

# MATHEMATICAL MODEL OF AN ION EXCHANGE COLUMN

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## Abstract

With the increasing need to reduce the colour of product white sugar from the Refinery a number of steps were taken to determine ways of improving colour removal across the ion-exchange resin plant.

A mathematical model of an ion-exchange column was derived to predict the optimum conditions for colour removal when varying the following parameters:

- (a) cycle time or number of cycles
- (b) resin bed height
- (c) feed liquor flowrate
- (d) initial colour of feed liquor
- (e) series or parallel operation

At Hulett's Refinery a series of comparison tests are being done in a Pilot Plant using acrylic/acrylic and acrylic/styrene columns in series. An evaluation is made on the advantages and disadvantages of operating columns in series and the performance of styrene and acrylic resins in the second columns.

In order to understand the mechanism of the colour removal process a series of laboratory batch experiments were done. The results agree well with the Langmuir absorption isotherm.

## Introduction

In order to improve the quality of white sugar from the Refinery a detailed study was made on the performance of ion-exchange columns and ion-exchange resins. The scope of the project included deriving a mathematical model of an ion-exchange column to determine optimum conditions for maximum colour removal.

As a result of this work, a decision was made to install a second stage of acrylic resin in series with the existing acrylic resins at Hulett's Refinery. Overseas experience suggests that a combination of acrylic and styrene resin yields the best results. An evaluation is being done at the pilot plant to compare the performance of acrylic/acrylic and acrylic/styrene columns in series; the results to date are outlined in this paper.

Finally, equilibrium batch tests were carried out to establish the mechanism of colour adsorption and properties of ion-exchange resins.

## Theory and Assumptions

The removal of colour bodies from the feed liquor by means of solid adsorbents is an unsteady state mass transfer process. A feed liquor is applied to the top of the resin bed and flows down through the bed with colour bodies progressively being adsorbed by the resin. The mathematical model was based on the assumption that the rate of transfer of material between the feed liquor and the small resin particles can be stated in terms of colour concentration  $Y$  in the fluid and  $X$  in the solid. This type of rate permits the introduction of a rate function  $F(Y,X)$  so that the rate equation can be written, for a differential element  $dz$  of the resin bed.

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$$\frac{dX}{dt} = F(Y,X) \quad \dots (1)$$

and leads to a system of partial differential equations.

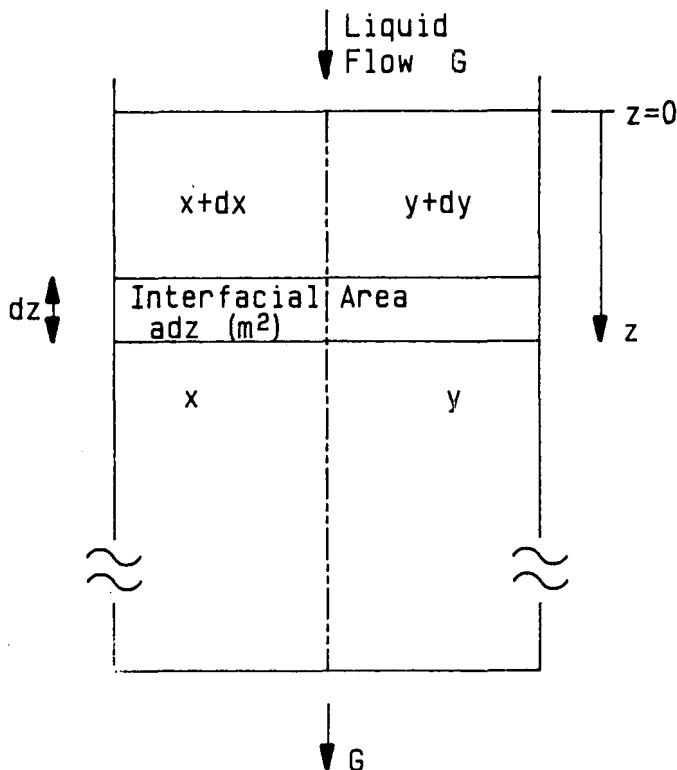


FIGURE 1 Element within the Packed Bed of Height  $dz$  and unity cross section.

The solving of these equations gives the final result relating colour in effluent liquor as a function of time, bed height and liquid flowrate.

$$Y(n, \tau) = (mX_0 + b) + (Y_0 - mX_0 - b) [1 - e^{-\tau} \int_0^{\tau} e^{-n_0} I_0(2\sqrt{m}n) dn] \quad \dots (2)$$

where:

- $Y$  = liquor colour/kg solution
- $Y_0$  = initial liquor colour/kg solution
- $m$  = defined by equilibrium relationship  $Y^* = mX^\beta + b$
- $\tau$  = dimensionless time variable =  $mt'/r\theta G$
- $n$  = no of transfer units =  $k_L a z/G$
- $I_0$  = modified Bessel function of first kind and zero order
- $X_0$  = initial colour in resin/kg resin
- $G$  = mass velocity of liquid stream ( $\text{kg/hr m}^2$ )

In deriving the model the following assumptions were made:

1. Since the ratio of bed height to particle diameter is large the effect of axial mixing is negligible in the bed and plug flow of the liquid phase is assumed.
2.  $\beta = 1$  in the equilibrium relationship  $Y^* = m X^\beta + b$ .

3. The concentration of adsorbed colour is uniform when  $t = 0$  and feed liquor entering the bed has constant colour.
4. After regeneration i.e. at  $t = 0$ , the concentration of the irreversibly adsorbed colour is uniform throughout the bed.
5. The feed liquor entering the bed has constant colour throughout the cycle.

Appendix 1 gives the theory and derivation of the model. Equation 2 is programmed in Regula which performs the integration using a six point Gaussian quadrature and the Bessel function is evaluated using a polynomial approximation.

#### Experimental Data for the Model

A series of tests were done on the resin pilot plant to generate experimental data for the mathematical model from which the unknown parameters,  $k_L a$  the mass transfer coefficient,  $X_0$  the initial colour loading of the resin and  $m$  defined by the equilibrium relationship  $Y^* = mX^b + b$ , could be evaluated.

Runs were done on acrylic resin which had been subject to different numbers of cycles in factory service, after being regenerated by an acid wash, to compare the decolourisation of feed liquor at flowrates varying from 1-5 bed volumes per hour (BV/HR).

The feed liquor was heated in a liquid/liquid heat exchanger, maintained at a constant temperature of 80°C and fed into a one litre column with a diameter 80 mm. All the runs were done at a set bed height with the bed volume flow rate being varied by changing the feed flowrate. Samples

were taken at two-hourly intervals and analysed at the STD laboratory.

