

SOLVENT EXTRACTION OF SUCROSE FROM SUGAR CANE PITH (COMFITH)

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Summary

A three parameter model has been presented to describe the process of solvent extraction from a packed bed of cellular plant material to a percolating solvent. An experimental programme of work extracting sucrose from sugar cane pith (Comfith), using water as solvent, has shown the model to be valid and values for the model parameters were determined. The rate of extraction was found to be strong function of water flowrate but not temperature. The work showed that the use of Comfith has certain advantages, over that of the whole cane stalk as a raw material for sugar production.

1. INTRODUCTION:

The sugar cane stalk is composed of three portions as follows:—

Rind — This is the hard outer portion of the plant consisting of a thick epidermis with a strong outer cuticle which is usually covered with a layer of wax. It provides rigidity and strength to the plant, also giving protection to the softer internal tissues.

Pith — This is the inner soft tissue made up of thin walled, easily ruptured, sheetlike cells; their main function being the storage of sucrose rich juice.

Vascular Bundles — These are groups of tissues of a more fibrous nature located between the rind and the pith. They serve to transport material for the plant metabolism.

In current sugar cane processing operations, the cane stalk containing all three portions identified above is prepared by knifing or shredding. The sucrose rich juice is then extracted by a series of three roll mills, or by a diffusion process, water being used as the extracting solvent in both cases.

A machine has however been developed in Canada [1] whereby sugar cane pith can be separated from the rest of the stalk. In the machine, the cane stalk is first directed towards a splitter blade where the cane is split longitudinally into two halves. Each half then passes to its own separator section where the pith is scraped out from the inside by a set of knives or scraper blades, in such a way that the pith, called Comfith, is collected separately from the rind. Since the Comfith contains the major portion of the sucrose content of the cane and the rind contains those substances (e.g. waxes, gums, inorganics) which give rise to processing problems during standard operations, it has a significant potential as a raw material for a sugar producing operation. In addition, Comfith is finely divided, thereby being ideally suited to a simple packed bed process, whereby the solvent, water, percolates by gravity through the material.

In the operation of such an extraction operation it may be assumed that a certain proportion of the sucrose is present in the Comfith in such a way that it would be washed away in the flowing solvent, the mechanism thus being one of convection.

The remainder of the sucrose will be held within the material and it will be necessary for this to be transferred initially to an area washed by the water before leaving the material. The most convenient transfer mechanism to apply to this portion of the sucrose is the diffusion model. This model for internal transfer has been used for plant materials [2], but it very much over simplifies the situation of internal transfer within the material of cellular structure, such as Comfith.

The basic aims of the work described in this paper were to:—

- (a) Develop a qualitative mechanism to describe sucrose transfer from Comfith during extraction with water.

- (b) Investigate the applicability of a generalized analysis for solvent transfer from a static packed bed material and to quantify the parameters.
- (c) Evaluate the suitability of Comfith as the raw material for sugar production.

2. THEORY

2.1 General Approach

The material is assumed to be composed of a network of cells. As a result of the action of the cane separation, it is likely that the cells on the surface of the material will be ruptured, as will a certain proportion of the cells in the interior of the material. The rest of the cells in the interior are assumed to be intact.

Extraction is proposed to occur in the following manner. A certain proportion of the solute will be associated with broken cells, the remainder being held in the intact cells. The fraction of the solute in the broken cells, α , will be readily extracted by a washing process, the rate constant being k_1 . The fraction of solute held in the intact cells, $(1 - \alpha)$, will be extracted at a much slower rate, the rate constant being k_2 . It may be anticipated that k_1 would be significantly greater than k_2 .

The process is considered to be one of unsteady state mass transfer in a static packed bed. The solvent, at zero solute concentration, is fed continuously to the top of the bed of material at a constant rate, and flows down through the bed by the action of gravity. At a particular position in the bed, the concentration of solute both in the solvent and in the material decreases continuously with time. At a particular point in time the concentration of solute both in the solvent and the material increases with distance from the top of the bed. These characteristics will apply until all the solute has been extracted from the bed. It is assumed that there are no stagnant zones in the bed and no lateral variations across the bed.

In the analysis, the bed is split up into a finite number of elements. A numerical analysis is carried out involving successive mass transfer calculations within each element, both as a function of distance from the top of the bed and also of time of extraction.

