APPLICATION OF ADAPTIVE CONTROL TO A RECIPROCATING PLATE LIQUID-LIQUID SOLVENT EXTRACTION COLUMN

by

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ABSTRACT

An experimental investigation of the application of direct digital control (DDC) has been carried out with continuous chemical processing equipment, namely a liquid-liquid solvent extraction column. The type of extractor used was the Karr reciprocating plate column.

Steady state simulations of the column were carried out using both a semi-empirical model for studying the hydrodynamics of the column, and a mechanistic model for studying the mass transfer characteristics of the column. The results of these simulations were used to interpret the closed loop response characteristics of the column. Two types of control schemes were studied; (i) an adaptive self tuning regulator (STR), (ii) the Dahlin algorithm.

Though the dispersed phase holdup is not the primary control variable, its control is essential to prevent the hydrodynamic instability. The frequency of reciprocation was used to control the holdup. The relationship between the frequency of reciprocation and the dispersed phase holdup is highly nonlinear therefore an adaptive controller (in this work, a self tuning regulator) has to be used to control this process. Previous work on holdup control has been extended

to include a wider range of operating conditions and prevention of covariance windup by using an empirical discounting factor.

The primary control objective was the control of the extract concentration. This was done in two ways. The first scheme was a cascaded control to manipulate the frequency of reciprocation whereby the inner loop was the holdup control. An STR-STR cascade arrangement was compared with cascade a PI-STR arrangement. It was found that the restrictions imposed on the outer loop meant that the PI-STR arrangement, was as effective as the STR-STR arrangement. The second control scheme manipulated the continuous phase flow rate with (MIMO) and without (SISO) the simultaneous control of the holdup via manipulation of the frequency of reciprocation. A variable dead time Dahlin controller was used to control the extract concentration in this second control scheme. In the MIMO case, decoupling was not necessary since one loop had a positive gain while the other had a negative gain and so the interactions were constructive.

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NOMENCLATURE

A	stroke (twice the amplitude), m
$\underline{\underline{A}}$	A matrix, equation 3.27
a	Specific interfacial area (m^2/m^3)
a_t	A random variable
b	Total dead time in the process
$\underline{b}$	A vector, equation 3.28
C_o	Discharge coefficient through the perforations
ΔC	Concentration difference ($kmol/m^3$)
c	fractional period of time delay
D	Axial dispersion coefficient (m^2/s)
$D(z^{-1})$	Controller equation in equation 4.30
D_m	Molecular diffusivity (m^2/s)
d_{32}	Sauter mean drop diameter (cm), equation 1.3
E	Expectation operator
e_t	error
f	Frequency of reciprocation (Hz)
g	Acceleration due to gravity (m/s^2)
$GH(z^{-1})$	Pulse transfer function
$\underline{\underline{G}}$	Matrix transfer function
$\underline{\underline{G}}(0)$	Steady state gain matrix
h	Centre to centre plate spacing (m)
K	Dimensionless drop circulation constant, equation 3.6

k	Mass transfer coefficient (m/s)
K	Overall mass transfer coefficient (m/s)
K_p	Process gain
K_t	Gain vector of the estimator, equation 4.24
$K(z^{-1})$	Closed loop transfer function, equation 4.29
L	Ratio U_d/U_c
ℓ_{ij}	Elements of Bristol relative gain array, equation 4.34
m	Distribution coefficient equation 3.18
N_t	Noise defined in equation 4.3
P_t	A matrix proportional to variance covariance matrix of the parameter estimates
ΔP	Differential pressure (kPa)
S	Fractional open area of each plate
s	Laplace variable
T	Control interval
$T(z^{-1})$	Polynomial in z^{-1}, numerator in equation 4.8
U	Superficial velocity (m/s)
$\underline{U}$	Input vector equation 4.33
U_k	Characteristic velocity (m/s), equation (3.4)
U_s	Slip velocity (m/s), equation 3.1
U^*	Velocity defined by equation (3.6) (m/s)
U_t	Input (manipulated) variable at time t
$\underline{w}$	A vector in equation 3.27

X	Solute concentration in the dispersed phase ($kmol/m^3$)
X^*	Solute concentration in equilibrium with Y ($kmol/m^3$)
X_F	Solute concentration in the feed ($kmol/m^3$)
X_t	A vector defined in equation 4.17
X_{t+b}	A vector defined in equation 4.18
Y_t	Controlled variable
Y	Solute concentration in the continuous phase ($kmol/m^3$)
Y_{in}	Solute concentration in the solvent inlet ($kmol/m^3$)
Z	Total length of the column in the active section (m)
ΔZ	Length of a well mixed compartment
z^{-1}	Backward shift operator

GREEK SYMBOLS

α_i	Controller parameter
$\alpha(z^{-1})$	Polynomial in z^{-1}, equation 4.12
$\beta(z^{-1})$	Polynomial in z^{-1}, equation 4.12
β_i	Controller parameter
γ	Interfacial tension Nm
η	A constant in equation 4.27
θ	A vector defined in equation 4.16
$\theta(z^{-1})$	Polynomial in z^{-1} numerator in equation 4.3 3

$\underline{\underline{\Lambda}}$	Bristol relative gain array
λ	Forgetting (discounting) factor
μ	Viscosity (Pa.s)
ξ	Constraint factor
ρ	Density (kg/m^3)
$\Delta\rho$	Mean density of dispersion kg/m^3, equation (3.10)
σ	Variance
τ	Time constant (s)
τ_c	Closed loop time constant (s)
τ_d	Process dead time (s)
ϕ	Volume fraction holdup of disperse phase
$\phi(z^{-1})$	Polynomial in z^{-1}, denominator in equation 4.
ϕ_t	Generalized output variable, equation 4.11
$\delta(z^{-1})$	Polynomial in z^{-1}, denominator in equation 4.3
ψ	Power dissipation per unit volume of dispersion (W/m^3)
$\psi(z)$	Polynomial in z^{-1} in equation 4.8
$\omega(z^{-1})$	Polynomial in z^{1} numerator in equation 4.1
∇	Backward difference operator

SUBSCRIPTS

c	Continuous phase
d	Dispersed phase
F	Flooding
t	Time index

SUPERSCRIPTS

$'$ Transpose of a vector or a matrix

CHAPTER 1

1. Introduction

1.1 General Introduction

Almost all the processes encountered in chemical engineering exhibit a nonlinearity in the sense that the transfer function obtained for control purposes is a function of the region in operating space. Therefore a fixed parameter controller designed on the basis of a transfer function obtained under one set of operating conditions may give unacceptable or even unstable control once the conditions of the process are changed. This might necessitate using an adaptive controller which is capable of readjusting the controller parameters on line so as to compensate for the changing process dynamics. Such controllers have become popular since the early 70's and have found increased use in the control of chemical processes.

In a typical chemical plant there are various process units wherein a physical or chemical change is accomplished. In most plants, the separation of a liquid mixture into its components may be done either by a distillation unit or a liquid-liquid extraction unit. The

former of these units has been explored extensively for applying direct digital control using classical and modern control theory, whereas extraction units have had little attention from the researchers. However Wilkinson and Ingham (1983), referring to computer control, found it necessary to control extraction columns as they are part of the overall chemical plant where direct digital control may be fully justified. They state that in the future such control will be indispensible.

It is the objective of this research to investigate the applications of modern control strategies to a Karr extraction column. The primary control objective in an extraction column is the regulatory and servo control of the outlet concentration(s). While this objective is being accomplished, the hydrodynamic stability of the two phase flow must also be preserved. This necessitates a secondary control objective so as to preserve the stability i.e. to prevent occurrence of flooding conditions in the column.

The following is the layout of the contents of each chapter in this thesis.

The later sections of this chapter introduce the concept of liquid-liquid solvent extraction as a separation process, and review earlier work on mathematical modelling and control of extraction columns. The literature survey shows that only a few control studies of extraction columns have been made. Chapter 2 describes the experimental Karr column apparatus and the peripherals such as measurement devices,

auxiliary control elements and the distributed computer system in use in the Chemical Engineering Department at McMaster University. The control objectives are presented, and finally experimental open loop response characteristics of the Karr column are given.

Steady state simulation results can be used to gain insight into the nonlinearities and operability of the process which can, in turn, be used to interpret the closed loop response characteristics of the system. This is investigated in Chapter 3 which is composed of two parts. In the first part the applicability of the Baird-Shen (1984) semi-empirical relation, based on the operating holdup data in a 5 cm diameter Karr column, is tested on the experimental apparatus and the model predictions are compared with the data. In the second part of this chapter, steady state mass transfer in the column is simulated using a mechanistic mathematical model presented in the previous chapter.

Chapter 4, which is composed of three parts, contains the theoretical background to the control schemes which were applied to the Karr column. In part 1, a brief summary of system modelling is followed by an account of the adaptive stochastic controllers relevant to this thesis. The logical foundations for the use of an ad hoc variable exponential discounting factor is also included. In part 2, the deterministic Dahlin controller is summarized. Finally a brief introduction to multi-input and multi-output (MIMO) systems is made and the interaction phenomenon is reviewed in part 3.

In Chapter 5, which is an extension of Kusuma's (1981) work, the experimental results of the control of the hydrodynamic holdup by manipulating the frequency of reciprocations with an adaptive implicit self tuning regulator (STR) are presented. The control is extended to a wide range of operating conditions. Also included in this chapter are the experimental results of the variable exponential discounting factor presented in the previous chapter.

Any input which has a significant causal relationship with an output variable can be used as a manipulated variable. Frequency of reciprocation has this relationship not only with the holdup of the dispersed phase but also with the extract (aqueous outlet) concentration and hence can be used to manipulate both of the outputs in a nested loop arrangement. This control scheme naturally leads to cascade control. Chapter 6 presents the results and discussions of such an arrangement whereby the inner loop is the dispersed phase holdup and the outer loop is the extract concentration. In this control scheme the presence of the inner loop guarantees that the hydrodynamically unstable condition known as flooding will not occur. STR-STR controller arrangement results are compared with results using the PI-STR arrangement.

Besides the frequency of reciprocation, the continuous phase flow rate also has a significant effect on the extract concentration. This is explored in Chapter 7 and the control of the extract concentration is done by manipulating the continuous phase flow rate. A deterministic Dahlin controller with variable dead time compensation is used both with and without the simultaneous control of the holdup.

Finally, Chapter 8 is devoted to conclusions drawn from this work and recommendations for future work.

1.2 Brief Overview of the Liquid-Liquid Extraction Process

Solvent extraction is a commonly used separation method in the process industries. Its nature is based on the "like likes like" principle. It depends on the transfer of a solute from its original solution into a second solvent which is immiscible or partially miscible with the solution. The solute transfer is completed when a thermodynamic equilibrium between the two phases is achieved. The ratio of the solute concentration in the newly added solvent, after equilibration, to that remaining in the original is called the distribution coefficient. When these two phases are separated, the original solution which is now lean in the solute concentration is called the raffinate; the phase which is now richer in the solute is called the extract. The original solution is referred to as feed and the second phase added to it is solvent. For unit volume of dispersion, the specific rate (kmol/m^3s) at which the solute will transfer from one phase into another is the product of the mass transfer coefficient k (m/s), the specific area a (m^2/m^3) of contact between the phases, and the concentration difference ΔC (kmol/m^3) between the actual concentration and the equilibrium concentration i.e.,

$$\text{Specific rate} = k\ a\ \Delta C \qquad (1.1)$$

To achieve high rates of mass transfer, the specific rate in

equation 1.1 has to be maximized. This is achieved by dispersing one of the phases into the other in the form of droplets (increasing a) and preferably maintaining a large concentration driving force (ΔC) by use of countercurrent contact.

The contactors used in liquid-liquid solvent extraction processes can be divided into two broad categories depending upon whether they operate in a stagewise or differential fashion. The difference lies in the fact that in a stagewise equipment such as mixer-settlers and multiple mixer columns, the spatial variation of concentration along the extractor is in a discrete manner, whereas, in a differential extractor such as a packed, spray, or reciprocating plate column the variation is continuous. In either case, the flow of each phase relative to each other can be cocurrent or (more usually) countercurrent. In both cases, the mass transfer takes place by diffusion and thus can be enhanced by means of mechanical energy input into the system. The mechanical energy supplied maintains turbulence in the continuous phase. Eddies formed in this turbulent field overcome the surface energy (i.e. interfacial tension) of the droplets hence creating smaller droplets. This increases the available interfacial area of the dispersed phase for mass transfer and also increases turbulence around the interface which, in turn, leads to an increase in the mass transfer coefficient. Energy input to the extraction process is done in various ways such as by pulsing the column, using rotating discs or impellers, or reciprocating a plate stack inside the column.

A useful concept concerning mass transfer equipment is the height of an equivalent theoretical stage (HETS) which provides a measure of mass transfer effectiveness. For a given height of a column, a small value of this quantity is an indication of high efficiency of the column. An important hydrodynamic variable in an extraction column is the holdup which is the volume fraction of the phase in question in the active part of the column. The specific area a is related to the holdup ϕ, and the Sauter mean droplet diameter, d_{32}, through the following equation:

$$a = \frac{6\ \phi}{d_{32}} \tag{1.2}$$

where

$$d_{32} = \frac{\Sigma\ d_i^3}{\Sigma\ d_i^2} \tag{1.3}$$

1.3 Mathematical Models of the Extraction Process

A mathematical model is a quantitative description of an underlying phenomenon. Although a model developed for a system is only an abstraction of a physical phenomenon, it provides some conceptual insight into the process in question by revealing the trends which otherwise require a large number of experiments.

A comprehensive review of modelling extraction columns is presented by Wilkinson and Ingham (1983). In this section a brief overview of different modelling techniques is given, and the control objectives are outlined.

1.3.1 Mechanistic Models:

When stagewise contactors are modelled by taking a transient material balance about a stage, two coupled difference differential equations (one for each phase) for the unsteady state behaviour of the column are obtained. If the backmixing effect (which is the analog of the axial dispersion coefficient defined in the sequel) is also incorporated in the model, the resulting difference part of the equation is second order (Jones and Wilkinson, 1973) and if the backmixing effect is neglected, then the resulting difference part is first order (Biery and Boylan, 1963). When differential type contactors are modelled by taking an unsteady state material balance over a differential control volume one obtains a set of coupled partial differential equations. If the residence time distributions in each phase are neglected, i.e. plug flow is assumed, then the governing equations will be of hyperbolic type. However, the plug flow assumption has been shown to be in general an unrealistic representation of differential columns, because of axial dispersion phenomena which cause a jump in the concentration profile at the entrance of the column and a reduction in axial concentration gradients which, in turn, reduce the driving force and hence increase the HETS. To incorporate axial dispersion in models, an axial dispersion coefficient, D, is defined, analogous to the diffusion coef-

ficient in Fick's Law. The axial dispersion coefficient lumps several effects, such as molecular diffusion, velocity gradients in the radial direction, turbulent eddies, drop size distribution for the dispersed phase, and channeling in the continuous phase. When a material balance is taken about a differential control volume (see Figure 1.1) in the column incorporating axial dispersion effects (assuming that mass transfer takes place from the dispersed phase to the continuous phase and the dispersed phase enters the column at z=0, and the continuous phase at z=Z where Z is the length of the column), one obtains the following coupled parabolic equations:

$$\phi \frac{\partial X}{\partial t} = U_d \frac{\partial X}{\partial z} + D_d \frac{\partial^2 X}{\partial z^2} - K_D\, a\, (X-X^*) \tag{1.4}$$

$$(1 - \phi) \frac{\partial Y}{\partial t} = U_c \frac{\partial Y}{\partial z} + D_c \frac{\partial^2 Y}{\partial z^2} + K_D\, a\, (X-X^*) \tag{1.5}$$

where the subscripts d and c denote the dispersed and the continuous phase respectively. U is the superficial velocity of the phase, X is the solute concentration in the dispersed phase and Y is the solute concentration in the continuous phase, X^* is the equilibrium solute concentration with respect to Y, and ϕ is the holdup of the dispersed phase.

An important quantity which provides a measure of the relative importance of axial dispersion is the Peclet number. This is the ratio of convective to diffusion transport (i.e. Pe=UZ/D for each phase). A

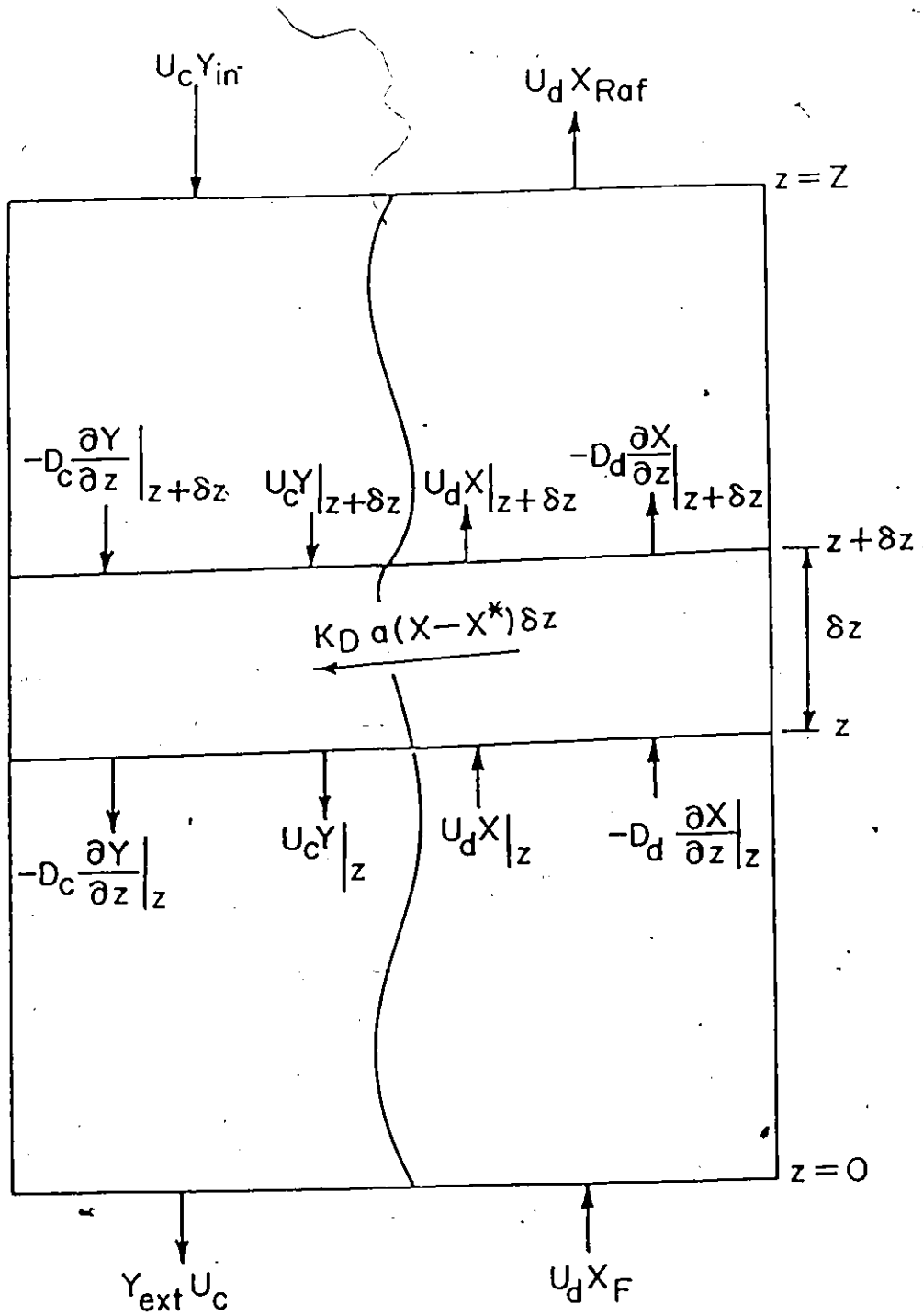

Figure 1.1 Schematic diagram of differential shell balance.

low value of Peclet number indicates that a significant amount of axial dispersion occurs, causing deviations from the "plug flow" assumption.

At the boundary surfaces at each end of the column, there is no capacity to accumulate; therefore time derivatives are not considered. The resulting boundary conditions are the celebrated Danckwerts (1953) boundary conditions i.e.:

$$z = 0 \qquad U_d X_F - U_d X - D_d \frac{\partial X}{\partial z} = 0 \tag{1.6a}$$

$$\frac{\partial Y}{\partial z} = 0 \tag{1.6b}$$

$$z = Z \qquad U_c Y_{in} - U_c Y + D_c \frac{\partial Y}{\partial z} = 0 \tag{1.7a}$$

$$\frac{\partial X}{\partial Z} = 0 \tag{1.7b}$$

The boundary conditions are discussed in the context of solvent extraction in the review by Pratt and Baird (1983).

An analytical approach to equations 1.4-1.7b is given by Burge and Clements (1972). Alternatively, equations 1.4-1.5 along with the boundary conditions can be solved numerically on the computer after replacing the spatial derivatives by their finite difference equivalents and obtaining 2N (where N is the number of the discretized stages) first order ordinary differential equations (Wang et al., 1977). Note that

replacing the spatial derivatives by their finite difference equivalents is tantamount to turning the differential column into a stagewise column where the spatial increment length represents the length of a well mixed compartment in which the concentration of the solute in both phases is constant.

Note that the steady state behaviour of the column is obtained from equations 1.4 and 1.5 by setting the time derivatives to zero. Detailed analysis of the steady state simulation of the Karr column is presented in Chapter 3.

1.3.2 Empirical Models:

An alternative way of quantifying a causal dynamic relationship between an input and an output variable can be obtained through an empirical approach. Such models do not provide any physical insight into the system, which is treated as a black box whose parameters are determined so as to fit the data. Empirical models are obtained by varying the input to the existing physical plant in a prescibed way and then observing the response of the output. There are various ways by which the input is varied; in this work, the so called step test method is employed. In this case, the input variable is varied in a stepwise fashion while the column is initially operating in steady state and the observed transient response of the output is utilized to obtain the parameters of the empirical model (Smith 1972, Rake 1980). The resulting model describes the plant response in the Laplace trans-

form domain and it can readily be transformed into other domains such as time or discrete z-domain. Relevant experimental step tests are presented in the following chapter.

1.4 Literature Survey on the Control of Extraction Columns:

As was stated earlier, researchers studying the application of modern control theory to separation processes have focussed mainly upon distillation. Solvent extraction, despite its lower energy cost and broader potential applicability, has received relatively little attention. Most of the work done covers the dynamics of the extraction process such as the response of the raffinate concentration to step changes in feed and solvent flow rates. Much of this work, as well as work on the control of extraction columns, was extensively reviewed by Pollock and Johnson (1974) and McDonald (1977). Most of the above mentioned work was carried out on pulsed, packed or Scheibel columns and almost always the reported method of control was by conventional analog PID controllers. Mills (1972) in his review article reports two computer control studies of extraction columns but he concludes that both of these works were essentially exercises involving a computer, rather than computer control. A recent article by Wilkinson and Ingham (1983) presents a useful survey of the control and dynamic behaviour of extraction columns. They ask the question of what is achieved by control objectives, and they respond as follows:

- to maintain product quality
- to avoid losses
- to maximize throughput

- to minimize operating costs
- to ensure safe operation

Wilkinson and McDonald (1977) discuss the optimal operating conditions for a multi-stage extraction column and present a table showing the control objectives, disturbances, control actions and measured variables. They note that an optimum operation would be given in terms of either (i) a maximum raffinate concentration above which the process would be uneconomic, or (ii) an extract concentration above some minimum value below which the operation would be uneconomical. From this point of optimality, the control objective is raffinate or extract solute concentration or sometimes both. The disturbances could be in the composition of feed and/or solvent or directly in the feed and/or solvent flow rates. For the control action, they suggest feed or solvent flow rates. They investigated feedforward and feedback control schemes as well as combinations of the two by using conventional analog controllers. The best control strategy found in their work is that of feedback/feedforward combination. The terminology used for the feed-forward control is not that normally used in the control descriptions but rather a strategy which is in between feedback and feedforward control. Raffinate concentration was measured and feed flow rate manipulated. In the feedback-only control scheme, the disturbance in feed concentration has to go all the way down the column before it is detected and the control action can be taken accordingly. This problem of sluggishness was circumvented by incorporating a measurement somewhere in the middle of the column and using a proportional-only controller for this measurement to manipulate the feed flow rate. Feed

back controllers based on raffinate concentration with PI action were retained to manipulate the feed flow for trimming. They also made open loop step tests in feed flow rate and concentration and compared the response of the raffinate concentration with that of the model prediction. Good agreement was found in step changes in feed concentration. However, for step changes in feed flow rate, the experimental response was slower than that predicted because of neglected hydrodynamic effects.

Zimmermann and Blass (1980) report a theoretical and experimental study on the dynamics and control of pulsed sieve plate extraction column. Their contribution was threefold. Firstly, they developed a mathematical model that describes the column operating in the mixer settler region and tested its validity against experimental data. Reasonably good agreement was obtained for low feed/solvent flow ratios. At high feed flow rates, model prediction leads the experimental data because of neglected hydrodynamic effects. Secondly, they have developed an optimal start up strategy to bring the column to steady state in the shortest possible time, and thirdly they used conventional analog controllers to keep the raffinate (dispersed phase) concentration at the desired value by manipulating the continuous feed flow rate. This work mainly deals with the dynamics of the column, rather then discussing the control problems.

Bonnefoi et al. (1977) report a simulated control study of a pulsed plate extraction column used in a nuclear fuel reprocessing plant. Uranium in acidic aqueous phase was extracted into the organic

phase. The column consisted of 2 parts, an extraction section and a scrubbing section. In their mathematical model, Bonnefoi et al. (1977) have neglected the axial mixing term and obtained 4 hyberbolic differential equations (2 for each section). They have applied modal control theory to regulate the concentration at a single point (the concentration being the mode of the system).

Ochiai (1977) reports a control study on an industrial liquid-liquid extraction column which was operating with lower throughputs than the design to meet the product specifications. In this work, the column also consisted of 2 parts, extraction and scrubbing sections, with a total of 50 trays. After studying the column, they have found that the throughput could be increased by 15% by controlling the temperature on the 40th tray by the solvent feed temperature, the interface position by the raffinate flow rate and the aqueous phase holdup by the scrub water flow rate.

Finally Kusuma (1981) has carried out direct digital control studies on the Karr reciprocating plate column. The control objective was the regulation and set point tracking of the holdup of the dispersed phase by manipulating the frequency of reciprocation. Step tests as well as the functional relationship between the holdup and the frequency of reciprocation showed that the system is highly nonlinear, non-minimum phase and asymmetric. The nonminimum phase response in the transfer function relating frequency and holdup was approximated as a dead time so an empirical first order plus dead time model was obtained. The same type of transfer function was also obtained between the holdup and the

dispersed phase flow rate. Holdup was estimated via a regression equation relating it to the differential pressure, frequency of reciprocation and flow rates.

Kusuma has tested and compared the performances of various controllers, namely:

i) Feedback PI for set point changes in holdup

ii) Feedback/feedforward on set point changes in holdup

iii) Modified feedback/feedforward on set point changes in holdup

iv) Feedback/feedforward for disturbances

v) Dahlin controller

vi) Minimum variance controller

vii) Adaptive self tuning regulator

Controllers (i) through (vi) are fixed parameter controllers and they have failed to handle the nonlinearity caused by large changes in the operating conditions, whereas the self tuning regulator (vii) moved the holdup to any desirable level. However Kusuma carried out most of her experiments with $U_c=0$ which is not realistic in industrial applications. This operating condition also does not permit consideration of the upper limit of holdup beyond which flooding occurs.