Figure 2 illustrates the % decolourisation against resin exhaustion at different flowrates using a cycle time of 18 hours. The results show no significant variation in % decolourisation between new resin and resin which had completed up to 165 cycles; however, after 165 cycles there is a marked decrease in % decolourisation obtained.

#### Determination of the Model Parameters

The model parameters are estimated from the best fit of the model to the experimental data using the Regula - Falsi and Secant Method.<sup>4</sup> The best fit value is that which minimises the sum of the squared values of  $\sum (Y - Y^1)^2$ . Since a strong correlation exists between  $X_0$  and the other two parameters  $m$  and  $k_L a$ ,  $X_0$  was set equal to zero in order for REGULA to converge. The agreement between the model's predicted results and the experimental data was found to be very good (average correlation coefficient = 0,91, no. of data pts = 35).

Figure 3 illustrates a plot between mass transfer coefficient  $k_L a$  and the number of cycles the resin has completed at different flowrates. The mass transfer coefficient, at a constant flowrate, increases for the first hundred and twenty cycles and then decreases fairly rapidly.

The reason for the initial increase is probably due to an increase in surface area caused by the destruction of the resin due to the caustic conditions in regeneration. The graph of parameter  $m$  vs number of cycles completed (Figure 4) suggests that  $m$  is also both a function of flowrate and age of resin.

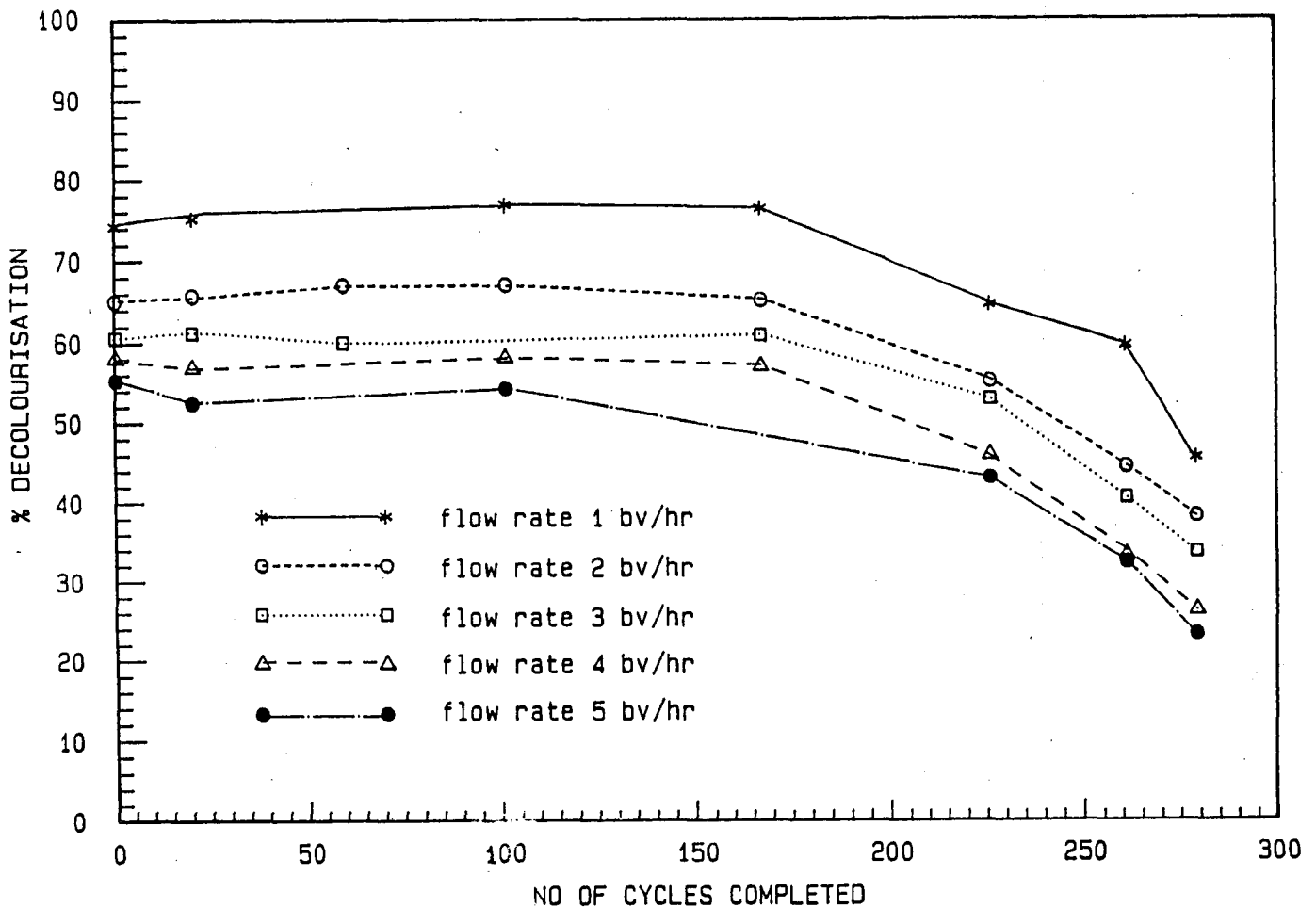


FIGURE 2 % Decolorisation versus Number of Cycles Completed.