2.2 Elemental Mass Balance

Consider an element within a packed bed of material of thickness dz and unit cross sectional area.

A solute balance on the liquid phase may be developed as follows:—

$$\text{Input to element} = LC_L$$

$$\text{Transfer within element} = k_1 [C_{m1} - C_L] dz + k_2 [C_{m2} - C_L] dz$$

$$\text{Output from element} = L [C_L + \frac{\delta C_L}{\delta z} dz]$$

..... (1)

This results in:-

$$L \frac{\delta C_L}{\delta z} = k_1 [C_{m1} - C_L] + k_2 [C_{m2} - C_L]$$

The rate transfer of solute from the broken cells may be given by:-

..... (2)

$$-\alpha H_s \frac{\delta C_{m1}}{\delta t} = k_1 [C_{m1} - C_L]$$

The rate of transfer of solute from the intact cells may be given by:-

..... (3)

$$-[1 - \alpha] H_s \frac{\delta C_{m2}}{\delta t} = k_2 [C_{m2} - C_L]$$

Dimensionless quantities are defined as follows:-

$$N = \frac{k_1}{L} z \quad \dots\dots (4)$$

$$\theta = \frac{k_1}{H_s} \left(t - \frac{H_D}{L} z \right) \quad \dots\dots (5)$$

$$R = \frac{k_2}{k_1} \quad \dots\dots (6)$$

The quantity N may be considered to be the bed height measured in terms of a number of transfer units.

The quantity $\frac{H_D z}{L}$ in Equation (5) is the time required to displace the dynamic holdup in the column of height z. Consequently, θ represents the dimensionless time during which a bed element, a distance z from the bed inlet, has been contacted by flowing liquid.

The basic Equations (1) to (3) may thus be written respectively as follows:-

$$\frac{\delta C_L}{\delta N} + C_L (1 + R) - C_{m1} - RC_{m2} = 0 \quad \dots\dots (7)$$

$$\alpha \frac{C_{m1}}{\delta \theta} + C_{m1} - C_L = 0 \quad \dots\dots (8)$$

$$(1 - \alpha) \frac{\delta C_{m2}}{\delta \theta} + R (C_{m2} - C_L) = 0 \quad \dots\dots (9)$$

This approach is based on that originally proposed by Rein [3]

2.3 Packed Bed Analysis

When using Equations (7) to (9) to develop the analysis for the static packed bed situation, it was assumed that the solute concentration in the material was initially constant throughout the bed and also that the solvent entered the bed solute free. The following boundary conditions therefore apply:-

$$\begin{aligned} \text{At } N = 0 \quad C_L &= 0 & \text{for all } \theta \\ \theta = 0 \quad C_{m1} &= C_{m2} & \text{for all } N \end{aligned}$$

The analysis splits the bed up into K sections as shown in Figure 1.

Equations (7) to (9) are rewritten respectively in a form suitable for numerical analysis as follows, reference being made to element J in Figure 1:-

$$C_L (J + 1) = C_L (J) + \Delta N \{ C_{m1} (J) + RC_{m2} (J) - C_L (J) [1 + R] \} \quad \dots\dots (10)$$

$$\frac{dC_{m1} (J)}{d\theta} = [C_L (J) - C_{m1} (J)] / \alpha \quad \dots\dots (11)$$

$$\frac{dC_{m2} (J)}{d\theta} = \frac{R}{1 - \alpha} [C_L (J) - C_{m2} (J)] \quad \dots\dots (12)$$

Equations (10) to (12) are solved for each section, the Runge - Kutta Fourth Order method being used for solving Equations (11) and (12).

The algorithm proceeds as follows:-

- (a) Equations (10) to (12) are solved at K points for $\theta < B$ in the programme, where B is the maximum dimensionless time.
- (b) $C_L (K + 1)$ (i.e. juice concentration at bed outlet) for $\theta = 0$ is obtained by extrapolation using seven points at $\theta = 1 \dots 7$.
- (c) The θ values are converted to real time and an interpolation carried out to compare experimental extract liquid concentrations with the calculated values.

In the use of the technique, a series of values of k_1 , k_2 and α were fed into the programme, the best fit between the model prediction and the experimental extraction curve being obtained by minimising the squares of errors between the two.

3. RAW MATERIAL CHARACTERIZATION:

In order to characterize the raw material, the physical and structural characteristics of the insoluble solids portion of the pith were examined by sieve analysis, determination of Preparation Index, and microscopic examination.

