

## *Computer Control of Perforated Plate Extractive Reactors*

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### *Abstract*

*Extractive reactors affected by feed flow rate and feed composition disturbances are liable to fluctuations in the fractional solute recovery which are reflected on the composition of the raffinate product. A distributed parameter mass transfer model relating the purification ratio to the number of actual plates and three dimensionless parameters is combined with existing hydrodynamic correlations to relate perforated plate extractive reactors performance to the feed and solvent flow rates. The model is used to suggest a computer control scheme for such reactors. The suggested scheme is based on coupling between feed forward rejection of feed flow rate disturbances and feedback cascade composition control. An adaptive scheme is suggested for adjusting the gains of the feed forward controllers when the gains of the process and other loop parameters are subject to deviations from their nominal model values.*

*Key Words:* Perforated plate extractors; Extractive reactions; Computer control

### **1. Introduction**

Liquid-liquid extraction equipment of quite different designs are used in a variety of applications covering the chemical, petrochemical, pharmaceutical, nuclear, fertilizers, and metallurgical industries. Phase contacting maybe effected with or without mechanical agitation depending on the emulsifying tendencies, availability of head room, unit capacity, and difficulty of phase separation. Phase separation may rely either on gravity or on mechanically induced centrifugal action. In many applications the mass transfer rate is enhanced by a chemical reaction in the extract phase<sup>(1)</sup>. Perforated plate liquid-liquid extraction columns are very effective both with respect to liquid handling capacity and extraction efficiency. This is because axial mixing of the continuous phase is confined to the region between trays and does not spread throughout the tower from stage to stage. Also the tendency to establish concentration gradients within the droplets is avoided because the

dispersed phase droplets coalesce and are reformed at each plate. Furthermore, perforated plate hydrodynamics are relatively simpler to analyze, since they are not affected by the stochastic elements<sup>(2)</sup> arising from droplet interactions associated with pulsations, mechanical agitation<sup>(3)</sup>, axial dispersion, and forward mixing<sup>(4)</sup>. However, perforated plate hydrodynamics limit the admissible range within which the phase flow rates maybe varied to bring about the required extract or raffinate composition control.

The present work addresses the steady-state control of perforated plate liquid-liquid extractors. The column response depends on the complex effects of changing phase flow rates on the system hydrodynamics and on the rate of the extractive reaction. In the case of physical extraction, it is sometimes possible to keep both the raffinate and extract compositions within narrow limits depending on the effect of phase flow rates on the overall mass transfer efficiency. In such cases the correction for feed composition disturbances would be through manipulation of both feed and solvent flow rates<sup>(5)</sup>. In the presence of an extractive reaction, the control variable of relevance is the raffinate composition which is chosen –based on economic considerations- to ensure sufficient depletion of the feed from the solute. In this case, the extract composition is less relevant since the solute transferred to the extract would continue reaction outside the column. In such cases, either or both feed composition and feed flow rate disturbances may be corrected by manipulation of the solvent feed rate.

A steady state model reflecting the interaction between mass transfer performance and hydrodynamic behaviour of such columns is developed by combining existing empirical hydrodynamic relations and a recently developed steady state mass transfer model<sup>(6)</sup>. An improvement over conventional single loop and cascade systems is suggested. An adaptive feed forward computer control scheme is proposed based on the developed non-linear model for the manipulation of the solvent flow rate to maintain raffinate composition despite feed flow rate and/or feed composition disturbances.

## 2. Mass Transfer Model

Referring to Fig. (1), considering a plate (n) on which the solute is transferred from the continuous L-phase to the dispersed extract V-phase where it undergoes a first order reaction, mass balances on the continuous and extract phases are given respectively by:

$$L(x_{n+1} - x_n) - V(y_n - y_{n-1}) = \int_0^h k_y \phi_D A_a dz \quad (1)$$

and

$$\int_{n-1}^n \frac{dy}{Ka' m x_n - (Ka' + k)y} = \int_0^h \frac{A_a \phi_D dz}{V} \quad (2)$$

Integration of equations (1) and (2) assuming that the solvent feed is solute free gives<sup>(6)</sup>

$$\frac{x_1}{x_f} = \frac{g(a_1 - a_2)}{(1 - e^{-\beta} + g)(a_1^N - a_2^N) - (1 - e^{-\beta} + g e^{-\beta})(a_1^{N-1} - a_2^{N-1})} \quad (3)$$

Where  $a_1$  and  $a_2$  are the roots of the auxiliary quadratic:  $a^2 + b a + c = 0$ .