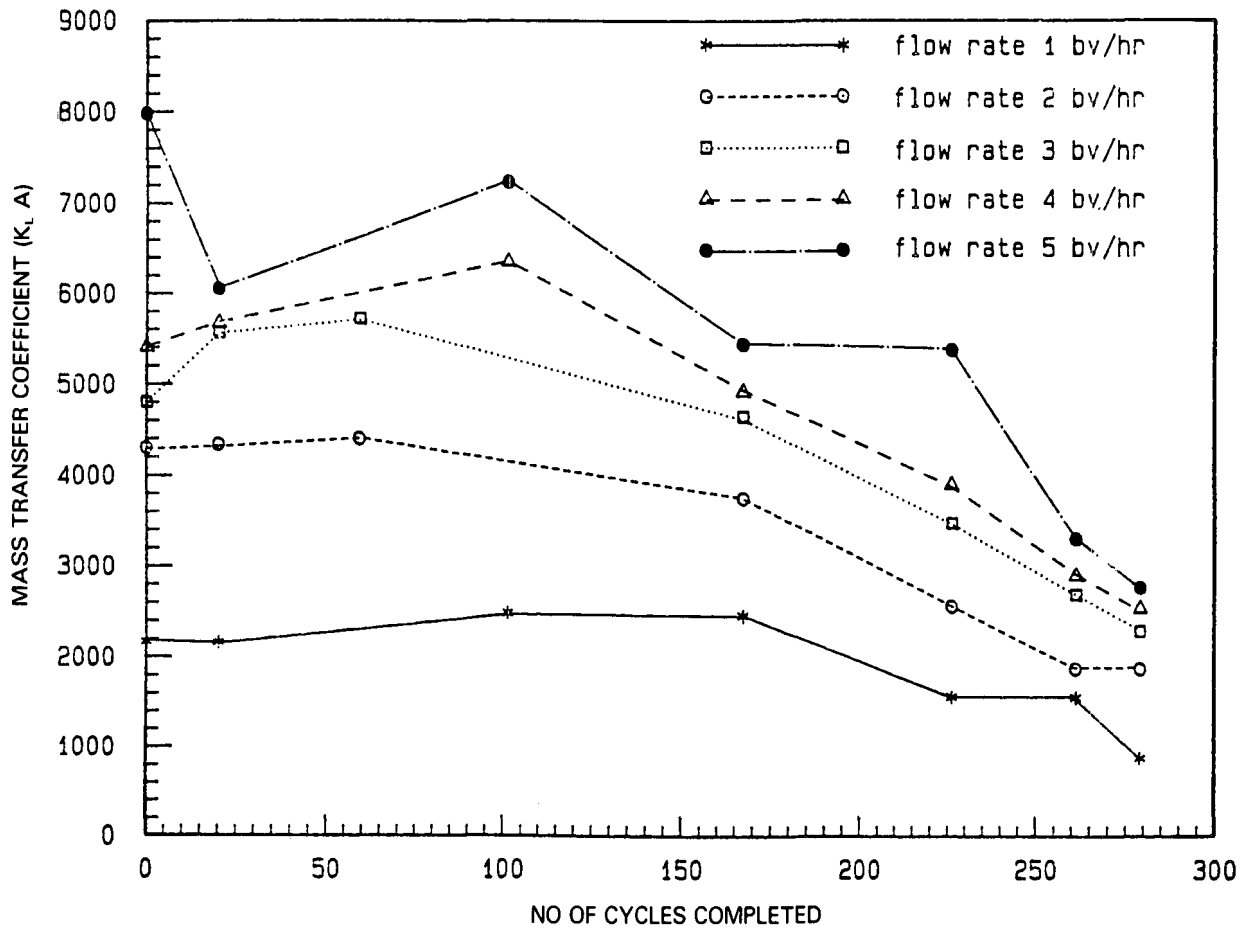


FIGURE 3 Mass Transfer coefficient versus Number of Cycles Completed.

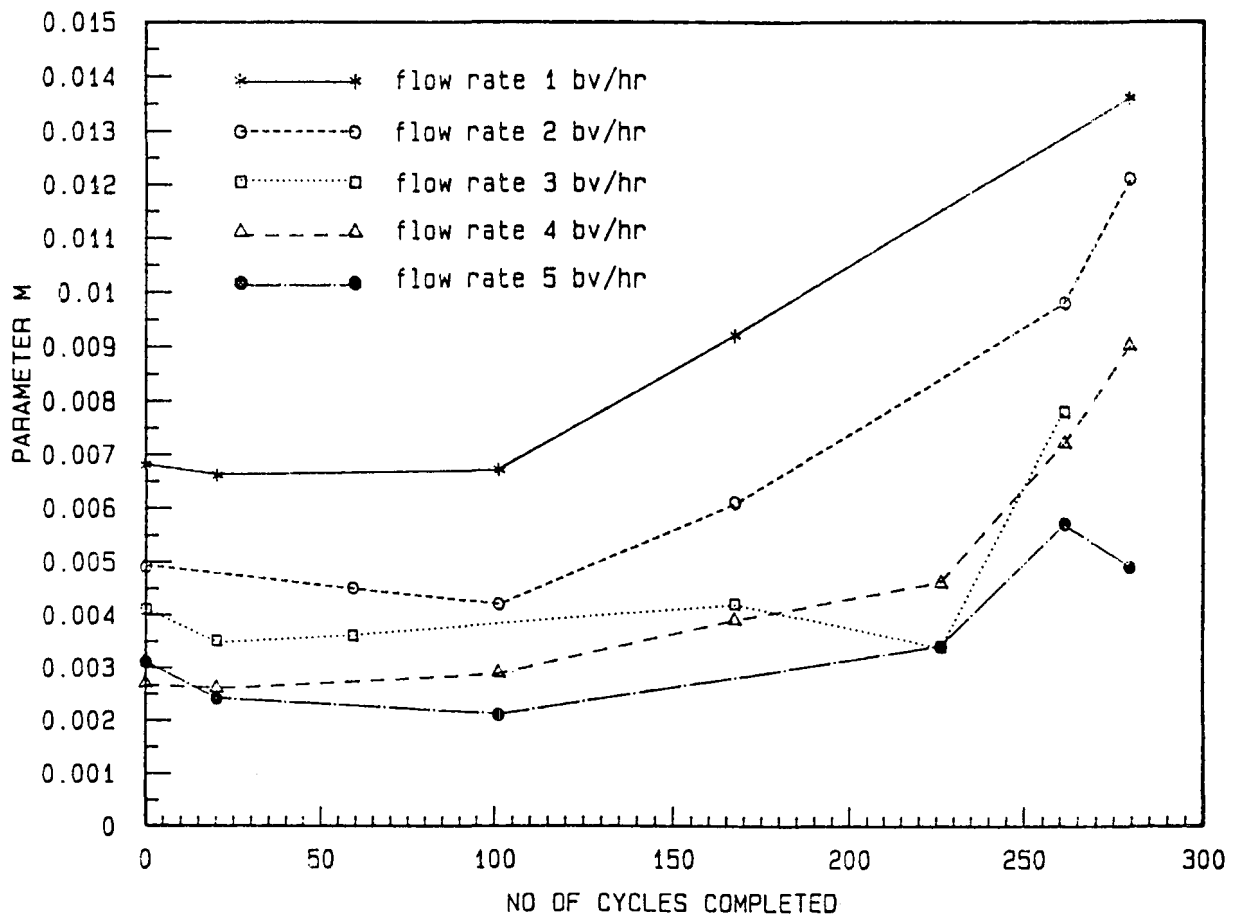


FIGURE 4 Parameter M versus Number of Cycles Completed.

As initial destruction of the resin occurs increasing the surface area, parameter  $m$  remains fairly constant at a specific flowrate, but after 100–120 cycles the value of parameter  $m$  increases as the resin becomes irreversibly fouled.

The higher the flowrate used in the experiment the greater the mass transfer. This is to be expected as the surfaces become more efficiently wetted. The value of  $m$  becomes smaller as flow rate increases and better solid/liquid contact is attained.

