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## World sugar pricas

As noted earlier, the London Daily Price for white sugar is now only quoted in US dollars while official prices are quoted in both sterling and dollars for raw sugar. This adds to problems of assessing sugar movements because, under present circumstances of plentiful supplies, currency movements and political factors assume a greater importance in price variation, and the US dollar has been more volatile than sterling in recent weeks. Nevertheless, in order to gauge the white sugar premium over raws, and the effect on this premium of different influences, more of the market observers are using the dollar quotation for the LDP instead of the formerly usual sterling price.

Following this trend we have to report that the LDP started the month of February at $\$ 179$ but rose to $\$ 184$ on February 3 on rumours that Cuba, the USSR and China had purchased substantial amounts of raws, as much as 300,000 tonnes. The rise was only temporary, however, and prices eased to $\$ 163$ by February 20, aided by the news that Coca-Cola would allow a higher blend of HFS in its soft drink. News of increased tension in the Middle East caused a recovery to $\$ 171.50$ but this again was shortlived and prices drifted downwards, to end the month at $\$ 160$ per tonne.

The LDP(W) started the month at \$205 per tonne but fell to $\$ 201$ on February 2 when it seemed likely that the USSR would not take up its option to buy $\mathbf{2 5 0 , 0 0 0}$ tonnes of Brazilian whites. It rose with the raw sugar price on the next day but also sank again, aided by news of a default on a white sugar contract for delivery to Nigeria. Following the coup in that country in January it was felt that completion was unlikely as current prices are lower than when the contract was first closed near the end of 1983. With the rise in the LDP, the white sugar price also rose on February 21. On February 23, however, F. O. Licht GmbH published their estimate of European beet areas, showing an increase of some 3\%, and this produced a reduction which continued so that the LDP $(W)$ ended the month at $\$ 179$ per tonne.

## International Sugar Agreement

A note of optimism emerged at the beginning of February in relation to negotiations for a new ISA ${ }^{1}$, with a report from an official of the leading French trade house, Sucres at Denrées, that the EEC had dropped most of its doctrinal requirements about the shape and basic mechanism of an agreement. However, the Community's insistence on a minimum export allowance is likely to be a snag; the EEC wants to be allowed to ship more than 5 million tonnes a year whatever the other exporters do, because it has been building up white sugar markets in Africa, the Middle East and particularly the geographically convenient USSR and does not want to give up any part of these outlets.
C. Czarnikow Ltd. reports ${ }^{2}$ that no final decision has been made as to the date for the third session of the UN Sugar Conference in Geneva, but there is a strong
impression that it will commence on Tuesday June 12 and run for a period of $3-4$ weeks.

## World sugar balance, 1983/84

Even at the beginning of the 1983/84 beet sugar campaign in Europe it had been expected that there would be a shortfall of production against cohsumption for the crop year of some millions of tonnes, with a corresponding fall in stocks, and this was reflected in the then current sugar prices. The recovery in sugar yields then became apparent, and the improvement in the USSR production level especially raised the forecast total outturn. F. O. Licht GmbH has now issued ${ }^{3}$ its second estimate of the world sugar balance for the period September 1983 to August 1984, based on assessments for the individual countries, and concludes that the diminution in world stocks is likely to be only some 160,000 tonnes, raw value. The figures appear below.

|  | 1983/84 | 1982/83 | 1981/82 |
| :---: | :---: | :---: | :---: |
|  |  | onnes, raw valu |  |
| Initial stock Production Imports | 38,024,000 | 32,190,000 | 24,226,000 |
|  | 95,860,000 | 100,496,000 | 100,605,000 |
|  | 27,995,000 | 29,010,000 | 31,573,000 |
|  | 161,879,000 | 161,696,000 | 156,404,000 |
| Exports Consumption | 28,405,000 | 29,557,000 | 31,933,000 |
|  | 95,607,000 | 94,115,000 | 92,281,000 |
| Final stocks | 37,867,000 | 38,024,000 | 32,190,000 |

World sugar consumption is forecast to advance by about 1.6\% from 1982/83, mostly from population increases but with a modest increase in the world economy and increased availabilities as additional factors. Consumption growth in developed countries is inhibited by substitutes while in many exporting countries higher domestic prices have been introduced because of the low returns from world market exports.