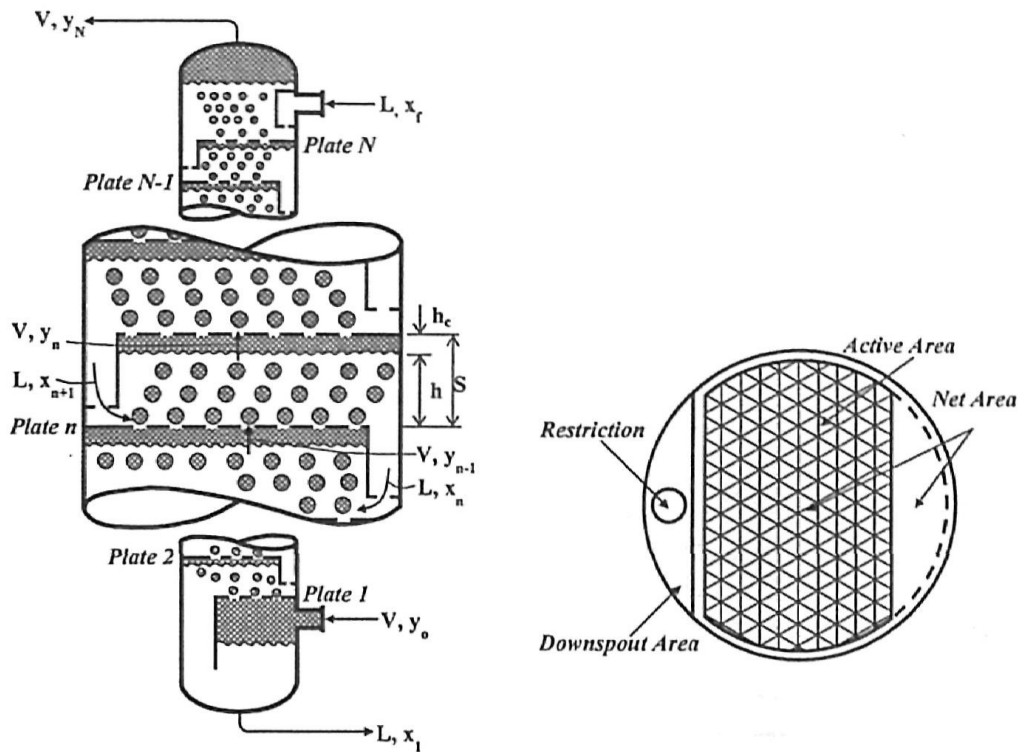


Fig. (1) The Model System

The dimensionless parameters (b), (c), (g), and ( $\beta$ ) are defined in the nomenclature section. Since the parameters (a) and (c) are functions of (g), ( $\beta$ ), and ( $\gamma$ ), the raffinate purification ratio is expressible in terms of the number of stages as well as (g), ( $\beta$ ), and ( $\gamma$ ). A Matlab program has been developed to generate generalized design charts enabling the estimation of the number of actual plates required to achieve a desired purification ratio for various values of the above three dimensionless parameters. Fig. (2) depicts such a chart.

The parameters (g), ( $\beta$ ), and ( $\gamma$ ) are combinations of a number of physico-chemical and hydrodynamic properties of the system which are sensitive to variations in the phase flow rates. The dimensionless parameter (g) depends on the phase flow rates and the slope of the equilibrium relation. The dimensionless parameter ( $\beta$ ) lumps the effect of the system hydrodynamics reflected in the values of the transfer area, transfer volume per stage, dispersed phase flow rate, and the effect of the overall mass transfer coefficient. The mass transfer coefficient is a function of the slip velocity and phase properties. In other words the calculation of the actual number of stages incorporates the effect of the individual stage efficiency on the overall cascade efficiency.

It is clear from Fig. (2) that for a given number of actual plates (N), the obtained purification ratio improves as the extraction factor (g) decreases and as the mass transfer parameter ( $\beta$ ) increases. The effect of chemical reaction is to increase solute recovery, thus the higher the value of ( $\gamma$ ), the ratio of the reaction rate constant to the mass transfer rate constant, the lower the value of ( $x_1/x_f$ ) for the same values (g) and ( $\beta$ )

