Haas and Johnson (8) and Goldberg and Rubin (7). However, these equations are not applicable for the present system for the reasons stated above. Thus an attempt is made here to obtain an empirical correlation for the HTU. This correlation contains parameters which could be determined experimentally.

In the present investigation, a correlation for HTU of the type given below was used and later tested using experimental data.

$$\text{HTU} = \phi \frac{Ld}{K_L S} \tag{5}$$

where d = average foam bubble diameter (m)
S = foam column cross-sectional area (m^2)
K_L = overall mass transfer coefficient (m/s)

In Eq. (5), ϕ is a dimensionless constant, the value of which is expected to differ from system to system. K_L can be evaluated from the equation for mass flux:

$$N_A = K_\mu \Delta\mu / \Delta C \tag{6}$$

or

$$K_L = K_\mu \Delta\mu / \Delta C \tag{7}$$

where K_μ = mass transfer coefficient ($kg \cdot kmol/kJ \cdot m^2 \cdot s$)
$\Delta\mu$ = chemical potential difference (kJ/kmol)
ΔC = overall concentration difference based on the liquid phase (kg/m^3)
$= X_F - \overline{X}_F^*$

The value of K_μ can be evaluated experimentally (4). The average foam bubble diameter, d, may be a calculated from [Lemlich (6)]

$$d = n_i d_i^3 / n_i d_i^2 \tag{8}$$

where n_i is the number of bubbles of diameter d_i in a representative portion of the foam. For nonuniform bubbles, a surface value for the diameter is employed in Eq. (8).

The chemical potential difference Δ_μ can be calculated from

$$\Delta\mu = RT \ln(X_B/\overline{X}_F^*) \tag{9}$$

where $\overline{X}_F^*$ = effective concentration of solute protein, $\overline{X}$, in equilibrium with X_B (kg/m^3)
R = gas constant (8.3143 kJ/kmol·K)
T = temperature (K)

Equation (7) then becomes

$$K_L = K_\mu \frac{RT \ln(X_B/\overline{X}_F^*)}{\Delta C} \tag{10}$$

Since the value of $\Delta\mu$ can be evaluated with the help of the equilibrium curve, all the parameters in Eq. (10) can be determined experimentally. If one gets reasonably identical values of ϕ for different known values of HTU on substitution of those parameters in Eq. (5), it may be concluded that Eq. (5) can represent the HTU satisfactorily.

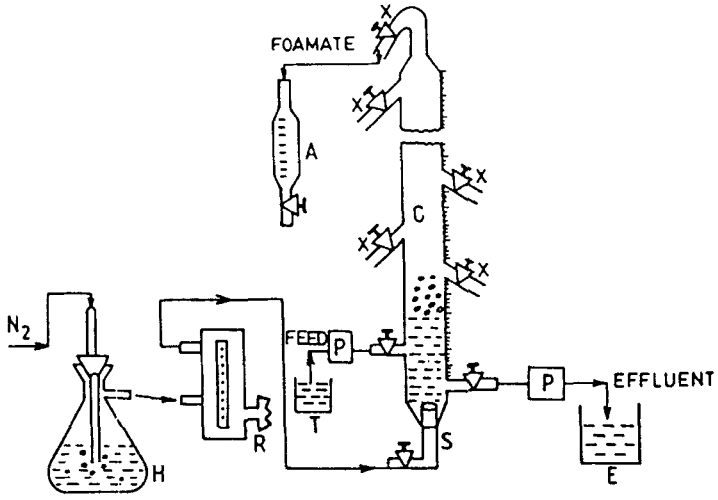
EXPERIMENTAL METHODS

Preparation of the Feed Solution

Human placenta was collected from Chittaranjan National Medical College, Calcutta, India. The tissue was kept in ice and was separated from the membrane. It was then cut into small pieces and washed with ice-cold distilled water to remove the blood. The tissue was then dried by absorbent paper and homogenized with a suitable amount of Tris-HCl buffer (pH 8.0) until the tissue dispersed homogeneously and only fats and cell debris were left behind. The homogenized solution was centrifuged in a cold centrifuge at 10,000 rpm for 30 minutes, and the supernatant solution (5% placental extract) was stored frozen until further use. This stock solution of placental extract was diluted to the desired concentration by adding buffer.

