

SOME NOTES ON THE EXTRACTION OF SUCROSE FROM CANE BY DIFFUSION

By C. VAN DER POL

By the term diffusion, as applied to the cane sugar industry, we understand the recovery of sugars from cane tissue by liquid extraction, as opposed to mechanical expression of the juices in conventional milling practice.

The sugar juices in the cane tissue are enclosed in cells, which in live cane are not permeable to the sugars. In order to allow the sugars to diffuse through the cell wall, the latter has to be made partially permeable, which can be most easily achieved by heating to above 75°C. At this temperature the colloids, of which the cell wall is made up, are precipitated and the true solutes of comparatively low molecular weight, are free to move through the tissue towards the surrounding extraction liquid. In this manner a juice free from impurities of high molecular weight can be recovered since the cell wall acts as a molecular sieve. This is a decided advantage over the mechanical expression method of extraction, where the cells are broken and the whole contents are squeezed out.

The driving force behind the movement of the solute molecules from within the cane tissue to the surrounding liquid is the difference in concentration between the juice inside the tissue and that surrounding the tissue. No useful diffusion can take place when these concentrations are equal. The movement of molecules through the solution inside the tissue is slow, and it is clear that the shorter the path along which the molecules have to move, the quicker the process. High temperatures increase the rate of movement of molecules and hence speed up the rate of diffusion. Also the larger the area of contact between the tissue and the surrounding liquid, the quicker the concentration difference can be equalised. Since diffusion stops when the concentration gradient has disappeared, it is necessary for rapid diffusion that the concentration of the liquid surrounding the cane is always kept at a minimum value. Also rapid movement of this solution past the exposed tissue surface minimises the possibility that a thin film of high concentration is built up in the immediate vicinity of the diffusion interface. Shortening the path of the diffusing molecules and increasing the diffusion interfacial area is simultaneously achieved by a fine preparation of the cane. This unfortunately cannot be accomplished without mechanical rupture of some juice cells which in turn allows the high molecular non-sugars to escape into the diffusion juice.

The above considerations enable us to lay down certain fundamental requirements for both the

condition of the cane and the design of a plant and give us the material with which to build up an equation which governs the diffusion process.

Plant Requirements

(a) *For preparation of the Cane*

As will be shown later, the rate of diffusion is inversely proportional to the square of the length of the path along which diffusion takes place. Hence not only should the cane dimensions be small for rapid diffusion but the regularity of the size must also be controlled with great exactitude for maximum efficiency. Coupling to this, the further requirement of minimum rupture of cells, it is not surprising that the problem of cane preparation for diffusion is still a long way from solved.

It would appear as if the general attitude of workers in this field towards this problem is to forego the advantage of higher purity of juices and to concentrate on a high efficiency of sucrose extraction. If this attitude is economically sound, a set of knives followed by a shredder of the type already used in the preparation of cane for milling should fulfill the requirements adequately. Furthermore since about 50 per cent of the juice in cane can be recovered in a single three-roller mill with comparative ease, it will undoubtedly be an advantage to place one mill in front of the diffuser. This would further assure even preparation and would reduce the load on the diffuser by 50 per cent, from which a substantial increase in extraction of sucrose can be expected, as will be shown later.

(b) *For the Diffuser and its Accessories*

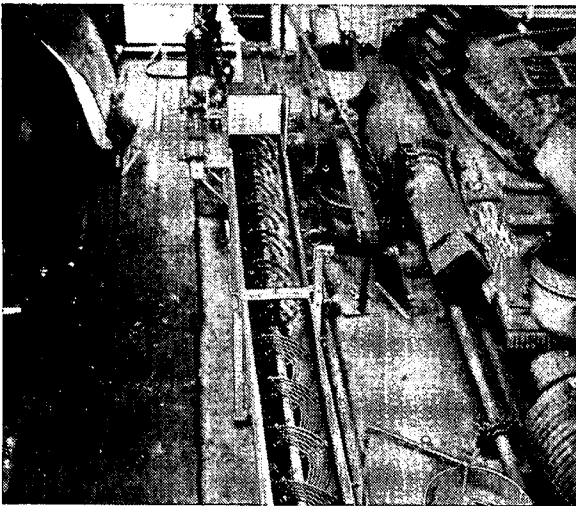
In order to kill any cells which have escaped rupture in the preparation of the cane for diffusion, the prepared cane must be brought to a temperature of not less than 75°C. There appears to be little reason, other than steam economy, why this temperature could not be taken to 100°C, since the structure of cane fibre is not such that it will soften as does beet fibre at temperature around 100°C. This scalding of the cane can be done in a variety of ways, either inside or outside the diffuser.