CHAPTER 2

2. Apparatus, Control Objectives, and Step Tests

2.1 Reciprocating Plate Extraction Columns

Reciprocating plate columns were introduced by Karr (1959) and are frequently refered to as Karr columns. Such a column consists of an assembly of perforated plates with relatively large holes (~15 mm) and high percentage open area (~60%), strung on a drive shaft in the form of a plate stack which is reciprocated vertically inside a shell. The stroke (twice the amplitude) ranges typically from 1 to 4.5 cm and reciprocation speed is in the range of 10-400 rpm. Columns up to 1 m in diameter and 12 m in height are reported (Karr, 1980). Karr columns are easy to scale up, and can handle high throughput with low HETS values. For given flow rates of the dispersed and the continuous phases, an increase in the frequency of reciprocation causes an increase in the holdup of the dispersed phase increase. In a Karr reciprocating plate column the functional relationship between holdup and agitation rate (i.e. product of stroke and frequency of reciprocation) is very nonlinear as shown in Figure 2.1. As the frequency of reciprocation

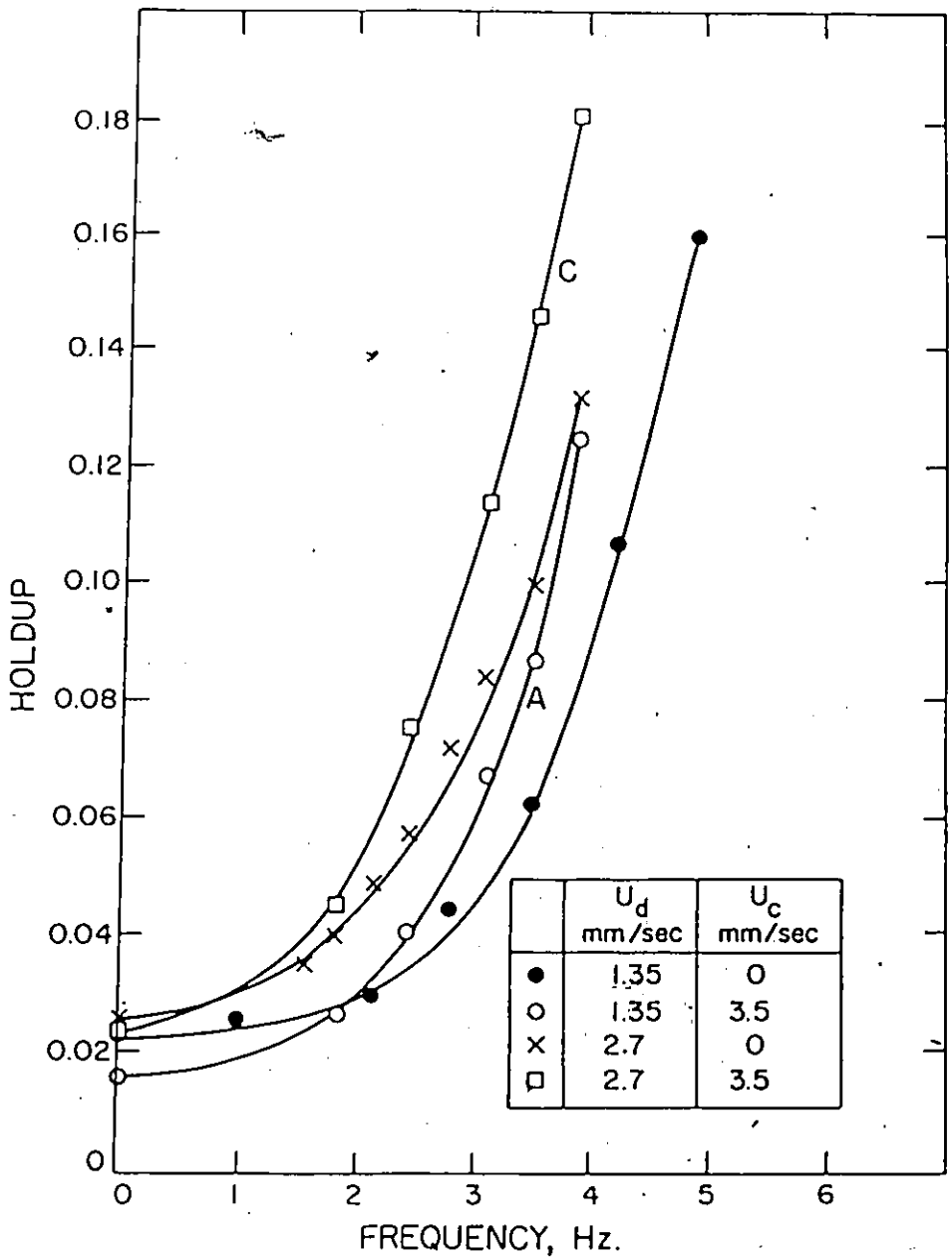

Figure 2.1 Holdup vs. frequency of reciprocation. (From Taylor et. al. (1982)).

increases at a constant stroke, the turbulence level increases and droplets get smaller. As the drops get smaller the drag force acting on them increases relative to buoyancy, and the velocity of the droplets decreases. This causes an increase in the residence time of droplets which in turn leads to an increase in the holdup. Further increase of the frequency of reciprocation eventually leads to the unstable condition known as flooding. When flooding occurs the stability of the two phase flow is no longer preserved.

In a small Karr column the HETS decreases with an increase in frequency of reciprocation (Karr, 1959), therefore it should be operated near its flooding point. However, a small change in the system parameters such as flow rates of either phase would shift the operating curve, say from A to C, shown in Figure 2.1, which will cause the column to flood. For this reason, the column is operated with some safety margin, which might increase the HETS drastically. From this point of view, a controller designed to keep the holdup at a certain value would be very economical, as in this way the column can be operated around the possible minimum HETS value which, in turn, increases the efficiency of the column (i.e. by extracting the solute from the feed at the maximum possible amount for a given heigth of the column). In large diameter Karr columns (1976) the HETS can reach a minimum at agitation levels below the flooding point. This is because axial mixing effects become serious at high agitation levels.

Besides frequency of reciprocation holdup is dependent upon the operating conditions i.e. the flow rates of the dispersed and continuous

phases, and the physico-chemical properties of the system. For a given frequency of reciprocation this dependence can be summarized as follows:

Flow rate of the dispersed phase: An increase in the dispersed phase flow rate results in an increase in the number density of the droplets therefore the holdup increases.

Flow rate of the continuous phase: an increase in this variable, as was stated earlier, increases the drag force acting on the droplet hence increases residence time of the droplets therefore the holdup increases.

Viscosity of the continuous phase: This has the same affect as increasing the continuous phase flow rate i.e. drag force around the droplet increases consequently the holdup.

Density difference: The higher the density difference the higher the bouyancy force therefore the droplets will rise faster resulting in a decrease in the holdup.

Interfacial Tension: High interfacial tension results in large droplet diameters and hence low holdup values. If there is a significant change in the interfacial tension in the column this will cause a variation in the holdup throughout the contactor. The interfacial tension in a liquid-liquid dispersion is influenced by the direction of mass transfer which, in turn, affects the coalescence behaviour of the

droplets (Shen et al., 1985). Further discussion of the mass transfer influence on the column hydrodynamics is given in Chapter 3.

2.1.1 The Extraction System and the Experimental Apparatus

The extraction system used was water, kerosene and acetic acid with acid being transferred from kerosene into water. Because of the wetting properties of the column kerosene was dispersed (stainless steel plate stack). Acetic acid is very soluble in kerosene as well as in water. It has a distribution coefficient of approximately 220 (Noh, 1981) in favour of water, yielding a high value of the extraction factor (i.e. the ratio of the equilibrium line slope to that of the ratio of the feed to the extract phase flow rate). During the experiments this created problems at high frequencies of reciprocation at which the exit kerosene phase was almost totally depleted in acid. The frequencies of reciprocation were 1.1–4.0 Hz, and the stroke was 2.54 cm. The superficial velocity ranges were 0.25–0.905 cm/s for the water (continuous phase) and 0.25–0.785 cm/s for the kerosene.

The present experimental apparatus has previously been used to study the hydrodynamic characteristics of liquid-liquid extraction systems (Hafez and Baird, 1978; Hafez et al., 1979) and more recently servo and regulatory direct digital control of the holdup of the dispersed phase (Kusuma, 1981; Taylor et al., 1982). A schematic diagram of the column is given in Figure 2.2. The column consists of three glass sections flanged together giving an overall height of 3.96 m. The internal diameter is nominally 0.15 m. The plate stack consists

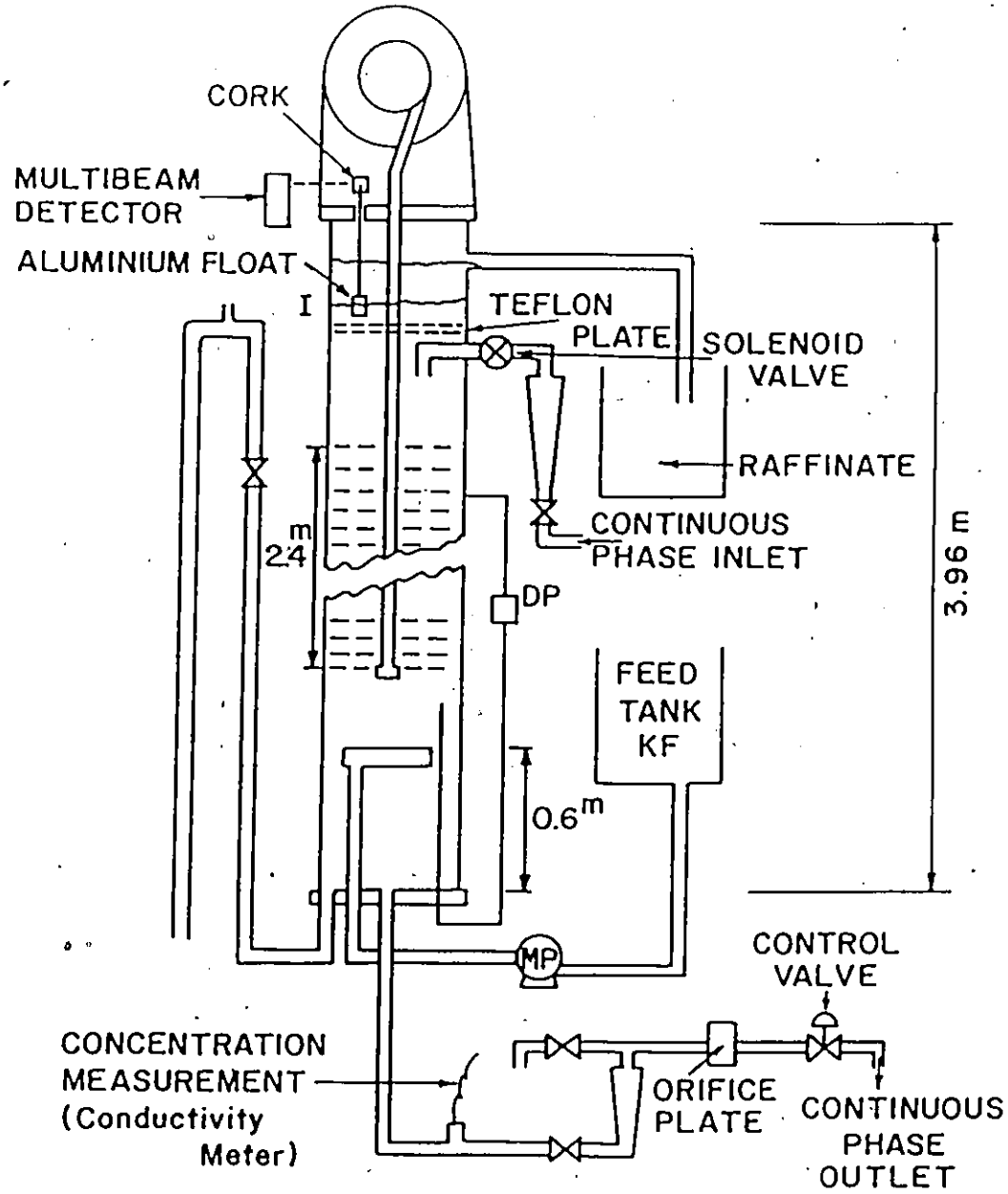

Figure 2.2 Schematic diagram of the experimental apparatus.

of 42 perforated stainless steel plates having a diameter of 0.147 m and spaced 0.0508 m apart. Each plate has 54 holes having a diameter of 14.29 mm. The height of the plate stack is 2.4 m and it is supported on a stainless steel shaft which is attached at the top to an adjustable yoke. Reciprocation is provided by a .373 W electric motor coupled to the yoke through a variable speed reducer. The amplitude (half stroke) is fixed at 0.0127 m and the frequency can be varied between 0.5-5.0 Hz. Tap water is fed as continuous phase to the top of the column from an overhead feed tank (not shown in Figure 2.2) while the dispersed kerosene phase containing acetic acid is pumped from the holding tank KF by a metering pump MP to the bottom of the column and dispersed by means of a perforated disk distributor. The outlet of the continuous phase is at the very bottom of the column about 0.6 m below the dispersed phase distributor.The outlet of the dispersed phase is about 0.5 m above the continuous phase inlet and the interface I is formed between the continuous phase water inlet and dispersed phase kerosene outlet (the control of the interface is discussed in section 2.2.1.2). The column was operated under the countercurrent regime.

2.1.1.1 Measurement Devices

Holdup Measurement: Holdup was inferred from a regression equation on: the differential pressure measured by a transducer DP (Figure 2.2) across a section of the column, the frequency of reciprocation, and the flow rates of the two phases. The last three variables are included into the regression equation to account for the frictional contributions of these variables. The differential pressure transducer

was a Pace Eng. Co., model P7D with a 0.1 psi diaphragm. The output was +10. V D.C. proportional to the differential pressure. It was calibrated so that a holdup of 0.4 would correspond to 10. V, and a holdup of 0.0 to 0.0 V. More details can be found in Kusuma (1981). The high frequency component on the differential pressure measurement which is due to plate stack oscillations was filtered through a fourth order Butterworth low pass filter with a cutoff frequency of 0.25 Hz. The output from the filter was then used as an input into the computer. The holdup measurement had a noise level with a standard deviation of 0.00024. It should be noted that holdup variations along the extractor are known to exist (Jiricny and Prochazka, 1980) but this method only gives an average holdup. Note that the parameters in the regression equation obtained by Kusuma (1981) were changed slightly in this work after carrying out a new set of 2 level factorial design experiments (Box et al., 1978). The regression equation used was:

$$\text{Holdup}=0.0002112\ \Delta P + 6.708\ U_d + 1.633\ U_c + 0.003824\ f \qquad (2.1)$$

Where ΔP (kPa), U_d (m/s), U_c (m/s) and f (Hz) are differential pressure, dispersed phase flow rate, continuous phase flow rate and frequency of reciprocation respectively.

Concentration measurement: The concentration of the acetic acid in the outlet continuous phase was measured by a conductivity cell. Output voltage was calibrated by using known concentrations of acetic acid. The noise level in the measurement had a standard deviation of 0.00025 moles/L.

Frequency of Reciprocation Measurement: This was done by a tachometer. Noise level in the frequency measurement is 0.005 Hz.

Continuous phase flow rate measurement: Flow rate was determined from the pressure drop across an orifice plate via a differential pressure transducer. The standard deviation of the flow rate measurement is 0.00001 m/s.

2.1.1.2 Auxiliary Control Elements

Interface Controller: the interface has to be controlled otherwise the dispersion band will move upwards or downwards thereby destabilizing the operation of the column. An aluminium float was designed which is heavier then kerosene but lighter then water so that it floated in the dispersion band. A solenoid valve was fitted to the continuous phase inlet. If the interface level was rising, a cork which was attached to the top of the float rod would block the light entering a multibeam detector which in turn deactivates the solenoid valve until the float returns to its original position. This control cycle was typically repeated 20-25 times per minute. This frequency was very high with respect to system time constants so it had no effect on the column operation. A fixed Teflon plate of 2.5 mm thickness having the same percent open area as the reciprocating plates was used to promote coalescence beneath the dispersion band so that the thickness of this band would be minimized.

2.2 Distributed Computer System

Data acquisition and control were done on a DEC VAX 11/750 host computer (available for general use) with a DEC DPM-23 frontend of the chemical engineering department. Besides the extraction column, a packed bed reactor and an extractive distillation column are also wired to the network and can all run simultaneously.

The high level control was done in the host VAX 11/750 with a single precision word length of 4 bytes. The main control program was written in FORTRAN-77. VAX 11/750 has 1.75 Mbyte memory with RM80 124 Mbyte disk and operates with VMS which provides several system service calls for real time services. For example, the hibernate ($HIBER) system service call halts the execution of the process temporarily. Hibernation is reversed by the schedule wakeup system service ($WAKE) called at prescribed time intervals, for instance in every 20 seconds to perform the calculations for obtaining the controller output. In between "hibernate" and "wakeup" the system is inactive but remains known to the system and can be interrupted, for example, to receive Asynchronous system traps (ASTs). An AST provides a change in any variable such as set point changes in the control variable.

The frontend is interfaced to the extraction column through A/D and D/A input/output boards. 4 analog inputs and 2 analog outputs were connected to the frontend screw terminals. The input signals were: differential pressure, frequency of reciprocation, flow rate measurement of the continuous phase, and the concentration of the extract outlet.

The output signals were: set point change of the frequency of reciprocation and the continuous phase flow rate.

The frontend is composed of an LSI-11/23 microcomputer with 192 Kbytes of memory, IP12 process interface I/O boards, and a power supply unit. DY32 communications link provides the communication between VAX and DPM-23 at a rate of 56 Kbaud. The single precision word length of the microcomputer is 2 bytes.

The function of the frontend is twofold: i) to receive the signals from the process, filter them if necessary, and send these signals to the host computer and receive signals from the host computer to be sent to the process. ii) to achieve low level control such as servo control of a manipulated variable.

Low level control of the frequency of reciprocation was done on a MACSYM 2 microcomputer. MACSYM 2 is endowed with A/D input/output channels and has a 256 Kbyte memory. The function of the MACSYM-2 microcomputer is twofold i) to convert the analog signal into a 14 bit digital signal ii) to perform servo control for the frequency of reciprocation. The set point of the frequency of reciprocation is received by MACSYM 2 microcomputer as an analog signal from the frontend. The controller was a PI controller and the algorithm was written in BASIC. The frequency control was done in every 0.5 seconds. The output of the microcomputer was received by a pulse generetor to be sent to the stepping motor controller attached to the agitator speed control.

For low level control of continuous phase flow rate a voltage signal from an orifice plate flowmeter was received by the frontend. A core resident PI controller in the frontend performed the low level control of the continuous phase flow rate every second. The set point was sent by the main control program from the host computer.

2.3 Control Objectives

The objective is to control the Karr column. By control the following is meant. (i) keeping one or both of the outlet concentrations at the desired set point despite disturbances (regulatory control), or (ii) changing the set point every so often because of a higher level optimization of the supervisory controller (servo control), or (iii) both (i) and (ii). Implicit in all of the foregoing is maintaining the stability of the hydrodynamics of the column while fulfilling the control objectives.

As stipulated by Wilkinson and McDonald (1977), the control strategy is based on the economy of the operation, and it is to keep the raffinate concentration below a maximum value or to keep the extract outlet concentration above a minimum value. Otherwise the process will be uneconomical. Holdup must also be controlled otherwise the column can flood. It is desired to operate at high holdup values in order to maximize the rate of mass transfer.

The input, state, intermediate, and output variables in the Karr column can be specified in the following way:

Input Variables: Inlet flow rates and inlet concentrations of the two phases, frequency and amplitude of reciprocation.

State variables: Concentrations of both phases along the column, holdup of the dispersed phase.

Intermediate variables: Interfacial area available for mass transfer, mass transfer coefficient and drop size distribution.

Output variables: Outlet flow rates and outlet concentrations of the two phases.

A close look at intermediate variables level, shows that they are all related to the frequency of reciprocations and none of these variables can readily be measured on line.

2.3.1 The Controlled and Manipulated Variables

In this work the extract outlet concentration was chosen to be the controlled variable since it was simple to measure on line, and the flow rate of the continuous phase was chosen to be the manipulated variable. The flow rate of the dispersed kerosene phase is left as a disturbance. The major reason for not using kerosene flow rate as the manipulated variable was that it was pumped by a positive displacement pump and it was technically diffucult to manipulate the flow rate. The frequency of reciprocation was used to control the dispersed phase holdup since holdup was most sensitive to this variable.

2.4 Step tests

As discussed in the previous section, there are 2 potential input variables, namely the frequency of reciprocation and the continuous phase flow rate; and two control variables, namely the holdup of the dispersed phase and the extract outlet concentration. In what follows the step responses between the above mentioned inputs and outputs are discussed.

i) Response of holdup to a step increase in frequency of reciprocation: A typical response is given in Figure 2.3. It has a wrong way (nonminimum phase) response. This is however, a measurement problem rather than an intrinsic process response. It is due to the fact that one tap of the differential pressure measurement is located in between two plates and the other is located about 10 cm below the bottom plate. Increasing the frequency of reciprocation creates velocity fields with different intensity on the tip of the probes thereby creating a sudden drop in the pressure measurement because of the Bernouilli effect.

ii) Response of extract concentration to a step increase in frequency of reciprocation: The time constant and the dead time of this process are inversely proportional to the continuous phase flow rate U_c. The dead time of the process is a result of the transportation lag which is the time required for the continuous phase to travel from the tip of the kerosene phase distributor to the point where the sensor is located. The dead time can hence be calculated if the flow rate of the continuous phase is known. For the reasons given in Chapter 1, an increase in the

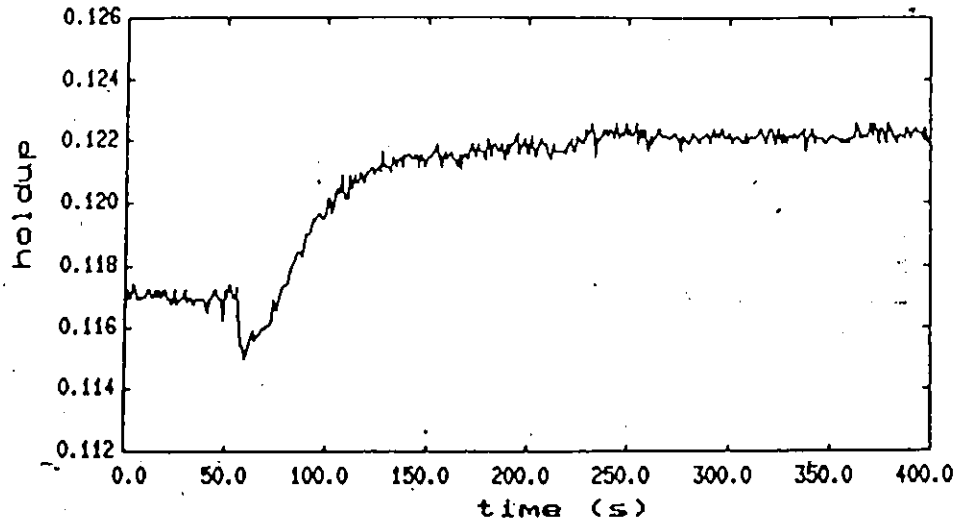

Figure 2.3 Holdup response to a step change in the frequency of reciprocation. (U_d = .32 cm/s, U_c= 0.4 cm/s, f: 1.9→2.3 Hz).

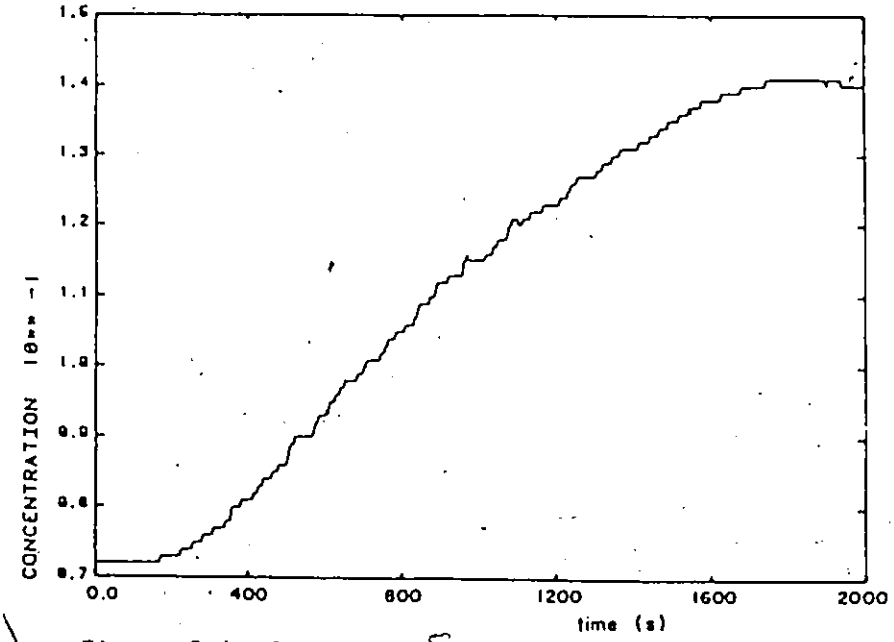

Figure 2.4 Extract concentration response to a step change in the frequency of reciprocation. (U_d = 0.3 cm/s, U_c = 0.32 cm/s, f: 1.3→2.3 Hz.)

frequency results in an increase in the extract outlet concentration, thereby the system shows a positive gain (Figure 2.4). The units of concentration in this and later figures are mol/L or kmol/m^3.

iii) Response of holdup to a step increase in continuous phase flow rate: This response is presented in Figure 2.5. The response of the holdup has two effective time constants, one for large droplets which is fast, and one for small droplets which is slow. This is because, for a given continuous phase flow rate the small droplets have a larger drag force acting on them, then do the larger droplets. However, the time constants of this process are relatively small in comparison to the time constants of the other processes; therefore the dynamics can be neglected.

iv) Response of extract concentration to a step increase in continuous phase flow rate: This response is given in Figure 2.6. It has a dead time which is again dependent on the flow rate. An increase in the flow rate results in a decrease in the concentration, thereby yielding a negative gain. The effect of increased holdup to give higher mass transfer is outweighed by the dilution effect of using more solvent. Further discussion is given in Chapter 3.

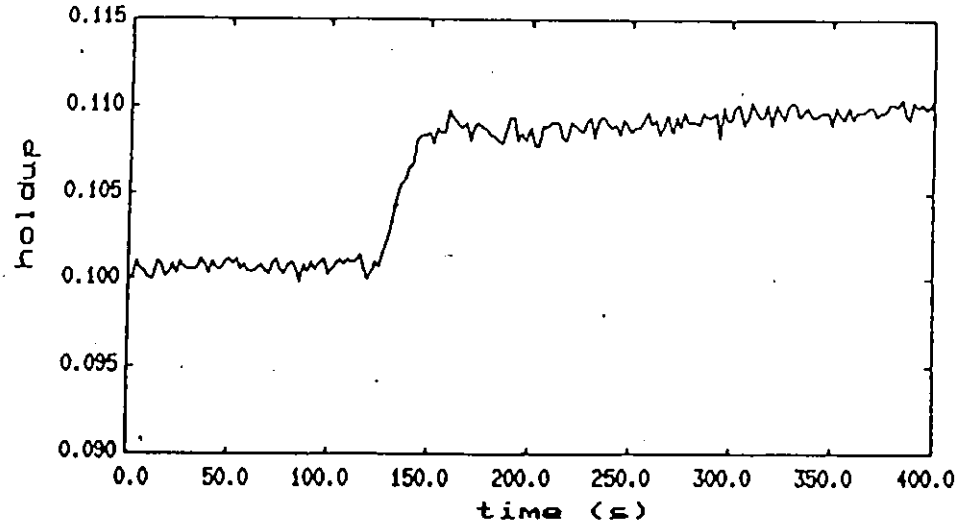

Figure 2.5 Holdup response to a step change in the continuous phase flow rate. (U_d = 0.32 cm/s, f = 2.0 Hz, U_c: 0.4→0.55 cm/s).

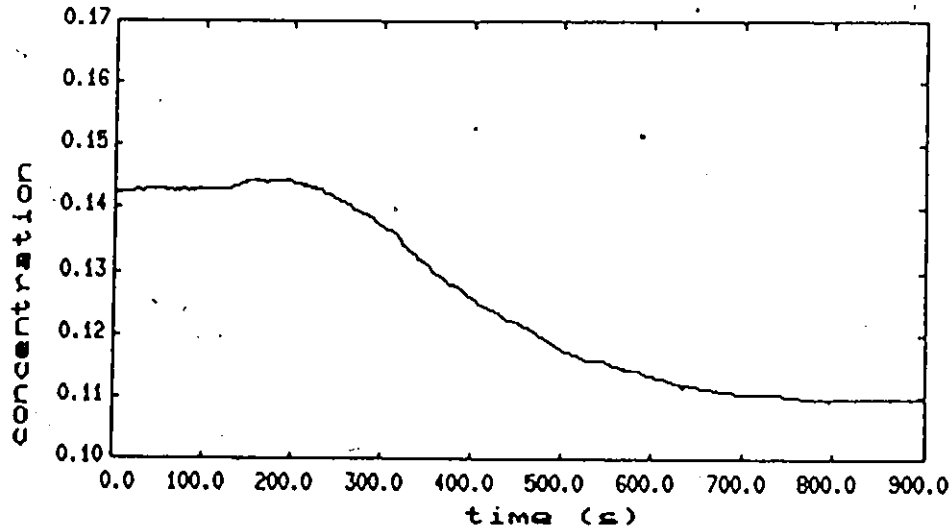

Figure 2.6 Extract concentration response to a step change in the continuous phase flow rate. (U_d = 0.32 cm/s, f = 2 Hz, U_c: 0.4→0.55 cm/s.)