Experimental Setup

A detailed diagram of the continuous foam fractionation column used in the present investigation is shown in Fig. 1. The graduated column, 50 cm high, with an inside diameter of 4 cm, was made of Corning glass. It was fitted with a sparger of 100 μm porosity, a feed inlet, an effluent



A — Foamate collector
C — Foam column
P — Peristaltic pump
T — Feed tank
R — Rotameter
H — Humidifier
S — Sparger
E — Effluent collector
X — Stop cocks for collection of foam at different heights

FIG. 1 Detailed diagram of the continuous foam fractionating column.

outlet, and a foam outlet nozzle. In addition, the column was fitted with six intermediate foam outlet nozzles at 5 cm intervals. The foam height was varied by using these intermediate nozzles.

Nitrogen, which was used as the carrier gas, was saturated by bubbling through water in a conical flask; its flow rate was measured by a rotameter. The liquid flow-rates were measured by volume collection. Two peristaltic pumps were used to change the liquid flow-rates. The foam outlet nozzles were kept inclined at an angle of 30°. The foam collapsed while passing through this bend. Hence, no separate arrangement for foam breaking was required.

Protein Concentration/Enzyme Activity Measurement

The surface tension vs concentration plot was established by measuring the surface tension of placental homogenate using the Wilhelmy method

(9). The threshold and critical micelle concentration limits of protein concentration in the placental extract were found to be 0.00435 and 0.625 kg/m^3, respectively, at 10°C. The total protease activity was determined by the casein digestion method as suggested by Leonard and coworkers (10). The protein measurement was done by the colorimetric method of Lowry et al. (11).

During the experiment a constant liquid height was maintained inside the column by proper adjustment of the effluent flow-rate. Samples of foamate and effluent were collected for analysis after a steady state was achieved for each run. The experiment was conducted in a constant temperature room maintained at 10°C.

Equilibrium Data Collection

For evaluating the equilibrium data the fractionator was used in the batch mode. The placental homogenate stock solution was diluted with buffer to a protein concentration of 0.204 kg/m^3. The initial height of the liquid was kept at 0.08 m in each run. Nitrogen gas saturated with water was allowed to pass at a moderate rate until no more foam rose up the column. Samples of foamate and effluent were collected for analyses.

Keeping the quantity and concentration of the feed the same as in the first run, subsequent experiments were carried out by gradually decreasing the nitrogen flow-rate until a further decrease ensured no rise of foam bubbles up the column. The above method was repeated for different feed concentrations. All the runs were repeated four times, and no significant changes in the experimental values were observed. The average of these four runs has been reported as a single set.

Bubble Diameter Determination

Bubble size was measured by the methods of Shih and Lemlich (12), Goldberg and Rubin (7), and Grieves et al. (13). For each operating condition, photographs of a section of the graduated column were taken above the liquid height, and the bubble size was obtained from the enlarged photographic prints. The diameters of several bubbles were measured on each print, and the mean of a normal distribution was obtained following the method of Lemlich (6).

Determination of Mass-Transfer Coefficient

The mass-transfer coefficient, K_μ, was determined experimentally by the method described by Sarkar et al. (4), and the value was found to be 8.82×10^{-11} kg·kmol/kJ·m^2·s.

RESULTS AND DISCUSSION

Equilibrium Curve

As mentioned earlier, it is difficult to construct a true equilibrium diagram for the present system because the solute protein is not a unique component. From our knowledge of human placental extract, it can be said that besides other nonproteinaceous substances, the main constituents are a mixture of nonenzymic proteins and enzymes. Fortunately, nonproteinaceous substances are not surface active, and thus will not be separated during foam fractionation. However, being surface active, all protein components will compete for adsorption on the foam bubble surface. This renders it difficult to construct a true equilibrium curve. On the other hand, remembering the importance of such an equilibrium curve in designing the column, an attempt was made in the present investigation to obtain a quasi-equilibrium curve showing the relationship between the protein concentration in the foamate and that in the effluent liquid. Total protein analyzed during the experiment was considered to be the solute concentration. It is believed that such a quasi-equilibrium curve will be of use in the absence of available data for the present system. While true equilibrium is a function of intensive thermodynamic properties only, the need for such a quasi-equilibrium curve is apparent because kinetics, flow behavior, hydrodynamics, and other rate effects are expected to alter the equilibrium condition, as in the present case.