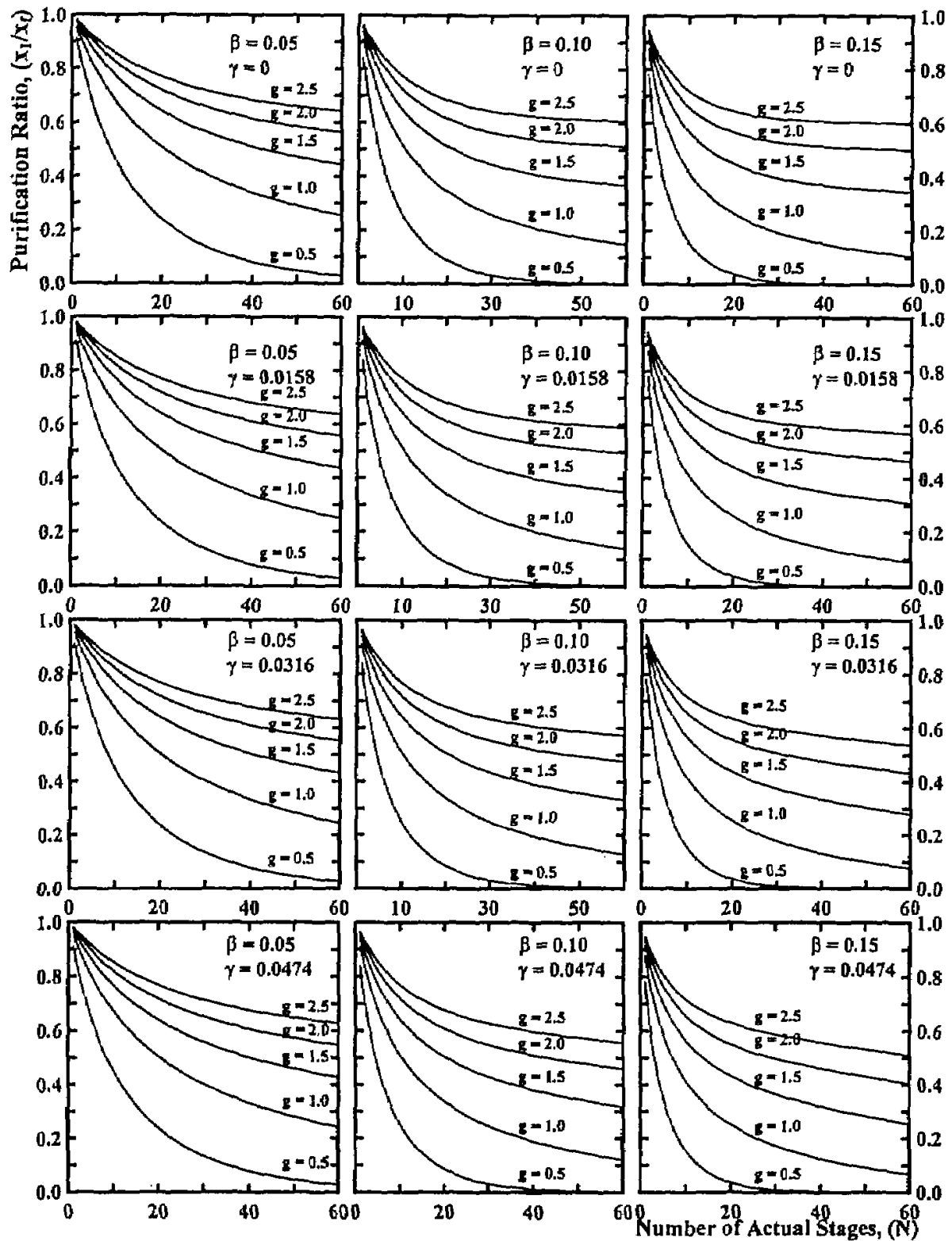


Fig. (2) Relation between purification ratio and number of stages for different values of  $(g)$ ,  $(\beta)$ , and  $(\gamma)$

### 3. Hydrodynamic Model

The hydrodynamic behaviour of perforated liquid-liquid extractors is covered by a number of empirical correlations enabling the design of plate and tower dimensions (e.g. perforation diameter, pitch, active area, downcomer area, plate spacing, etc.), to ensure stable contacting patterns<sup>(7)</sup>. The range of hydrodynamic stability of an existing column is obviously constrained by the column dimensions. Also within the stable operating range, changing phase flow rates are reflected on the mass transfer performance through changes in the values of the relevant transport parameters.

Referring to Fig. (1), the depth of the coalesced layer below each plate is the sum of the head losses of the dispersed and continuous phases obtained on crossing a plate. It is therefore a function of phase velocities<sup>(8)</sup>, which depend on column design and flow rates as well as on the phase densities. The effective mass transfer height ( $h$ ) appearing in the dimensionless parameter ( $\beta$ ) is the difference between the plate spacing and the height of the coalesced layer as shown in Fig. (1). The dispersed phase holdup and slip velocity are also obtainable by iterative solution of existing correlations for the mechanics of counter flowing liquid-liquid systems<sup>(9, 10)</sup> together with the Zenz<sup>(11)</sup> correlation. The net dispersed phase velocity based on net cross sectional area, drop diameter, continuous phase viscosity, phase densities, and interfacial tension are the required input data for calculating phase holdup and phase velocities.