CHAPTER 3

3. Steady State Behaviour of the Extraction Process:

This chapter is composed of two parts. In the first part (3.1) some simulation and experimental results on the hydrodynamics of the Karr column are presented. In order to control a reciprocating plate extraction column so that flooding is prevented, it is useful to have a mechanistic model for the effects of operating variables upon the holdup. Such a model has recently been proposed (Baird and Shen, 1984) based on extensive earlier work on column hydrodynamics. This model has been compared with data obtained from the experimental column. In the second part (3.2) steady state simulations of the column using mechanistic models presented in chapter 1 are carried out. These are useful for gaining insight into the column behaviour and interpreting the closed loop response characteristics.

3 .1 Hydrodynamics of the Column

3 .1.1 Background Information on the Hydrodynamics of the Karr Column

The slip velocity of a droplet is the velocity one would observe while travelling with the speed of the droplet in relation to the

counterflowing continuous phase and it is related to the superficial velocities of the dispersed and continuous phase via the relationship (Lapidus and Elgin, 1957):

$$U_s = \frac{U_d}{\phi} + \frac{U_c}{1-\phi} \tag{3.1}$$

The slip velocity may be regarded as the vectorial difference between the dispersed and continuous phase velocities.

Thornton (1956) treated holdup as an independent variable, and the velocities U_c and U_d as dependent variables and assumed that flooding occurs when U_d or U_c reaches a maximum with respect to holdup.

$$\frac{\partial U_c}{\partial \phi} = 0 \text{ or } \qquad \frac{\partial U_d}{\partial \phi} = 0 \text{ at flooding} \tag{3.2}$$

By assuming that the ratio U_d/U_c (=L) was constant, Thornton showed analytically that the holdup at flooding was given by:

$$\phi_F = \frac{(L^2 + 8L)^{0.5} - 3L}{4\ (1-L)} \tag{3.3}$$

The slip velocity was expressed (Lapidus and Elgin, 1957) in terms of a characteristic velocity U_k which is the slip velocity at zero holdup is:

$$U_s = U_k \ (1.0-\phi) \tag{3.4}$$

Baird and coworkers (Baird et al., 1971; Baird and Lane, 1973; Hafez et al., 1979; Baird and Shen, 1984) developed a modified relationship between the slip velocity and a velocity analogous to characteristic velocity for the Karr columns based on experiments with 5 and 15 cm diameter Karr columns under well agitated conditions.

$$U_s = U^* \ (1.0 - \phi)/\phi^{1/3} \tag{3.5}$$

where

$$U^* = d_{32} \left[\frac{g \ \Delta\rho}{\rho_c \ \mu_c}\right]^{1/3} K^{-2/3} \tag{3.6}$$

This relationship (equation 3.5) becomes unrealistic as holdup tends to zero, but in the normal operating range of holdups it was found to be consistent with observations.

The Sauter mean droplet diameter d_{32} is calculated from the agitation rate and the interfacial tension (Baird and Lane, 1973). The circulation parameter K in the above equation is 30 for the rigid drops and 15 for circulating drops. In the present system, the rigid value is taken in view of the high degree of contamination.

By combining equations (3.1),(3.4) and (3.5) it can be shown that

$$U_c = \frac{(1-\phi)^2 \, U^*}{\phi^{1/3} \, (1-L) + L \, \phi^{-2/3}} \tag{3.7}$$

When Thornton's criterion (equation 3.2) is applied to equation (3.7) the following modified Thornton relation is obtained for the holdup at flooding:

$$\phi_F = \frac{(9L^2 + 54 \, L + 1)^{0.5} - 7L - 1}{10 \, (1-L)} \tag{3.8}$$

For a given set of operating conditions, i.e. superficial velocities of the two phases, frequency and stroke of reciprocations, circulation parameter K, interfacial tension, continuous phase viscosity, and phase densities, the Sauter mean droplet diameter and the holdup ~~can~~ be calculated as follows.

Power dissipation is calculated from:

$$\psi = \frac{2\pi^2}{3} \frac{(1 - S^2)}{h \, C_0^2 \, S^2} \bar{\rho} \, (A \, f)^3 \tag{3.9}$$

In this work the fractional open area S is taken as 0.6, the orifice coefficient C_0 is taken as 0.6, and the plate spacing h is 0.05 m. The average dispersion density $\bar{\rho}$ is given by:

$$\bar{\rho} = \rho_d \ \phi + (1-\phi) \ \rho_c \tag{3.10}$$

The Sauter mean drop diameter d_{32} is given by the equation of Baird and Lane (1973)

$$d_{32} = 0.36 \ \frac{\gamma^{0.6}}{\bar{\rho}^{0.2} \ \psi^{0.4}} \tag{3.11}$$

In this work, the interfacial tension γ was found to be 0.0175 Nm^{-1} for kerosene/water. This value is lower than most reported measurements for this system, indicating a fairly high contamination level. The densities of kerosene and water (ρ_d, ρ_c) were respectively 812 and 1000 kg/m^3.

Holdup is obtained from the rewritten form of equation (3.7)

$$\frac{U_c}{U^*} = \frac{(1 - \phi)^2}{\phi^{1/3} \ (1-L) + L\phi^{-2/3}} \tag{3.12}$$

The above equation as written is unwieldy for finding the root ϕ. However after multiplying the numerator and denominator of the right hand side by $\phi^{2/3}$ and then taking the third power of the resulting equation, the following rational polynomial in the holdup ϕ is obtained.

$$\left(\frac{U_c}{U^*}\right)^3 = \frac{(1-\phi)^6 \ \phi^2}{(1-L)^3 \ \phi^3 + 3(1-L)^2 \ L \ \phi^2 + 3(1-L) \ L^2 \ \phi + L^3} \tag{3.13}$$

The above equation could further be simplified to obtain an 8th order polynomial in ϕ, but in the above form it is simple enough for the root to be sought in the interval 0-0.4 by using the Newton-Raphson method. The initial estimate value of the holdup was found by the bisection method as the convergence was found to be sensitive to this value.

3.1.2 Experimental Procedure

Superficial velocities and the frequency of reciprocation were set for each run. In the absence of mass transfer, the operating holdup was determined after about 15-20 minutes of steady operation by "freezing" the column i.e. simultaneous closing of all inlet and outlet valves. When mass transfer occurred, holdup was inferred from the differential pressure measurements, using the regression equation discussed in chapter 2. The interface was manually controlled at a fixed position by adjusting the aqueous exit flow. Repeated runs gave up to 5% variation in the holdup measurements. All but 6 data points were obtained under conditions of no mass transfer with acid free kerosene fed to the column. The remaining data points were obtained with mass transfer of acetic acid from the kerosene feed to the aqueous phase. The interfacial tension between kerosene and water was measured by the Wilhelmy plate method.

3.1.3 Results and Discussions

The experimentally found holdups are compared with the theoretical holdups obtained from the computer program as plotted in Figure 3.1. Analysis of variance (ANOVA) shows no evidence of lack of fit (within 95% confidence limit).

The data obtained with mass transfer gave more scattered results as expected. It is now well known that in dispersed to continuous phase mass transfer, the surface tension gradients formed around the two approaching droplets cause a rapid drainage of the continuous phase and hence enhance coalescence. This is known as Groothuis-Zuiderweg effect (1960) and has not been accounted for in the present model. Surface tension also varies significantly throughout the column which in turn changes the equilibrium drop size according to equation (3.11). However it is to be noted that whether or not mass transfer occurs, the holdup value at flooding should remain unchanged since this value, unlike values below the flooding point, is a function only of the ratio of the flow rates but not any physical properties (see equation 3.3 or equation 3.8).

In the absence of mass transfer, data at low holdup values fall above the 45 degree line. These points correspond to low agitation rates and Baird and Lane (1973) had found that equation 3.11 tended to overpredict the drop size at low agitation i.e. to underpredict the holdup. Figure 3.2 shows a plot of holdup obtained from the model-based

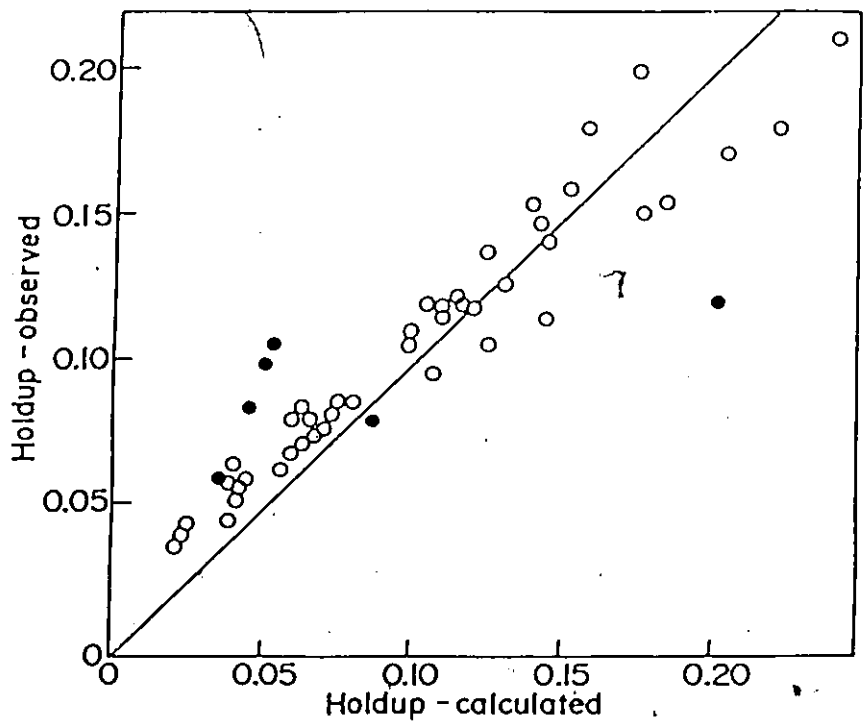

Figure 3.1 Comparison between observed and calculated holdup values
o data without mass transfer
• data with mass transfer

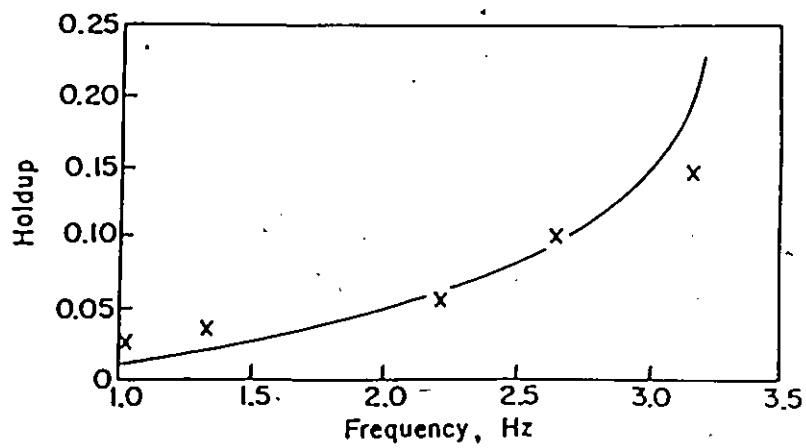

Figure 3.2 Typical data compared with model.
$U_d = 0.26\ cm\ s^{-1}$ $U_c = 0.33\ cm\ s^{-1}$
× data points without mass tranfer
— Model prediction ($\phi_F = 0.233$)

computer program versus frequency of reciprocation, for particular fixed flow rates of dispersed and continuous phases. The x points correspond to experimentally found values. This confirms the tendency of the model to underpredict holdup at low agitation levels. The lower limit of agitation for reasonable accuracy has been suggested to be A*f=3 cm/s. (Baird and Lane, 1973).

It may be concluded that in the absence of mass transfer, and at high agitation rates, the mechanistic model is satisfactory. However in the presence of mass transfer in this work, the model is not reliable for the prediction of holdup (Figure 3.1). A similiar conclusion was reached by Shen et al. (1985) for dispersed to continuous phase mass transfer in a 5 cm diameter column, although the mechanistic model was found to be reasonable for the reversed direction of mass transfer (continuous to dispersed phase). In general, the model is useful for approximately estimating the column characteristics for a given system.

In developing the control system for extraction in the Karr column, it has been decided to use equation (3.8) as the basis for the upper limit of holdup, since it is independent of system properties. The control system is prevented from setting the holdup any higher than 60-70% of the value of ϕ_F calculated from equation (3.8). The estimation of holdup at sub-flooding conditions using the mechanistic model cannot be used in the control system because of the effects of mass transfer as discussed above, and because the mechanistic model has only been developed for the steady state. Instead, holdup was inferred, as discussed in Chapter 2, from a regression equation relating measured differential pressure across a section of the column, the frequency of

reciprocation and the flow rates of the two phases. This holdup estimate was used to implement the control strategies.

3.2 Mass transfer Characteristics of the Column

In the first part of the simulation program, discussed in the previous section, the holdup in the column was calculated from the given operating conditions and the system physical properties. In this part the steady state concentration profile in the column is calculated by numerically solving the differential equations that describe the solute material balance in the column. The steady state equations are obtained from equations (1.4)-(1.5) in chapter 1 by setting the time derivatives to zero as follows:

$$D_d \frac{d^2X}{dz^2} - U_d \frac{dX}{dz} - K_D\, a(X-X^*) = 0 \qquad (3.14)$$

for the dispersed kerosene phase, and

$$D_c \frac{d^2Y}{dz^2} + U_c \frac{dY}{dz} + K_D\, a(X-X^*) = 0 \qquad (3.15)$$

for the continuous water phase.

With boundary conditions

$$z = 0 \qquad U_d X_F - U_d X - D_d \frac{dX}{dz} = 0, \qquad \frac{dY}{dz} = 0 \tag{3.16}$$

$$z = Z \qquad U_c Y_{in} - U_c Y - D_c \frac{dY}{dz} = 0, \qquad \frac{dX}{dz} = 0 \tag{3.17}$$

And the linear equilibrium relationship is given by

$$Y = m X^* \tag{3.18}$$

Where m is the distribution coefficient and X^* is the solute concentration in equilibrium with Y.

An analytical solution to these equations was originally developed by Miyauchi and Vermeulen (1963) who showed how the concentration profile could be calculated from entry conditions for a given length of column. More recent developments are included in the review by Pratt and Baird (1983).

For the simulation purposes these equations can also be solved numerically on the computer after writing the finite difference equivalent of the equations (3.14)-(3.17) and solving the resulting set of algebraic linear equations as is done in the following way.

The column length is divided into N stages. Where N is the number of sections in the column in which the concentrations are uniform or equivalently the number of well mixed compartments. The length of the column is taken to be the length of the plate stack. Disengagement

sections are taken to be 10 cm regions below and above the plate stack. The following equations are obtained in this way:

$$\frac{D_d}{\Delta z^2}(X_{n+1} - 2X_n + X_{n-1}) - \frac{U_d}{\Delta z}(X_n - X_{n-1}) - K_D\, a(X_n - \frac{Y_n}{m}) = 0 \qquad (3.19)$$

$$\frac{D_c}{\Delta z^2}(Y_{n+1} - 2\,Y_n + Y_{n-1}) + \frac{U_c}{\Delta z}(Y_{n+1} - Y_n) + K_D\, a(X_n - \frac{Y_n}{m}) = 0 \qquad (3.20)$$

$$U_d\, X_F - U_d\, X_0 + \frac{D_d}{\Delta z_1}(X_1 - X_0) = 0 \qquad (3.21)$$

$$Y_0 = Y_1 \qquad (3.22)$$

$$X_{N+1} = X_N \qquad (3.23)$$

$$\frac{-D_c}{\Delta z_2}(Y_{N+1} - Y_N) - U_c\, Y_{in} - U_c\, Y_{N+1} = 0 \qquad (3.24)$$

Where the subscripts 0 and N+1 refer to the disengagement sections and Δz is the length of a well mixed compartment.

The axial dispersion coefficients in each phase were taken to be equal and calculated from the correlation given by Hafez and Baird (1979). The dispersed phase mass transfer coefficient k_d is calculated from an equation proposed by Treybal (1963). This equation is valid for

rigid spheres with no internal circulation and in the absence of interfacial turbulence and drop oscillations, and when the mass transfer takes place by molecular diffusion.

$$k_d = \frac{2\pi^2 D_m}{3\, d_{32}} \tag{3.25}$$

$$k_d a = \frac{4\pi^2 D_m \phi}{d_{32}^2} \tag{3.26}$$

The resistance in the aqueous phase was neglected in this work because of high affinity of acetic acid to water; thus the overall mass transfer coefficient K_d is taken to be equal to k_d. Holdup was calculated from the hydrodynamics of column by the method described in the previous section. Molecular diffusivity D_m was obtained from the Wilke-Chang correlation assuming that kerosene has the effective molecular properties of dodecane (Noh, 1981) and was taken as 1×10^{-9} m^2/s.

The equations (3.19-3.24) can be written compactly as:

$$\underline{\underline{A}}\ \underline{w} = \underline{b} \tag{3.27}$$

$\underline{\underline{A}}$ is a square matrix with 2N+2 rows and columns containing constants as its elements, $\underline{w}$ is an augmented vector of concentrations

X_i's and Y_i's i=0,N+1 i.e $w=(X_i,Y_i)'$ and b is a vector of inputs to the column i.e.

$$\underline{b} = (U_d\ X_F,\ 0,\ 0.....0,\ U_c\ Y_{in})' \tag{3.28}$$

Because of the high dimensionality of the $\underline{\underline{A}}$ matrix, equations were solved by iteration as opposed to matrix inversion method. Different N values were used namely 21, 42, 84 and 168. The outlet concentration of the continuous phase for different N values was calculated. As N increases, the number of iterations before the concentration converges increases drastically. However the change in concentration as N increases was also negligible. Therefore it was decided to use N equal to 21. Note that the concentration profile obtained from the matrix inversion method for a small N value of 5 gave negligibly different concentration profile from that of the iteration method.

Because of the reasons given in the first part of this chapter, the influence of variations in the interfacial tension on drop size is substantial but unpredictable in quantitative terms; consequently no direct quantitative comparison between simulated and experimental results had been sought. The mass transfer coefficient had to be adjusted until agreement was reached between the experimental and the model prediction of the continuous phase outlet concentration. However, modelling the column from a mechanistic sense is helpful in elucidating the extraction process and its nonlinearities. The simulation results

obtained are used in the following chapters to interpret the experimental response characteristics of the column under automatic control. Experimental step tests also confirmed the trends revealed from the simulation.

3.2.1 Simulation Results and Discussion

In the simulation results presented here the dispersed phase flow rate and acetic acid concentration in the feed were kept constant and the simulation was done for two different continuous phase flow rates U_c namely 0.3 and 0.4 cm/s. In each figure presented, the dotted line corresponds to the operating condition when U_c is 0.4 and the solid line to 0.3 cm/s.

Figure 3.3 shows what will happen when only the holdup is controlled in the column so that flooding will not occur. For a given holdup the outlet continuous phase extract concentration is lower for higher flow rate. The reason for this is twofold: i) The dilution effect of the high flow rate. ii) Lower agitation intensity for the high flow rate when holdup is fixed. In Figure 3.3 it can be also seen that it is thermodynamically feasible to obtain the same outlet concentration (~ 0.18 in the figure) at higher U_c, but the holdup required would be very close to the flooding limit i.e. hydrodynamically infeasible. Note that the rate of change of concentration w.r.t. holdup, i.e. the steady state gain relating the holdup to the output concentration, is higher at low holdup values and becomes less pronounced at high holdup values.

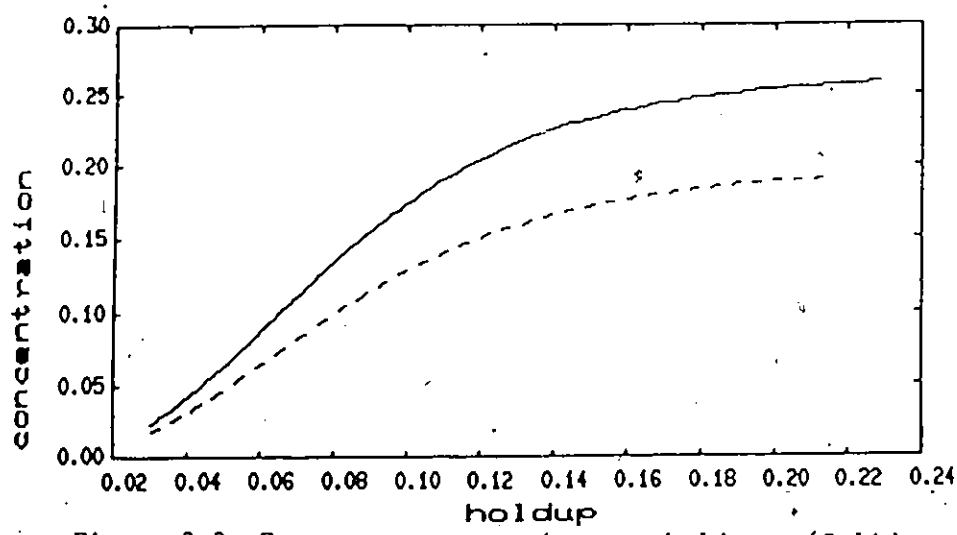

Figure 3.3 Extract concentration vs. holdup. (Solid curve: U_c = 0.3 cm/s, U_d = 0.4 cm/s dotted curve: U_c = 0.4 cm/s, U_d = 0.4 cm/s.)

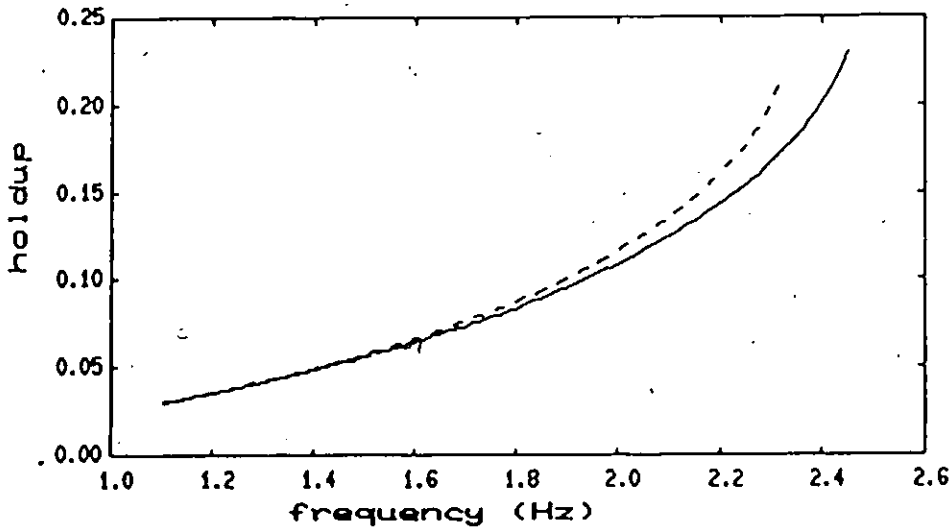

Figure 3.4 Holdup vs. frequency of reciprocation. (Solid curve: U_c = 0.3 cm/s, U_d = 0.4 cm/s dotted curve: U_c = 0.4 cm/s, U_d = 0.4 cm/s.)

This results in a distortion of the amplitude of the output concentration when the holdup is varied sinusoidally around a fixed value. More will be discussed on this in chapter 7.

When the continuous phase flow rate is low, the frequency is high in order to keep the holdup at this desired value (Figure 3.4); this high agitation yields a drop size distribution in which the mean droplet diameter is small (Figure 3.5). This gives high interfacial area and hence high rates of mass transfer. The small droplet diameters also lead to low rise velocities and high residence times which in turn increase the contact time, consequently increase the degree of extraction. The extract concentration at the two flow rates approaches the limiting values for complete extraction.

3.3 Conclusions

In the second part of this chapter the steady state mass transfer characteristics of the column are simulated. Because of the complex nature of the mass transfer induced droplet droplet interaction no quantitative comparison has been made between the simulated and the experimental results. Instead, the nonlinearities and operability of the column were demonstrated and the simulation results are used in the following chapters to interpret the data obtained from the column under automatic control.

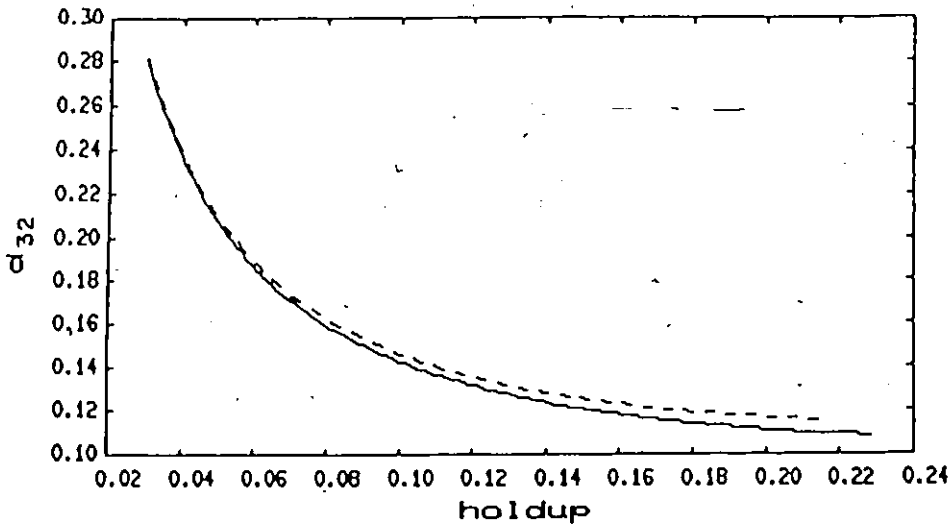

Figure 3.5 Sauter mean drop diameter, d_{32} vs. holdup. (Solid curve: U_c = 0.3 cm/s, U_d = 0.4 cm/s dotted curve: U_c = 0.4 cm/s, U_d = 0.4 cm/s.)

CHAPTER 4

4. Pertinent Control Theory

The purpose of this chapter, which is composed of three parts, is to present an overview of the pertinent control theory. In the first part the stochastic controllers relevant to this thesis are discussed. In the second part a brief summary of the deterministic Dahlin controller is given. The third part contains the necessary background on the multivariable systems and the interaction problem associated with them.

4.1 Stochastic Controllers

In this part the stochastic controllers are reviewed. This is done mostly within the context of practical applications. The mathematical formalism of the topic can be found in Astrom (1978).

4.1.1 Prolegomenon to control

The transfer function of a system is that which maps an input

variable U to an output variable Y in a transform domain. Direct digital control deals with sampled data systems, consequently it is relevant to consider the transform domain as the discrete z-domain (Astrom and Wittenmark, 1983) in this work.

A system is understood to be stable if it produces a bounded output to a bounded input. The stability region is a subset of the complex z-plane and it is the interior of the unit disc.

Hereafter it is assumed that an input/output relationship, for a given plant, is available and the discrete model is obtained either from a mechanistic modelling of the underlying physical and chemical principles of the system, or directly from plant experiments using some identification method. The final parametric model obtained can then be represented, in the complex z plane, as a rational polynomial in the following way:

$$Y_t = \frac{\omega(z^{-1})}{\delta(z^{-1})} U_{t-b} \qquad (4.1)$$

Where Y_t is the deviation, at the sampled time t, of the measured process output from its set point, and U_t is the deviation of the input from the corresponding equilibrium value. b is the total number of periods of delay necessary before the effect of the input on the output can be observed and is the sum of the integer part of the pure process delay plus one which is introduced by the zero order hold, i.e. $b=\text{integer}(\tau_d/T)+1$. z^{-1} is interpreted as the backward shift

operator such that $z^{-1} Y_t = Y_{t-1}$.

Furthermore if it is assumed that the measured output of the process Y_t is contaminated with some noise, N_t, which is additive to the output, then the confounded Box-Jenkins (Box and Jenkins, 1976) type transfer function plus disturbance model is:

$$Y_t = \frac{\omega(z^{-1})}{\delta(z^{-1})} U_{t-b} + N_t \tag{4.2}$$

N_t represents the combined effect on the output Y_t of the disturbances of stochastic nature which are uncorrelated with the input variable U_t, and is parsimoniously modelled by an Auto Regressive Integrated Moving Average (ARIMA) time series.

$$N_t = \frac{\theta(z^{-1})}{\phi(z^{-1})\ \nabla^d} a_t \tag{4.3}$$

$$Y_t = \frac{\omega(z^{-1})}{\delta(z^{-1})} U_{t-b} + \frac{\theta(z^{-1})}{\phi(z^{-1})\nabla^d} a_t \tag{4.4}$$

Where a_t is a random variable, $\nabla = 1-z^{-1}$ is the backward difference operator, and d represents the nonstationary behaviour of the noise. If the noise is drifting in a stepwise manner then d is equal to 1; if the drift is rampwise then d is equal to 2. If the noise is stationary then d is equal to 0. The optimal controller will contain an

integral action if d is 1 and a double integrator if d is 2 so as to eliminate the steady state offset.