The quasi-equilibrium relationship between the protein or solute concentration in the foamate and in the effluent is shown in Fig. 2. The procedure for obtaining the relevant data has already been described. The temperature was kept constant at 10°C during experimental runs. Conceptually, equilibrium condition within the foam column is attained when the residence time of an individual foam bubble is sufficiently high. This can be achieved by maintaining the lowest possible gas flow-rate. In the present investigation, some foam bubbles collapsed and some ascending foam bubbles entrained in the liquid while the lowest gas flow-rate was maintained.

Transfer Units

The operating line (Eq. 4) has been superimposed on the equilibrium diagram (Fig. 2). For a particular set of runs, the limits of the integral in Eq. (1) are known. In order to solve the equation for several known values of protein concentrations in the effluent, the corresponding equilibrium concentrations of solute protein ($\overline{X}^*_B$) were computed from the equilibrium curve. In order to obtain values of $\overline{X}^*$ and $\overline{X}$, once again the equilibrium

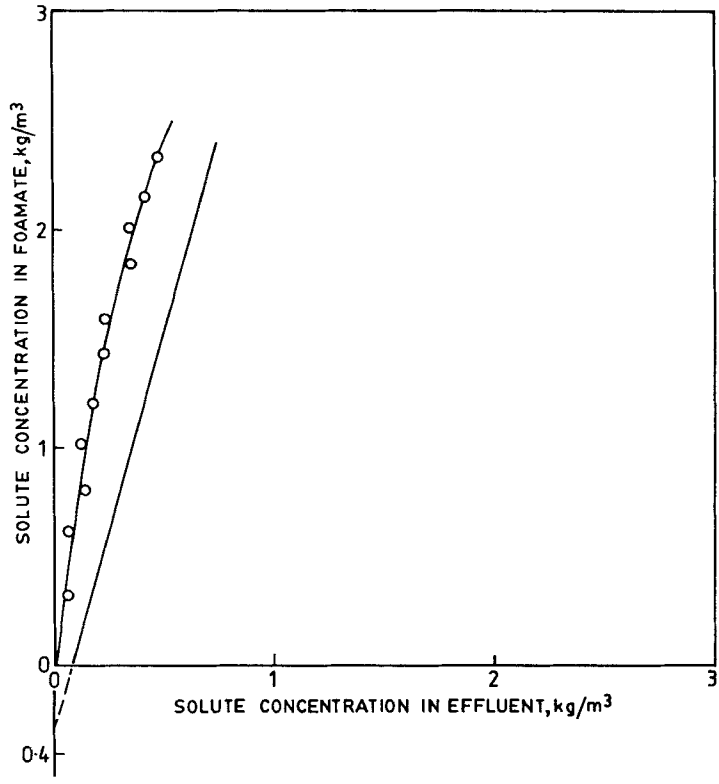


FIG. 2 Equilibrium curve and operating line (for Run 5) for the present system.

curve and the operating line, respectively, were used. This set of values was used to integrate Eq. (1) using Simpson's Rule. The integral provided the number of transfer units. The effects of different physicochemical parameters on separation efficiency have been published (5). Table 1 further shows that at constant feed flow rate (L) and increasing gas flowrate (G), bubble diameter decreases. This is probably due to the fact that at higher gas velocity, large foam bubbles collapse. On the other hand, under a similar situation the mass transfer coefficient (K_L) increases (as is evident from the table) due to higher turbulence. It is to be remembered, however, that in the case of a nonlinear interactive system like the present one, it is difficult, if not impossible, to explain the effect of individual physicochemical parameters on the system effectiveness. Sometimes one has to depend on experimental observation. The height of a transfer unit

TABLE 1
NTU and HTU Determination for a Foam Column[a]