The key growth area for consumption is Asia with a forecast growth of 5\%; in India, especially, the government apparently intends to keep consumer prices low. The government takes 65\% of production at a fixed, relatively low price and releases it at low retail price. The sugar factories can sell the remainder for whatever price they can get. However, with high production over the last two years, the government has considerable fixedprice stocks and has not hesitated to release these, which prevents the free-sale sugar price from rising too much.

For the future, Licht foresees at best a small deficit of production against consumption; world stocks are likely to remain excessive and free market prices low. "Serious efforts to curb production are essential; it is not enough to rely on a combination of bad weather and other natural disasters."

## Commercial sugar stocks

For many years the assessment of whether sugar production was in surplus was calculated on consumption and the necessary commercial stocks which were based rather arbitrarily but on a basis of previous experience, and generally reckoned as $25 \%$ of consumption. World Sugar Journal has recently carried out a survey, based on records going back to 1965/66 and eliminating years of excess production or shortage, of the stocks held by different countries grouped as importers and exporters and each group sub-divided into developed and developing countries ${ }^{4}$.

[^0]
## Notes and comments

As a consequence of this survey, WSJ concludes that, contrary to a previously-held view, exporters have to maintain commercial stocks at a higher level than importers; that developing countries maintain them at a higher level than developed countries so that developed importers maintain the lowest possible stocks amongst all countries; it took developing importers and, to some extent, developing exporters longer to come out of recession in the early 1980's; the ending stocks in 1980/81 were lower than commercial stocks, disproving the view maintained by some that there was no statistical justification for the price to behave as it did during that year; and that the only two crop years during the past eighteen years at the end of which stocks were lower than commercial stocks were 1973/74 and 1974/75. The results of the study confirm the validity of the assumption that commercial stock level is equal to the weighted average of ending stocks in these two years, expressed as a percentage of consumption, calculated by WSJ as $\mathbf{1 7 . 9 \%}$, against $14.09 \%$ for importers and $\mathbf{2 2 . 8 4 \%}$ for exporters. The lowest commercial stocks are maintained by developed importers ( $11.03 \%$ of their consumption) and highest by developing exporters (25.26\% of their consumption).

## World sugar production estimates, 1983/84

F. O. Licht GmbH recently published their second estimate of world sugar production for the current crop period ending in August $1984^{1}$. Since the first estimate in October 1983 a further estimate of European beet sugar production has been published ${ }^{2}$ and these are almost the same in the latest estimate, apart from the USSR crop figure which has been raised 300,000 tonnes, raw value, to 8.4 million tonnes. World cane sugar production has been set almost a million tonnes lower than in October, at 59,887,000 tonnes against 60,748,000 tonnes (and 63,752,000 tonnes in 1982/83); this stems largely from smaller crops expected in Asia. The Indian crop is set 400,000 tonnes lower at 7.7 million tonnes, that of Indonesia is reduced by 156,000 tonnes to $1,650,000$ tonnes, and the Philippines crop from 2.5 to 2.0 million tonnes, all owing to adverse weather.

Total world sugar production is set at $95,137,000$ tonnes against $94,483,000$ tonnes in the first estimate and a $1982 / 83$ outturn of $101,440,000$ tonnes, raw value.

## Portugal sugar beet experiments ${ }^{3}$

Before their independence in the mid-1970's, Portugal's colonies supplied all of the country's sugar needs. Since then, Portugal has imported all its sugar (currently about 300,000 tonnes annually) except for a small production of about 5000-6000 tonnes of beet sugar in the Azores.

With a view to reducing its import dependence, Portugal has been experimenting with sugar beets in the Tagus basin north-east of Lisbon, according to a USDA report. Based on the results of the small experimental plots of winter-and spring-sown beets over several years, commercial production of this crop could be feasible, according to Portugal's sugar monopoly AGA which, with the Ministry of Agriculture, has been conducting the research. AGA believes that farmers could produce $50-90$ tonnes of beet per hectare, depending on the variety, with the lower-yielding varieties containing more sugar.