In equations 4.2 and 4.3 $\omega(z^{-1})$, $\delta(z^{-1})$, $\theta(z^{-1})$, $\phi(z^{-1})$ are polynomials in the backward shift operator, i.e.

$$\omega(z^{-1}) = \omega_0 - \omega_1 z^{-1} - \omega_2 z^{-2} \ldots\ldots - \omega_s z^{-s}$$

$$\delta(z^{-1}) = 1.0 - \delta_1 z^{-1} - \delta_2 z^{-2} - \ldots\ldots - \delta_r z^{-r}$$

$$\theta(z^{-1}) = 1.0 - \theta_1 z^{-1} - \theta_2 z^{-2} \ldots\ldots - \theta_q z^{-q}$$

$$\phi(z^{-1}) = 1.0 - \phi_1 z^{-1} - \phi_2 z^{-2} \ldots\ldots - \phi_p z^{-p}$$

where s,r,q and p are the orders of the respective polynomials. Moreover, $\omega(z)$ and $\delta(z)$ are coprime (i.e. have no common roots); as are $\theta(z^{-1})$ and $\phi(z^{-1})$.

For open loop stability the roots of polynomials $\delta(z^{-1})$, $\theta(z^{-1})$, $\phi(z^{-1})$ are assumed to lie inside the unit disc in the z-plane while no restrictions are placed on the roots of $\omega(z^{-1})$ polynomial. This in fact provides a means to model the noninvertable (nonminumum phase) systems.

When the sampled data contains a fractional period of time delay, c, then the effect of this is to introduce a zero into the z-transformed model. This may be a non-minimum phase zero if the fractional time delay c satisfies an inequality (Macgregor et al., 1983).

The process is said to be nonlinear if coefficients of polynomials $\omega(z^{-1})$, $\delta(z^{-1})$ and/or b are dependent upon operating conditions. In this case the linear transfer function, in equation 4.1 is a locally linear approximation for the underlying process. For the nonlinear systems principle of superposition does not hold, and with two different inputs, a nonlinear system can exhibit two drastically different outputs.

4.1.2 Optimal Controllers

The so called "one-step" optimal regulator in its self tuning form is used for the control studies on the Karr column. In this section this algorithm as well as minimum variance and linear quadratic controllers (as a background to this algorithm) are reviewed briefly.

The design of an optimal controller, requires not only the transfer function and the disturbance model of the process that is to be controlled, but also a quantitative measure for judging the performance. The selection of different performace indexes will lead to different class of controllers. However they all entail the minimization of the given performance index. In what follows the disturbance is assumed to

have one root on the unit disc and therefore the expected value of ∇U_t is considered rather then U_t itself since the latter has theoretically an infinite variance.

a) Minimum Variance Controller:

If the performance index is chosen as

$$\min_{U_t} E\{Y^2_{t+b}\} \qquad (4.5)$$

where E denotes the expectation operator. The controller synthesized by means of the above criteria is called a minimum variance controller since, at steady state, the output of this controller will be the one with the smallest variance produced amongst all classes of controllers. In effect minimum variance controller cancels the effect of the b-step ahead minimum mean square forecast on the output so that the error left on the output is the forecast error. Frequently this controller calls for large variations in the manipulated variable and for this reason it is not always practical to use. Further details on the minimum variance controller can be found in Astrom (1970).

b) Linear Quadratic Controller:

An alternative to minimum variance control is to choose the performance index as:

$$\min E\ \{Y_t^2 + \xi(\nabla U_t)^2\} \tag{4.6}$$

i.e. minimize the variance of the output Y_t subject to a constraint on the magnitude of the ∇U_t. The constraining parameter ξ can be interpreted as the cost per unit control action taken. The controller obtained from this criteria is called the linear quadratic (LQ) controller since its based on a linear process model and a quadratic performance index is minimized. Solution of this constrained minimum variance control problem requires performing a spectral factorization algorithm (Wilson, 1970). The constraint factor ξ appears implicitly in the controller equation; for this reason spectral factorization has to be carried out for a range of values of ξ until both the variance of the output Y_t and the variance of the input ∇U_t are acceptable. It should be noted that, in linear quadratic controller, the effect of the present control action on all future values of the variance of the output is taken into account. For this reason this controller is also called the infinite step controller.

c) "One-step" Optimal Controller:

A simpler solution to the problem of minimizing the variance of the output Y_t subject to a constraint on the variance of ∇U_t, which also eliminates the necessity of spectral factorization, was proposed by Clarke and Hasting-James (1971). They suggested minimization of a quadratic performance index such that at every control interval the resulting controller drives the b-step ahead minimum mean squared forecast of the output to zero. This controller is short-sighted as it

does not take into account the present control action effect on the variance of the output beyond the deadtime b but only sets the instantaneous b step ahead forecast of the output to zero.

The performance index of "one-step" optimal controller is

$$\min \{Y^2_{t+b/t} + \xi/\omega_0(\nabla U_t)^2\} \tag{4.7}$$

where $Y_{t+b/t}$ is the b-step ahead forecast of the output Y_t.

To solve this problem the disturbance part is separated into two parts:

$$N_{t+b} = \psi(z^{-1})\ a_{t+b} + \frac{T(z^{-1})}{\phi(z^{-1})\nabla}\ a_t \tag{4.8}$$

where the first term on the RHS of the equation (4.8) is the forecast error (unforecastable part) and the second term is the minimum mean square forecast. After inserting the equations (4.4) and (4.8) into the equation (4.7) and differentiating it w.r.t. U_t and setting the resulting derivative to zero to obtain the minimum the following controller equation results.

$$\nabla U_t = \frac{\delta(z^{-1})\ T(z^{-1})}{\psi(z^{-1})\ \omega(z^{-1})\ \phi(z^{-1}) + \frac{\xi}{\omega_0}\ \delta(z^{-1})\ \theta(z^{-1})}\ Y_t \tag{4.9}$$

Note that the constraint factor appears explicitly in the control law. Also note that the constraint factor can be interpreted as the pole shifting parameter which gradually shifts the poles of $\omega(z^{-1})$ towards those of $\delta(z^{-1})\nabla$. For $\xi = 0$ the "one-step" optimal controller reduces to minimum variance controller. A one step optimal controller can be viewed as cancelling some fraction of the minimum mean square forecast; therefore, for a given variance of the input, the variance of the output is slightly larger then the corresponding LQ design. Nevertheless the simplicity of the algorithm makes it popular.

4.1.3 Self Tuning Regulators

4.1.3.1 General Introduction

The theory of self tuning control (STR) was originally proposed by Kalman (1958) and later expounded by Astrom and his coworkers whose work produced the seminal papers (Astrom and Wittenmark, 1973, Astrom et al., 1977). A comprehensive review of self tuning regulators, their design principles and applications are given in Astrom (1980).

As was stated earlier, in designing an optimal controller the process and the disturbance model have to be known a priori, but for self tuning regulators no such knowledge is necessary since the controller converges to the optimal controller that would have been obtained had the process and disturbance been known (the self tuning property). STRs are based on an on line combination of identification and control. Unknown controller parameters are estimated through using

a recursive parameter estimation routine. It is based on certainty equivalence i.e. uncertainties associated with parameter estimates are not taken into account. There are many types of STRs resulting from different design procedures and different estimation routines. If the process dynamics is time varying then an adaptive version of STRs may be necessary to meet the model parameter variations since an adaptive controller can retune itself to changing dynamics. If the process has operating point dependent nonlinearity then this is handled by the STR as if it is a time varying problem. STRs can be implemented as true self tuning algorithms in that they use all the process information available to determine the optimal settings for the tuning parameters. They can also be implemented as adaptive controllers, in that they only use a part of the past process information to tune the parameters; they can then re-tune as the process evolves because they "forget" very old process information. Another approach to adaptive control is gain scheduling. This method can be applied if the process parameters (dead time, time constant and process gain) can be determined as functions of an auxiliary measured variable. This relationship can be obtained experimentally (Dennis-Germuska et al., 1984). Note that this adaptive scheme is open loop in that no corrective action is taken for the error resulting from the difference between the calculated and the real process parameter. The third alternative to the adaptive control schemes, namely Model Reference Adaptive Control (MRAC) as well as gain scheduling are discussed in a survey paper by Astrom (1983). In the self-tuning, non-adaptive implementation, the parameters typically converge after an initial learning period and then remain constant. From this time onwards the non-adaptive STR can be considered to be a

fixed parameter controller since its parameters will remain at their converged values.

A control loop operating under STR is composed of two parts; (i) the controller and the process and (ii) an on line estimator and a controller design part. The information that the estimator receives about the dynamics of the process and the disturbance are the present and past values of the input (i.e. the manipulated variable) and the output (i.e. the controlled variable) deviations from their respective steady state values. The estimator uses this information to estimate the transfer function and the disturbance model and passes these estimates to the controller design block which operates on this data to carry out the controller parameter calculation such as a spectral factorization or pole placement and, in turn, feeds this information to the controller. Finally the controller calculates the present control action. This estimation form is being referred to as explicit identification because the process and the noise model are estimated explicitly. A simpler version of an explicit STR is the one whereby the controller parameters are updated directly bypassing the intermediate algorithmic computations. This STR algorithm is referred to as implicit. It must be emphasized that the rate at which the estimator adapts to varying process dynamics must be faster then the rate at which the process parameters change. Note that the parameters are estimated under closed loop conditions. This is possible since a closed loop system with STR is a nonlinear and time varying stochastic system.

Information content of a signal is directly related to its variability. For good estimation, "richness" of the frequency content of the signal is necessary. On the contrary, for good control, input/output signals have to be at steady state. The controller which is able to inject disturbances into the input signal, when the signal is poor, in order to increase the information content of the signal is referred to as a dual controller (Fel'dbaum, 1965). In the self tuning regulator the control of the process is done independently from the estimation, i.e. estimation and control are separated. This is called the separation principle; in other words the estimator is forced to be content with the information that it receives. Nevertheless the algorithm has desirable asymptotic properties.

4.1.3.2 Clarke-Gawthrop Self-Tuner

Clarke and Gawthrop (1975), by using Clarke and Hasting-James (1971) performance index, developed the implicit self tuning version of "one-step" optimal controller as a straight forward extension of the stochastic minimum variance self tuning controller proposed by Astrom and Wittenmark (1973). However as pointed out by Macgregor and Tidwell (1977) an assumption made regarding the expectation operator in Clarke and Hasting-James (1971), and Clarke and Gawthrop (1975) is incorrect. A corrected version of the detailed derivation can be found in Harris (1977). It can be shown that minimizing equation 4.7 is equivalent to minimizing:

$$\min E\{(Y_{t+b} + \xi' \nabla U_t)^2\} \tag{4.10}$$

Where $\xi' = \xi/\omega_0$ and can be a negative or a positive number depending on the sign of ω_0 such that ξ in equation 4.7 is positive.

Defining a generalized output variable of the form:

$$\phi_{t+b} = Y_{t+b} + \xi' \nabla U_t \tag{4.11}$$

it can be shown that, if the transfer function plus the disturbance model are known, the control law is:

$$\alpha(z^{-1}) \nabla U_t + \beta(z^{-1}) Y_t = 0 \tag{4.12}$$

and would minimize equation 4.10 if the controller parameters converge. The polynomials $\alpha(z^{-1})$ and $\beta(z^{-1})$ are the denominator and the numerator of the equation 4.9 respectively.

There are a variety of methods that could be used for parameter estimation (Ljung and Soderstrom, 1983). A commonly used method, for its simplicity, is the recursive least squares estimation. The unknown parameters of the control law, equation 4.12, are estimated via the recursive least squares estimation algorithm, from the regression equation:

$$\phi_{t+b} = \alpha(z^{-1}) Y_t + \beta(z^{-1}) \nabla U_t + e_{t+b} \tag{4.13}$$

Where e_{t+b} is the error term associated with the estimation, and is assumed to be uncorrelated with the regressors Y_t and VU_t. The generalized loss function minimized by the estimation algorithm is:

$$J = \sum_{i=1}^{t} \lambda^{t-i} \cdot e^2_i \ (t) \tag{4.14}$$

In this equation λ is the exponentially discounting factor (forgetting factor) whose domain is defined on the interval $0 < \lambda \leq 1.0$. This generalized form of recursive least squares estimation, for λ less then 1.0, naturally leads to adaptive control whereby the controller parameters are adapted to changes in process transfer function parameters. If λ is equal to 1.0 then the estimation algorithm is reduced to ordinary least squares and when it is less then 1.0 then the algorithm will discount the information of the distant past by putting lesser weights on these past data points. A discounting factor between 0.99 and 0.95, provided that the disturbances are persistent and that the controller is not overparametrized, works well in practical applications. The number of past observations used in estimation is called the effective window length (Clarke and Gawthrop, 1975) and is given by:

$$\frac{1.0}{1-\lambda} \tag{4.15}$$

A large value of λ will lead to slow but smooth adaptation, whereas a small value of λ will lead to quick but noisy adaptation. λ

equal to 1.0 corresponds to infinite memory length where all the present and past information are weighted equally.

The estimation routine is done in the following way:

Define the θ, X_t and X_{t-b} vectors as:

$$\theta = (\alpha_0, \alpha_1, \ldots, \alpha_{m+1}, \beta_0, \beta_1, \ldots, \beta_{l+1})' \tag{4.16}$$

$$X_t = (Y_t, Y_{t-1}, \ldots, Y_{t-m}, \nabla U_t, \nabla U_{t-1}, \ldots, \nabla U_{t-l})' \tag{4.17}$$

$$X_{t-b} = (Y_{t-b}, Y_{t-b-1}, \ldots, Y_{t-b-m}, \nabla U_{t-b}, \ldots, \nabla U_{t-b-l})' \tag{4.18}$$

where m is the order of the polynomial $\alpha(z^{-1})$, and l is the order of the polynomial $\beta(z^{-1})$ in equation 4.12. Note that the dimensions of the vectors defined in equations 4.16 through 4.18 are equal to m+l+2.

The equation 4.13 can then be rewritten in compact vector notation as

$$\phi_{t+b} = X'_t \, \theta + e_{t+b} \tag{4.19}$$

or,

$$\phi_t = X'_{t-b} \, \theta + e_t \tag{4.20}$$

Note that the controller equation is given by:

$$X'_t \; \theta = 0 \tag{4.21}$$

or,

$$\beta_0 \; \nabla U_t + \beta_1 \; \nabla U_{t-1} + \ldots + \beta_\ell \; \nabla U_{t-1} + \alpha_0 \; Y_t$$

$$+ \ldots + \alpha_m \; Y_{t-m} = 0 \tag{4.22}$$

The elements of the θ vector are updated at every control interval from:

$$\theta_t = \theta_{t-1} + K_t \; (\phi_t - X'_{t-b} \; \theta_{t-1}) \tag{4.23}$$

~~The term~~ in the parenthesis is an estimate of the one step ahead prediction error, and K_t is the gain vector for updating the parameters. The gain vector is related to the P_t matrix which is proportional to the variance covariance matrix of the parameter estimates i.e. the θ vector. The gain vector K_t and the P_t matrix are given by:

$$K_t = \frac{P_{t-1} \; X_{t-b}}{\lambda + X'_{t-b} \; P_{t-1} \; X_{t-b}} \tag{4.24}$$

$$P_t = [P_{t-1} - \frac{P_{t-1} \; X_{t-b} \; X'_{t-b} \; P_{t-1}}{\lambda + X'_{t-b} \; P_{t-1} \; X_{t-b}}] / \lambda \tag{4.25}$$

The diagonal elements of the P_t matrix are a measure of the uncertainty of the parameter estimates. A low value is an indication of the certainty and conversely a high value is an indication of uncertainty for the corresponding parameter. The second term in equation 4.25 is a measure of the reduction in the parameter uncertainty attained from the last measurement. Therefore, when the discounting factor is equal to 1, the elements of the P_t matrix decrease monotonically as the parameters converge. This is because at every interval more information is obtained, consequently confidence over the parameter estimates is increased. The effect of the discounting factor on the P_t matrix, for $\lambda < 1$, is that the elements of the P_t matrix and hence the gain of the estimator is kept larger; thereby the elements of the P_t matrix will not tend to zero and the algorithm will always be alert to track changing process dynamics.

Consider the case when the parameters of the process and the disturbance model are time invariant, and λ is 1.0. As more information is coming to the estimator the parameters converge to a constant value. This is reflected in the P_t matrix by the fact that the elements tend towards zero because of the increased confidence over the parameter estimates. This in turn causes the gain of the estimator to approach zero. At this point the estimator should be switched off to avoid numerical problems.

If the transfer function plus the noise model structural configurations can be conjectured without any knowledge as to the parameter values, then the orders of the $\alpha(z^{-1})$ and $\beta(z^{-1})$ polynomials can readily

be obtained, from equation 4.9. If, however, neither the structural configuration nor the parameters are known then the order of $\alpha(z^{-1})$ and $\beta(z^{-1})$ polynomials can be guessed and then the optimality of the controller can be checked by means of the two theorems due to Astrom and Wittenmark (1973). According to one of these theorems, if the controller structure is optimal then certain cross and auto correlations of the generelized output variable ϕ_t with the input variable U_t will be statistically insignificant.

The parameters α_i and β_i are estimated at each control interval. A close look at equation 4.12 shows that one parameter is redundant and hence can be fixed initially by zeroing out the corresponding rows and columns of the P_t matrix. Since a zero element of this matrix reflects an absolute certainty over this parameter it will not be updated. It is shown by Astrom and Wittenmark (1973) that the fixed numerical value of β_0 must the satisfy the inequality $0.5\ \omega_0 < \beta_0 < \infty$ for the closed loop stability.

It should be emphasized that STRs are complex regulators. Contrary to what the name "self tuning" might imply, a number of a priori estimates have to be provided so that a stable and optimal controller can be achieved. This requires system insight and engineering judgement to implement a self tuner in a control loop. Order of the $\alpha(z^{-1})$ and $\beta(z^{-1})$ polynomials, sampling period, number of periods of delay, discounting factor λ, and the initial estimates of the θ vector and the P_t matrix are required. A detailed discussion regarding the choice of the tuning parameters of the STR is given in Wellstead and Zanker (1982).

4.1.3.3 Some Problems Associated With the Estimation Algorithm

In the recursive least squares estimation algorithm, when a discounting factor of less then 1.0 is used, the data points beyond the effective window length are discarded. For the systems which are nearly deterministic and noise free, and the disturbances or set point changes are infrequent then a problem occurs due to a lack of information in the estimator, coined as covariance windup (Astrom 1980). A heuristic argument for covariance windup is as follows. The elements of X_t vector, in equation 4.17, are the deviations from their steady state values. After a step change in the desired output the process reaches steady state and X_t vector becomes a null vector. The second term in equation 4.25 becomes a null matrix which in turn reduces this equation to:

$$P_t = \frac{1}{\lambda} P_{t-1} \tag{4.26}$$

Since λ is less then 1.0 the P_t matrix will grow exponentially while no disturbances are coming to the process under control. This will cause the uncertainty in the parameter estimates to increase, and as a result the parameters will vary erratically. The large values of the elements of the P_t matrix may cause numerical problems as well as increasing the sensitivity of the parameters to small disturbances. This undesirable result can be avoided by using a variable discounting factor. In the literature there are two theoretical studies on the variable discounting factor reported. Fortescue et al. (1981) in their work developed an estimation algorithm which preserves the hypervolume

of the confidence region of the parameter estimates. This forces the estimator to keep the old data unless new information arrives. The tuning parameter for the recursive estimation algorithm with variable discounting factor is this hypervolume of the confidence region. They report a successful application of this algorithm on a computer controlled CO_2 absoption-desorption pilot plant. Dumont (1982) also successfully applied this algorithm to self tuning control of a chip refiner motor load. However difficulties associated with finding an appropriate value for the tuning parameter are reported in this work. Hagglund (1983) proposed an algorithm whereby the past data is discounted so as to retain a constant desired amount of information in the estimator. The proposed algorithm yielded successful simulation results though it was not applied to any process experimentally. There are also ad hoc methods proposed by various workers. These include, for example, keeping the trace, i.e., the sum of all diagonal elements of a square matrix, bounded. However fixing the trace whenever it reaches an upper bound can not be related to any physical entity in the process.

In this work the covariance windup problem is avoided via an empirical variable discounting factor. The quantity $(Y-Y_{set})/Y_{set}$ is a measure of how far the measured variable is from the set point. A large value of this quantity indicates that a long way has yet to be gone to reach the desired value, therefore small weighting can be placed on the past data since the process parameters will change while traversing this nonlinear region. From this heuristic argument the following time varying discounting factor based on the physical conditions in the column can be suggested.

$$\lambda = 1.0 - \eta \text{ abs } [(Y-Y_{set})/Y_{set}] \tag{4.27}$$

$$\text{abs } (Y-Y_{set}) < B\ \sigma \tag{4.28}$$

Where η is a constant, σ is the standard deviation of the measurement noise and B is a constant which determines, in relation to measurement noise, the size of the window in which λ is set to 1.0. Also a lower bound to λ is needed and is taken as 0.95. This algorithm will stop the adaptive characteristic of the STR whenever the measured value is within some specified window, as determined by equation 4.28 and will start the adaptation when vice versa. For easy reference Figure 4.1 is used to illustrate the manner in which the discounting factor varies when the absolute value of $Y-Y_{set}$ is greater then the prescibed value. The experimental application of this stopper starter criteria on discounting the past data is presented in the following chapters.

The shortcoming of this algorithm is that if the set point changes or the disturbance is a ramp with a very small slope, or steps of small magnitude in the same direction stopper starter will then not be activated i.e. the estimation algorithm will be non adaptive since the discounting factor will stay at the value 1.

4.2 Dahlin Controller

In this part a model based, deterministic, pole placement

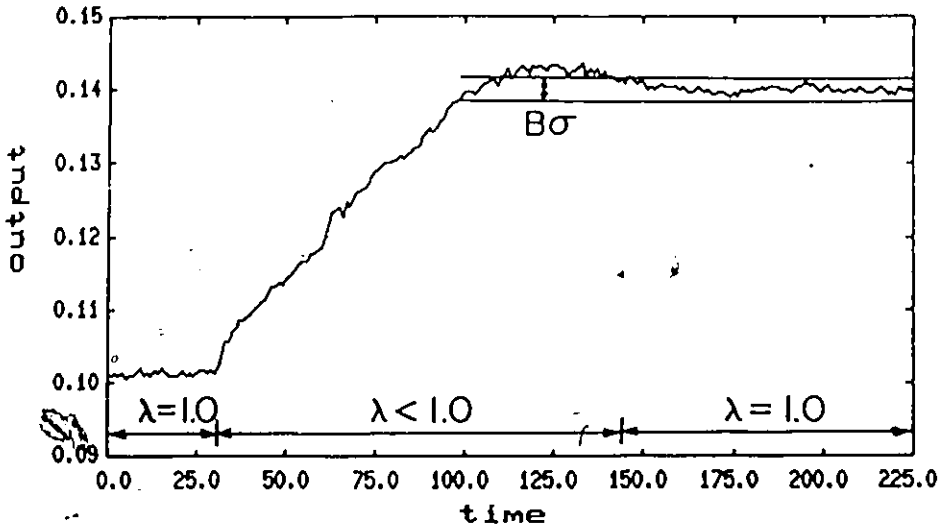

Figure 4.1 The manner in which the empirical discounting factor varies.

control algorithm namely Dahlin controller is reviewed. The basic idea behind this controller is that the closed loop transfer function is prescribed and then the controller is designed which yields this specified response.

The simplest version of the pole placement controllers is the so called deadbeat controller. This controller places the closed loop transfer function pole to the origin in the complex z plane. However this controller calls for large changes in the manipulated variable. Dahlin (1968) proposed a smoother closed loop response by specifying its closed loop transfer function as a first order plus dead time in the continuous domain.

If $K(z^{-1})$ denotes the closed loop transfer function from process set point to process output in the complex z-domain then.

$$K(z^{-1}) = \frac{1.0 - \exp(-T/\tau_c)}{1.0 - \exp(-T/\tau_c)z^{-1}} z^{-n-1} \qquad (4.29)$$

where τ_c is the specified closed loop time constant, T is the control interval and n is the dead time of the desired closed loop response. Note that dead time in the desired response can not be smaller than the process dead time for physical realizeability of the controller. Basic block diagram algebra will show that the necessary controller transfer function $D(z^{-1})$ to achieve the desired closed loop transfer function is:

$$D(z) = \frac{K(z^{-1})}{1.0-K(z^{-1})} \frac{1.0}{GH(z^{-1})} \tag{4.30}$$

Where $GH(z^{-1})$ is the process transfer function with a zero order hold. After substituting equation 4.29 into 4.30, and defining $p = \exp(-T/\tau_c)$ it can readily be shown that

$$D(z^{-1}) = \frac{(1-p)\ z^{-n-1}}{(1-z^{-1})\ [1 + (1-p)\ z^{-1} + (1-p)\ z^{-2} + \ldots + (1-p)\ z^{-n}]} \frac{1}{GH(z^{-1})} \tag{4.31}$$

The closed loop time constant τ_c is the design parameter and for a given control interval determines the location of the closed loop pole and hence the closed loop bandwidth. A small value of this parameter renders a fast response and becomes sluggish as this value is increased.

The Dahlin controller intrinsically contains dead time compensation and integral action. The dead time compensator adds phase lead into the system whose net effect is to cancel the phase lag produced by the dead time of the process. The integral action enters the controller via the term:

$$\frac{K(z^{-1})}{1-K(z^{-1})} \tag{4.32}$$

The fact that the poles and zeros of the process are cancelled by the regulator, requires that the model of the process exactly matches

the process. A problem arises if the poles and zeros of the process are located near the instability region. However the problems associated with the model accuracy can be obviated by increasing the closed loop time constant i.e. by decreasing the bandwidth of the closed loop at the expense of rendering the response sluggish.

4.3 Multivariable Systems and Interaction Measure

Hitherto single input/single output (SISO) control systems, whereby one input variable is manipulated to control one output variable, were discussed. A multivariable system is a multi-input multi-output (MIMO) system in which more then one variable is manipulated to control one or more output variables in one control system. In such a system, any set point change or disturbance entering the system may activate some or all controllers if it causes changes in some or all outputs. If there are m inputs and l outputs then denoting the input $\underline{U}$ as an m-dimensional vector valued function of the complex variable s and the output $\underline{Y}$ as an l-dimensional vector function of the complex variable s (i.e. the elements of the $\underline{U}$ and $\underline{Y}$ vectors are the Laplace transforms of the input and the output vectors in the time domain), the input/output relation of the dynamical system can be represented as:

$$\underline{Y} = \underline{\underline{G}}\,\underline{U} \tag{4.33}$$

Where $\underline{\underline{G}}$ is a m*l matrix valued function of the complex variable s and is referred to as the transfer function matrix whose elements g_{ij} are the individual transfer functions relating the ith control variable

Y_i to the jth manipulated variable U_j.

A matrix of concern is the steady state gain matrix $\underline{\underline{G}}(0)$, the elements of which represent the steady state channel from the input U_j to the output Y_i.

There exits an interaction between the variables of the process if the input U_j significantly affects the output Y_i for $j \neq i$. If the controller design procedure, if any, does not take the interaction into account, then it might be impossible to control the process since individual controllers will "fight" each other.

Bristol relative gain array method (RGA) proposed by Bristol (1966) shows the degree of steady state interaction in a multivariable process between different loops. It also serves as a guide to provide the "best" pairing of the controlled and the manipulated variables. In this method a matrix $\underline{\underline{\Lambda}}$ is defined whose elements l_{ij} are the ratio of two gains. The numerator of l_{ij} is obtained when all loops are open, i.e. the corresponding element g_{ij} of the steady state gain matrix $\underline{\underline{G}}(0)$. The denominator is obtained when all other loops except U_j and Y_i are open loops. The $\underline{\underline{\Lambda}}$ matrix can be obtained quantitatively, provided that $\underline{\underline{G}}(0)$ is available, without any need of experiments from the following formula:

$$\underline{\underline{\Lambda}} = [\underline{\underline{G}}\ (0)\ (\underline{\underline{G}}^{-1}\ (0))'] \tag{4.34}$$

Then the pairings of input and output are established by

inspecting each row of the Λ matrix and choosing U_j for controlling Y_i such that l_{ij} , in that row, is positive and the largest. A numerical value of 1.0 on the diagonal elements of Λ matrix is an indication of an non-interacting system. Note that l_{ij} is invariant under input output scaling.