The requirement of maximum concentration difference between the juice inside the cane tissue and that surrounding it, is best fulfilled in continuous countercurrent operation. The cane must be transported along the diffuser in such a manner that feed-back is prevented and also no channelling of juice should occur, since both these factors mitigate

against the existence of a maximum concentration difference at all points within the diffuser. The cane must further be fully submerged in the diffusing liquid at all times to ensure maximum area of contact between the cane and the surrounding juice. The volume of the diffuser must also be a minimum to ensure a maximum rate of movement of the diffusing liquid past the cane surfaces.

These requirements have already been fulfilled, partly or wholly, in diffusers in commercial use in the beet sugar industry. Since cane fibre is an entirely different material from beet pulp, the mechanical properties of fibre, such as its greater hardness and its tendency to felt, will have to be given special consideration when modifying a sugar beet diffuser design to the cane sugar industry.

PILOT PLANT DIFFUSER



Top view of Diffuser showing Single Scroll Screw Conveyor

The extracted cane has a fibre content of about the same order as the original cane and it will have to be brought to the composition of bagasse before it can be used as fuel in the boilers. The most convenient way of achieving this is to pass it through a three-roller mill, although other types of expressors are available. The expressed liquid will contain some sugars in concentration varying with the degree of efficiency obtained up to that point, and it can be returned to the diffuser at the appropriate point.

The Diffusion Equation

Fick's fundamental diffusion law states:

$$ds = k \cdot \frac{T}{\eta} \cdot A \cdot \frac{dc}{dx} \cdot dt,$$

in which ds is the weight of sugar diffusing in the time interval dt across an area A ; dc is the difference

in concentration across a diffusion path of length dx ; T is the absolute temperature, η the viscosity of the solution and k a proportionality constant.

Upon integration and substitution the equation can be developed to:

$$S = K \times \frac{T}{\eta} \times \frac{t}{d^2} \times \frac{(C_1 - J_1) - (C_2 - J_2)}{\log \frac{C_1 - J_1}{C_2 - J_2}} \dots (A)$$

in which S = weight of sucrose diffused per unit weight of cane

T = absolute temperature at which diffusion takes place

η = viscosity at the diffusion temperature

t = time interval in minutes during which diffusion takes place

d = average thickness in cm. of the cane chips in the direction of diffusion

C = concentration of sucrose in juice in cane

J = concentration of sucrose in juice surrounding the cane

suffix 1 = conditions at the cane inlet end of the diffuser

suffix 2 = conditions at the cane outlet end of the diffuser

K = proportionality constant independent of the other variables.

Equation (A) can be further simplified and written in terms of variables more easily assessed than the above. For counter-current conditions, adding pure water as the solvent,

$$C_n = \frac{n-1}{n} \times \frac{C_0}{\left[\text{Antilog} \left(K \times \frac{T}{\eta} \times \frac{t_2}{d} \times \frac{(n-1)}{n} \right) \right] - 1} \dots (B)$$

where C_n = Pol per cent extracted cane as it leaves the diffuser

C_0 = Pol per cent cane

n = $\frac{\text{weight of diffusion juice}}{\text{weight of extractable juice in cane}}$

= $\frac{\text{water added per cent cane}}{100 - \text{fibre per cent cane}}$

= draft

and other symbols as before.

Now $(1 - \frac{C_n}{C_0}) \times 100$ = per cent extraction, which

from equation (B) can be completely evaluated from the draft n , and the physical variables T , η , t and d , provided K is known.