Sieve analyses indicated that the bulk of the material was between 3.35mm and 0.5mm with a mean size of ~ 1.2 mm.

The Preparation Index is effectively a measure of the percentage of broken cells in the material. This was found to vary between 95 and 98% for the material used in the experimental programme.

Slides of dried material from the various fractions in the screen analysis were made up and examined at a magnification of about 100. The slides were prepared in such a way as to provide both longitudinal and cross section views of the cell structure. A typical photograph of a cross section is shown in Figure 2 and a side view in Figure 3.

More complete details of the material characterization have been reported elsewhere [4].

4. EXPERIMENTAL METHOD

4.1 General Description of Extraction System

The experimental work was carried out in a simple 1m long vertically oriented 38mm internal diameter glass column. The pith, which virtually filled the column for each run was supported by a mesh screen at the column base. The solvent, water, was heated to the required temperature in a water bath prior to being pumped to the top of the column, where it was distributed over the full cross section of the bed by use of a nozzle. In order to prevent heat losses when operating above ambient temperatures, an electrical heating tape was wrapped around the column. All experiments were carried out on a 'once through' basis with continuous addition of water at the top of the bed and continuous removal of sucrose juice from the base of the bed.

4.2 Experimental Measurements

The temperature of extraction was measured by the use of thermocouples placed at the top of the bed and on the mesh screen at the base of the bed. The flow rate of water into the column was measured by a rotameter and the outlet flow of juice using measuring cylinders and a stop watch. The bed depth was measured with a ruler.

The soluble solids content of the juice associated with the pith used in each experiment was determined by expressing the juice using a small press and measuring the brix (percentage soluble solids) value with a refractometer. The pith moisture content was determined by use of an infrared moisture balance.

Extraction curves were determined by taking samples of extract juice leaving the bed at various times intervals. The brix value of each sample was measured with the refractometer.

5. EXPERIMENTAL RESULTS

Three series of experiments were carried out as follows:-

- (i) Fixed Temperature 27°C — Varying flowrate 0.0029 to 0.0118 m³ s⁻¹ m⁻² cross section.
- (ii) Fixed Temperature 80°C — Varying flowrate 0.0029 to 0.0118 m³ s⁻¹ m⁻² cross section.
- (iii) Fixed flowrate 0.0059 m³ s⁻¹ m⁻² cross section. Varying temperature 27°C to 80°C.

The highest flowrate used was just about at the flooding point of the system.

Because of experiment to experiment variations in the initial brix readings the results are presented as reduced brix (extract brix at given time/first extract brix) against time. Zero time was when the first extract left the bed of material. The effect of water flow rate on the extraction curves as carried out in the first series of experiments is shown in Figure 4, where it is seen that the rate of extraction increases significantly with increasing flow rate.

Duplicate experiments were carried out in this series in order to examine the reproducibility of results using the same raw material. Typical comparisons for the highest and lowest flow rates are also shown in Figure 4.

Some variation in extraction curves was noted at different temperatures, but reference to Figure 5 shows no definite trends.

In order to apply the model to the experimental results it was necessary to have values for the static hold-up, H_S , and the dynamic hold-up, H_D . Attempts to obtain hold-up values from residence time distribution determinations using tracer techniques failed due to the tracer being picked up by the material. It was therefore necessary to make some approximations. The static hold-up was taken as the juice content of the pith fed to the bed for each experiment. The dynamic hold-up was taken as the difference between the amount of water fed to the bed and the amount of juice collected during each experiment, both being cumulative.

Values of k_1 , k_2 and α were fed into the computer programme and the best fit values obtained by minimising the squares of errors between the model prediction and the experimental curve in each case. A typical comparison is shown in Figure 6. Calculated values of the parameters are shown in Table 1. Values are not quoted for the highest flow rate because of the proximity to flooding conditions.

Reference to Table 1 shows k_1 to be a strong function of flow rate but not of temperature. This is further demonstrated in Figure 7 where values of k_1 are plotted against flow rate of all temperatures. Dynamic head measurements showed similar trends.

Reference to Table 1 shows a significant variation between calculated values of k_2 but no identifiable trends except perhaps for the possibility of a slight reduction at higher temperatures.

The calculated values of α were always high, between 0.9 and 0.98, no trends being apparent.