CHAPTER 5

5. Control Studies on Holdup Alone

The control problem investigated in this chapter is the servo control of the hydrodynamic holdup of the dispersed phase by manipulating the frequency of reciprocation. The functional relation between holdup and the frequency of reciprocation has a multifactorial nonlinearity and it has been shown by Kusuma (1981) and Taylor et al., (1982) that adaptive controllers are necessary when controlling the holdup by manipulating the frequency of reciprocation. This work is an extension of Kusuma's work (1981) on the control of the dispersed phase holdup. The control was extended to different operating regions, and an empirical discounting factor described in chapter 4 was implemented to circumvent the burst phenomenon of the P_t matrix.

5.1 Modelling the System

As was stipulated in chapter 2, The response of the dispersed phase holdup to a step change in frequency of reciprocation has a non-

minimum phase response. Following Kusuma's (1981) work the nonminumum phase response was modelled as a pure time delay. A first order plus dead time transfer function model was fit to the response by the reaction curve method (Smith, 1972). The transfer function obtained, in the Laplace domain, from the input frequency of reciprocation to the output holdup has the form:

$$G(s) = \frac{K_p \exp(-\tau_d s)}{\tau s+1} \tag{5.1}$$

or in discrete z^{-1} domain

$$GH(z^{-1}) = \frac{(\omega_0-\omega_1 z^{-1})z^{-b}}{1.0 - \delta z^{-1}} \tag{5.2}$$

If the process is assumed to be subject to randomly occurring deterministic step disturbances, then the Box-Jenkins type transfer function plus disturbance model is as follows.

$$Y_t = \frac{\omega_0-\omega_1 z^{-1}}{1-\delta z^{-1}} U_{t-b} + \frac{1}{1-z^{-1}} a_t \tag{5.3}$$

Where a_t represents random shocks whose magnitude is zero most of the time and non-zero occasionally. The standard deviation of a_t, for nonzero values, is a measure of the magnitude of up or down step

changes. The duality between purely stochastic disturbances and randomly occurring deterministic disturbances is shown by Macgregor et al. (1984).

The corresponding Clarke-Gawthrop self tuner (cf. equation (4.9) in chapter 4) can readily found to be:

$$\nabla U_t = \frac{1.0-\delta\ z^{-1}}{(\omega_0-\omega_1 z^{-1}) + \frac{\xi}{\omega_0}(1-\delta\ z^{-1})} Y_t \tag{5.4}$$

$$\nabla U_t = \frac{\alpha(z^{-1})}{\beta(z^{-1})} Y_t \tag{5.5}$$

Where $\alpha(z^{-1}) = \alpha_0 + \alpha_1\ z^{-1}$ and $\beta(z^{-1}) = \beta_0 + \beta_1\ z^{-1}$ i.e. the controller has 2 α and 2 β parameters which are functions of the constraint factor, and $\omega(z^{-1})$and $\delta(z^{-1})$ i.e. the pole and the zero of the transfer function.

A mechanistic mathematical model, based on the fundamental physical laws, to obtain a dynamic relationship between the holdup and the frequency of reciprocation, with the flow rates of the two phases as parameters, if at all possible, is very complex. Therefore such a model was obtained empirically by carrying out step tests at different operating regions to gain insight into the process. Table 5.1 summarizes the

results of the step tests. Relevant representive step test response is given in Chapter 2. Note that this step test data was obtained under mass transfer conditions.

F (Hz)	U_c (Cm/s)	U_d (Cm/s)	Gain $(Hz)^{-1}$	Dead time (s)	Time constant (s)
1.3→2.3	0.320	0.301	0.021	18	56
1.3→2.0	0.509	0.301	0.024	16	50
2.0→1.3	0.509	0.301	0.024	22	28
1.1→1.8	0.310	0.560	0.029	20	36
1.1→2.0	0.509	0.560	0.040	16	64
2.0→1.1	0.509	0.560	0.038	29	36
1.3→2.3	0.490	0.405	0.035	13	74
2.3→1.0	0.490	0.405	0.035	32	48

Table of step tests for the effect of frequency of reciprocation on the dispersed phase holdup

TABLE 5.1

The gain, time constant, the dead time are dependent upon the operating conditions. This dependency has no correlation which precludes using a simple adaptive controller namely gain scheduling (see Chapter 4 in section 4.1.3.1). Consequently a sophisticated adaptive controller, self tuning regulator, was implemented to control the holdup.

Selecting the control interval is a compromise between the quality and the speed of the control. A small control interval makes it possible to observe high frequency disturbances and the optimal controller tries to eliminate them, thereby complicating its function. On the other hand, a very large control interval may bring very little information on the process and the control quality decreases. In this work the control interval was chosen to be 20 seconds on the basis of the need to protect the control algorithm from the changing dead times and the constraint factor ξ, for the no mass transfer case, to be 1.0 after trial and error. For zero constraint factor the control was unstable with the manipulated variable banging in between upper and lower bounds.

In all experiments the P_t matrix was initialized by setting it to P_t=10000*I where I is the unit matrix. The controller parameters were set to some nonzero value (obtained from a previous experiments) and β_0 was fixed to be 1.0. In all experiments the column was first brought to steady state and then the control program was started. The maximum holdup set point was fixed at 70% of the flooding value, as at 80% unstable operation was produced. The reason for this is that the slope of the holdup vs frequency of reciprocation curve increases very rapidly as it approaches flooding conditions, thereby the rate at which gain changes is very high. As was stated in chapter 4, this violates the condition that the process parameters should be changing slowly relative to adaptation rate of the controller parameters.

5.2 Results and discussions

Experiments were carried out under no mass transfer conditions and under mass transfer conditions. Table 5.2 summarizes the operating conditions in these experiments.

Figure No	U_c (cm/s)	U_d (cm/s)	Mass Transfer Occurring	Discounting Factor
5.1-5.5	0.49	0.32	No	0.95
5.6-5.9	0.49	0.32	No	Variable
5.10-5.11	0.49	0.32	Yes	Variable

Table 5.2

Operating conditions for the holdup control experiments.

5.2.1 Servo Control of the holdup Under No mass Transfer Conditions

Herein 2 sets of experimental results of the servo control of the dispersed phase holdup is presented namely; i) A fixed discounting factor ii) A variable discounting factor. Disturbance rejection experiments that were carried out on the holdup only control are not included in this chapter as these experiments are presented in the proceeding chapters.

i) Set point changes with constant discounting factor:

The operating conditions were as follows: continuous phase flow rate was 0.49 cm/s, the kerosene feed flow rate was 0.32 cm/s, frequency of reciprocation was 1.9 Hz. Discounting factor was set to 0.95. The corresponding flooding holdup value as calculated from the Baird-Shen relation was 0.219. Figure 5.1 shows the controlled variable holdup. The dotted lines in the figure are the set points. At the beginning frequent set point changes were made. Then no step change was made for a long period of time. During this time, as shown in Figure 5.2, the trace of the P_t matrix grows exponentialy and, in turn, renders the gain of the estimator very large; even very small information, i.e. a small deviation from the set point, will have a large affect on the parameters. This information also drops the elements of the P_t matrix. During this period, when no disturbance was entering the system, the controller parameters are varying erratically as can be seen in Figures 5.3-5.5. Note that the controller parameters α_0 and α_1 are varying about zero since for no disturbance the optimal control law is that of no control. An interesting point to note is that the control law equation 5.5 will have an unstable pole if β_1 has an absolute value which is greater then 1.0. This happens occasionally as can be seen from Figure 5.5 but yet no instability of the controlled variable was encountered. Later another set point change was made and the controller successfully brought the holdup to the desired set point value.

Even though the stability of the controller was preserved while the controller parameters were varying erratically it is not wise to implement this controller as it is. It is shown (Fortescue et al.,

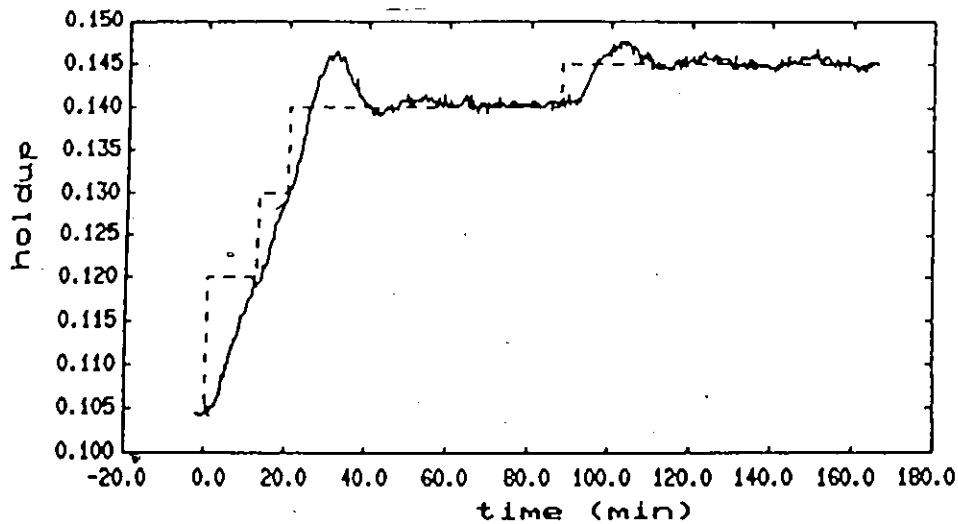

Figure 5.1 Response of the controlled variable holdup when a constant discounting factor (0.95) was used.

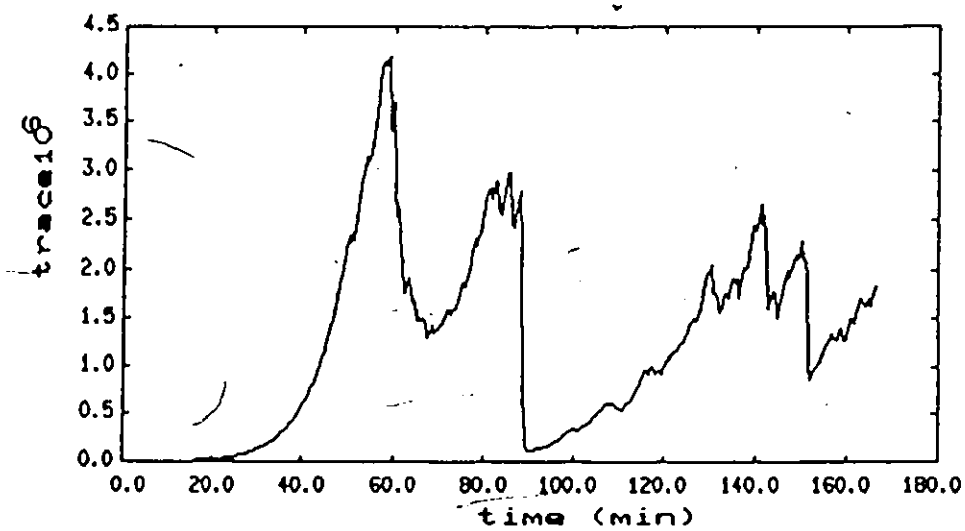

Figure 5.2 The trace of the P_t matrix when a constant discounting factor (0.95) was used.

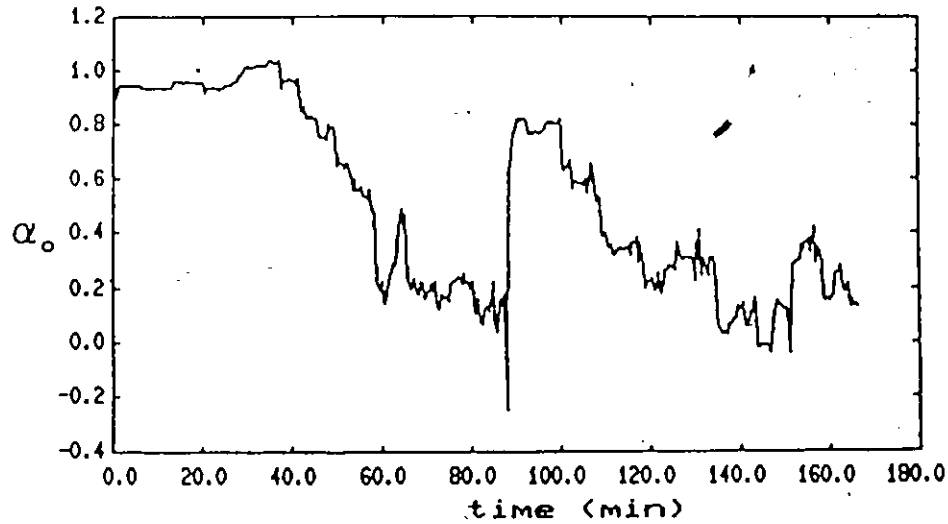

Figure 5.3 Controller parameter α_0 when a constant discounting factor (0.95) was used.

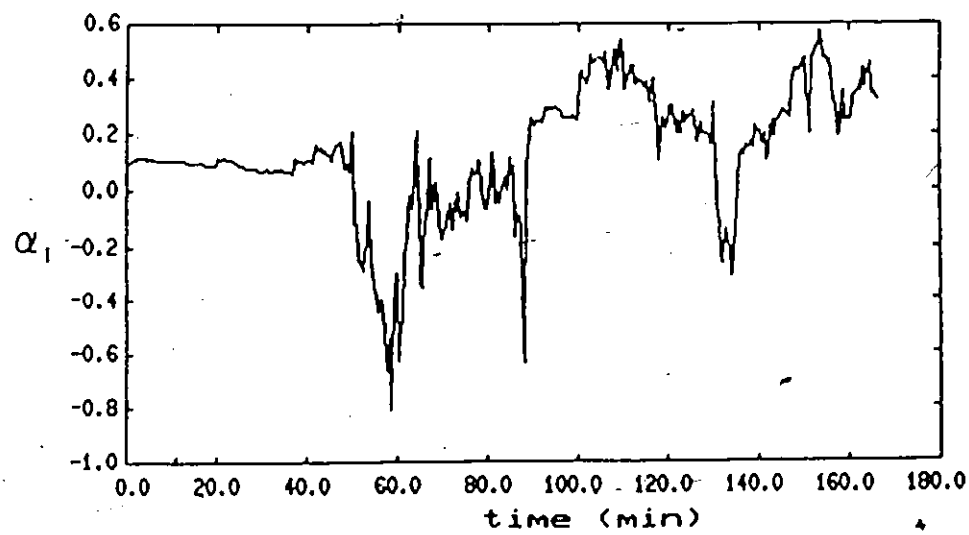

Figure 5.4 Controller parameter α_1 when a constant discounting actor (0.95) was used.

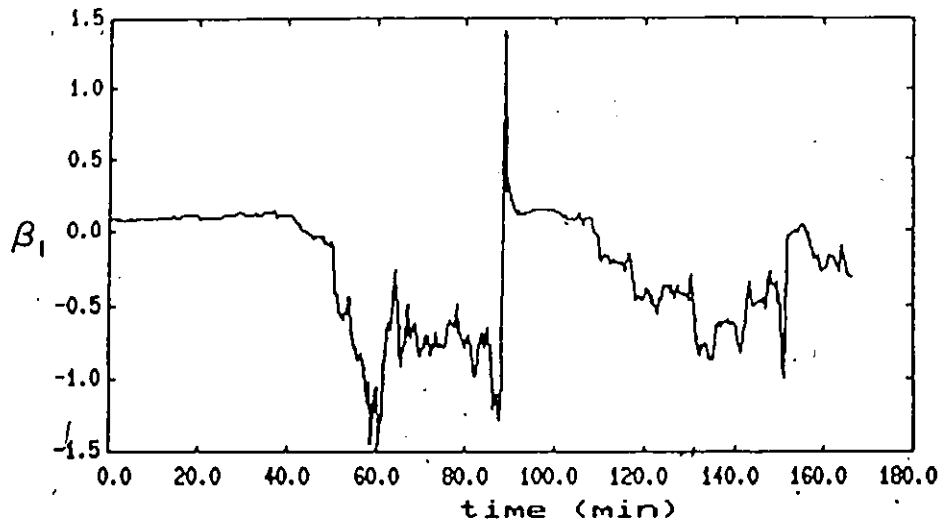

Figure 5.5 Controller parameter β_1 when a constant discounting factor (0.95) was used.

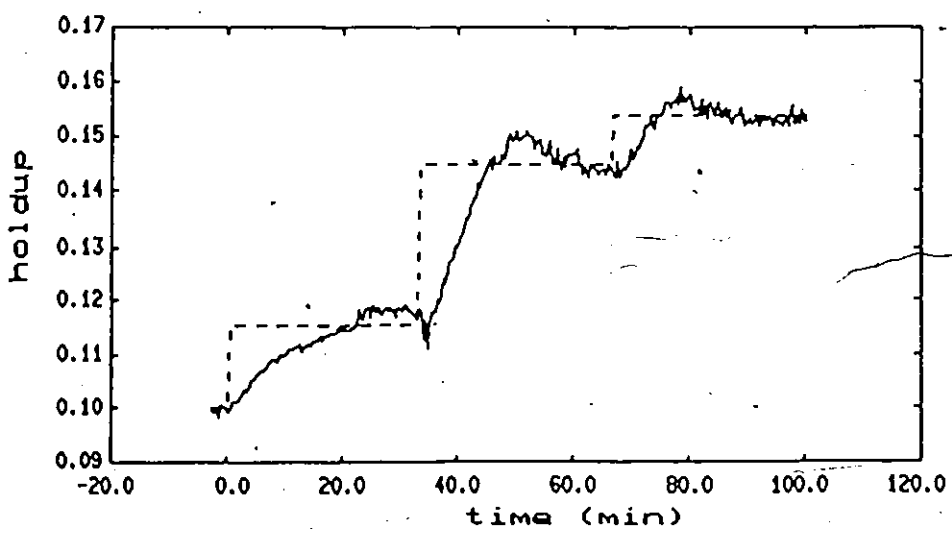

Figure 5.6 Response of the controlled variable holdup when a variable discounting factor was used.

(1981)) that instability will perhaps set in if the unit is left like this for longer periods of time.

ii) Set Point Changes With Variable Discounting Factor:

In this section the results of similar experiments that were carried out with variable discounting factor are presented. The set point changes were made relatively infrequently (once in every 100 control intervals). This was done so as to observe the behaviour of the trace of the P_t matrix and hence the controller parameters. The response of the holdup is given in Figure 5.6. At higher values of the holdup, due to an increase in the steady state gain of the process (cf. holdup vs frequency of reciprocation curve), the response becomesu ome-what underdamped. Note that the control of the holdup at 70% flooding value (=0.154) can be seen to be stable. In Figure 5.7 the trace of the P_t matrix is given. If the trace of the P_t matrix reported in (i) above is compared to that reported here, it can be seen that in the former is 1000 times higher for the same initial values. As can be seen in Figure 5.7, the trace levels off and starts to decrease monotonically each time the measured value holdup is within the specified window in which the discounting factor is set to 1.0. Discounting factor is given in Figure 5.8. Whenever the measured value is outside the specified window the discounting factor becomes less then 1.0 in proportion to the percent deviation from its set point thereby forcing the P_t matrix to be alert to parameter variations by making its elements grow in magnitude. The controller parameters as presented in Figure 5.9 illustrates their smooth behaviour under variable discounting factor.

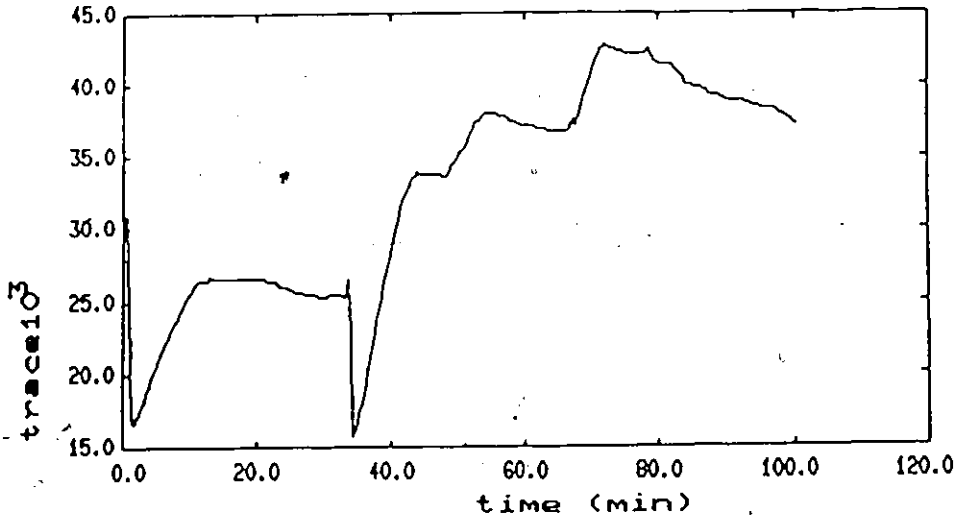

Figure 5.7 Trace of the P_t matrix when a variable discounting factor was used.

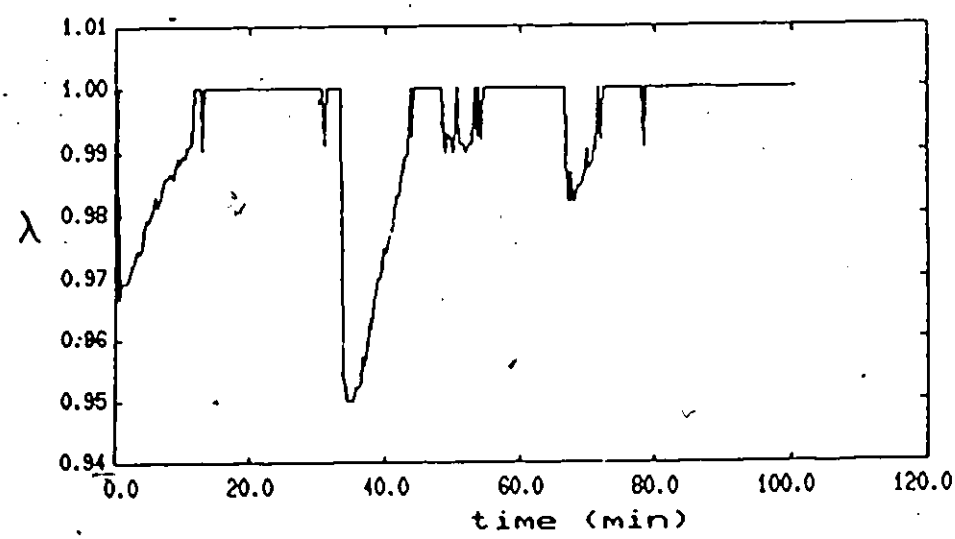

Figure 5.8 Response of the variable discounting factor.

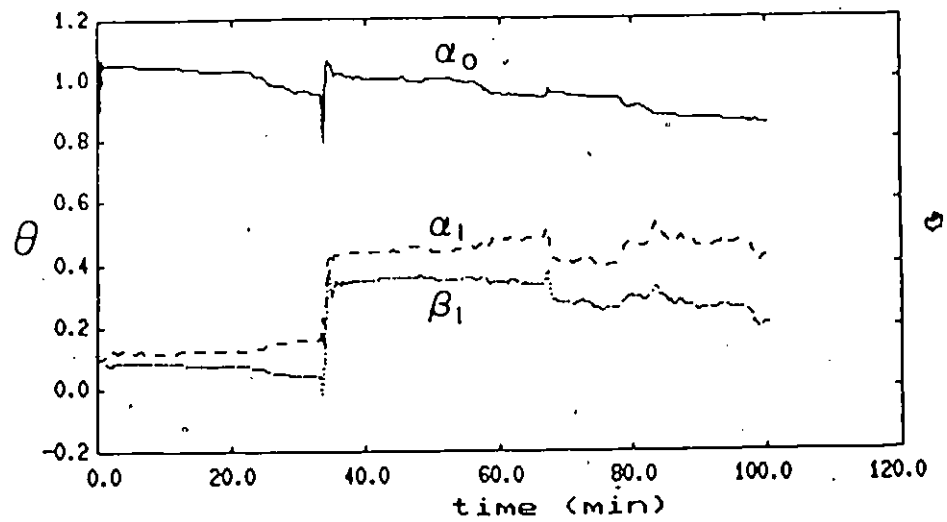

Figure 5.9 Controller parameters when a variable discounting factor was used.

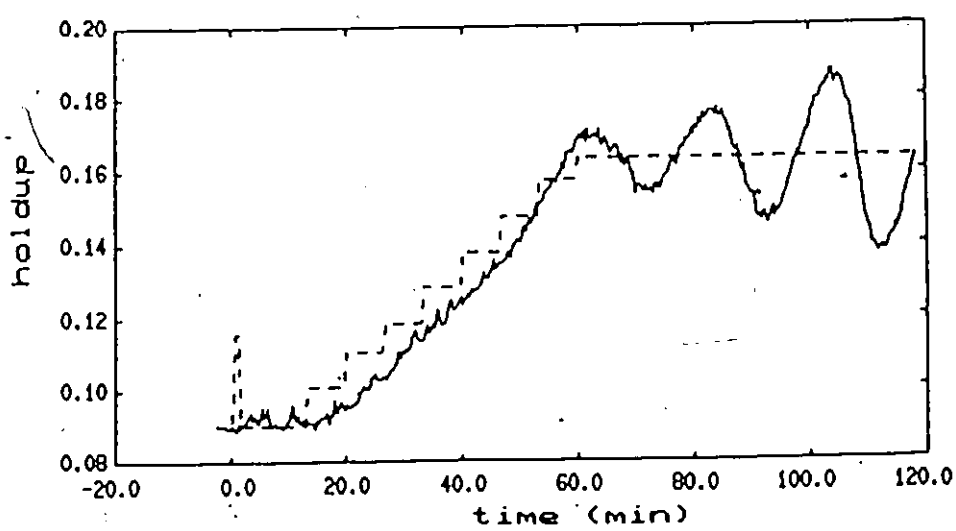

Figure 5.10 Servo response of the controlled variable holdup with mass transfer taking place.

5.2.2 Servo Control of the Holdup Under Mass Transfer Conditions

As is described in the following chapter the extract outlet concentration was controlled in a cascade arrangement with the inner loop being the holdup. This means that the holdup set point is coming from the outer concentration loop. Figure 5.10 shows what happens to the controlled variable under mass transfer conditions. The set point of the holdup was varied every 20 control intervals (i.e. 400 seconds). The upper bound of the holdup set point was fixed at 70% flooding value. The constraint factor that was found experimentally to be reasonable for the holdup under no mass transfer conditions can be seen to be too high when there is mass transfer; thereby yielding sluggish control. For this reason the constraint factor was further reduced to 0.5 by trial and error and this value was used during the proceeding experiments. Note that control also becomes unstable at the 70% flooding value of the holdup which was stable under no mass conditions. A plausible explanation is as follows. In the case of mass transfer the shape of the holdup versus the frequency of reciprocation curve changes because of mass transfer induced coalescence. At low frequencies the curve becomes flattened and elongated in other words the gain is smaller then that of no mass transfer case. Conversely at high frequencies of reciprocation with mass transfer the holdup/frequency curve has a sharper slope i.e. higher gain. This corresponds to a large change in steady state gain. Under mass transfer conditions also the rate at which the equilibrium drop size is approached is lessened. A heuristic reasoning for this phenomena is as follows:

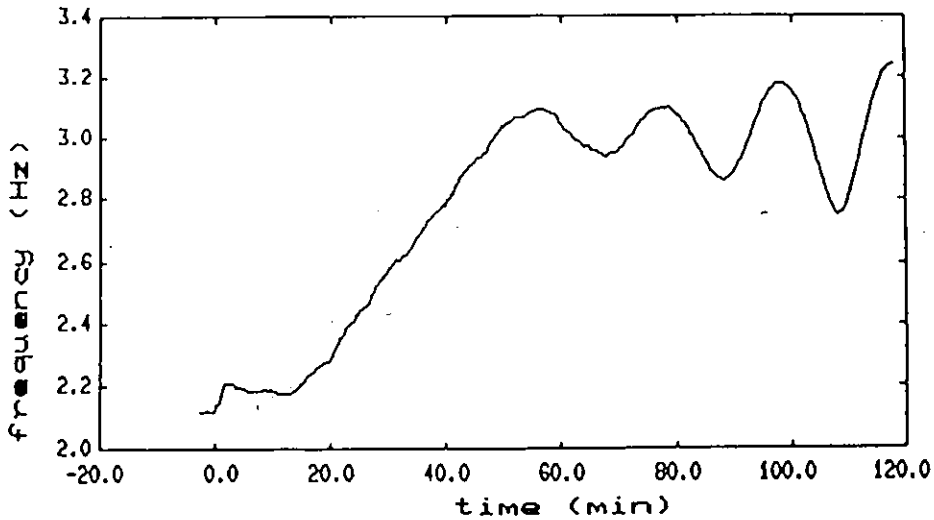

Figure 5.11 Servo response of the manipulated variable frequency of reciprocation with mass transfer taking place.

drop sizes in a liquid-liquid dispersion evolve as a consequence of droplet breakage and coalescence and have been extensively studied by Tavlarides (1981) and others. Following a step change in the energy input (i.e. an increase in the frequency of reciprocation) the droplets will break up creating daughter droplets of smaller size as a result of the increased kinetic energy of the turbulent eddies of the continuous medium. Finally an equilibrium between the breakage and the coalescence rates will be reached. When mass transfer is taking place, the rate at which this equilibrium condition is reached is slowed down mainly due to (i) enhanced coalescence frequency (or equivalently the coalescence probability when two droplets approach each other), and, in part, (ii) the high number density of droplets at high holdup values. This reduction in the rate corresponds to an increase in the time constant of the process. Consequently the gain, the time constant and perhaps the dead time are changing very rapidly as the column is approaching the flooding condition. As a result the condition that the process parameters should be changing slowly relative to the adaptation rate of the controller parameters is again violated. Hence even though the controller has stable poles the control is unstable caused by a large phase lag i.e. the Bode stability criteria is violated. Phase lag can be seen by comparing the response of the input frequency of reciprocation, (Figure 5.11), with the holdup response (Figure 5.10).