### Ion Exchange Column Performance Simulation

A programme RESIN SIM was developed to simulate the performance of the Refinery resin columns. The values of parameters determined from the experimental data by the Regula programme were used in the predictive mode to establish how the model responds to changes in the independent variables.

The effect of changes to the following variables were considered.

- (a) initial colour of feed liquor
- (b) volume of resin used in the column
- (c) change in flowrate
- (d) length of cycle

The results of predicted % decolourisation vs number of cycles the acrylic resin has completed, are shown in Figure 5 for five different flowrates. The following base case values of the variables were used:

- (a) bed height of the resin  $z = 0,131$  m
- (b) length of cycle  $t = 18$  hours
- (c) initial brown liquor colour 900 ICUMSA units
- (d) brown liquor density  $h = 1290,6$  kg/m<sup>3</sup>
- (e) resin bulk density  $H = 610$  kg/m<sup>3</sup>
- (f) brown liquor brix 66 brix
- (g) initial brown liquor  $Y_0 = 594$  liquor colour/kg solution.

The model predicts the same % decolourisation is achieved at different bed heights, providing the flow in BV/HR remains the same. Varying the initial feed colour alters the final colour, but the predicted % decolourisation remains the same. The model is not sensitive to large fluctuations in brown liquor density.

### Equilibrium Batch Tests

The mathematical model was derived using the assumption that the equilibrium curve  $Y^* = mX^\beta + b$  is a straight line (ie  $\beta = 1$ ).

In order to confirm this assumption and to determine the mechanism of acrylic and styrene anion exchange resins, a series of batch tests were completed using new styrene resin, acrylic resin 3 cycles old and acrylic resin 313 cycles old. Samples of resin were placed in a 1 000 ml flask in a shaking water bath for eight hours at a temperature of 70°C, with a series of sugar solutions ranging in colour from 35 to 5 700 units, until equilibrium was reached. Previous tests showed

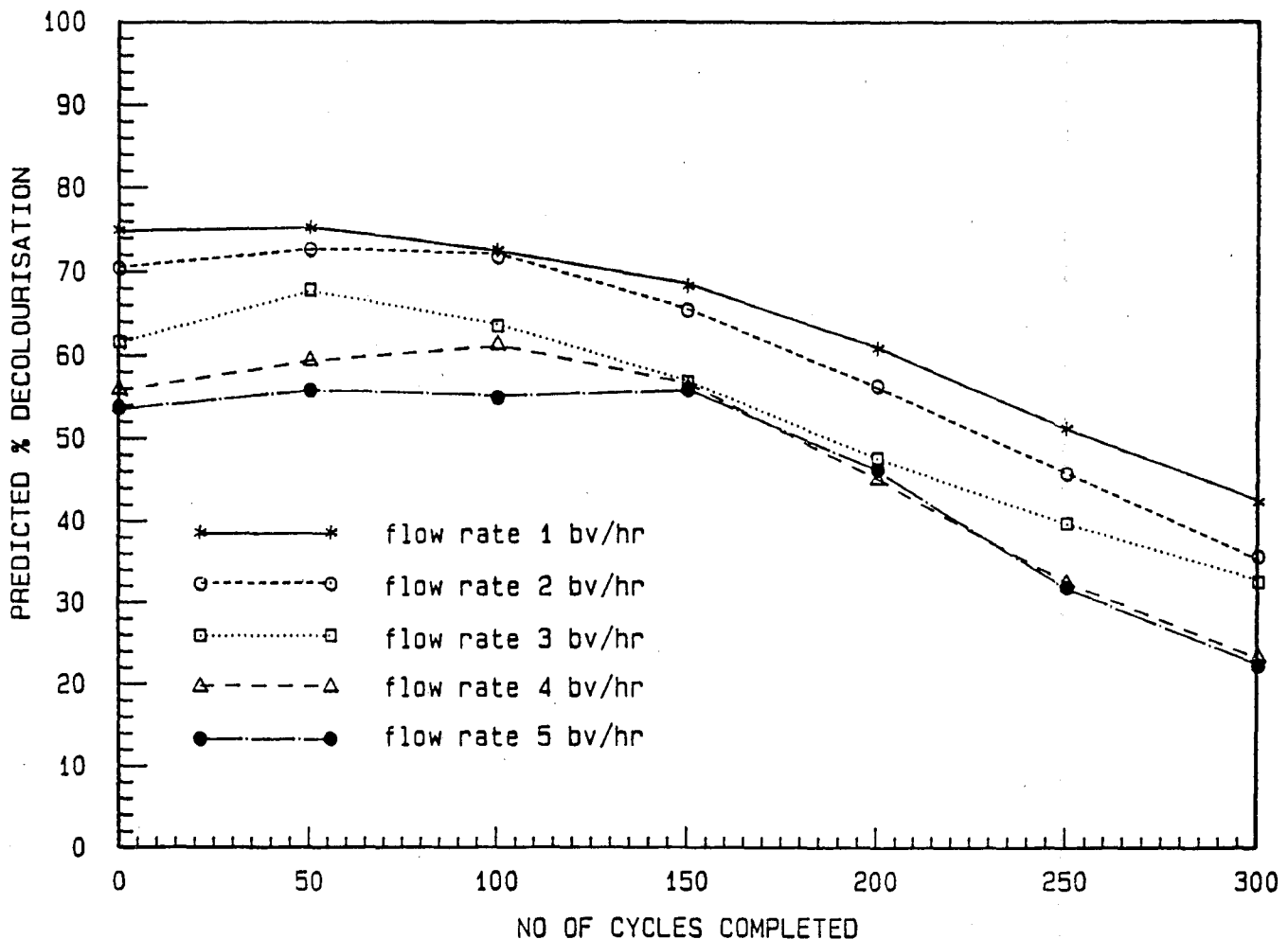


FIGURE 5 Predicted % Decolorisation versus Number of Cycles Completed.