The constant K is extremely useful in diffusion work in that it can be used to define the efficiency with which a diffusion operation is carried out. Once an absolute value for K has been determined, that is, under perfectly ideal conditions, the deviation of any other value of K obtained with a particu-

lar diffusion process from this absolute value is a measure of the inefficiency of the process.

The equation also involves the term d , i.e. the average thickness of the cane chips in the direction of the diffusion. For shredded cane it will be impossible to determine this variable directly and some inferential method will have to be found for its evaluation. However, the equation can still be used to calculate the effect of changes in the other variables on the diffusion process, provided d can be assumed to remain constant.

Laboratory Investigations

The aim of the laboratory investigations was to establish the validity of the equation for diffusion in cane and also to determine an absolute value for the constant K . Neither of these aims were fully realised. The difficulties encountered were twofold. Firstly the extremely high degree of accuracy required made reproducibility of results very difficult and secondly the role played by brix-free water in determining the concept "extractable juice in cane" has not been fully elucidated. However, the work will be continued and the results so far obtained do suggest the equation to be valid over a wide range of extraction values. The value of K was found to be about 2×10^{-5} . Using this value of K it was deduced that a cane chip thickness not exceeding 0.20 cm. would be required to reach 96 per cent extraction in one hour, with a diffusion juice ratio of 110 percent on cane, and 15 per cent fibre in cane. (Australian workers have reported 95 per cent extraction by diffusion at 180°F for 60 minutes with a slice thickness of $\frac{1}{16}$ inch.)

If now 50 per cent of the juice in cane is extracted by crushing prior to the diffusion process, 98.9 per cent overall extraction could be expected, other conditions being the same.

Pilot Plant Investigations

The plant used in these investigations was the one designed and built by Mr. Saville of Entumeni Sugar Milling Co. This plant consists essentially of a single scroll horizontal screw conveyor and with shredded cane has a capacity of about 600 lb. cane per hour. The first half of the trough is steam jacketed which allows the whole of the contents of the diffuser to be maintained at 95°C.

The cane was prepared by shredding in a type of fixed-blade hammer mill, since no other device was available. The nature of the prepared cane varied considerably with the type of cane; hard fibred canes yielded a coarser product than soft- or low fibred canes. It is estimated that on the average at least 10 per cent of the feed to the diffuser consisted of cane chips with a minimum dimension greater than half an inch.

It did appear that the single scroll horizontal screw conveyor is not an ideal design for transportation of cane through a diffuser. There was a marked tendency for the cane to be dragged with the screw to the one side of the diffusion trough and channelling of the extracting liquid could not be avoided. Due to the felting characteristic of the cane, this movement of the cane along the diffuser was irregular and some tendency towards back-feed was observed.

The drainage wheel whereby the extracted cane is removed from the diffuser, was of inadequate capacity to cope with the rate of feed required to keep the diffuser full, which resulted in a residue of a much higher water content than would normally be expected.

In spite of these disadvantages, very useful information on the diffusion process as applied to cane was obtained, which can be summarised as follows:

The results of the last 15 experiments, each of about 7 hours duration, gave the following average values in the range indicated:

	Average per cent	Range per cent
Per cent Pol Extraction	86.2	80.9 — 92.7
Per cent Inversion	0.87	0.04— 2.21
Corrected Pol Extraction	87.1	82.4 — 93.4
Pol per cent Extracted cane	1.38	0.75— 2.13
Diffusion Residue per cent cane	135	128 — 145
Juice per cent Cane	118	98 — 173
Residence time (min.)	53	42 — 65

It is obvious from the pol extraction data that the results obtained by diffusion only are considerably lower than one would expect from conventional milling. The question now arises whether a substantial increase in pol recovery could be expected if:

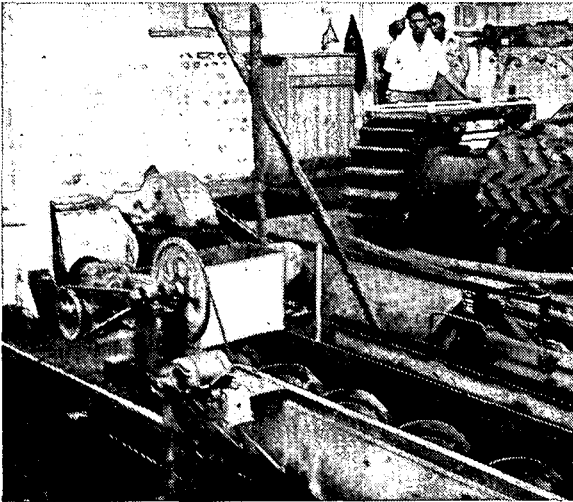
- (a) cane preparation was improved;
- (b) the juice expressed in the mill from the diffusion residue were to be returned to the diffuser;
- (c) the design of the diffuser was improved.

(It was already shown that partial crushing of the cane before diffusion will result in a substantial improvement in pol extraction.)

(a) Cane Preparation

On analysis of the coarse and finer cane particles in the extracted cane as it left the diffuser, the great importance of even preparation of the cane fed to the diffuser was forcibly demonstrated. It was shown that 72 per cent of the pol in the residue was present in the coarse pieces. Assuming that the coarse pieces made up 10 per cent of the cane fed to the diffuser, the pol extraction could have been raised from 87 per cent to 96 per cent, if the coarse pieces were entirely eliminated, other conditions being the same.

PILOT PLANT DIFFUSER



View of Feeding Box and Cane Shredder in the background

(b) Return of Expressed Juices

A number of tests were carried out to determine the pol distribution in the diffuser residue. The residue was passed through a hand mill, after allowing the surplus liquid to drain off. The following average distribution of pol was found:

	Per cent on Pol in Residue	Per cent on Pol in Cane	Purity
Pol in drainings ...	7.6	1.0	82.5
Pol in expressed juices	61.3	7.9	85.8
Pol in final bagasse...	31.1	4.0	44.5

It is clear that the return of both drainings and expressed juices to the diffusion process will considerably improve the pol recovery. Using average data, the pol extraction could be increased from 87.1 per cent to 96.0 per cent, other conditions remaining the same.

The high purity of the expressed juice is not as surprising as it seems, when it is remembered that the bulk of the pol in residue is in the larger bits of cane.

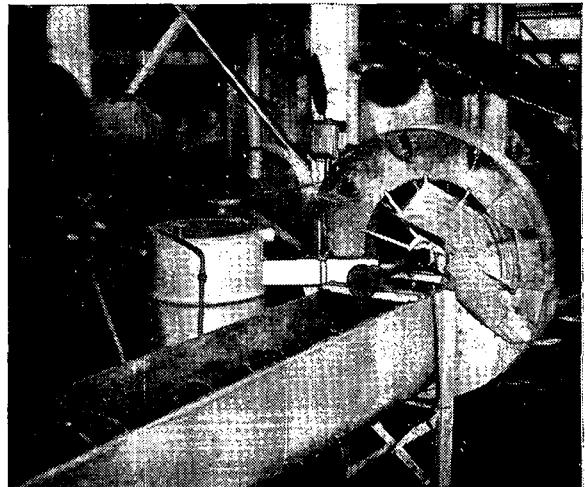
Furthermore, the efficiency of the hand mill was comparatively low, moisture in the final bagasse being as high as 63.5 per cent. It is hence reasonable to expect that, when the bagasse is milled to 50 per cent moisture, a proportionally larger amount of pol will be recovered from the diffuser residue.

(c) Design of the Diffuser

Whether or not changes in the design of the diffuser can be expected to improve the efficiency of the process can only be deduced from the magnitude of the diffusion constant K , when applying the diffusion equation to the process. Unfortunately d , the average length of a cane chip, cannot be measured directly with shredded cane, and the equation cannot be solved for K . Attempts were made to infer d from a measure of the percentage pol in opened cells, but more work will have to be done before an equation is established which relates the average particle size to the pol in opened cells. In these investigations it was established that the shredded cane used as diffuser feed had at least 70 per cent of its cells ruptured. This introduces a further uncertainty in applying the equation to shredded cane.