The phase lag was firstly overcome by increasing the control interval of the inner loop to effectively detune the loop. However this makes the closed loop response very sluggish so the maximum allowed operating holdup was set to 65% and finally 60% of the flooding value

calculated via the Baird-Shen relation, instead of 70%.

5.3 Conclusions

In this chapter experimental application of the Clarke Gawthrop self tuner is reported. An ad hoc discounting factor, based on the physical conditions in the column, is implemented to circumvent the covariance windup problem and is found to work satisfactorily. The difference in the hydrodynamical conditions between with and without mass transfer brought about by mass transfer induced coalescence was also found to have a significant influence on the response characteristics of the adaptive controller.

CHAPTER 6

6. Control of Extract Concentration by Manipulating the Frequency of Reciprocation; Cascaded Control

Previous work on the direct digital control of the Karr column examined only the control of the hydrodynamic holdup. The main objective of this work was the regulatory and servo control of the extract outlet concentration. While the primary control objective is the control of the concentration, the holdup must also be controlled in order to prevent the column from flooding. There are, then, two variables, holdup and concentration, which are to be controlled. From the control point of view this problem lends itself to a multivariable control scheme with the possible manipulated variables being the frequency of reciprocation, and the continuous phase flow rate. If the frequency of reciprocation is chosen to be the only manipulated variable then the control scheme needs to be a cascade arrangement with the hydrodynamic holdup controlled by the plate frequency in order to prevent flooding, and the holdup setpoint controlled by an outer loop concentration controller.

Transfer functions relating the frequency of reciprocation to outlet concentration of the continuous phase were obtained by performing step tests at different operating points and fitting a first order plus dead time model; the parameter values are given in Table 6.1 (relevant representative step test response is given in Chapter 2). The process as seen by the outer loop controller is the closed loop transfer function of the inner loop (i.e. holdup setpoint to inferred hydrodynamic holdup). However as a first approximation, the following conclusions on the effects of the frequency of reciprocation on continuous phase exit concentration can be drawn. It is clearly seen that the process parameters vary considerably within the operating region and as was stated in the Chapter 2, the time constant and the dead time of this process vary inversely with the continuous phase flow rate.

F (Hz)	U_c (Cm/s)	U_d (Cm/s)	Gain mol/L Hz	Dead time (s)	Computed dead time(s)	Time Constant(s)
1.3→2.3	0.320	0.301	0.069	172	187	852
1.3→2.0	0.509	0.301	0.047	128	117	448
2.0→1.3	0.509	0.301	0.049	108	117	458
1.1→1.8	0.310	0.560	0.147	164	193	784
1.1→2.0	0.509	0.560	0.086	112	117	496
2.0→1.1	0.509	0.560	0.087	112	117	462
1.3→2.3	0.490	0.405	0.060	124	122	404
2.3→1.0	0.490	0.405	0.064	126	122	418

Step tests input: frequency of reciprocation output: extract concentration.

TABLE 6.1

6.1 The Control Scheme

One of the requirements of cascade control is that the inner loop should be faster then the outer loop. In this application the inner loop had to be detuned by increasing the constraint factor, ξ, so that it would not oscillate and thereby drive the process, even for short periods of time, into the flooding region. The inner loop control interval was chosen to be 20 seconds and the outer loop control interval, by trial and error, 400 seconds (i.e. 20 inner loop control intervals). This control interval for the outer loop allowed the inner loop to settle between changes of the holdup setpoint.

An experimental difficulty concerning the inventory of the kerosene was encountered. With 650 L of kerosene it was only possible to run the experiments for 20 control intervals of the outer loop. However this time duration was sufficient to draw conclusions as to the efficiency of the control algorithm.

The following were the characteristics of the STRs used in the cascade control scheme:

a) Both regulators were implicit Clarke-Gawthrop self tuners.

b) Both STR's contained integral action to eliminate the offset from constant disturbances and the control law was:

$$\alpha_0 Y_t + \alpha_1 Y_{t-1} + \beta_0 \nabla U_t + \beta_1 \nabla U_{t-1} = 0 \qquad (6.1)$$

The inner loop STR set point (i.e. holdup set point) was clamped to prevent flooding. The flooding holdup value was calculated on line via the Baird-Shen (1984) semi-empirical model. The initial parameter estimates for the STRs were the values found from previous experiments and the initial covariance matrix for both loops was taken to be 10000*I where I is the unit matrix. The β_0 parameter was fixed to be 1.0.

6.2 Experimental Procedure

The column was initially brought to steady state, and then the control algorithm started in the following manner. The outer loop was initially bypassed and up and down step changes in the holdup set point were made so that the inner loop regulator parameters would converge. Also, a bumpless transfer was provided by forcing the measured value of the control variable to be the set point. After 20 inner control intervals the outer loop was started and a bumpless transfer also provided. It was during the second iteration of the outer control loop that load disturbances or set point changes were made. The dispersed phase flow rate was used as a disturbance and the continuous phase flow rate was constant in the experiments reported.

6.3 Results and Discussions

In these experiments an STR-STR cascade arrangement was compared with a cascade PI-STR arrangement. In Table 6.2 a summary of operating conditions of these experiments is given.

Figure No	U_c (cm/s)	U_d (cm/s)	Type	Controller
6.1-6.8	0.40	0.32→0.40	Regulatory	STR-STR
6.9-6.10	0.40	0.32	Servo	STR-STR
6.11-6.12	0.40	0.32→0.40	Regulatory	PI-STR

Operating conditions for the cascade control experiments.

TABLE 6.2

6.3.1 STR-STR Cascade Arrangement

Two types of tests were applied to the cascaded control scheme namely: a disturbance in kerosene feed flow rate, and a concentration set point change for the outlet continuous phase.

i) Disturbance Rejection Characteristics of the Cascaded Regulators: The objective of this experiment was to test the performance of the cascaded self tuning regulator to a step change in the kerosene flow rate. After the column had reached the steady state and the inner loop regulator parameters had settled, the kerosene flow rate was increased manually from 0.32 cm/s to 0.40 cm/s. Figure 6.1 shows the regulation of this step disturbance in kerosene flow rate; the controller is able to bring the concentration back to the set point. In Figure 6.2 the corresponding inner loop (i.e. the holdup) response is presented. The dotted lines correspond to the holdup set point which is the output

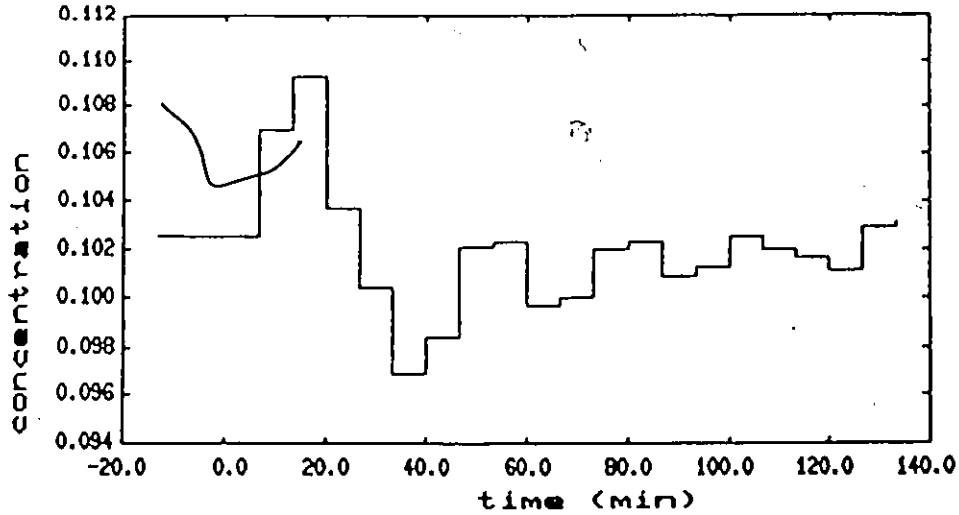

Figure 6.1 Regulatory response of the controlled variable extract concentration (outer loop) to a step disturbance in kerosene feed flow rate

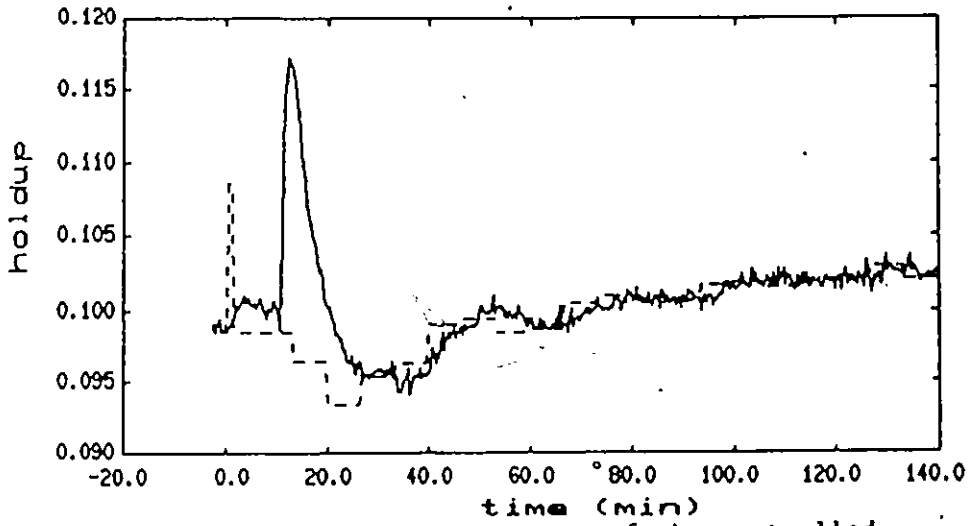

Figure 6.2 Regulatory response of the controlled variable holdup (inner loop) to a step disturbance in kerosene feed flow rate.

of the outer loop controller. In Figure 6.3 the manipulated variable (i.e. the frequency of the reciprocation) is shown. Figure 6.4 shows the trace (i.e. sum of the diagonal elements of the covariance matrix) of the inner loop controller. Up and down step changes in holdup were made so that the inner loop regulator would converge. After the initial holdup set point changes the trace falls considerably because of the information the estimator has obtained through these step changes. This corresponds to the initial learning period where the outer loop is in over-ride. Figure 6.5 shows the inner loop regulator parameters which adapt during the learning period and then adapt again when the kerosene flow rate disturbance moves the process to a new operating condition. Figures 6.6, 6.7 and 6.8 show the concentration as seen by the inner loop, the trace of the outer loop and the outer loop regulator parameters respectively during the learning period and disturbance.

ii) Step Change in the Concentration Set Point:

The performance of the controller was also tested for servo response to a step change in the continuous phase outlet concentration set point. The magnitude of the step was 0.012 moles/L. Figure 6.9 shows that the controller brings the concentration to the desired value in approximately 10 control intervals. The overshoot in the concentration response results from the inventory of extracted solute present in the column. As the frequency increases, the interfacial area increases, and hence the rate of solute transfer increases. In the transient state, this produces a high level of extraction at the bottom of the column where fresh kerosene enters. The extracted acetic acid is

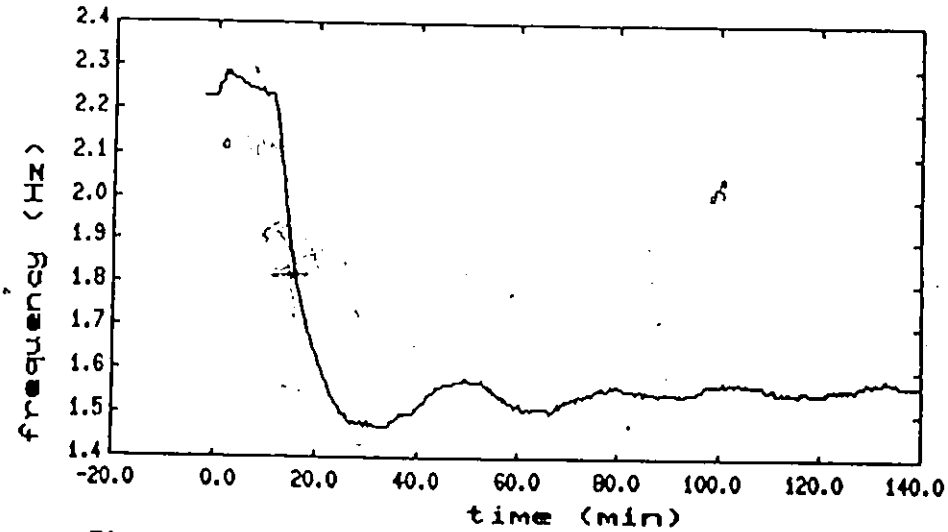

Figure 6.3 Regulatory response of the manipulated variable frequency of reciprocation to a step disturbance in kerosene feed flow rate.

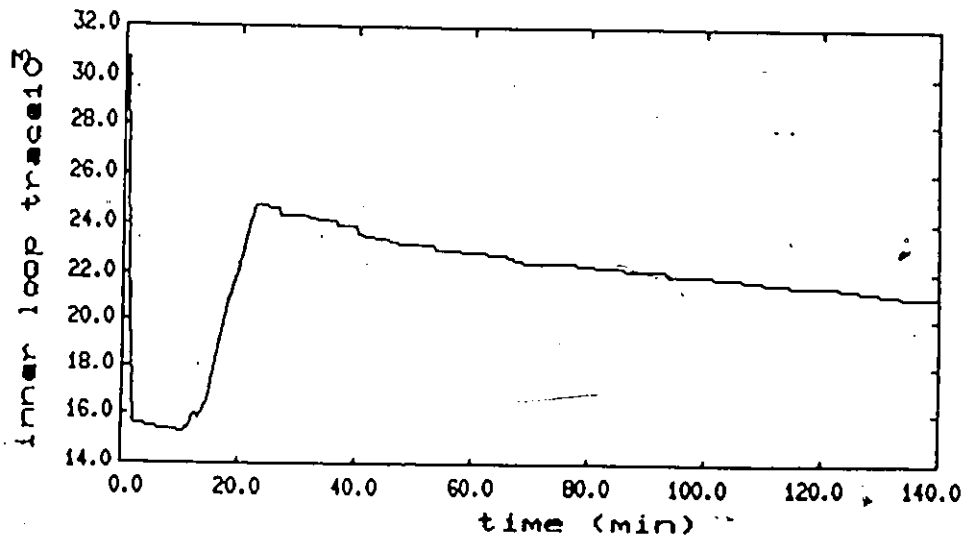

Figure 6.4 Trace of the inner loop variance-covariance matrix.

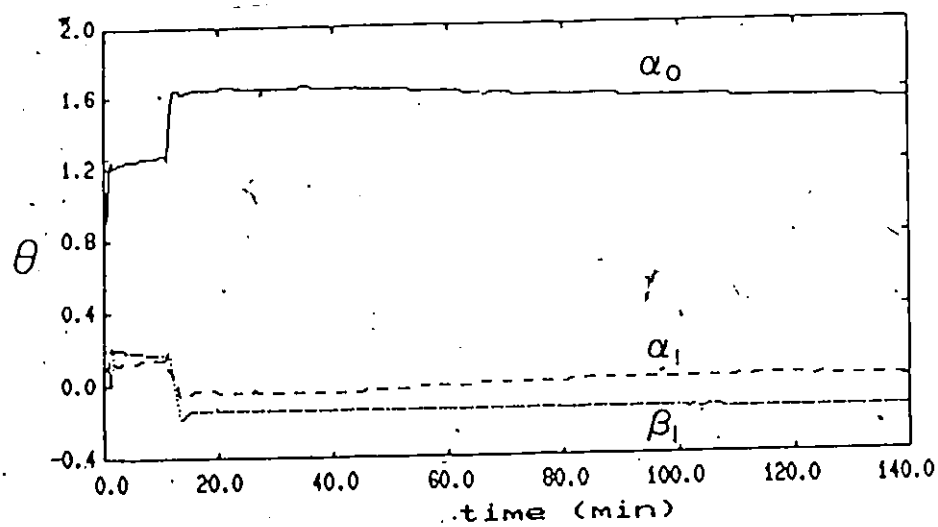

Figure 6.5 Inner loop controller parameters.

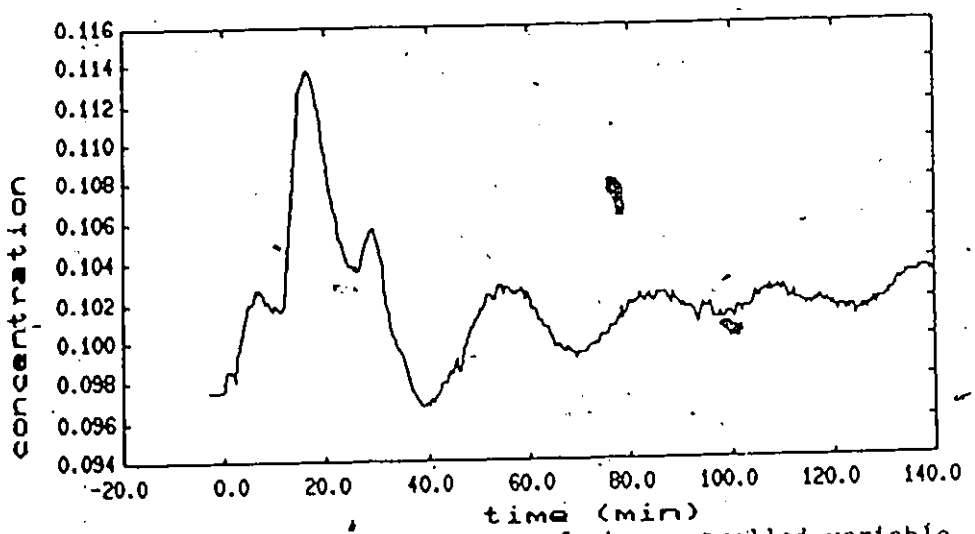

Figure 6.6 Servo response of the controlled variable extract concentration as seen by the inner loop.

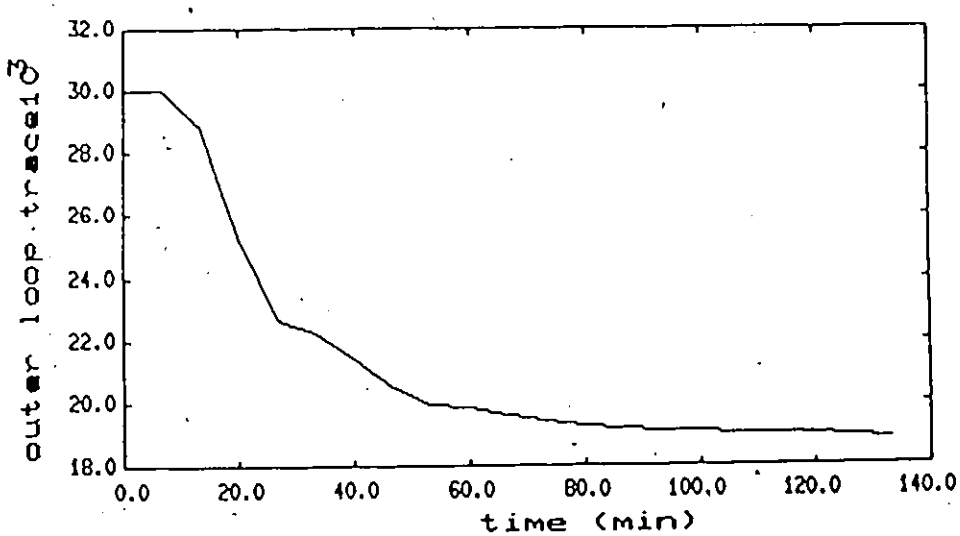

Figure 6.7 Trace of the outer loop variance-covariance matrix.

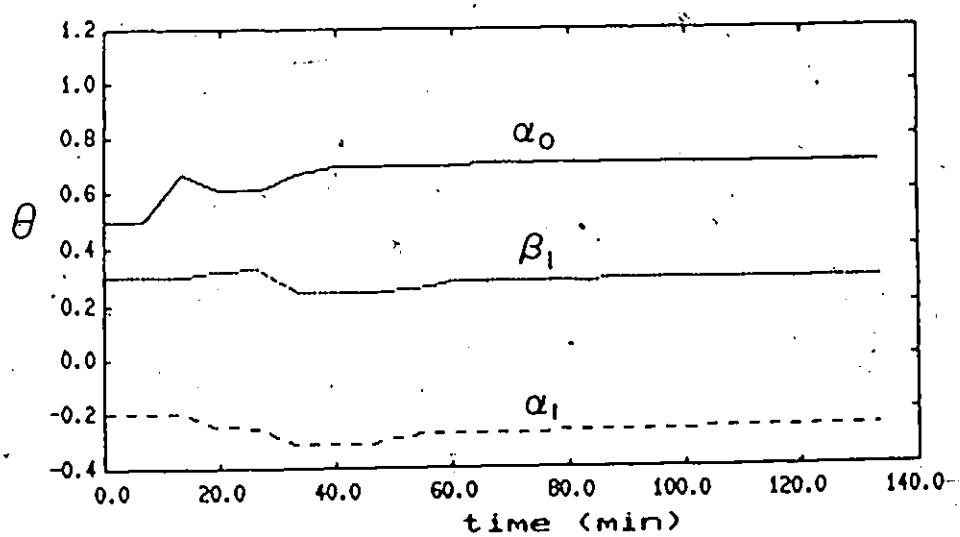

Figure 6.8 Outer loop controller parameters.

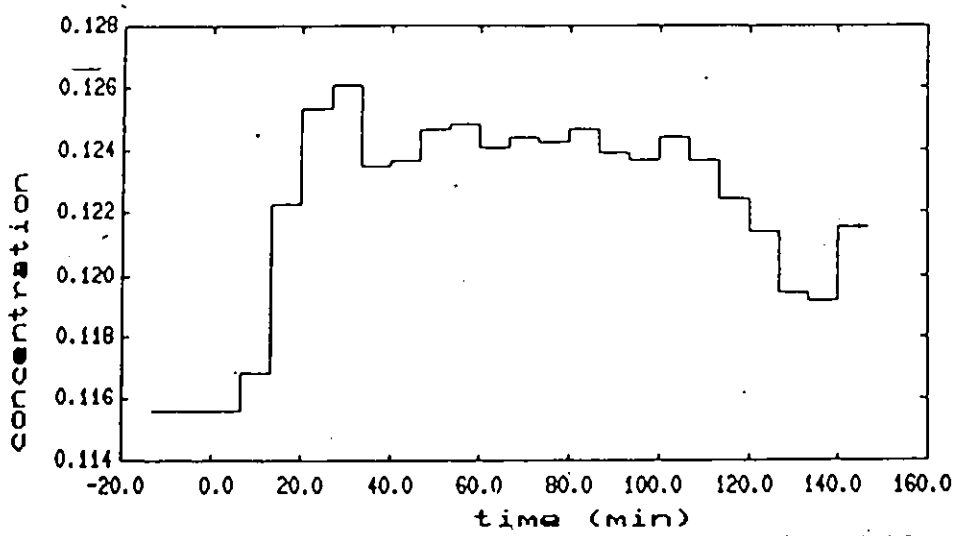

Figure 6.9 Servo response of the controlled variable extract concentration to a step change in the concentration set point.

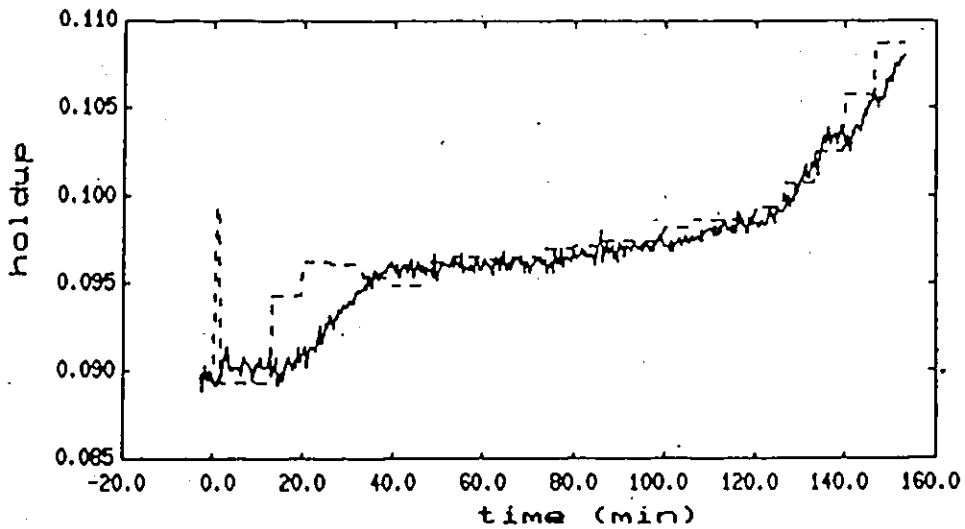

Figure 6.10 Response of the controlled variable holdup to a step change in the concentration set point.

quickly swept out of the column causing the overshoot. The corresponding inner loop behaviour is shown in Figure 6.10 where the dotted line indicates the holdup set point and the continuous line gives the measured holdup value. Later in the run a disturbance in the kerosene feed concentration enters the column and the controller is able to respond to this by increasing the holdup in the column.

6.3.2 Performace of a PI Outer and STR Inner Control Arrangement

The operating constraints on the outer loop controller (i.e. 400 second control interval to allow the inner loop to settle, clamping of the holdup setpoint so as to prevent flooding, and constraining the rate of change of the holdup set point in order to provide smooth operation across the highly nonlinear hydrodynamic holdup operating space since the settling time of the inner loop is dependent upon the magnitude of the set point change) meant that the parameters of the outer loop controller were not significantly affected by either the concentration set point change or the kerosene flow rate disturbance. In fact, the physical operating constraints and the constraints imposed by the cascade control scheme forced us to detune the outer loop controller to the extent that it hardly needed to be an adaptive controller. In light of this observation, the outer loop adaptive STR was replaced with a discrete equivalent of conventional PI controller in its velocity form and the disturbance rejection experiment was repeated. The gain and the integral action of the PI controller were obtained from the converged values of the STR of previous experiments in the following way:

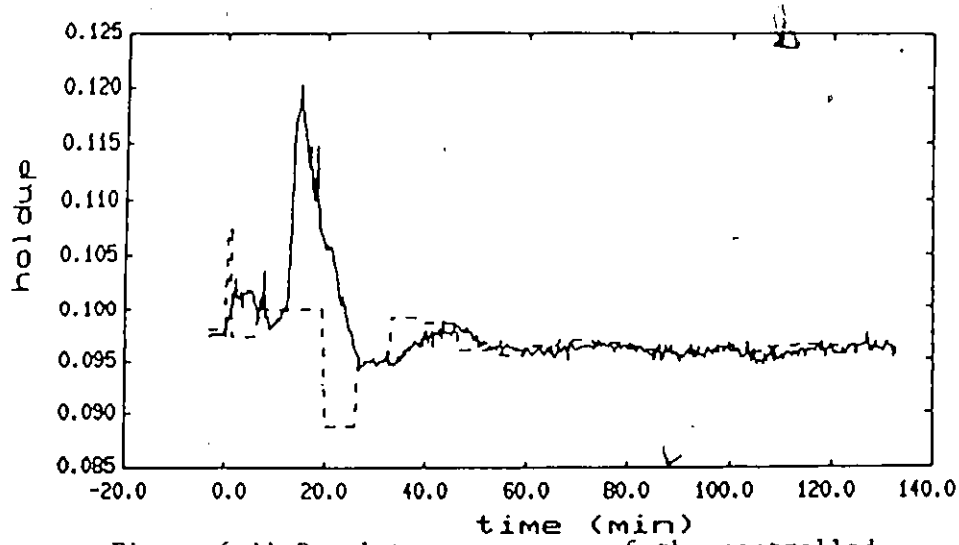

Figure 6.11 Regulatory response of the controlled variable holdup to a step disturbance for PI outer STR inner loop configuration.