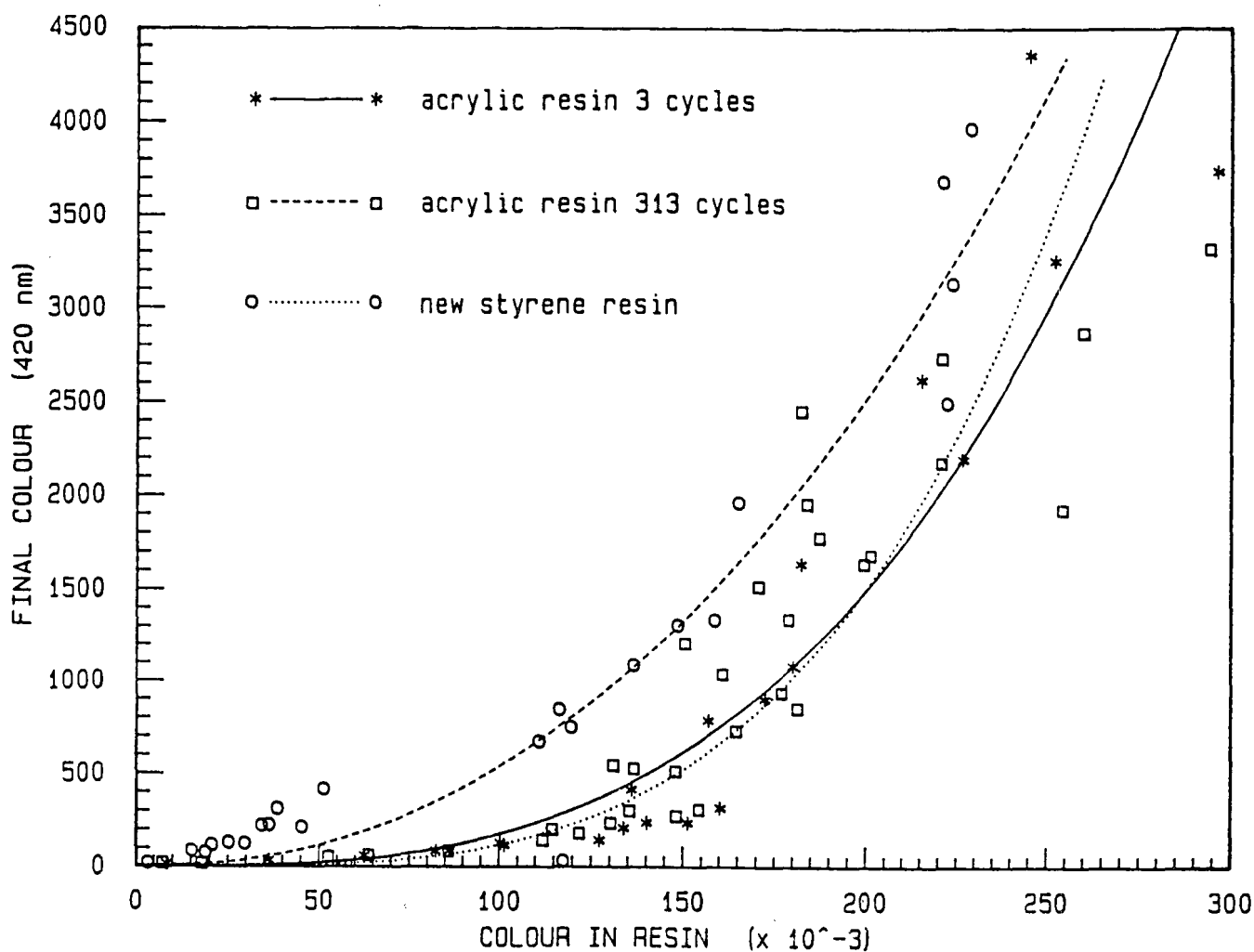


FIGURE 6 Equilibrium Curves for Acrylic and Styrene Resins.

that equilibrium was achieved after six hours. A temperature of 70°C was used to simulate conditions as closely as possible to the Refinery without the destruction of the resin.

Feed liquor was diluted with a white sugar solution to act as feed material at a low colour range (ie feed colour <850) and concentrated with factory syrup for higher colours.

The equilibrium curves for product colour (420 mm) against colour absorbed by the resin are plotted in Figure 6. The tests show that the equilibrium relationship  $Y^* = mX^\beta + b$  for both acrylic and styrene resins is a curve, with  $\beta$  varying from 2,23 to 3,8 depending on the type of resin and the age of the resin.

Table 1

The Equilibrium Curve Parameters

Type of Resin	No. of Cycles Completed	$\beta$	m
Styrene	0	3,8	$5,00 \times 10^{-17}$
Acrylic	3	3,0	$8,47 \times 10^{-14}$
Acrylic	313	2,2	$3,75 \times 10^{-7}$

However, the model has not been altered since, over the limited range of interest (ie final colour <400) a straight line gives a reasonable approximation of the curve, and if  $\beta \neq 1$  the solving of the equations in the model and numerical

calculations would present problems. The values of parameter m obtained using a straight line approximation over the range final colour <400 are shown in Table 2, and compare favourably with those attained at high flow rates from the mathematical model and shown in Figure 4. This is probably due to good liquid-solid contact.

Table 2

Equilibrium Parameters Obtained using Linear Approximation

Type of Resin	No. of Cycles Completed	$\beta$	m
Styrene	0	1	0,0023
Acrylic	3	1	0,0024
Acrylic	313	1	0,0046

### Langmuir Adsorption Isotherms

The adsorption isotherm for acrylic and styrene resins (Figure 7) typifies the group of isotherms which can be described by the Langmuir Equation. The adsorption mechanism for the resins is primarily chemisorption ie the bond between the solid and the first sorbed layer involves the sharing of electrons. Because electrons are shared between solid and sorbate molecules, no more chemisorption can occur when the surface of the solid has been covered with enough sorbate to satisfy the residual valency requirement of the surface atoms.

The derivation of the equilibrium sorption isotherm requires:

1. All the surface has the same activity for adsorption ie energetically uniform.
2. There is not interaction between adsorbed molecules. This means that the number of molecules on a site has no effect on the rate of adsorption to that site.
3. That only one molecule can be accommodated on a sorption site and that the molecules are free to move on the surface.
4. Extent of adsorption is less than one complete monomolecular layer on the surface.

The Langmuir isotherm is derived in Appendix 2 and given by

$$q_e = \frac{QKc_e}{1 + Kc_e} \quad \dots (3)$$

- where  $q_e$  = concentration of adsorbed solute on the sorbent (m mol/g)  
 $Q$  = asymptotic maximum solid phase concentration (m mol/g)  
 $K$  = the adsorption equilibrium constant  
 $c_e$  = the concentration of solute in equilibrium solution (m mol/g)

Plotting  $1/q_e$  versus  $1/c_e$  allows us to determine the two constants  $Q$  and  $K$  in the Langmuir adsorption isotherm equation.

The Langmuir isotherms are

1. new styrene resin

$$q_e = \frac{1670,8c_e}{1 + 29416,3c_e} \quad \dots (4)$$

2. acrylic resin 3 cycles old

$$q_e = \frac{1674,4c_e}{1 + 28851c_e} \quad \dots (5)$$

3. acrylic resin 313 cycles old

$$q_e = \frac{208,77c_e}{1 + 2545,95c_e} \quad \dots (6)$$

and are now plotted in Figure 7. From Equation (3) the fraction of the resin surface covered by the adsorbed molecules is given by

$$\frac{q_e}{Q} = \frac{Kc_e}{1 + Kc_e} \quad \dots (7)$$

Due to the good agreement between the Langmuir adsorption isotherm and the batch test results, it seems that the mechanism of the acrylic and styrene resins is adsorption of a colour molecule on a sorption site, with negligible interaction between adsorbed molecules.