6. DISCUSSION

Reference to Figures 4 and 5 shows that the rate of extraction increased significantly with increasing water flow rate, but was not affected by varying temperature. Reproducibility of experiments with the same raw material, demonstrated in Figure 4, was considered to be reasonably satisfactory. The differences between extraction curves were attributed to sampling deviations, errors in measurement and especially differences in packing from run to run, giving rise to varying water irrigation patterns. The additional problems of raw material variation, as demonstrated by the differences in expressed brix readings in Table 1, probably accounted for the wider, seemingly random, variation shown in the experiments at varying temperature.

The rate coefficient k_1 , which is associated with that portion of sucrose transferred by convection, was found to be a strong function of solvent flow rate. This is demonstrated in Figure 7. The rate coefficient k_2 , associated with transfer from intact cells, was not a function of solvent flow rate and, in addition, was found to be significantly lower than that of k_1 . These general trends in the rate constants are predictable from the model. The values of α , the fraction of solvent held in broken cells, as determined from the application of the model were generally in the range of 0.94 to 0.98. These values are directly comparable with the experimental determinations of Preparation Index giving broken cells counts in the range 95 to 98%. All these factors together with the ability of the model to give a very good curve fit, as shown typically in Figure 6, serve to demonstrate the application of the model. The spread in the values of k_1 and k_2 may be attributed to the problem of having to balance three (3) parameters to give the best curve fit to experimental curves which are subjected to some experimental error in addition to raw material variation.

The values of the Preparation Index and α indicate that about 95% of the cells in the material are ruptured. Reference to Figures 2 and 3 only show ruptured cells on the surface of the material. The percentage of ruptured cells on the surface, as indicated by the microscope photographs, as however very much less than the total, varying with the size of material, but typically being 40 to 50%. It appears that the magnification was not sufficient to show ruptured internal cells. The fact that there was a significant proportion of ruptured cells in the interior of the material serves to imply that there are three (3) steps in the extraction process as follows:-

- (i) The rapid displacement of sucrose from the broken cells on the surface by the solution flowing across the material.
- (ii) Displacement of sucrose from broken cells within the material by solution passing through from ruptured cell to ruptured cell. It is postulated that increasing solution flow rate increased the thickness of liquid on the material providing a higher head and thereby a greater flow through the broken cells.
- (iii) Transport of solute through the cell walls of the intact cells. It has been shown by electron microscopy [5] that sugar cane tissue has minute pores in the cell normally used for the transport of matter into the cell. It is postulated that, in extraction, these pores are used to transport matter out of the cell.

The first step probably accounts for 40 to 50% of the extraction thereby being associated with the reduction of the Reduced Brix from unity down to 0.5 or 0.6. The second step accounts for the region from a Reduced Brix of 0.5 or 0.6 down to 0.5. The third step is the slowest of the three and is associated with the extraction of the final 5% of the solution through the walls of the intact cells. In the model k_1 combined (i) and (ii), and k_2 quantified (iii).

It is to be noted that the rates of extraction were extremely rapid, the bulk of the sucrose being extracted within two (2) minutes. This compares very favourably with the extraction of sucrose from knifed or shredded whole cane where extraction times in excess of fifteen (15) minutes were found to be necessary using similar equipment [6]. This large reduction in extraction residence time together with the lack of waxes, gums, inorganics etc. in the juice makes Comfith a potentially attractive raw material for sugar production when compared with shredded whole cane. The analysis presented, together with the parameters determined, can be used as the basis for the design of a full scale extractor.

CONCLUSIONS

The analysis presented can be used to describe the process of mass transfer of solute from a packed bed of cellular material to percolating solvent. The rate constant for transfer of solute from broken cells was found to be a strong function of solvent flow rate but not of temperature whereas that for transfer of solute from intact cells was not shown to vary with either of these variables. Because of its finely divided nature, rates of extraction from sugar cane pith are significantly greater than from whole cane. The analysis in conjunction with the calculated values of the parameters can be used as the basis for the design of a full scale extractor.