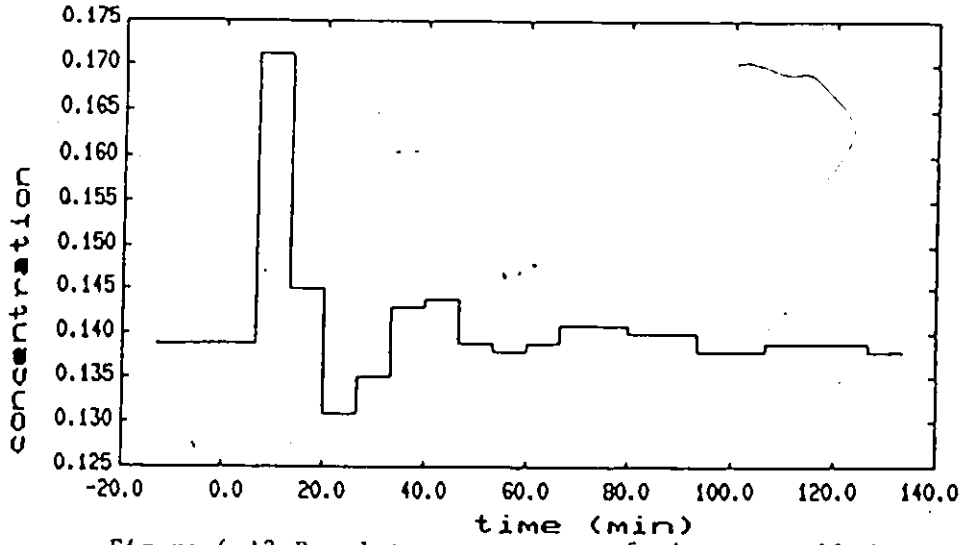

Figure 6.12 Regulatory response of the controlled variable extract concentration to a step disturbance for PI outer STR inner loop configuration.

The STR with 2 α and 2 β parameters, equation 6.1, has a PI controller structure with a memory term of the control action to provide dead time compensation. If this equation is multiplied by the operator ∇^{-1}

$$U_t = -[\beta_1 U_{t-1} + \alpha_0 \nabla^{-1} Y_t + \alpha_1 \nabla^{-1} Y_{t-1}]/\beta_0 \tag{6.2}$$

and since

$$\nabla^{-1} = \frac{1}{1-z^{-1}} = 1 + z^{-1} + z^{-2} + z^{-3} + \ldots \tag{6.3}$$

This can be written as

$$U_t = -[\beta_1 U_{t-1} + \alpha_0 \Sigma Y_t + \alpha_1 \Sigma Y_{t-1}]/\beta_0 \tag{6.4}$$

or after rearranging

$$U_t = -[\beta_1 U_{t-1} + (\alpha_0 + \alpha_1) \Sigma Y_t - \alpha_1 Y_t]/\beta_0 \tag{6.5}$$

The first term in the brackets of equation (6.5) is the memory term, the second term is the integral action term, and the last term is the proportional term. Comparing equation (6.5) with the position form of a discrete PI algorithm where the integral is approximated by a rectangular integration one could obtain the corresponding settings of a PI controller (i.e. the gain and the integral action). Since the gain and the integral action in equation (6.5) are overtuned, due to the dead

time compensation term, the controller settings used in the experiments were detuned slightly. From figure 6.8 the detuned PI settings are 0.2 and 200 for the gain and the integral action respectively. Figures 6.11 and 6.12 show the inner and outer loop responses for this configuration. Following the arguments above, the performance of this combination of an adaptive inner loop and a fixed parameter outer loop controller proved to be as effective as the cascaded adaptive STR control scheme.

McDonald and Wilkinson (1977) in their analog PI regulator investigations of a multiple-mixer solvent extraction column, controlled the heavy phase outlet concentration by manipulating the heavy phase flow rate into the column and have reported successfull results. Their result confirms our conclusions that: detuned controllers on the continuous phase outlet concentration do not need to be adaptive, and that the need for the dead time compensation inherent in the STR is reduced when long control intervals (relative to dead time) are used.

6.4 Conclusions

In this chapter an experimental application of cascaded adaptive STRs is reported. The cascaded scheme performed well for both disturbance rejection and servo control and preventing the column from flooding. A fixed parameter conventional PI controller for the outer loop was found to be an adequate replacement for the outer loop STR. However, Taylor et al. (1982) showed that adaptive control of the holdup is necessary for stable control. The outer loop STR converged parameters were used to calculate the controller settings for the PI regulator in

the outer loop. For the cascaded arrangement of concentration control the choice of the control interval is found to be dependent upon the settling time of the inner holdup loop. However, when concentration is controlled by manipulating the continuous phase flowrate U_c then the control interval can be chosen independently.

CHAPTER 7

7. Control of Extract Concentration by Manipulating Continuous Phase Flow Rate; SISO and MIMO Case

In the control studies in Chapter 6, only the frequency of reciprocation was used as the manipulated variable to control the extract concentration. In this chapter another variable, namely the continuous phase flow rate, is also included in the control loop. Now, there are two input variables; the frequency of reciprocation and the continuous phase flow rate, and two controlled variables; the dispersed phase holdup and extract concentration. In the sequel it will be shown through BRGA method that the pairing of the control loop is that of controlling the extract concentration with the continuous phase flow rate and controlling the holdup with the frequency of reciprocation. As was shown in the steady state simulation part in chapter 3, the process steady state gain is dependent upon the operating range of the input signal; i.e. the continuous phase flow rate. Later in the sequel this will be confirmed experimentally and it will be shown that not only the gain but also the dead time and the time constant of the process are also dependent upon the input signal. This suggests the application of an adaptive control scheme to compensate for the varying process dynamics in a manner similiar to the control of the holdup by manipulat-

ing the frequency of reciprocation. Note that the variations in the dead time as the manipulated variable spans the operating space can be calculated on line as was discussed in Chapter 2. Before proceeding with an adaptive controller, a fixed parameter controller (in this application a Dahlin controller, because of the significant process dead time) was used for servo and regulatory control both with SISO and MIMO (i.e. with and without simultaneous holdup control).

7.1 Experimental Identification of the System, Step Tests

In order to synthesize a Dahlin controller to control the extract concentration by manipulating the continuous phase flow rate, an open loop transfer function relating these two variables is required. A single step test experiment was designed in which a step change in the continuous phase flow rate was made from 0.4 to 0.55 cm/s. The transfer function between the flow rate and the extract concentration obtained from this step test is as follows:

$$\frac{C}{U_c} = -\frac{0.23\ \exp(-110\ s)}{240\ s+1} \tag{7.1}$$

Later four more step tests for a given kerosene feed flow rate and frequency of reciprocation were carried out in order to gain more insight into the nonlinearities of the process. In all the step tests the dispersed phase flow rate was 0.32 cm/s, frequency of reciprocation was 2.0 Hz, and acetic acid concentration in the kerosene feed was 0.1

moles/L. Table 7.1 summarizes these step test results (relevant representive step test response is given in Chapter 2).

Test No	U_c initial (cm/s)	U_c final (cm/s)	dead time (s)	Gain $(\frac{mol\ s}{L\ cm})$	Time constant (s)
1	0.450	0.600	100	-0.08	204
2	0.600	0.450	126	-0.10	272
3	0.450	0.300	200	-0.19	360
4	0.300	0.450	125	-0.17	270

Table of step tests for the effect of continuous phase flow rate on extract concentration.

TABLE 7.1

The following can be deduced from Table 7.1. Increasing the continuous phase flow rate results in a decrease in the residence time of this phase and hence the time constant of the process decreases in parallel to the residence time. Conversely, an increase in the flow rate results in a decrease in the time constant of this process. In step tests number 2 and 4 since these two experiments have the same flow rate they also have the same time constant (within experimental error). As was predicted in Chapter 3 through steady state simulations the gain of the process decreases at high flow rates.

In addition to the step changes in the continuous phase flow rate, the following transfer function was obtained from a step change in

kerosene feed flow rate from 0.32 to 0.4 cm/s while continuous phase flow rate was kept at 0.45 cm/s and frequency of reciprocation at 2.0 Hz.

$$\frac{C}{U_d} = \frac{0.19\ \exp(-130s)}{121\ s+1} \tag{7.2}$$

7.2) Bristol Relative Gain Array for the Karr Column

The approximate transfer matrix of the process obtained from the step tests with the input variables being the the continuous phase flow rate (U_1), and the frequency reciprocation (U_2), and the controlled variables being the extract concentration (Y_1) and the dispersed phase holdup (Y_2) given in the Laplace domain is:

$$g_{11} = \frac{-0.23\ \exp(-110.\ s)}{(240.0\ s + 1.0)} \qquad g_{12} = \frac{0.05\ \exp(-108.\ s)}{(460.0\ s + 1.0)}$$

$$g_{21} = \frac{0.06}{(5.0\ s + 1.0)} \qquad g_{22} = \frac{0.03\exp(-20.\ s)}{(40.0\ s + 1.0)} \tag{7.3}$$

The steady state gain matrix obtained from the above is:

$$G(0) = \begin{matrix} -0.23 & 0.05 \\ 0.06 & 0.03 \end{matrix} \tag{7.4}$$

The corresponding relative gain array can readily be shown to be:

$$\Lambda = \begin{matrix} 0.67 & 0.33 \\ 0.33 & 0.67 \end{matrix} \tag{7.5}$$

Following can be inferred from the RGA:

As to the pairing of the manipulated and output variables, the continuous phase flow rate should be used to control the extract concentration, and the frequency of reciprocation should be used to control the dispersed phase holdup. This conclusion is arrived at from the fact that the diagonal elements of the relative gain array should be dominant in the pairing. This is consistent with the experimental results obtained in sensitivity analysis in chapter 2.

7.3 Dahlin Controller

The Dahlin controller as implemented in the proceeding sections was synthesized from the nominal transfer function model given by equation 7.1. The pulsed transfer function corresponding to this equation, for a control interval of 20 seconds, is:

$$GH(z) = \frac{-0.0184\ z^{-6}}{1.0-0.92\ z^{-1}} \tag{7.6}$$

In the Dahlin controller, for a fixed control interval and dead time, the controller parameters are functions of the specified closed loop time constant τ_c. Although different τ_c values were used in the control studies a value of 60.0 seconds is chosen to show the numerical

values of the controller parameters. For any given value n of the closed loop dead time, if taken equal to the process dead time, the control law as obtained from equation (4.31) of chapter 4 is:

$$\nabla U_t = \frac{0.283-0.26\ z^{-1}}{-0.0184-0.0052\ z^{-1} - 0.0052\ z^{-2} \ldots -0.0052\ z^{-n}}\ Y_t \qquad (7.7)$$

As can be seen from this equation, the dead time only effects the number of poles, n, of the controller. The numerical value of (n-1) is equal to the number of dead time compensation terms in the controller i.e. the previous control actions. Therefore provided that n can be measured on line, as was the case for the Karr column, the dead time compensation terms can be varied on line (Dennis-Germuska et al., 1984).

The following are the salient features of the Dahlin controller. Nominal operating values were obtained from the transfer function model given in equation 7.1. No fractional delay was included in the discrete model since the control interval is fast relative to process dynamics. However, neglecting the fractional period of delay was compensated for by an addition of an extra dead time. The Dahlin controller intrinsically contains dead time compensation as was shown in Chapter 4. A variable dead time was used by calculating the dead time on line and incorporating it into the controller equation.

The control interval was chosen to be 20 seconds to match the holdup control interval. A clamp was imposed on the manipulated variable between 0.25 and 0.65 cm/s. The dead times corresponding to

maximum and minimum flow rates are 5 (for the flow rate 0.65 cm/s) and 12 (for the flow rate 0.25 cm/s) and at the nominal operating point (for flow rate 0.45 cm/s) the dead time is 7 control intervals. In all experiments inlet feed concentration was in the range of 0.10-0.20 moles/L. In all experiments the extractor was brought to steady state and then the control program was started. In either case of SISO or MIMO control, the controller was tested for set point changes in the extract concentration, and a disturbance in kerosene feed flow rate. Another possible disturbance is a change in the acetic acid feed concentration. However this experiment was not done for the following reason. A disturbance in kerosene feed flow rate upsets both manipulated variables directly and is therefore a severe disturbance; whereas a disturbance in the concentration only upsets the extract concentration directly and therefore is not as severe. Consequently, the severe case was tested first and because the result was successful the less severe case was not attempted.

Nominal operating point corresponds to the following conditions. Continuous phase flow rate was 0.45 cm/s, kerosene feed flow rate was 0.32 cm/s, frequency of reciprocation was 2 Hz, and the flooding holdup value was 0.2256.

7.4 Results and Discussions

The experiments reported include: (i) Concentration only

control (SISO); and (ii) concentration control along with the dispersed phase holdup control (MIMO). Table 7.21 summarizes the operating conditions in the proceeding experiments.

Figure No	U_c (cm/s)	U_d (cm/s)	τ_c (s)	case	type	dead time (s)
7.1-7.3	0.45	0.32	240	SISO	servo	variable
7.4-7.6	0.45	0.32	60	SISO	servo	variable
7.7	0.45	0.32	100	SISO	servo	fixed
7.8-7.10	0.45	0.44→0.30	60	SISO	regulatory	variable
7.11-7.16	0.45	0.44→0.30	60	MIMO	regulatory	variable
7.17-7.18	0.45	0.32	60	MIMO	servo	variable

Operating conditions for the concentration control experiments.

TABLE 7.2

7.4.1 Control of Extract Concentration by Manipulating Continuous Phase Flow Rate; SISO case

7.4.1.1 Servo Control

Different closed loop time constant τ_c values were used to test the response of the controlled variable. Initially the closed loop time constant τ_c was chosen to be equal to the open loop time constant of the process i.e. 240 seconds (12 control intervals). A set point change in the extract concentration from 0.063 to 0.085 moles/L was made. Controlled variable response (i.e. extract concentration) is shown in

Figure 7.1. Manipulated variable response is given in Figure 7.2. The concentration reaches the set point value in about 40 control intervals. Approximate closed loop time constant is about 20 control intervals as opposed to the theoretical closed loop time constant of 12 control intervals. The difference arises from the fact that the estimated transfer function parameters are different from that of the real process. Part of this variation in the transfer function parameters is expected and due to the fact that transfer function parameters were estimated from a step test where the manipulated variable was increased from 0.40 to 0.55 cm/s. Whereas in this experiment a step change in the set point of the extract concentration was intentionally made to drive the flow rate to a lower value than the nominal value of 0.45 (new steady value of the flow rate can be seen to be 0.37 cm/s from Figure 7.2). This low flow rate, since the flow rate and the time constant are inversely proportional, results in a higher value of the time constant than the one used in the process model. Figure 7.3 shows the response of the uncontrolled dispersed phase holdup. Since the flow rate was decreased the holdup was also decreased. Figure 7.4 shows the response of the extract concentration when the closed loop time constant was reduced to 60 seconds i.e. 3 control intervals. The set point change was made very large in this case and was from 0.0687 to 0.12 moles/L. Figures 7.5 and 7.6 show the response of the manipulated flow rate and uncontrolled dispersed phase holdup respectively. The closed loop time constant is almost the same as the previous case, Figure 7.1 with τ_c = 240, because the control variable reached the lower limit of 0.25 cm/s.

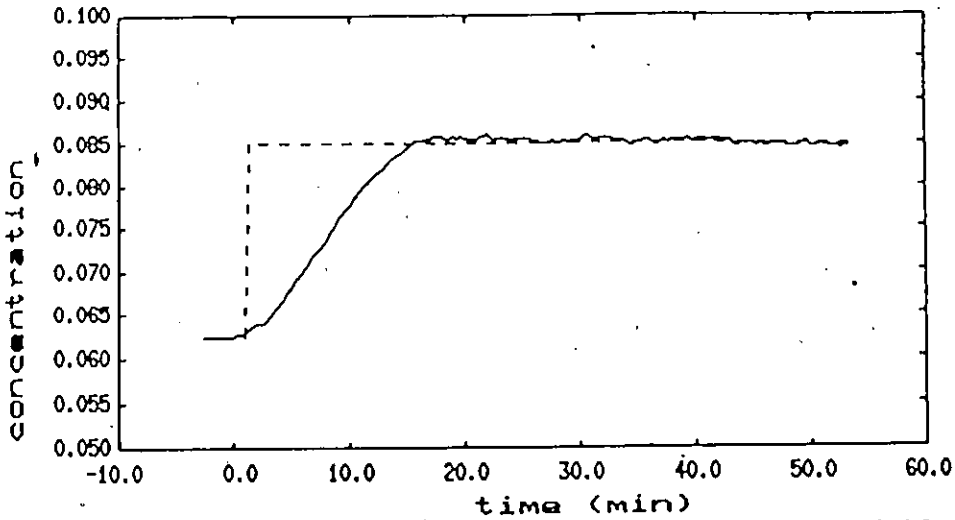

Figure 7.1 Servo response of the controlled variable extract concentration.

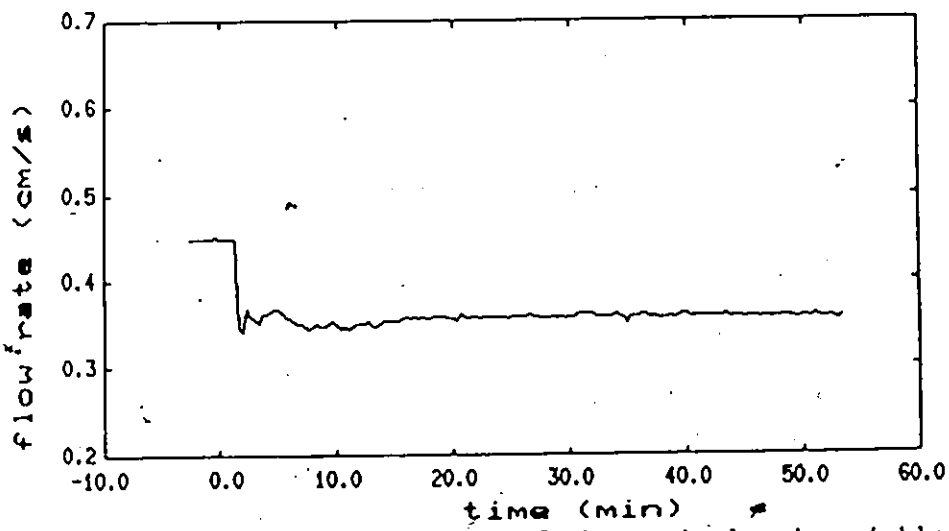

Figure 7.2 Servo response of the manipulated variable continuous phase flow rate.

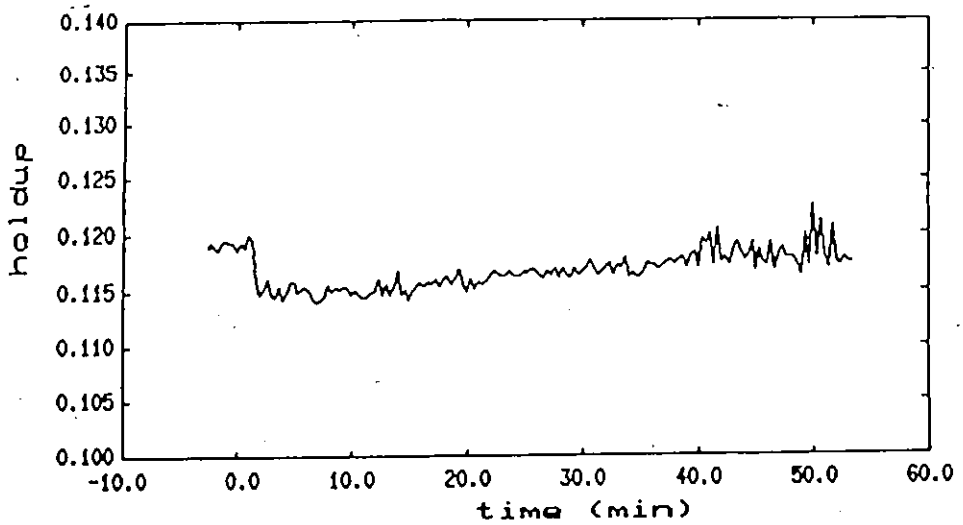

Figure 7.3 Response of the uncontrolled holdup.

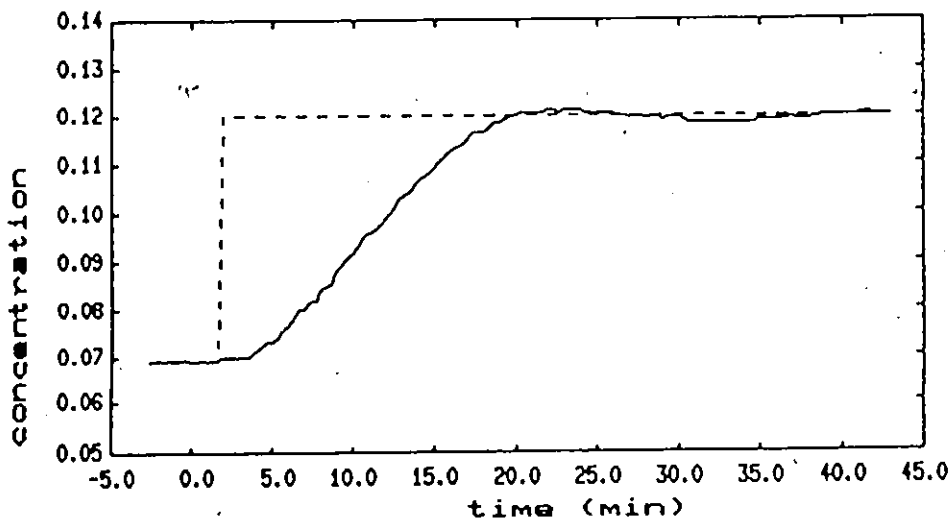

Figure 7.4 Servo response of the controlled variable extract concentration.

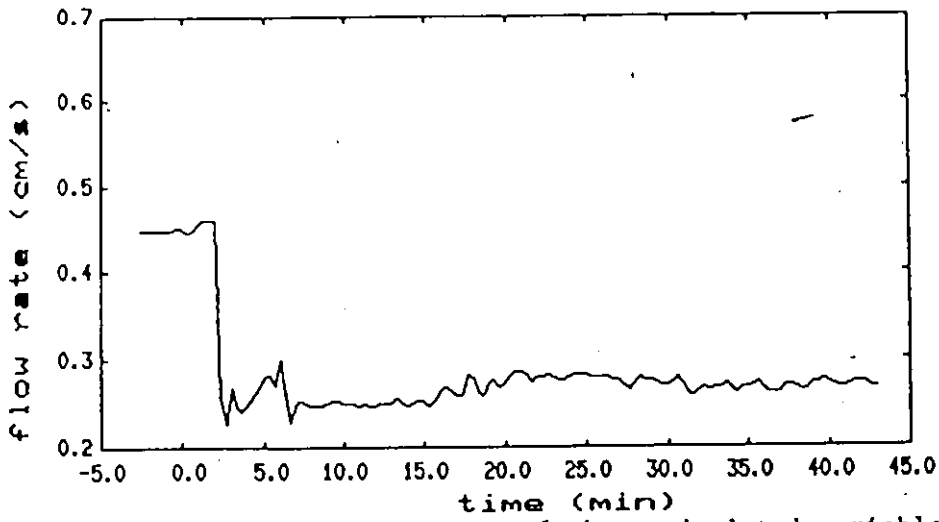

Figure 7.5 Servo response of the manipulated variable flow rate.

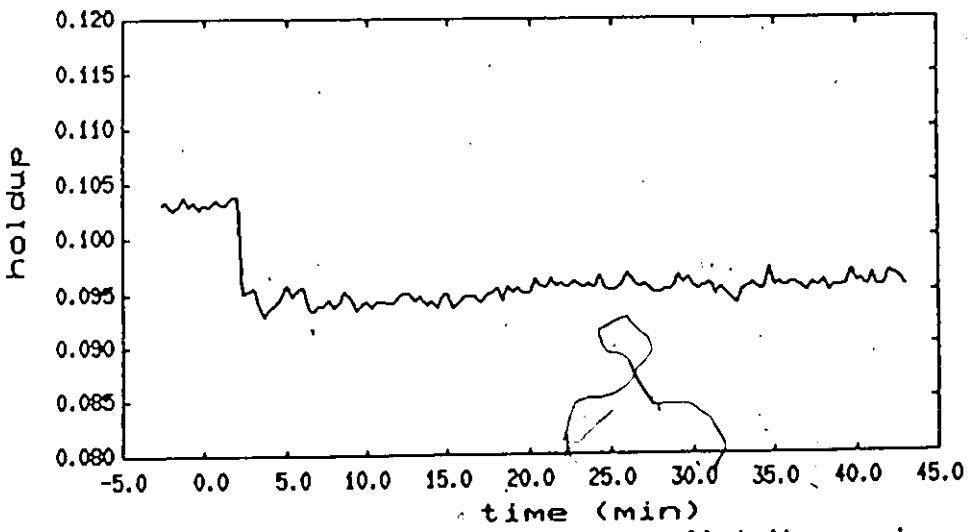

Figure 7.6 Response of the uncontrolled dispersed phase holdup.

An experiment was also carried out with a controller with a fixed dead time compensation of 7 control intervals obtained from the nominal value of a flow rate of 0.45 cm/s. The response to this control is underdamped but yet still stable. The closed loop time constant was 100 and the process dead time changed from 7 to 9 control intervals. The extract concentration response is shown in Figure 7.7. The oscillations in the output reveals the destabilizing effect of constant dead time.

7.4.1.2 Regulatory Control

The Dahlin controller obtained from the nominal transfer function for servo control was tested for a disturbance in the kerosene feed flow rate with variable dead time. The kerosene flow rate was decreased from 0.44 to 0.3 cm/s. The response is satisfactory and is shown in Figure 7.8. The controlled variable goes through a long lasting deviation and then is damped. Also included in Figures 7.9 and 7.10 are the manipulated variable (flow rate), and the uncontrolled dispersed phase holdup respectively.

7.4.2 Control of Extract Concentration Along With Holdup Control; MIMO Case

As was stipulated in chapter 6, controlling the extract concentration alone is not sufficient since the column might flood if holdup is left uncontrolled. In this section experimental results are reported when also the holdup control is included by manipulating the frequency of reciprocation. This inclusion along with the control of the extract

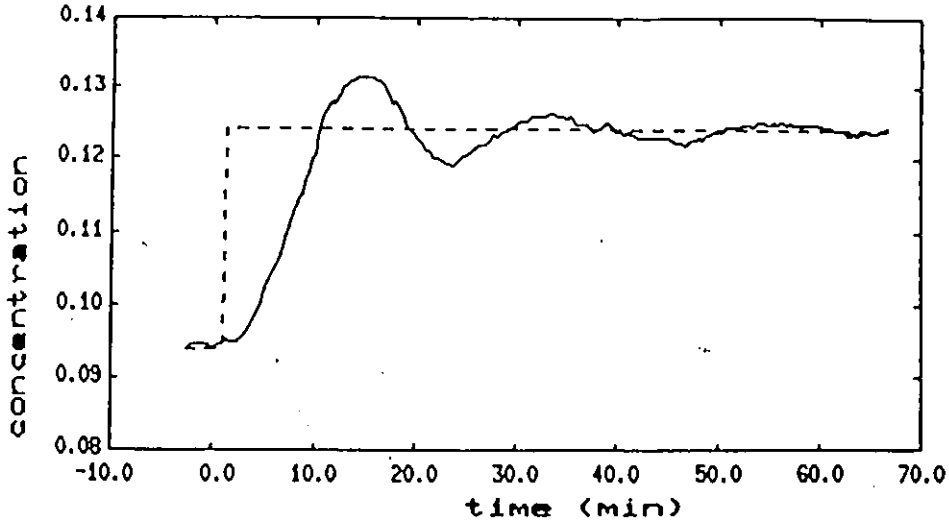

Figure 7.7 Servo response of the controlled variable extract concentration with fixed dead time.