#### Decolourising of Feed Liquor using Columns in Series

At the Refinery, a series of comparison tests are being done in the pilot plant using acrylic/acrylic and acrylic/styrene columns in series. Acrylic resin which had completed

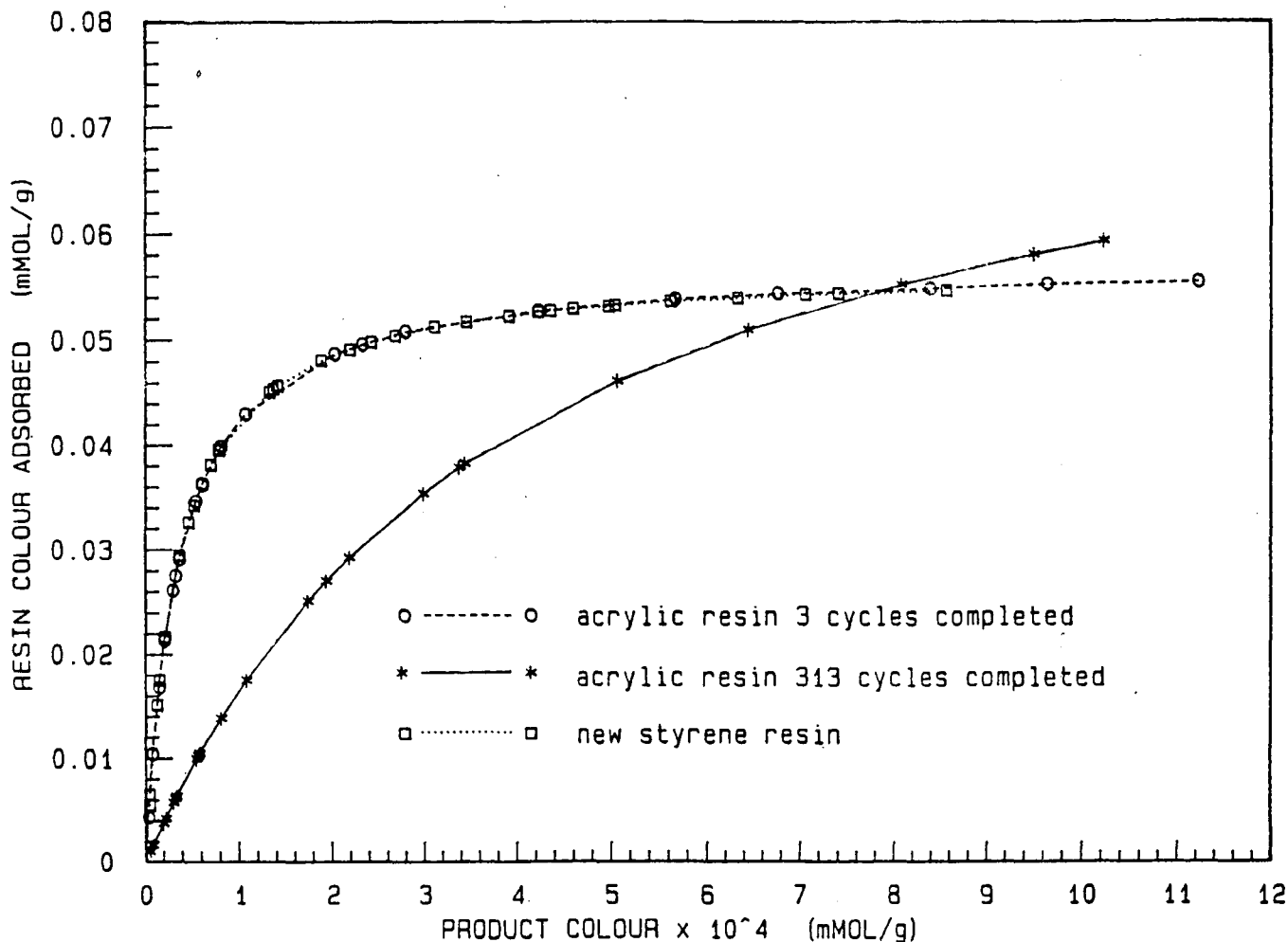


FIGURE 7 Langmuir Adsorption Isotherms.

106 cycles prior to the beginning of the tests is used in both first columns. New acrylic resin (IRA-958) and styrene resin (IRA-900) were used initially in the second column.

The objectives of the runs are:

1. determine the advantages and disadvantages of operating columns in series
2. compare the performance of the styrene resin and acrylic resin in the second column during the life span of the resins.

Feed liquor is fed to the first column, at 3BV/HR and at 80°C. Liquid/liquid heat exchangers are installed at the entrance to each column, to ensure a constant feed temperature at 80°C.

The results obtained from the acrylic/acrylic and acrylic/styrene columns are shown in Figure 8. It is apparent from the graphs that the acrylic/styrene columns perform better straight after acid wash than the acrylic/acrylic columns; but tend to foul more rapidly and as a result need to be regenerated with acid more often. The effect of the more frequent acid washes on the life span of the styrene resin still has to be determined. The acrylic/acrylic pair seems to give a more predictable and stable performance.