The Hayworth<sup>(12)</sup> correlation enables the expression of the drop diameter in terms of the hole diameter, hole velocity, interfacial tension, continuous phase viscosity, and on the densities of the two phases. The overall mass transfer coefficient maybe estimated from the drop diameter, slip velocity, equilibrium partition coefficient, solute diffusivities in both phases, interfacial tension, and phase densities<sup>(11)</sup>. The interfacial area per unit volume ( $a'$ ) is readily calculable from the dispersed phase holdup and the drop diameter.

It is recalled that the model parameters ( $g$ ), ( $\beta$ ), and ( $\gamma$ ) are sensitive to the phase flow rates. The above hydrodynamic correlations are quite involved and often require an iterative solution for their calculations. Matlab programs have been developed for generating the effect of phase flow rates, column dimensions, and physico-chemical parameters on the mass transfer model parameters.

The application of these programs is illustrated by considering an extractive reactor of given plate dimensions handling a system of known physico-chemical properties. The data of this system is given in Table (1). The effects of varying the feed rate, ( $L$ ) between 0.0022 to 0.003 m<sup>3</sup>/s and solvent rate, ( $V$ ) between 0.0075 to 0.012 m<sup>3</sup>/s are shown in Figs. (3) and (4). The phase holdup, slip velocity, drop diameter, overall volumetric mass transfer coefficient, effective contact height per plate, and the parameters ( $\beta$ ), ( $g$ ), and ( $\gamma$ ) are displayed as a function of the dispersed phase flow rate ( $V$ ) for different values of the continuous phase flow rate ( $L$ ). The data of Figs. (3) and (4) have been also formulated in the form of simple correlations to facilitate their processing in subsequent computer programs.

Table (1) Tower dimensions and physico-chemical properties

Tower Dimensions		Physico-Chemical Properties	
Hole diameter ( $d_h$ ), m	$6 \times 10^{-3}$	Dispersed phase Density, $\text{kg/m}^3$	730
Pitch (p), m	$15 \times 10^{-3}$	Continuous phase Density, $\text{kg/m}^3$	1009
Number of perforations	2151	Dispersed phase viscosity, $\text{kg/m.s}$	$0.9 \times 10^{-3}$
Active area ( $A_a$ ), $\text{m}^2$	0.4189	Continuous phase viscosity, $\text{kg/m.s}$	$3.1 \times 10^{-3}$
Minimum downcomer area ( $A_r$ ), $\text{m}^2$	0.0131	Acid diffusivity in continuous phase, $\text{m}^2/\text{s}$	$1.24 \times 10^{-9}$
Total tower area ( $A_t$ ), $\text{m}^2$	0.8057	Acid diffusivity in dispersed phase, $\text{m}^2/\text{s}$	$1.96 \times 10^{-9}$
Tower diameter (T), m	1	Interfacial tension, $\text{N/m}$	0.013
Net area ( $A_n$ ), $\text{m}^2$	0.6973	Slope of equilibrium relation (H)	0.26
Actual number of plates (N)	40	Reaction rate constant (k), $\text{s}^{-1}$	0.001