The equation was developed and tested for diffusion from killed but unruptured cells, and whereas leaching of juice is also a diffusional process, it need not necessarily obey the same diffusion equation. The value of the diffusion constant K will certainly be different for the two processes. Hence overall values of K for shredded cane cannot be compared with absolute values of K for cut cane discs to determine whether the process is efficient or not. Only a value of the diffusion constant determined over that part of the process where leaching has been completed could be used for this purpose. Thus unfortunately it is not yet possible to say

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View of Residue Discharge Wheel and Hot Water Supply Tank

whether improvements in the design of the diffuser can be expected to result in a substantial improvement in pol extraction.

General Conclusions

During the investigations a serious loss of sucrose by inversion was found, amounting to an average of 0.87 per cent of pol in juice. This is not surprising since no steps were taken to increase the natural pH of the juice in cane. The pol extraction was corrected for this loss of sucrose and hence inversion does not enter into the evaluation of the efficiency of the process, as there is no reason to believe that it can not be entirely eliminated by careful adjustment of the pH of the contents of the diffuser.

As is to be expected from the very high percentage of ruptured cells in the cane fed to the diffuser, the purity of the diffusion juices was but little higher than that which would have resulted if the cane had been milled in the usual way.

This paper is but a brief summary of some aspects of the work carried out on the diffusion process. A detailed report on this work is in preparation, and will be presented once the laboratory investigations have been concluded. Even the latter report is not expected to give the final answer to the question whether or not diffusion is a superior process to milling. The very most that can be said at this stage is that diffusion as an adjunct to milling, as used at Entumeni at present, can be expected to give results superior to those generally obtained by milling only, provided conditions are carefully controlled.

The Author wishes to express his appreciation to the Management and Staff of Illovo Sugar Estates for facilities put at the disposal of the S.M.R.I. The experimental work was carried out with the assistance of Messrs. E. Beesley and C. Young, both of the S.M.R.I.

Dr. A. McMartin demonstrated diagrammatically on the blackboard the construction of a plant cell. He pointed out in the structure of the mature cell of sugarcane it often appeared as though the cell had already been ruptured or was altered anatomically and wondered if the high osmotic pressure due to the sucrose inside the cell had caused some alteration in the structure. He asked if any work had been done to find out if this was so.

Dr. C. Van der Pol said that work had been done in Australia and it showed that heating to 75°C was necessary to kill the cell so that the sucrose could diffuse rapidly. He thought that Dr. McMartin's remarks threw light on the behaviour of partially crushed cane when this is extracted with cold water to determine the juice in ruptured and unruptured cells respectively.

Dr. K. Douwes Dekker said that it had been found that SO₂ substances which had anaesthetising effect on human beings could kill the cell at low temperatures.

Mr. Walsh commented upon the plant description given in the paper which stated that a set of knives followed by a shredder would be sufficient. He queried this because in America they were starting to slice cane. He said that a beet sugar diffuser would not be suitable for sugarcane. He had had experience in the past of using a beet sugar diffuser where the material was passed directly from cell to cell and the water introduced on the counter-current principle. He had found that with a three-roller mill it was not possible to reduce the moisture down to 50 per cent in the final bagasse. The plant he had used handled not 600 lb. of cane per hour but 60 tons of cane per hour. He would have liked to have seen various types of diffuser tried. The passage of the cane through a diffuser must be positive and he thought it would be very difficult to do this with a scroll conveyor. He considered that the type of diffusion tried was dangerous because of the enormous amount of inversion which took place.

Dr. Van der Pol queried Mr. Walsh's statement that Americans were trying out slicing of cane. He said that he would have liked Mr. Walsh to have enlarged upon his experiences, particularly the extraction, etc. At Entumeni where a similar plant was operating on a big scale no trouble was obtained in draining the product from the diffuser. The same thing applied in Egypt. He may have been slightly optimistic in his statement that the pH of the juice could be controlled to avoid inversion because as the juice inside the cell was acid perhaps the damage could be done before it could be neutralised by lime.