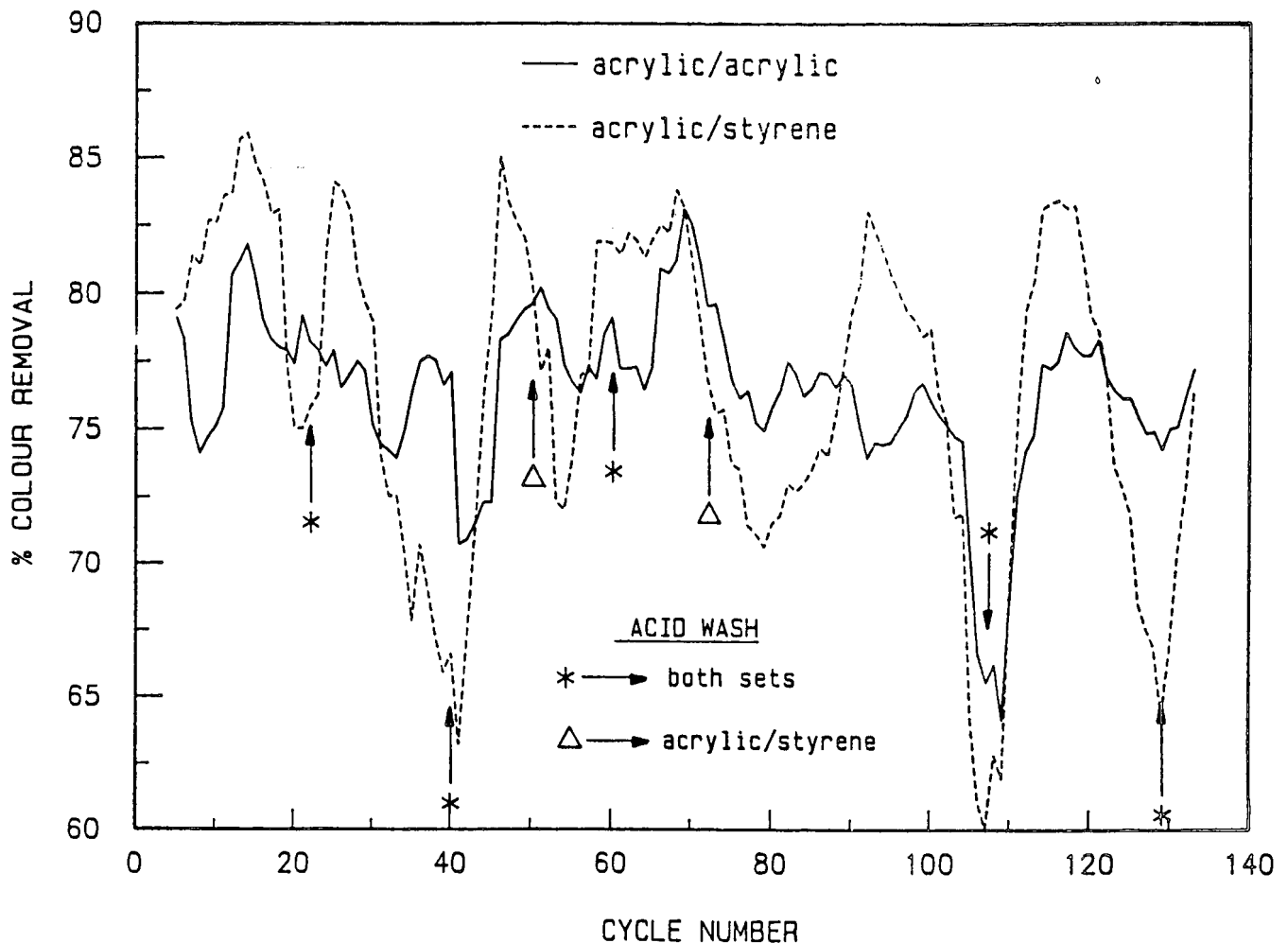
During a few selected runs samples were taken at regular time intervals after both the first and second columns, and analysed for colour. These data were then used in Regula to determine parameters for columns in series. The results from the model are given below in Tables 3 and 4.

**Table 3**  
Results of REGULA on Acrylic/Acrylic Data

Column No.	No. of cycles completed	Length of Cycle Hrs	Parameter m	Parameter $k_t a$	Correlation Coefficient
1st Acrylic	121	40	0,0035	5120	0,92
2nd Acrylic	15	40	0,0040	2503	0,97
1st Acrylic	173	20	0,0042	6811	0,97
2nd Acrylic	67	20	-0,0081	1131	0,89
1st Acrylic	181	20	0,0049	5392	0,90
2nd Acrylic	75	20	0,0029	1739	0,98
1st Acrylic	185	20	0,0040	5827	0,95
2nd Acrylic	79	20	0,0010	1703	0,98
1st Acrylic	225	20	0,0061	4907	0,99
2nd Acrylic	119	20	0,0047	1866	0,99

**Table 4**  
Results of REGULA on Acrylic/Styrene Data

Column No.	No. of cycles completed	Length of Cycle Hrs	Parameter m	Parameter $k_t a$	Correlation Coefficient
1st Acrylic	121	40	0,0029	5020	0,84
2nd Acrylic	15	40	-0,0001	2959	0,91
1st Acrylic	173	20	0,0100	8601	0,85
2nd Acrylic	67	20	-0,0092	2054	0,74
1st Acrylic	181	20	0,0065	5696	0,97
2nd Acrylic	75	20	0,0138	904	0,98
1st Acrylic	185	20	0,0084	1193	0,97
2nd Acrylic	79	20	0,0037	5851	0,97
1st Acrylic	225	20	0,0055	4720	0,98
2nd Acrylic	119	20	0,0007	1988	0,95



**FIGURE 8** % Colour Removal of Columns in Series (5 Cycle average).

The values of parameter  $m$  and  $k_L a$  for the 1st acrylic columns are in line with those predicted for a single column (Figures 3 and 4), when there has been a consistent decrease in % decolourisation with time as expected. If this is the case, the parameters of the 2nd column show the predicted trend, but have lower values of  $m$  and  $k_L a$ . However, if the performance of the 1st column is erratic the value of the parameters for both columns become nonsense, since the performance of the 2nd column depends on the first.

A further advantage of acrylic resin in this situation is that it is cheaper than styrene resin and offers more flexibility, in that old resin can be cascaded from secondary to primary stage, with newer resin being used in the final stage of colour removal.

As a result of this work, it was decided to install a second stage of acrylic resin after the existing acrylic resin columns.

### Conclusions

A mathematical model of an ion-exchange column has been derived and found to be an accurate description of the actual behaviour of a column. The model predicts the % decolourisation obtained in the column when varying certain operating parameters.

At present Hulett Refineries operate their ion-exchange plant using two stages of acrylic resin in series with a flowrate of 2,4 BV/HR in the 1st column and 1,9 BV/HR in the 2nd column due to different resin bed heights. From the results obtained from the model and pilot plant this would seem to be in the region of optimum colour removal.

The equilibrium batch test results agree well with the Langmuir Adsorption Isotherm and the mechanism of the acrylic and styrene resin seems to be adsorption of a molecule on a sorption site with negligible interaction between adsorbed molecules.

The results obtained from the pilot plant show that the acrylic/styrene columns in series initially performed better than the acrylic/acrylic columns; but as the number of cycles increases the acrylic/acrylic columns are out performing the acrylic/styrene columns. The styrene resin appears to be fouling irreversibly more quickly and has to be acid regenerated more often.

### Acknowledgements

The co-operation and assistance of the staff of Huletts' Refinery and the Operation Research Department is gratefully acknowledged.