NOMENCLATURE

- C_L — Concentration of solute in percolating liquid — kg solute/kg solution
 C_{m1} — Concentration of solute in broken cells in material — kg solute/kg solution
 C_{m2} — Concentration of solute in intact cells in material — kg solute/kg solution
 H_s — Static hold-up of solution — kg solution/m³ column
 H_D — Percolating Liquid hold-up in column — kg solution/m³ column
 k_1 — Mass transfer coefficient for solute from broken cells —

$$\frac{\text{kg solute}}{\text{sec m}^3 \text{ column}} \frac{\text{kg solute}}{\text{kg solution}}$$

- k_2 — Mass transfer coefficient for solute in intact cells —

$$\frac{\text{kg solute}}{\text{sec m}^3 \text{ column}} \frac{\text{kg solute}}{\text{kg solution}}$$

- L — Mass flow rate of percolating liquid —
kg solution/sec m² cross section
- α — Fraction of solute held in broken cells
- z — Depth of bed — m

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TABLE 1
CALCULATED PARAMETER VALUES

EXPERIMENT NO.	FLOW RATE	TEMPERATURE	EXPRESSED JUICE Brix	H _s	H _D	k ₁	k ₂	α
	$\frac{m^3}{s m^2}$	°C	% Soluble Solids	$\frac{kg}{m^3}$	$\frac{kg}{m^3}$	$\frac{kg}{s m^3}$	$\frac{kg}{s m^3}$	—
01 - 29 A	0.0029	27	19.0	196	397	7.0	1.5	0.97
B	0.0029	27	19.1	201	359	5.0	1.4	0.90
02 - 02 A	0.0059	27	16.8	226	456	14.0	2.1	0.94
B	0.0059	27	17.0	232	428	13.5	1.5	0.94
06 - 01 A	0.0088	27	19.6	220	535	24.0	1.5	0.94
B	0.0088	27	19.5	220	579	25.0	2.2	0.94
05 - 18	0.0029	80	20.8	197	328	6.8	1.2	0.95
05 - 09	0.0059	80	22.3	195	448	12.8	1.4	0.97
05 - 14	0.0088	80	20.0	210	532	22.0	1.5	0.94
03 - 07	0.0059	40	20.2	196	506	11.0	2.3	0.98
03 - 09	0.0059	50	19.2	204	478	17.0	2.2	0.98
05 - 01	0.0059	60	20.0	195	455	11.0	1.2	0.97