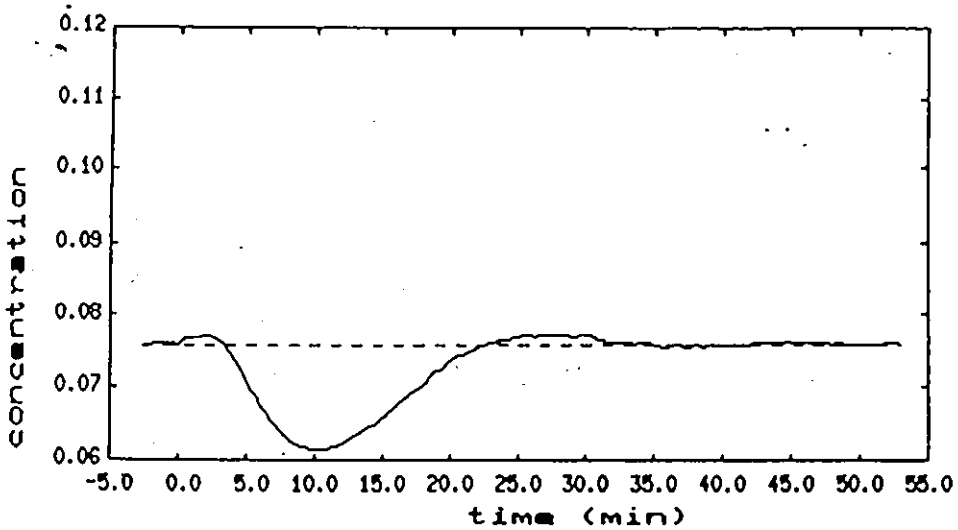

Figure 7.8 Regulatory response of the controlled variable extract concentration to a disturbance in kerosene flow rate.

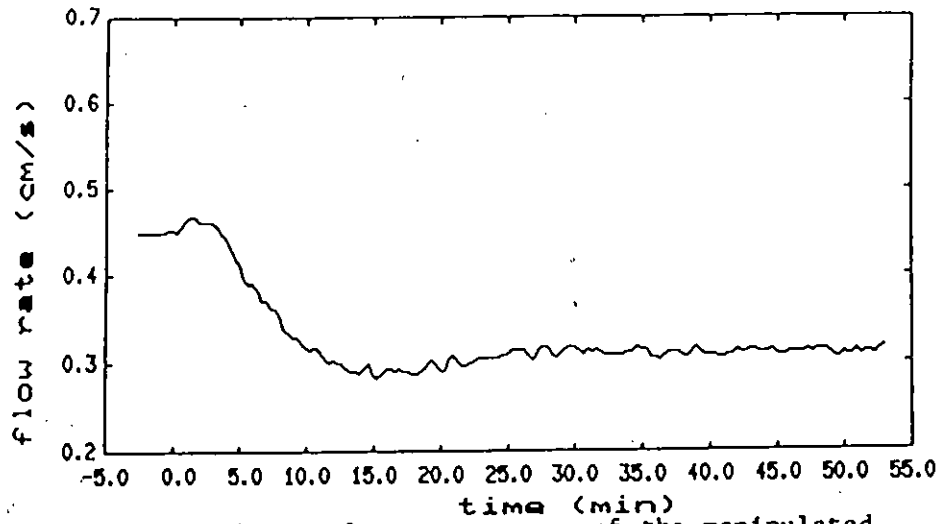

Figure 7.9 Regulatory response of the manipulated variable continuous phase flow rate to a disturbance in kerosene flow rate.

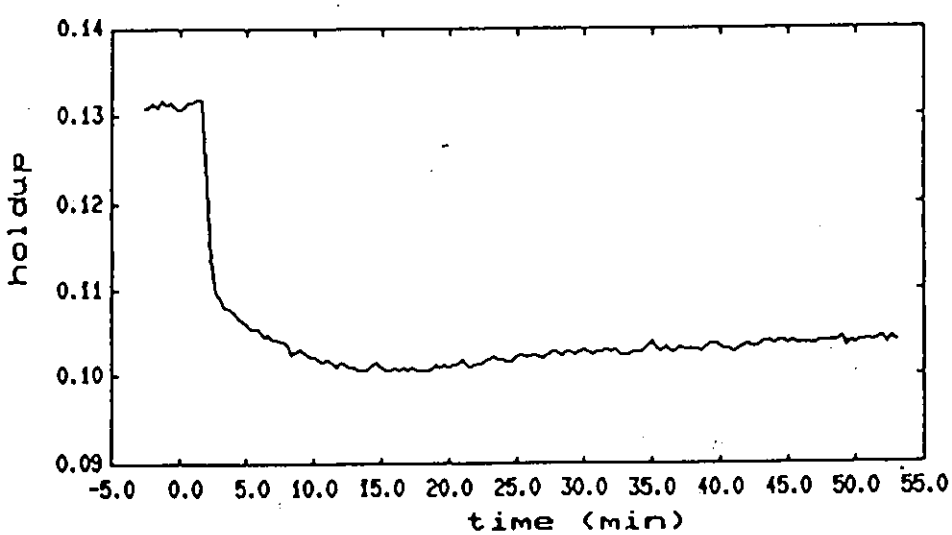

Figure 7.10 Response of the uncontrolled dispersed phase holdup to a disturbance in kerosene flow rate.

concentration by manipulating the continuous phase flow rate renders the control scheme into a multivariable control scheme.

In these set of experiments the Dahlin controller was used again for controlling the extract concentration by manipulating the continuous phase flow rate together with a STR which was used for controlling the dispersed phase holdup by manipulating the frequency of reciprocation.

The following a priori considerations suggest that for the multivariable control of the Karr column, with the given input/output pairing, decoupling is not necessary. While the column is operating under the steady state conditions if an upward set point change is made in the extract concentration, the manipulated variable i.e. continuous phase flow rate will go down in order to bring the concentration to its new desired value. This will reduce the dispersed phase holdup as a result of the reduction in the drag force acting on the droplets. If holdup is also controlled by manipulating the frequency of reciprocation then in response to this decrease in the holdup the manipulated variable i.e. the frequency of reciprocation will increase to restore the holdup back to its set point. This will also yield higher rates of mass transfer. Following the preceding lines of rationale the same conclusion can also be reached for a disturbance in the kerosene feed flow rate. Consequently, the two controllers help each other in their interaction rather then compete once the set point of one of them deviates from its steady state value.

The control law of the STR is the same as the one used for the single loop holdup only control i.e. the Clarke-Gawthrop algorithm in its self tuning form with 2 α, 2 β parameters with a constraint factor of 0.5 and a control interval of 20 seconds. Control law for the Dahlin algorithm is that the control interval is again 20 seconds with a closed loop time constant of 60.0 seconds i.e. 3 control intervals.

7.4.2.1 Regulatory Control

The coupled Dahlin and STR controller was tested for regulatory response to a disturbance in kerosene feed flow rate and the data obtained were compared with that of obtained from the single loop data. Disturbance in kerosene flow rate was from 0.44 cm/s to 0.30 cm/s. The extract concentration response as can be seen from Figure 7.11, is oscillatory, asymmetric, and very underdamped in comparison to the response obtained from the experiment with SISO controller. The asymmetry is a result of the nonlinearity of the process. The small second overshoot in the holdup, shown in Figure 7.12, results in a large overshoot in concentration. As was discussed in Chapter 3, the graph of concentration versus holdup, obtained from simulations, is steep for lower values of the holdup (i.e. high gain), and levels off at higher values of the holdup (i.e. low gain). The graph shown in Figure 7.13 is given as an aid to illustrate what follows. If the holdup is varied sinusoidally around a fixed value, as a result of the nonlinearity of the process, the amplitude of the output, i.e. concentration, is distorted. This explains the reason for the second large overshoot in

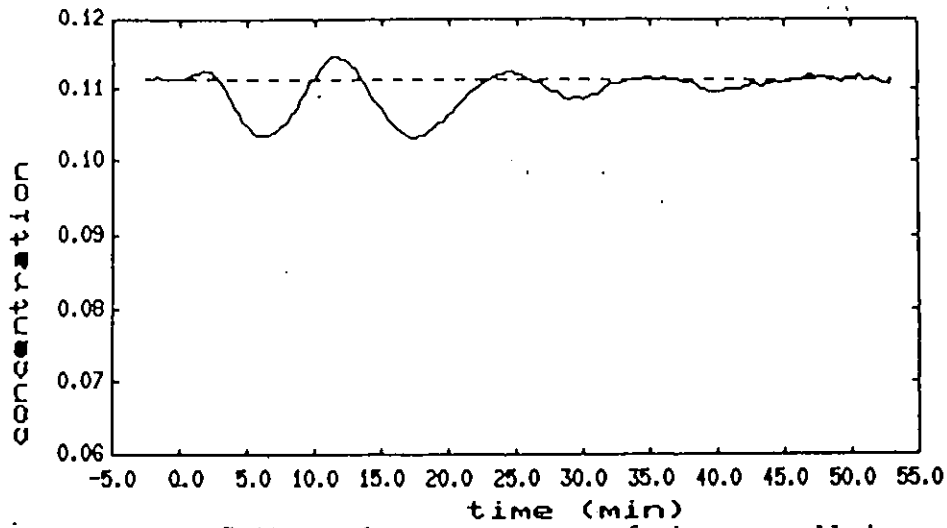

Figure 7.11 Regulatory response of the controlled variable extract concentration to a disturbance in kerosene feed flow rate.

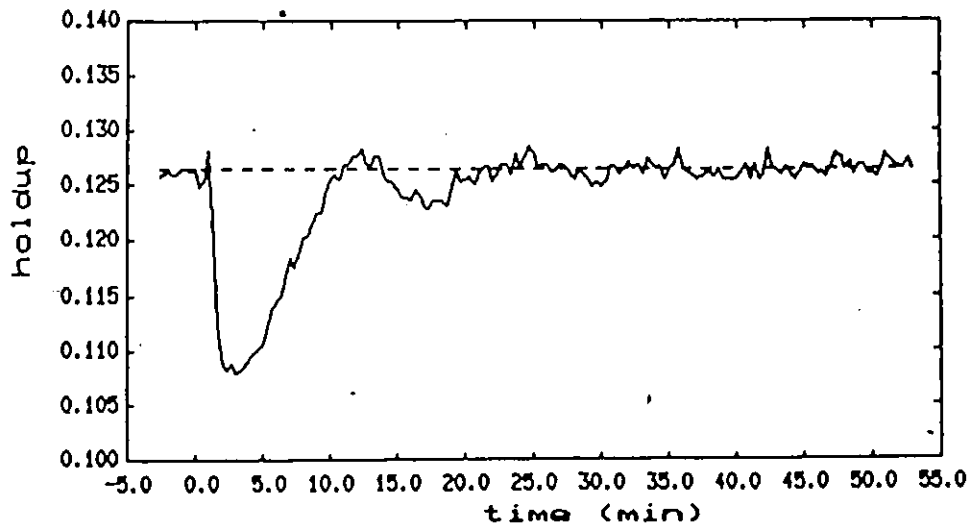

Figure 7.12 Regulatory response of the controlled variable holdup to a disturbance in kerosene feed flow rate.

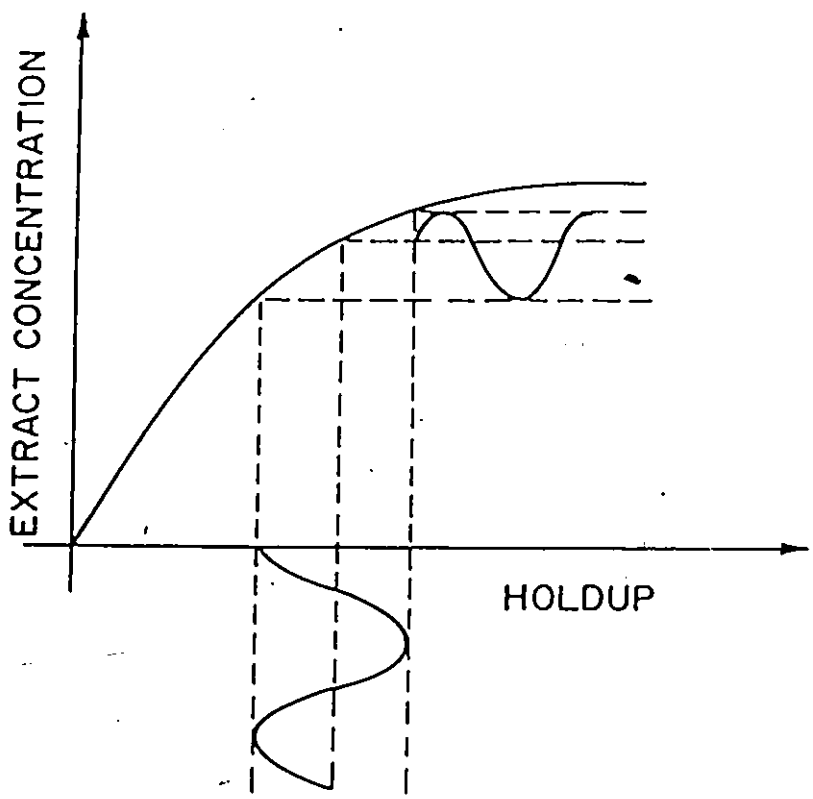

Figure 7.13 Amplitude distortion effect as a result of nonlinearity.

the concentration with an underdamped holdup response. Note that the period of oscillation is approximately twice the SISO case. The two controllers, despite the fact that they are not decoupled, are successful in keeping the individual set points at their desired values and their interactions help them as was conjectured earlier. This can be seen from the steady state value of the flow rate. In the SISO case the new steady state value of the flow rate is approximately 0.33 cm/s from the nominal startup value of 0.45 cm/s giving a net change of 0.12 cm/s; whereas in this case, for the same amount of disturbance the new equlibrium value of the flow rate is settled at 0.37 cm/s (Figure 7.14) giving a net change of 0.08 cm/s. The difference is compensated by the increase in the frequency of reciprocation, Figure 7.15, which is increased to bring the holdup to its set point. This suggests a saving in operating costs whereby an objective can be set to use the frequency of reciprocation as much as possible since it is much cheaper than the solvent flow rate.

The controller parameters of the STR in Figure 7.16 can be seen to have been adapted to new values. Stopper starter criteria as was discussed in Chapter 4 section 4.1.3.3 allows the parameters to adapt to changes in the process by setting the discounting factor less than 1.0 and once the process is settled making it 1.0 again so as to prevent covariance windup.

7.4.2.2 Servo Control

In this experiment a set point change was made in the extract

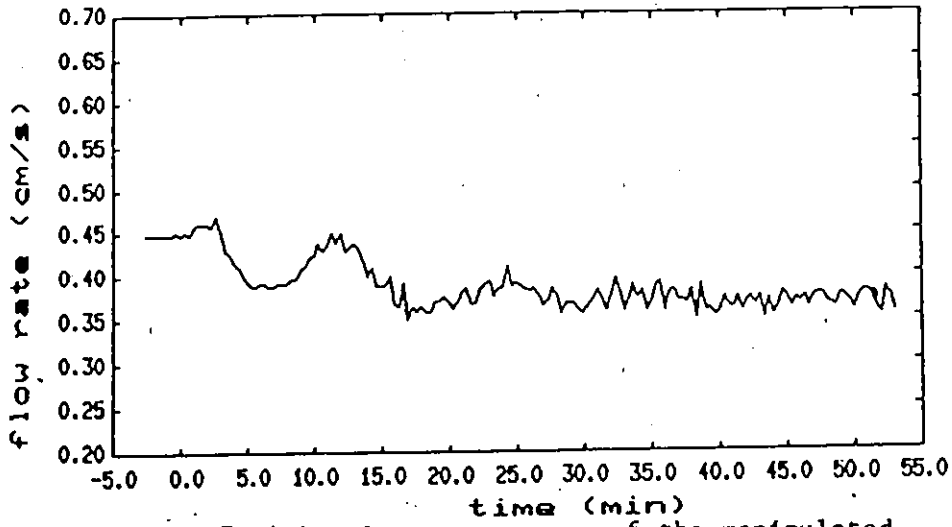

Figure 7.14 Regulatory response of the manipulated variable continuous phase flow rate to a disturbance in kerosene flow rate.

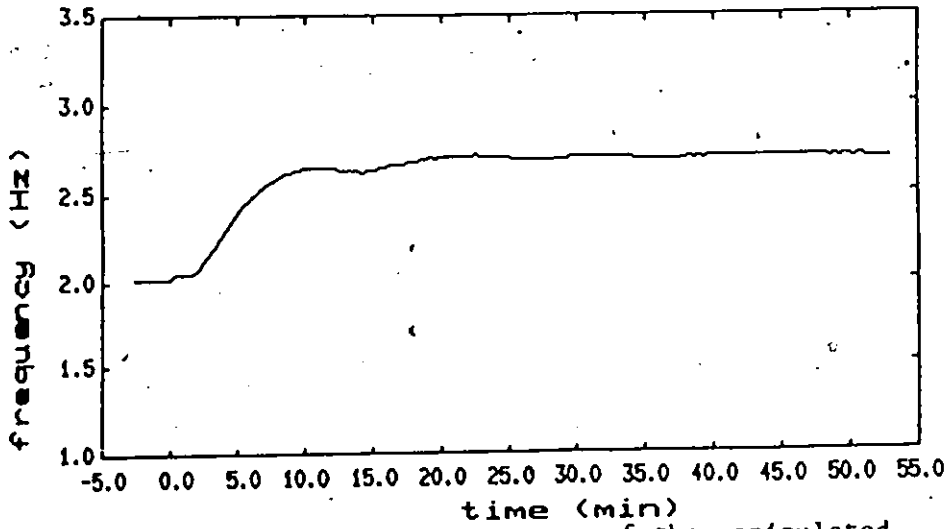

Figure 7.15 Regulatory response of the manipulated variable frequency of reciprocation to a disturbance in kerosene flow rate.

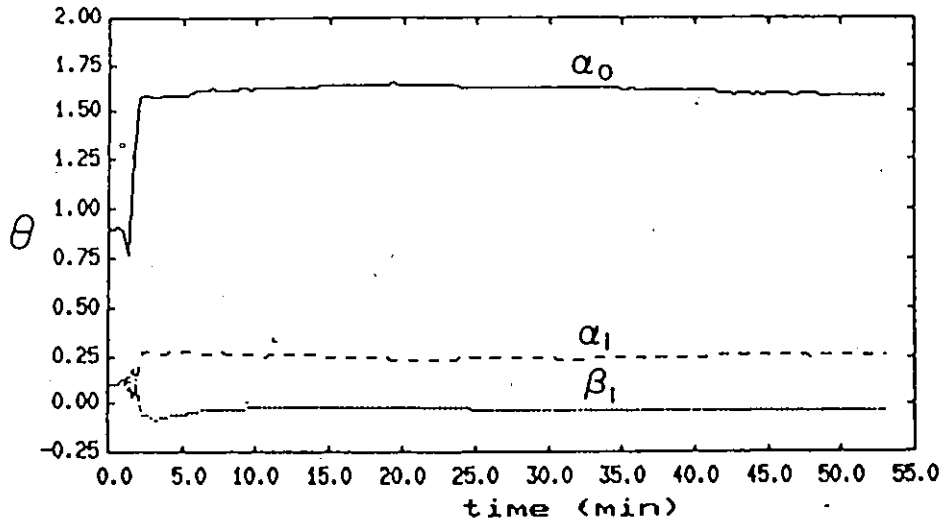

Figure 7.16 Self tuning regulator parameters.

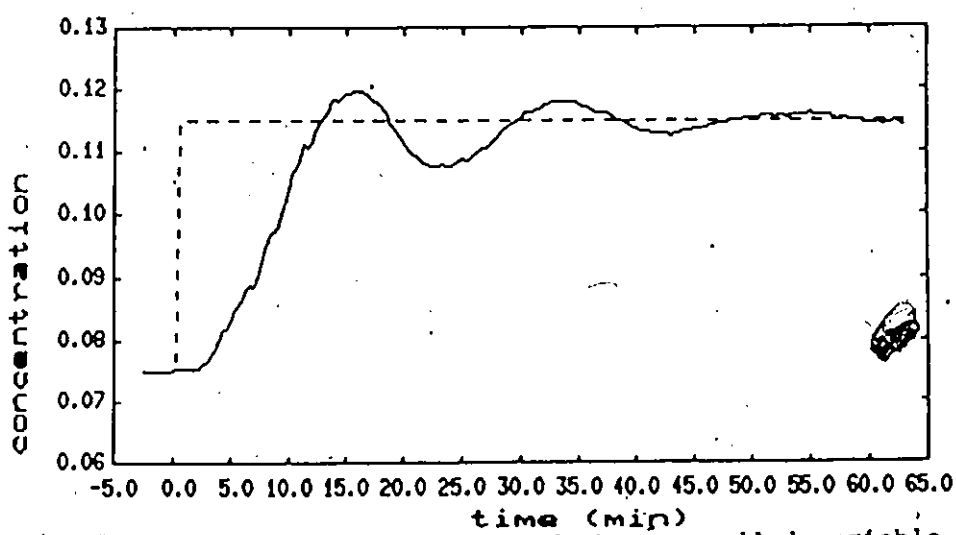

Figure 7.17 Servo response of the controlled variable extract concentration.

concentration from 0.075 to 0.115 moles/L. The concentration response is underdamped in the MIMO case as opposed to overdamped response of the SISO case for the same closed loop time constant of 60 seconds and is again due to interaction. The responses of the concentration and the holdup are given in Figures 7.17 and 7.18 respectively.

7.5 Conclusions

In this chapter the control objective was to achieve servo and regulatory control of the extract concentration by manipulating the continuous phase flow rate in the cases of SISO and MIMO control. This control objective is successfully achieved. Despite the nonlinearities of the system a fixed parameter controller, namely Dahlin algorithim with variable dead time compensation, has accomplished this objective. Within the operating region the variation in the dead time was between 5-12 control intervals, the gain and the open loop time constant of the process has varied approximately by twofold within the operating region. The fact that one steady state gain is negative, from the input of continuous phase flow rate to the output of extract concentration, while the other steady state gain, from the input of frequency of reciprocation to the dispersed phase holdup, is positive means that the interactions help both controllers to achieve their objectives rather than causing them to compete with each other. This interaction has made the response oscillatory and may be improved by one way decoupling; is recommended for the future work.

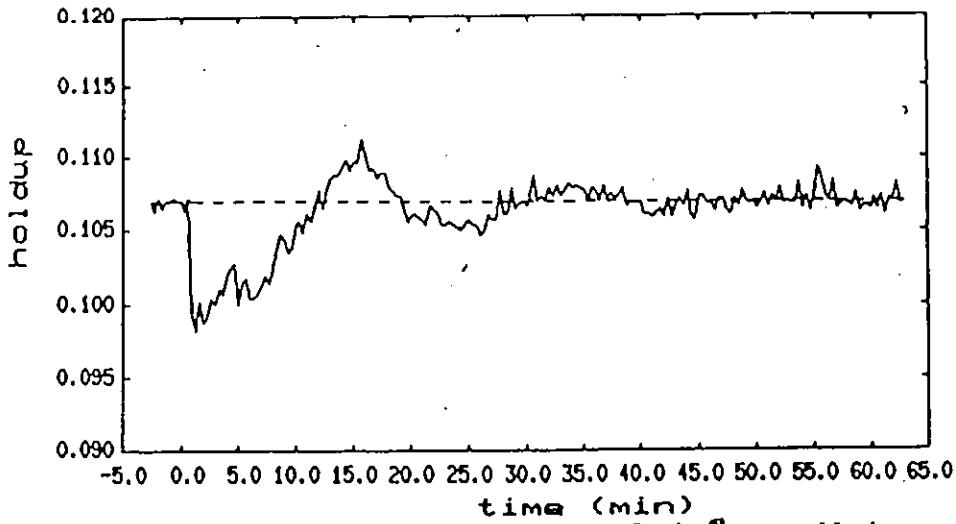

Figure 7.18 Regulatory response of the controlled variable holdup.

CHAPTER 8

8. Conclusions and Recommendations for Future Work

8.1 Conclusions

Steady state simulations, based on a semi-empirical model proposed by Baird-Shen, were done on the hydrodynamics of the holdup and it was found that the data agree quite well with prediction at conditions approaching flooding, but at low agitation rates the holdup values exceed prediction by about 25%. This is to be expected because the mechanistic model is based on dispersion behaviour in a well agitated system at high holdup.

In the presence of mass transfer (acetic acid from the dispersed kerosene phase to the continuous water phase), the holdup data are not well predicted by the model due to enhanced droplet coalescence which was not accounted for in the model. However the equation which predicts the flooding holdup is independent of the physicochemical properties of the extraction system but is a function of the ratio of the flow rates and therefore can be used to estimate the flooding holdup value.

Steady state mass transfer characteristics of the column were also simulated using a mechanistic model and the results were used to interpret the closed loop response characteristics of the extraction process. No direct quantitative comparison between simulated and experimental results had been sought because of the complex coalescence behaviour of the droplets as a result of droplet to continuous phase mass transfer, and the difficulty associated with the estimation of parameters such as the mass transfer coefficient.

The multifactorial nature of the nonlinearity of the hydrodynamics of the column necessitates an adaptive regulator for servo control and disturbance rejection. Consequently an implicit adaptive Clarke-Gawthrop self-tuner was used to control the dispersed phase holdup. The control in this work was an extention of Kusuma's (1981) work covering a larger operating space. Successful control was achieved, under no mass transfer conditions, up to holdup values of 70% of the flooding value as calculated from the Baird-Shen relation. The holdup control at 80% of the flooding value was unstable due to the variations of the process parameters at a rate which was faster then the ability of the controller to adapt. In the case of conditions under which mass transfer was taking place, mass transfer induced coalescence had severely affected the stability of the control system and conditions which were stable without mass transfer became unstable with mass transfer taking place. Therefore the maximum permissible holdup value was reduced to 60% of the flooding value by trial and error. Also an ad hoc variable discounting factor, based on the prevailing physical conditions in the column, was tested experimentally and found to be

successful in preventing the covariance windup problem. The shortcoming of this variable discounting factor is that if the set point variations are steps with small size in the same direction or a ramp with a small slope, then the "stopper starter" will not be activated.

The control of the extract outlet concentration was implemented using two different control schemes i) A multi loop arranged in a cascaded fasnion and ii) SISO and MIMO control. The cascade arrangement was that the inner loop was the holdup control, to guarantee that flooding will not take place, and the outer loop was the extract concentration control, with the frequency of reciprocation as the manipulated variable. Because of the various restrictions placed upon the outer loop controller a PI-STR arrangement was found to be as effective as an adaptive STR-STR controller arrangement.

The Dahlin controller was used to control the concentration by manipulating the continuous phase flow rate with (MIMO) and without (SISO) the presence of the control loop of the holdup by manipulating the frequency of reciprocations. A variable dead time was incorporated into the Dahlin controller to account for the dead time variations due to flow rate changes. The Dahlin controller was succesful despite a twofold variation in the time constant and the gain of the process. In the MIMO case, even though the controllers were not decoupled their interaction had helped them to achieve the control objectives. This was because one of the forward path transfer functions had a positive gain while the other had a negative gain.

This study has indicated that the potential for greatly improved control of extraction columns is possible by operting close to flooding point. Some further work is needed to confirm this, as indicated in the following section.

8.2 Recommendations

1) Change the solute from acetic acid to i-butyric acid which has a dissociation constant close to acetic acid but the distribution coefficient is only .3:1 in favour of water as opposed to the 220:1 distribution coefficent of acetic acid in favour of water. This way the column can be operated at high frequency and holdup values with less depletion of the acid in the kerosene phase (lower extraction factor).

2) Move the location of the bottom differential pressure probe to in between the two bottom plates so that the nonminimum phase response of the holdup to frequency of reciprocations due to the Bernouilli effect is eliminated. The holdup control can then be done in less than 20 seconds. This way, the cascaded control loop can be repeated much faster then was possible in this work.

3) The MIMO case can be extended to include a one way decoupling between the holdup loop and the concentration loop such that as long as the operating holdup value in the column is less then the set upper bound the manipulated variable of the concentration loop will not be activated. This will save operating costs since the cost of manipulating the frequency of reciprocation is much less than that of the solvent.

4) Other control schemes which inherently contain dead time compensators such as the Smith predictor, should be evaluated as alternatives to the Dahlin controller.

5) On the basis of present work, it will be possible to carry out optimal control studies to determine how to use the frequency of reciprocation and the flow rate to achieve the lowest cost operation of exit concentration while maintaining safe holdup control.

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