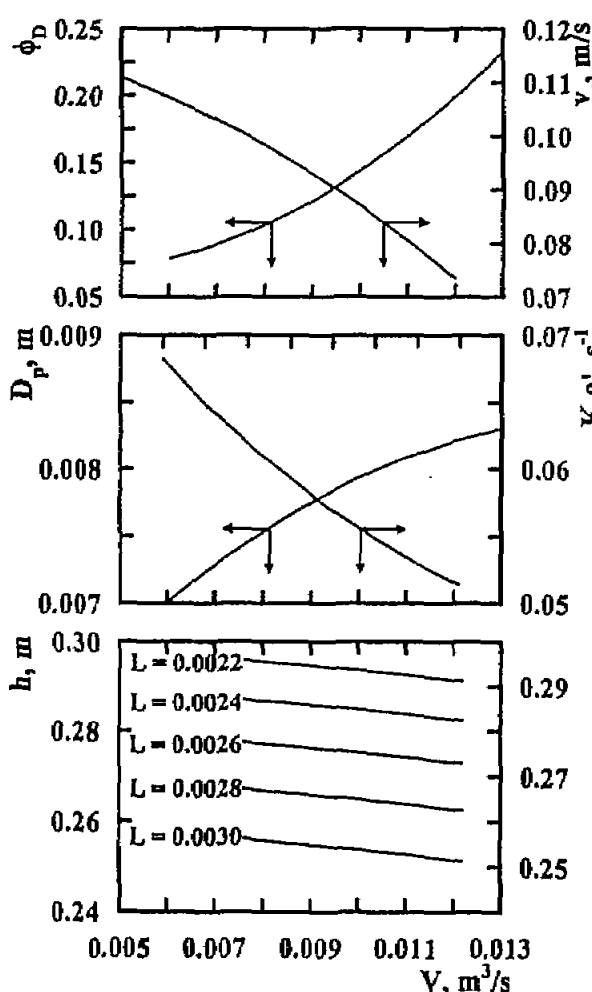


Fig. (3) Effect of phase flow rates on hydrodynamic and mass transfer parameters

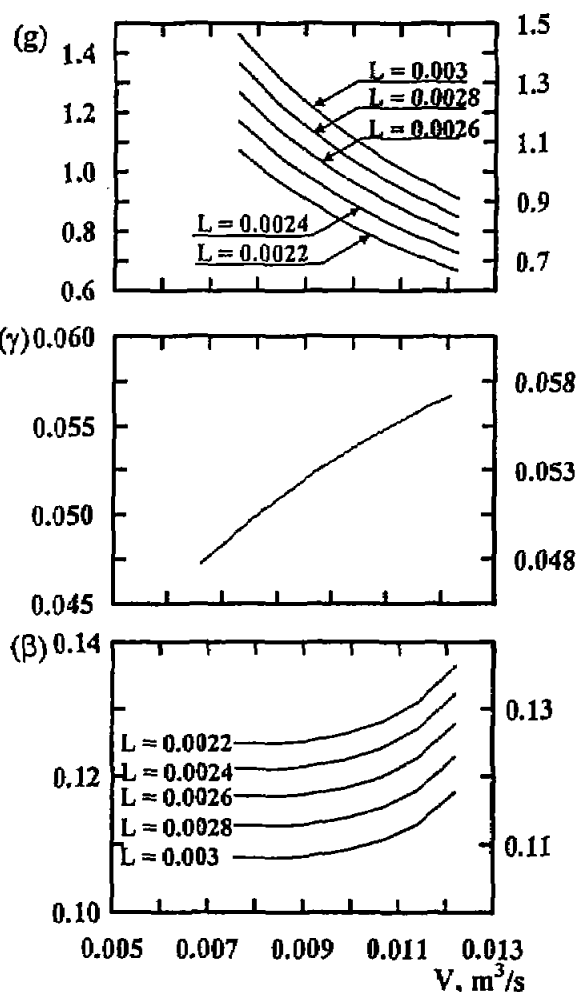


Fig. (4) Effect of phase flow rates on the dimensionless parameters, (g), (β), and (γ)

#### 4. Column Control

When an existing extractive reaction column with fixed number of stages and plate design is subjected to changes in feed composition ( $x_p$ ) or continuous phase flow rate ( $L$ ), the mass transfer driving force and column efficiency are expected to change. This will be obviously reflected on the purification ratio ( $x_1/x_f$ ). The required raffinate composition ( $x_1$ ) maybe restored by effecting an appropriate change in the dispersed phase flow rate ( $V$ ). This is illustrated by considering the steady state control of the 40 plate column, the basic data of which is given in Table (1).

##### 4.1. Static Response

It is clear from Fig. (4) that  $(g)$ ,  $(\beta)$ , and  $(\gamma)$  are functions of  $(L)$  and  $(V)$ , accordingly Eq. (3) maybe modified on the basis of the data in Figs. (3) and (4) to generate  $(x_1/x_p)$  as a function of both  $(L)$  and  $(V)$  for this particular column. This is shown in Fig. (5).