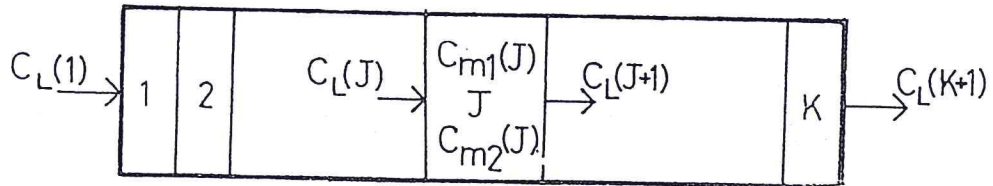


Fig. 1: Sections for Numerical Analysis

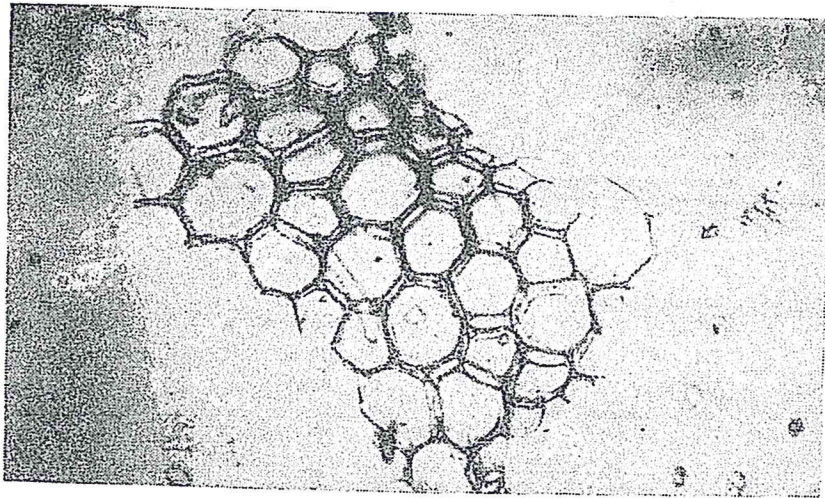


Fig. 2: Cross Section of Sugar Cane
Pith: Magnification 100

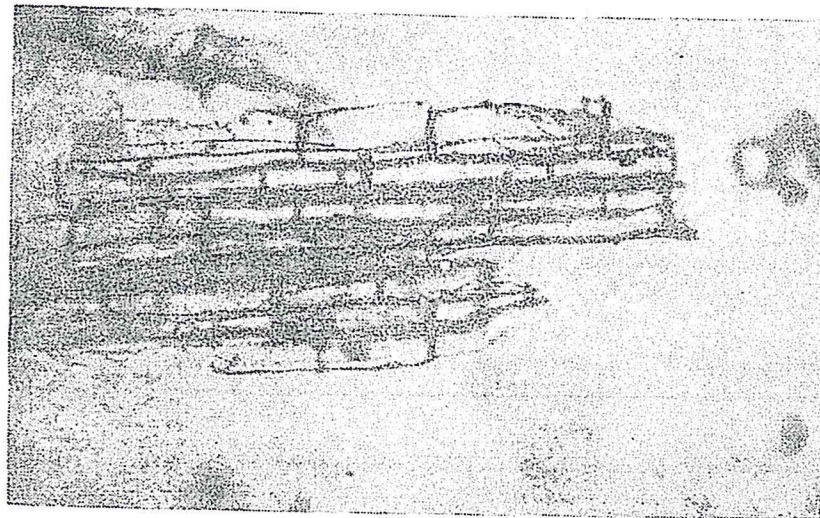


Fig. 3: Side View of Sugar Cane
Pith: Magnification 100

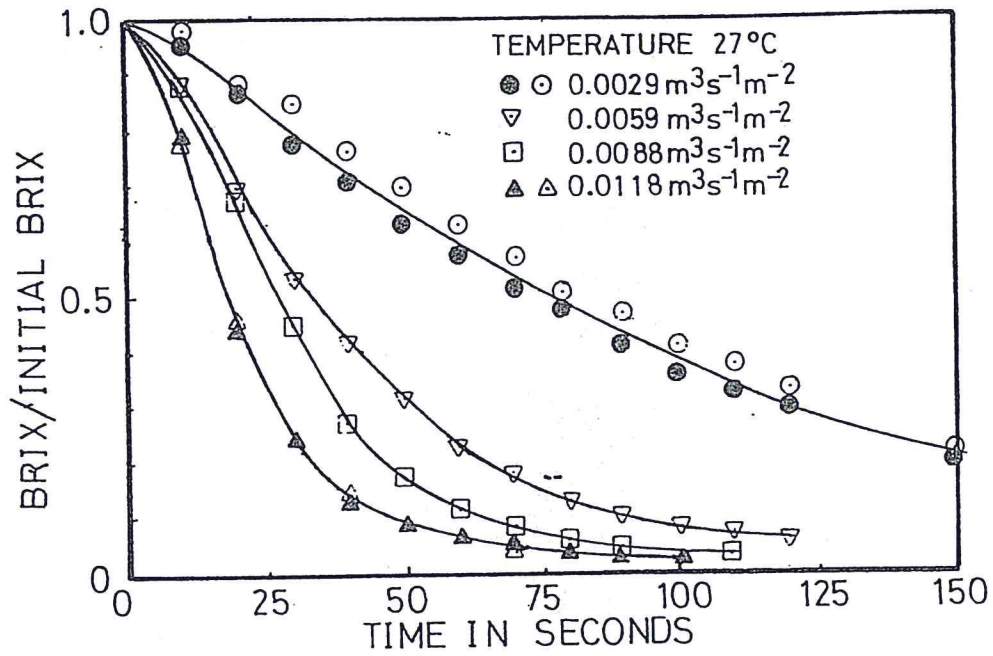