Run	$L \times 10^6$ m^3/s	$h \times 10^2$ m	$G \times 10^6$ m^3/s	X_F (kg/m^3)	X_B (kg/m^3)	NTU	Actual HTU m	$K_L \times 10^5$ m/s	$d \times 10^2$ m	$\phi \times 10^4$	Predicted HTU	Percent deviation in HTU
1	0.347	15	4.167	0.442	0.106	1.750	0.0857	0.238	0.85	868.97	0.0916	−6.88
2	0.347	15	8.333	0.284	0.109	1.956	0.0767	0.298	0.83	997.24	0.0714	6.91
3	0.347	15	10.000	0.263	0.113	1.993	0.0753	0.306	0.82	1017.58	0.0687	8.76
4	0.347	10	4.167	0.423	0.12	2.312	0.0433	0.243	0.82	907.22	0.0443	−2.31
5	0.347	20	4.167	0.421	0.119	2.261	0.0885	0.245	0.88	892.27	0.0921	−4.07
6	0.347	25	4.167	0.430	0.126	2.670	0.0936	0.233	0.89	887.38	0.0980	−4.70
7	0.347	30	4.167	0.415	0.134	2.977	0.1008	0.232	0.90	940.96	0.0995	1.29
8	0.347	35	4.167	0.408	0.139	3.244	0.1079	0.221	0.95	908.99	0.1102	−2.13
9	0.347	42	4.167	0.407	0.143	3.399	0.1236	0.210	1.0	939.95	0.1221	1.21
10	0.300	15	4.167	0.433	0.100	1.606	0.0934	0.263	1.18	871.96	0.0995	−6.53
11	0.300	15	5.000	0.38	0.098	1.705	0.088	0.277	1.15	887.85	0.0921	−4.66
12	0.300	15	8.333	0.285	0.102	1.790	0.0838	0.313	1.13	972.27	0.08	4.53
13	0.250	15	4.167	0.428	0.093	1.427	0.1051	0.276	1.6	911.27	0.1071	−1.90
14	0.250	15	5.000	0.378	0.092	1.560	0.0961	0.286	1.55	974.75	0.1001	4.76
15	0.250	15	8.333	0.289	0.095	1.687	0.0889	0.323	1.53	943.34	0.0875	1.57
16	0.180	15	8.333	0.295	0.087	1.514	0.0991	0.336	2.5	929.82	0.0990	0.10
17	0.180	15	10.000	0.282	0.092	1.611	0.0931	0.329	2.3	929.70	0.0930	0.11

[a] $X_L = 0.204$ kg/m^3, $S = 12.566 \times 10^{-4}$ m^2. The data points represent a mean of four experimental readings.

was calculated from the known value of foam height and the corresponding NTU using the following equation:

$$h = (\text{HTU})(\text{NTU})$$

The mass-transfer coefficient, K_L, and the bubble diameter, d, have been found by the methods already discussed. By using these parameters and with the help of Eq. (5), the values of ϕ for different experimental runs have been calculated. The results are given in Table 1 with foam height, gas flow-rate, and liquid flow-rate as the operating variables. From the results it is apparent that the deviation of actual HTU and predicted HTU is not appreciable, and the following empirical correlation for HTU can be used for foam bed height computation:

$$\text{HTU} = 928.72 \times 10^{-4}\, Ld/K_L S \tag{5}$$

NOMENCLATURE

L	flow rate of the feed solution (m^3/s)
B	flow rate of the effluent (m^3/s)
F	flow rate of the foamate (m^3/s)
G	gas flow rate (m^3/s)
X_F	concentration of protein in the overflowing foam on a gas-free basis (kg/m^3)
X_B	concentration of protein in the effluent (kg/m^3)
$\overline{X}_B^*$	effective concentration of solute protein, X, in equilibrium with X_B (kg/m^3)
$\overline{X}_F^*$	effective concentration of solute protein, X, in equilibrium with X_F (kg/m^3)
X	protein concentration in the liquid (kg/m^3)
$\overline{X}^*$	effective upflow protein concentration in equilibrium with X on a gas-free basis (kg/m^3)
$\overline{X}$	effective upflow protein concentration on a gas-free basis (kg/m^3)
NTU	number of transfer units in the foam, based on the upflowing stream (dimensionless)
HTU	height of a transfer unit (m)

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