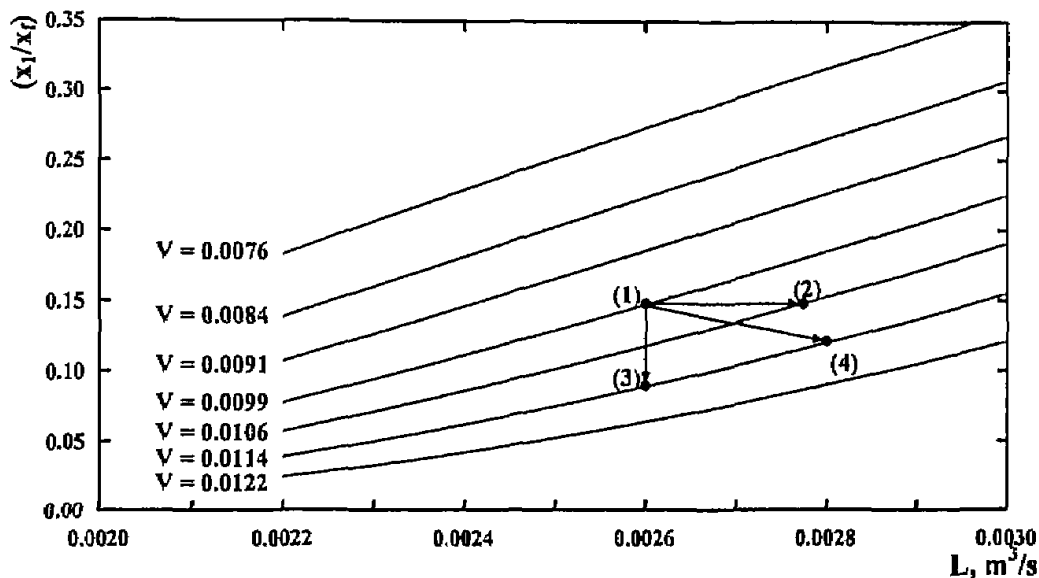


Fig. (5) Manipulation of  $(V)$  to maintain  $(x_1)$  despite disturbances in  $(L)$  and  $(x_1/x_p)$

Assuming that the plant is operating at the normal steady state operating level represented by point (1) on Fig. (5), where  $(x_1/x_p) = 0.147$ ,  $(L) = 0.0026 \text{ m}^3/\text{s}$ , and  $(V) = 0.0099 \text{ m}^3/\text{s}$ , if  $(L)$  increases to  $0.00276 \text{ m}^3/\text{s}$ ,  $(x_1/x_p)$  maybe maintained by increasing  $(V)$  up to  $0.0106 \text{ m}^3/\text{s}$  as indicated by point (2) on Fig. (5). If on the other hand,  $(x_p)$  is increased and the same  $(x_1)$  is to be maintained, the desired value of  $(x_1/x_p)$  would drop to 0.09 and  $(V)$  should be increased up to  $0.0114 \text{ m}^3/\text{s}$  as indicated by point (3) on Fig. (5). If both  $(L)$  and  $(x_p)$  are subjected to disturbances such that  $(L)$  is increased to  $0.0028 \text{ m}^3/\text{s}$  and  $(x_1/x_p)$  is reduced to 0.0123, the solvent rate  $(V)$  should be manipulated to  $0.0114 \text{ m}^3/\text{s}$  in order to satisfy the operating conditions as indicated by point (4) on Fig. (5). It is recalled that the above calculations implicitly take into account the reflection of the continuous and dispersed phase flow rates on the overall column efficiency.

The non-linear relation  $(x_1/x_f) = \psi(L, V)$  presented in Fig. (5) maybe linearized around the steady state operating level by retaining the first terms of its Maclaurin series expansion. This gives the linearized relation between the perturbation variables as

$$(x_1/x_f) - (x_1/x_f)_{s,s} = (\partial\psi/\partial L)_{s,s}(L - L_{s,s}) + (\partial\psi/\partial V)_{s,s}(V - V_{s,s}) \quad (4)$$

For the column under consideration,

$$(\partial\psi/\partial L)_{s,s} = K_L = 184.42 \text{ s/m}^3$$

and

$$(\partial\psi/\partial V)_{s,s} = K_V = -45.34 \text{ s/m}^3$$

#### 4.2. Control Schemes

Since the raffinate product composition ( $x_1$ ) is the control variable of relevance, while ( $L$ ) and ( $x_f$ ) are the likely disturbances, the simplest control scheme would be a single loop where ( $V$ ) is manipulated on the basis of measurements of ( $x_1$ ). In order to cope with downstream and upstream solvent pressure fluctuations, a cascade composition control would result in better performance. Fig. (6) outlines the above two schemes along with the block diagram of the cascade system.