Experimentally, sugar contents have averaged as high as $16-17 \%$. AGA believes that the area sown to winter and spring beet could reach 10,000 ha after three years,
producing at least 400,000 tonnes of beets yielding something over 60,000 tonnes of sugar, based on very conservative yield estimates. AGA considers 400,000 tonnes to be the minimum crop needed for one sugar factory to operate profitably for 100 days a year.

AGA expects the government to decide within a year whether to introduce commercial production, at which time it would establish a guaranteed producer price, perhaps based on Italy's producer prices. However, AGA is reported to see two major considerations against commercial introduction of a sugar beet crop. (1) Portugal does not have a sugar factory and the government may not want to approve such an investment at present when difficult economic austerity measures are in effect, and (2) EEC opposition to expansion of sugar production in a potential EEC member country.

## EEC farm prices

The Agriculture Ministers of the ten member nations in the European Economic Community met in Brussels in early February to discuss the proposals of the EEC Commission in regard to farm prices for 1984/85 ${ }^{4}$. At a second session agreement was reached in respect of dairy products but not of other crops, including sugar, and the question was deferred until the meeting of heads of government in late March.

## UK sugar imports and exports ${ }^{5}$

Two decades ago annual UK exports regularly exceeded half a million tonnes and in some years were more than 700,000 tonnes. This was before the full thrust of competition from European beet sugar, first from the east but more latterly from the west, came to be felt and the outlet for the in-transit refiner was much larger than it is at present. Over the years British exports dwindled until eventually it was mainly only markets where a very high quality product was required that remained. In some recent years, this has resulted in exports from the United Kingdom falling below 100,000 tonnes.

The recent very good crops of sugar beets in the United Kingdom have led to the establishment of surpluses of beet sugar; in part it has been possible to carry this forward from one season to another, but it has also been practicable to export a large percentage of it. Accordingly exports from the UK more than doubled to 316,000 tonnes in 1983. Among the major outlets Iran figured with 86,000 tonnes and China with 67,000 tonnes; neither of these countries had taken any British sugar in the previous year. The other large outlet was Israel, taking 76,000 tonnes, compared with 62,000 tonnes in 1982.

Imports into the UK come into three categories nowadays. The bulk consists of raw sugar originating in the ACP countries which is destined for refining and domestic consumption. This is followed by imports of direct consumption supplies from other EEC countries, while finally there is the small quantity needed for refining and re-export. As usual, the main supplier was Mauritius, with a little less than 400,000 tonnes. The only other suppliers to exceed 100,000 tonnes were Fiji and Guyana with 142,000 and 136,000 tonnes, respectively. At $1,144,000$ tonnes, imports in 1983 were the lowest since 1945.

Details of UK imports and exports appear elsewhere in this issue.

1 International Sugar Rpt., 1984, 116, 39-47.
2 I.S.J., 1984, 86, 65-66.
3 F. O. Licht, International Sugar Rpt., 1984, 116, 12.
4 I.S.J., 1984, 86, 65.
5 C. Czarnikow Ltd., Sugar Review, 1984, (1690), 41-42.

# The kinetics of sugar crystallization by cooling 

## Theory and mathematical solution of a model based on Kukharenko's velocity data with calculation of the time-optimal law of cooling

By JORGE GUERRA DEBEN<br>(Professor, Faculty of Chemical and Food Processes, Instituto Superior Politécnico "José Antonio Echeverria", La Habana, Cuba)

## Introduction

As a preliminary phase of a broader study of some kinetic aspects of the sugar crystallization process as generally carried out nowadays in vacuum pans it has seemed advisable to tackle at the outset the much simpler problem posed by the kinetics of sugar crystallization by cooling. It is felt that such knowledge as may be gained from both the theoretical as well as the experimental side of this simpler situation will provide insight into the operation as carried out industrially. This article presents theory, pertinent calculations, numerical data and some experimental results relating to one single initial condition of the system. The presentation will show that this approach readily permits extension to other sets of initial conditions.