Fig. 4: Extraction Curves Showing the effect of flow rate

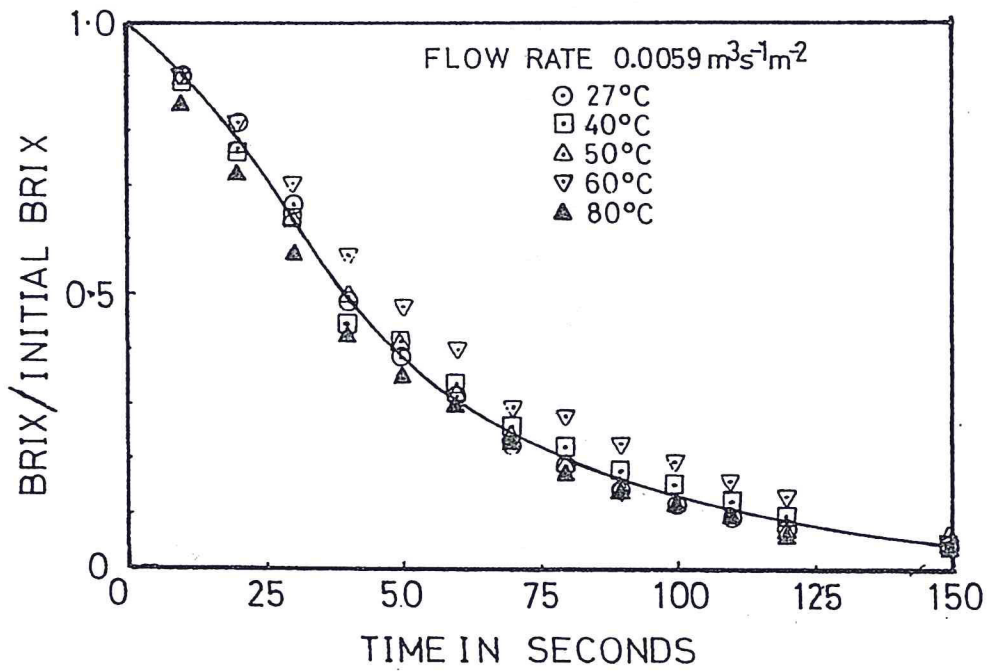


Fig. 5: Extraction Curves Showing The effect of temperature

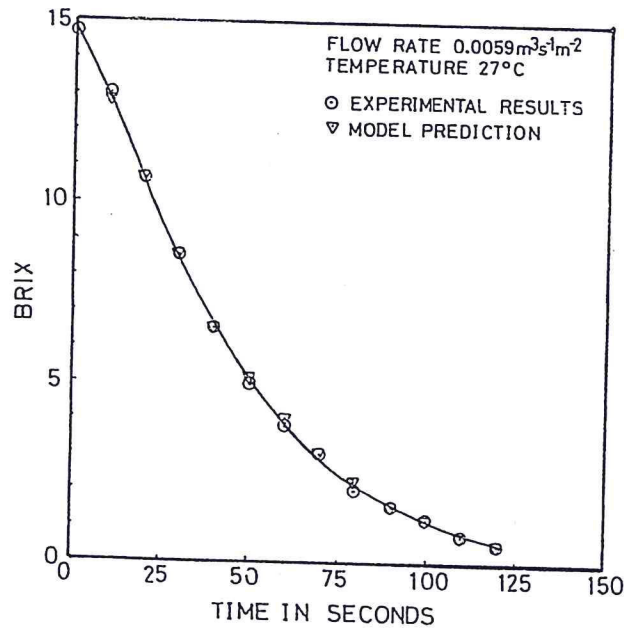


Fig. 6: Comparison of Model Prediction With Experimental Curve

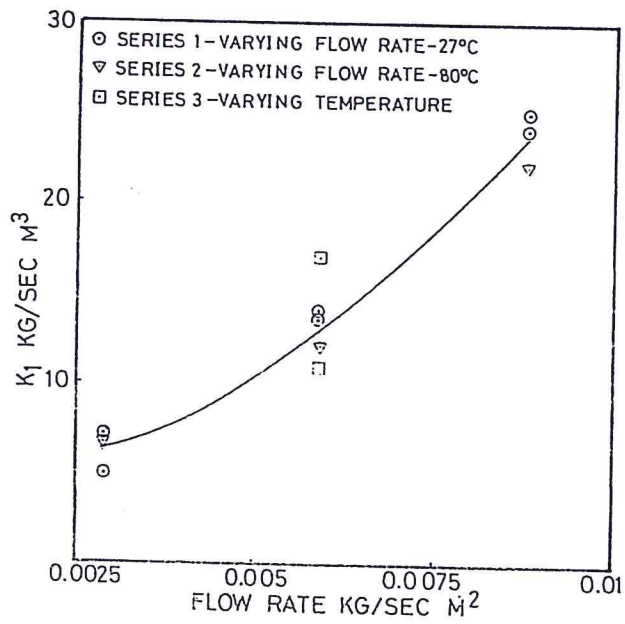


Fig. 7: Plot of K_1 Against Flow Rate