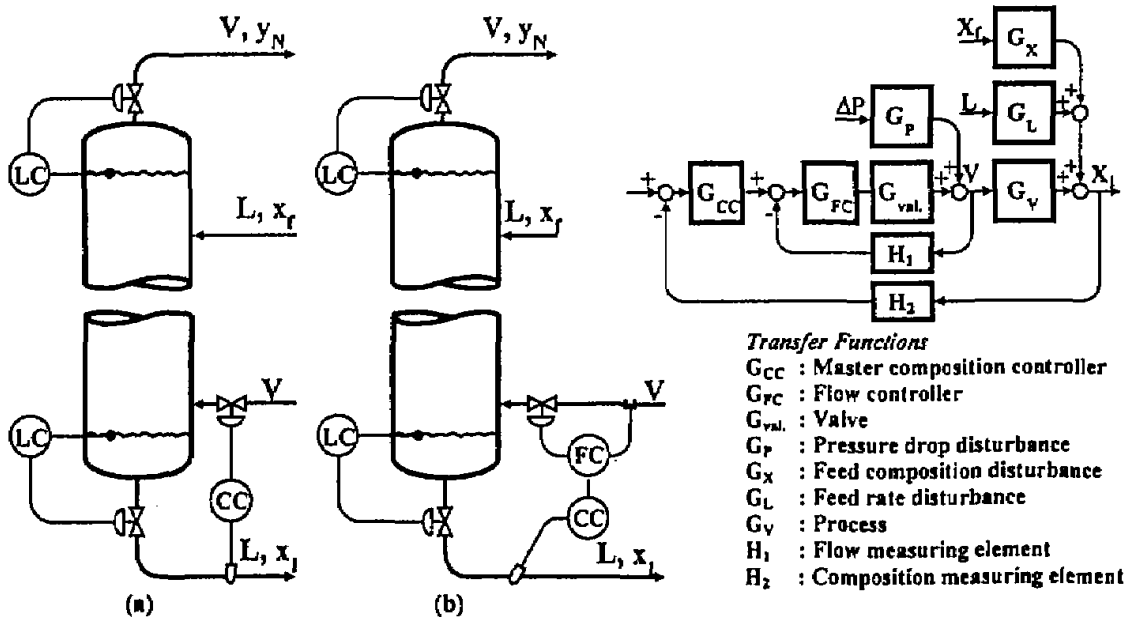


Fig. (6) Simple extractive reactor control schemes  
 (a) Single Loop, (b) Cascade

Computer control presents some advantages with respect to such systems. It can provide for limiting the feed and solvent rates within the limits of hydrodynamic stability of the column. Using the purification ratio ( $x_1/x_f$ ) as the controlled variable, the reference input ( $x_1$ ) and disturbance ( $x_f$ ) are lumped into one and the same variable. Computer control also enables the incorporation of feed forward action to cope with feed rate fluctuations.



Fig. (7) outlines the incorporation of a simple feed forward computer control scheme into the feedback cascade control scheme shown in Fig. (6). It is recalled that the transfer functions of the feed forward controllers for disturbance rejection and set point tracking depend on the transfer functions of the measurement transducers, control valve, load, and process. Owing to possible changes in the system parameters and deviations from the model hydrodynamics and mass transfer coefficient correlations, the gains of ( $G_{SP}$ ) and ( $G_{FF}$ ) are adjusted through a steady state optimizing computer control scheme. The nominal values of ( $K_L$ ) and ( $K_V$ ) maybe updated depending on the actual processing conditions through an adaptive actuation scheme.

On the basis of measurements of ( $x_1/x_2$ ), ( $L$ ), and ( $V$ ), the model predicted ( $x_1/x_2$ ) is compared to the actual value. The deviation between the actual and measured values is used to modify the model values of ( $K_L$ ) and ( $K_V$ ) to obtain a match between the values of the measured and predicted purification ratio. The new values of ( $K_L$ ) and ( $K_V$ ) are then used to modify the gains of the set point tracking and disturbance rejection feed forward controllers. A simplified outline of the computer control block diagram is also shown in Fig. (7).