Mr. Rault said that the experiments at Mount Edgecombe were very costly and disappointing. The trouble was fundamentally that the plant was unsuitable and inadequate for the cane throughput. They had long delays and it was necessary to produce sugar, so only a small part of the production was put through the diffusion plant. The company unwisely started off without having carried out preliminary experiments on a small scale.

Mr. R. A. Carter said that the Entumeni plant had run over a period of fifty-two hours only. The process now used was to crush the cane through a crusher and then a mill with the result that big particles of cane were already broken and ruptured, thus enabling the sucrose to be extracted. Prior to the use of the process where pre-liming took place in the diffuser, they had trouble with the filters, but this disappeared when using the diffusion process. The last mill, after the diffuser, had given no trouble, the moisture was reduced to 50-54 per cent despite the fact that cane carried over on the inter-carrier

between Nos. 1 and 2 mills had to go into the last mill with the diffusion product. This carry-over had up to 10 per cent sucrose, but the extraction obtained by this process was as good as obtained by their mills and was from 90-92 per cent.

In general he was extremely optimistic because the juice was good and so was the sugar, and they had no trouble in the filters.

Mr. J. W. Main said that a tremendous amount of experience on cane diffusion was available from other countries such as Egypt, Australia, etc. He himself had had some experience and he found a big problem was to cool down diffused cane so that it could be handled by the mill. In many places where diffusion had been tried it had eventually been discarded and he wanted to know from Dr. Van der Pol what had prompted the design of the present plant used by him.

Dr. Van der Pol said that the Sugar Milling Research Institute had been asked to accumulate some data on diffusion and as this plant was already available it had been used. It was hoped to obtain other kinds of plant so the different performances could be compared. Cold water was used at the end of the diffuser at Entumeni and this resulted in heat economy as it was heated up by the hot residue from the diffuser. There was no difficulty in milling such diffusion residue at Entumeni.

Mr. C. A. Narbeth said there were many different types of diffuser and there should not be much trouble with diffusion itself. The chief trouble would be in the preparation of cane for diffusion. Recently in the beet sugar industry a tremendous amount of work had been done on preparation of beet slices and great progress had been made.

Dr. H. H. Dodds said that he was very interested to see this paper presented as it described a process which might become eventually universal in the future. He knew some years ago of some factories in Java which practised some system of diffusion and they invariably obtained better extraction than those

factories using the conventional milling process. He wondered if some modification of the process used by Dr. Van der Pol could be reduced to laboratory scale and then utilized for a direct determination of sucrose in cane at the factories, for which there was evidently much need as a routine method in this country.

Dr. C. Van der Pol said that the diffusion process was not rapid and not really suitable for laboratory work. To speed up the process one had to reduce the size of the cane particle and one could hardly talk of it as being diffusion any more, but rather as leaching or extraction. He pointed to other laboratory plant which could be more suitably used for the purpose suggested by Dr. Dodds.

Mr. Leclezio said that it was obvious from Dr. Van der Pol's paper that the increased purity claimed by others could not always be obtained by diffusion. He asked if the correction of pH by lime might not delay diffusion.

Dr. C. Van der Pol said that the purity of juice obtained was a little higher than one could expect from milling and he considered that this was due to some extent to the heating which gave a partial defecation of the juice. As far as the quantity of water required was concerned it had to be one hundred per cent of the juice in the cane but if the diffuser was preceded by a crusher then the amount of juice in cane would be so much lower that the water requirement could perhaps be reduced.

Mr. C. G. M. Perk said that the process Dr. Dodds referred to, was known in Java as Nobel's Hot Maceration. It simply consisted in pumping up juice at a high temperature, several times, on to the bagasse. This resulted in better extraction. Since the steam consumption was rather high, the application was limited to three or four mills. Like the hot maceration process used in Australia, Nobel's hot maceration may not be called diffusion, because the time of contact between bagasse and juice is too short, for proper diffusion.