### Nomenclature

- $a$  = specific interface surface area/packed volume ( $m^2/m^3$ )
- $C_e$  = equilibrium product colour (m Mol/g)
- $C_i$  = feed colour (m Mol/g)
- $G$  = mass velocity of liquid stream ( $kg/hr m^2$ )
- $h$  = hold up liquid phase in the tower ( $kg/m^3$ )
- $H$  = hold up of solid phase in the tower ( $kg/m^3$ )
- HTU = height of transfer unit (m) ( $HTU = G/k_L a$ )
- $I_0$  = modified Bessel function
- $k$  = rate constant of resin
- $K$  = equilibrium constant =  $k/k'$
- $k_L$  = mass transfer coefficient

$$\left[ \frac{(\text{colour transferred})/m^2hr}{\left( \frac{\text{colour transferred}}{\text{total colour}} \right)} \right]$$

- $m$  = defined by the equilibrium relationship  $Y^* = mX^{\beta} + b$
- $n$  = no of transfer units
- $Q$  = asymptotic maximum solid phase concentration (m Mol/g)
- $qe$  = concentration of absorbed solute on the sorbent (m Mol/g)
- $r$  = hold up ratio  $H/h$
- $t'$  = time measured from the instant the liquid flow first reaches the point in question  $n$  units below the top of the column,  $n. t' = t - n\theta$  (hr)
- $\tau$  = dimensionless time variable
- $X$  = absorbed colour/kg solid
- $X_0$  = initial colour units/kg solid
- $Y$  = colour units/kg solution
- $Y_0$  = initial colour units/kg solution
- $Y^1$  = predicted final colour units/kg solution
- $Y^*$  = colour units/kg solution in equilibrium with the resin
- $z$  = height of resin packing (m)
- $\beta$  = exponent on  $X$  in equilibrium relationship
- $\theta$  = time to displace hold up in liquor phase contained in one transfer unit

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### APPENDIX 1

#### Derivation of the Mathematical Model

Consider an element within the packed bed, of height  $dz$  and unit cross-section. Liquid of mass velocity  $G$  ( $kg/hr m^2$ ) flows vertically downwards into this section and the concentration of colour bodies  $Y$  decrease as a result of mass transfer from liquid to resin before flowing out the element (see Figure 1). The concentration of colour bodies in the liquid and resin vary with distance from the top of the bed and time. Radial concentration differences are assumed to be negligible.

A material balance for the solid phase is then

$$H \frac{dX}{dt} = k_L a (Y^* - Y) \quad \dots (8)$$

A material balance for the liquid phase is then

$$h \frac{dY}{dt} = G \frac{dY}{dz} + k_L a (Y^* - Y) \quad \dots (9)$$

The following dimensionless variables are defined:

1. Distance down the column measured by number of transfer units

$$n = \frac{z}{(H.T.U.)} = \frac{k_L a z}{G}$$

where H.T.U. = height of transfer unit (m)

2. Time required for the liquid flow to displace the hold up in the liquor phase contained in one transfer unit

$$\theta = \frac{h (H.T.U.)}{G}$$

3. The hold up ratio  $r = H/h$

Equation (8) and (9) can be rewritten as

$$\theta r \frac{dX}{dt} = - (Y^* - Y) \quad \dots (10)$$

$$\theta \frac{dY}{dt} = \frac{-dY}{dn} + (Y^* - Y) \quad \dots (11)$$

4. These equations may be simplified using a time variable

$$t' = t - n\theta$$

where  $t'$  represents time measured from the instant the liquid flow reaches the point in question  $n$  units below the top of the packed bed. In terms of the new independent variables, equations (10) and (11) become respectively

$$r\theta \frac{dX}{dt'} = -(Y^* - Y) \quad \dots (12)$$

$$\frac{dY}{dn} = (Y^* - Y) \quad \dots (13)$$

Equations (12) and (13) can be simplified by the introduction of the dimensionless time variable

$$\tau = \frac{mt'}{r\theta} = \frac{mG(t - n\theta)}{H(H.T.U.)}$$

which leads to the differential equations

$$m \frac{dX}{d\tau} = -(Y^* - Y) \quad \dots (14)$$

$$\frac{dY}{dn} = (Y^* - Y) \quad \dots (15)$$

$m$  is defined by the equilibrium relationship  $Y^* = mX^\beta + b$

Assume  $\beta = 1$ :  $Y^* = mX + b \quad \dots (16)$

It is assumed the concentration of adsorbed colour is uniform when time equals zero and the feed liquor entering the bed has a constant colour.

The boundary conditions are

$$X(n, 0) = X_0 = \text{const} \quad \dots (17)$$

$$Y(0, \tau) = Y_0 = \text{const} \quad \dots (18)$$

It is required to solve for  $Y(n, \tau)$  from the set of partial differential equations (14) and (15) for the boundary conditions equations (17) and (18). The final result is

$$Y(n, \tau) = (mX_0 + b) + (Y_0 - mX_0 - b) [1 - e^{-\tau}] \int_0^\tau e^{-n} I_0(2\sqrt{\tau n}) dn \quad \dots (2)$$

## APPENDIX 2

### Derivation of Langmuir Adsorption Isotherm

In the system of solid surface and liquid, the molecules of the liquid will be continually striking the surface and a fraction of these will adhere. However, because of their kinetic, rotational and vibrational energy, the more energetic molecules will be continually leaving the surface. An equilibrium will be established such that the rate at which molecules strike the surface and remain for an appreciable length of time, will be balanced by the rate at which molecules leave the surface.

If  $Q$  represents the concentration corresponding to a complete molecular layer on the resin,  $C_e$  is the concentration of solute in product solution and  $q_e$  is the concentration of adsorbed solute on the sorbent, then the rate adsorption is

$$r_a = k C_e (Q - q_e) \quad \dots (19)$$

where  $k$  is the rate constant of the resin

The rate of desorption will be proportional to the fraction of covered surface

$$r_d = k_1 q_e \quad \dots (20)$$

At equilibrium the rates given by equations and are equal and solving for  $q_e$  the result is called the Langmuir isotherm.

$$q_e = \frac{Q K C_e}{1 + K C_e} \quad \dots (21)$$

where  $k/k_1$  is the adsorption equilibrium constant.

In order to convert concentrations in colour units ( $X, Y, Y_0$ ) into concentrations of  $m$  mol/g ( $q_e, C_e, C_i$ ) an average conversion factor of 7.75 ICUMSA/ppm of phenolics was used. This number was derived from data reported by Clarke<sup>2</sup> and an average molecular weight of 50 000 was taken for the phenolic colour compounds.<sup>3</sup> Clarke<sup>2</sup> *et al* stated that approximately two thirds of the colour in raw sugar came from phenolics and the flavonoid group. The molecular weight value used was the best figure obtained from a literature survey at the time.