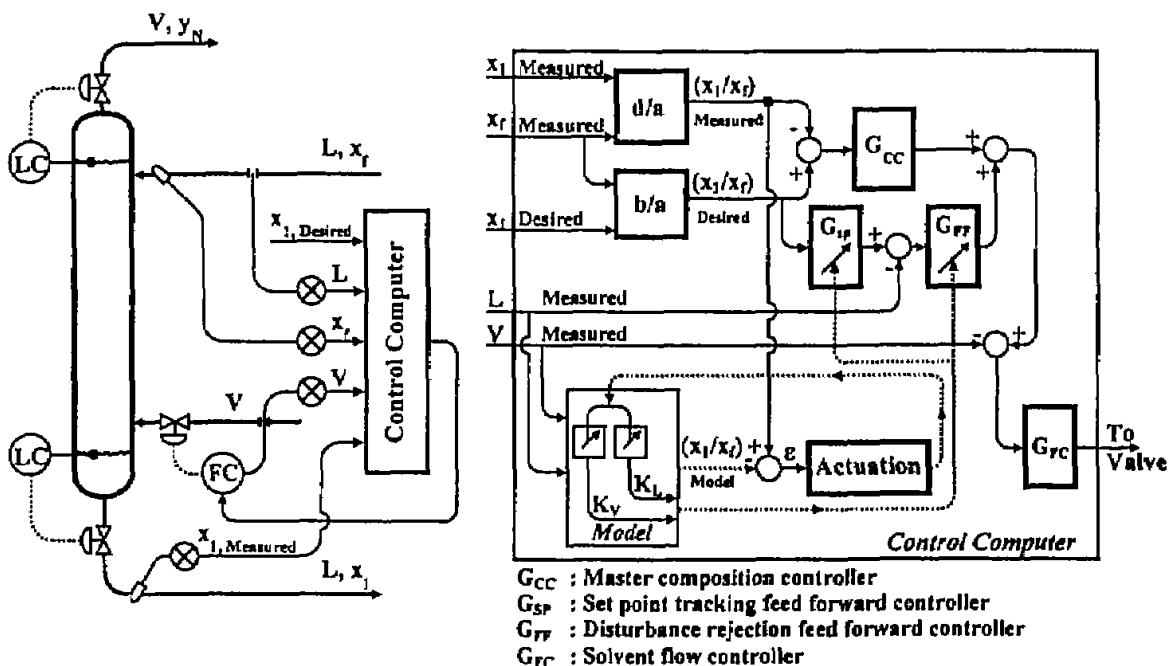


Fig. (7) Computer control scheme

## 5. Conclusions

1. The static response of perforated plate extractive reaction columns has been analyzed in the light of a distributed parameter steady state model. The model accounts for the interaction between perforated plate hydrodynamics and mass transfer performance associated with changes in phase flow rates in an extractive reactor of given design parameters.

2. The numerical solution of the non-linear model enabled the expression of the obtained purification ratio in terms of the phase flow rates. A linearized version of the model has been developed to study the control of the raffinate composition when the column is subjected to feed flow rate and composition disturbances.
3. A combined feed forward-feed back control scheme has been suggested to enable the system to cope with both types of disturbances. An adaptive gain scheduling computer control scheme has been suggested for updating the feed forward controller parameters.
4. The considered control problem assumed that the raffinate product composition is the main control variable of interest. This is because the extract reaction is likely to proceed after leaving the extractor till the solute is practically exhausted.
5. In the case of physical extraction, it is possible to keep both extract and raffinate compositions if both (L) and (V) are manipulated. If conventional feedback loops are used to control the compositions of both products, the two loops are bound to interact since both manipulated variables affect both of the controlled variables. Decoupling of the two interacting loops would rely also on feed forward decouplers the transfer functions of which depend on the nominal process transfer functions. In such cases, the adaptive scheme discussed above may be resorted to for ensuring optimum steady state control of the unit.

### *Nomenclature*

$a_1, a_2$	:	Roots of the quadratic $a^2 + b a + c = 0$ .
$a'$	:	Interfacial area per unit volume of dispersed phase, $m^2/m^3$
$A_a$	:	Active plate perforated area, $m^2$
$A_n$	:	Net tower area, $m^2$
$b$	:	Dimensionless parameter given by $b = \left( -1 - e^{-\beta(1+\gamma)} - \frac{(1 - e^{-\beta(1+\gamma)})}{g(1+\gamma)^2} - \frac{\beta}{g} \frac{\gamma}{(1+\gamma)} \right)$
$c$	:	Dimensionless parameter given by $c = \left( e^{-\beta(1+\gamma)} + \frac{(1 - e^{-\beta(1+\gamma)})}{g(1+\gamma)^2} + \frac{\beta}{g} \frac{\gamma}{(1+\gamma)} e^{-\beta(1+\gamma)} \right)$
$d_h$	:	Hole diameter, m
$D_p$	:	Droplet diameter, m
$g$	:	Extraction factor, $L/mV$
$h$	:	Effective stage height, m, $h = S - h_c$
$h_c$	:	Height of coalesced layer, m
$k$	:	Reaction rate constant, $s^{-1}$
$K$	:	Mass transfer coefficient, m/s
$K_L$	:	Gain in the linearized ( $\psi$ ) function, $s/m^3$
$K_V$	:	Gain in the linearized ( $\psi$ ) function, $s/m^3$
$L$	:	Volumetric flow rate of the continuous phase, $m^3/s$
$m$	:	Slope of the equilibrium relation
$N$	:	Number of actual perforated plates
$p$	:	Pitch, m
$S$	:	Tray spacing, m
$V$	:	Volumetric flow rate of the dispersed phase, $m^3/s$

$v_s$	:	Slip velocity, m/s
$x_1$	:	Solute concentration in the final raffinate, kmole/m <sup>3</sup>
$x_f$	:	Solute concentration in the continuous phase feed, kmole/m <sup>3</sup>
$x_n$	:	Solute concentration in the continuous phase on plate (n), kmole/m <sup>3</sup>
$y$	:	Solute concentration in the dispersed phase, kmole/m <sup>3</sup>
$y_o$	:	Solute concentration in the fresh solvent, kmole/m <sup>3</sup>
$y_N$	:	Solute concentration in the final extract, kmole/m <sup>3</sup>

### Greek Symbols

$\beta$	:	Dimensionless mass transfer parameter, given by $\beta = Ka' A_a \phi_D h / V$
$\phi_D$	:	Dispersed phase holdup
$\gamma$	:	Dimensionless enhancement factor, given by $\gamma = k / Ka'$
$\psi$	:	Non-linear function of (L) and (V)

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