Theory and calculations, some notation and general remarks
$V_{\mathrm{K}}=\quad$ Specific superficial mass velocity of crystallization, $\mathrm{mg} \cdot \mathrm{m}^{-2} \cdot \mathrm{~min}^{-1}$, as measured by Kukharenko ${ }^{1}$;
$F M S_{\text {Ssat }}=$ molar fraction of sucrose in supersaturated solution at temperature $\theta\left({ }^{\circ} \mathrm{C}\right)$, from primary data of Kukharenko;
$F M S_{\text {sat }}=$ molar fraction of sucrose in saturated solution at temperature $\theta$; calculated from the formula of Charles ${ }^{2}$ for the solubility of sucrose as a function of temperature;
$\triangle F M S=F M S_{\text {sat }}-F M S_{\text {sat }}$, with both the supersaturated solution and the saturated solution at the same temperature; this is the "driving force" of the crystallization process;
$V=\quad$ Specific superficial mass velocity of crystallization, g.cm ${ }^{-2} \cdot \mathrm{~min}^{-1}=$ as $V_{\mathrm{K}} \cdot 10^{-7}$.

Of the two sets of data on specific superficial mass velocity of crystallization available to us, viz. those of Kukharenko and of Smythe ${ }^{3}$, the former was chosen because it is felt that the experimental conditions employed by Kukharenko are more likely to approximate to the conditions obtaining in a well-mixed, circulating massecuite, wherein the crystals are kept

J. Guerra Deben
in motion as a consequence of the hydrodynamic drag forces exerted upon them by the flowing liquid phase. It is difficult to visualize a circulating massecuite in which the crystals remain fixed in position while a rapidly moving stream of solution impinges upon them, as was the case in Smythe's experiments.

The utilization of $\triangle F M S$ as an index of the degree of supersaturation is advantageous, by contrast with the supersaturation coefficient commonly in use for technical calculations, in that (a) it allows for direct connexion with thermodynamic studies of solvent and solute activities and (b) it provides an easy and rational way of introducing the presence of additional system components such as glucose, fructose, invert sugar and others to which definite molecular weights may be assigned. With regard to other more complex components of various molecular weights, this method of treatment may indeed perhaps reveal the feasibility of imputing to them mean "effective" molecular weights in so far as their effects on sucrose solubility and velocity of crystallization are concerned.

A log-log plot of $V_{K}$ vs. $\triangle F M S$ from the primary data of Kukharenko was drawn with temperature as parameter. With the exception of the data at $30^{\circ} \mathrm{C}$, the points for the other temperatures, after deletion of occasional outlyers, seemed to define rather nicely a set of nearly parallel straight lines. The points for these temperatures were fitted by least squares to a linear model and their intercepts and slopes were calculated; these were then fitted by least squares to polynomial models in temperature. This preliminary work led to the formula

$$
\log _{10} V_{K}=b_{0}+b_{1} \cdot \log _{10} \triangle F M S
$$

in which

$$
\begin{gathered}
b_{0}=3.9075+0.0539 \theta-0.0001545 \theta^{2} \quad \text { and } \\
b_{1}=1.0799+0.0120 \theta-0.000057125 \theta^{2} .
\end{gathered}
$$

It is assumed that the crystallization process begins at time $t=0$ upon adding to the supersaturated solution a definite number of seed crystals. For the sake of simplicity, it is also assumed at this stage of the work that (a) the seed crystals are cubic in shape and identical in size, (b) the degree of circulation and mixing is such that at any time the temperature is uniform throughout and the sugar from the liquid phase deposits uniformly upon each and every crystal at whatever rate is determined at the time by the temperature, the prevailing value of the "driving force" $\triangle F M S$ and the surface area of the crystals themselves and (c) no new nuclei or centres of crystallization appear in the course of the process.

[^1]```
I = length of edge of one crystal (cm) and defines the
    size of the crystal;
\(\mathrm{s}=\) surface area of one crystal \(\left(\mathrm{cm}^{2}\right)=61^{2}\);
density of sucrose crystal \(=1.588 \mathrm{g.cm}{ }^{-3}\);
\(\mathrm{p}=\) mass of one crystal \((\mathrm{g})=1.588 \mathrm{I}^{3}\);
\(\mathrm{n}=\) number of crystals added as seed.
```

From geometrical considerations it is easy to show that $s=4.408 \mathrm{p}^{2 / 3}$; this relation is the so-called "twothirds" law.

With regard to assumption (c) above it may be stated that conflicting opinions are found in the literature. The position of Lyle ${ }^{4}$ is quite definite when he asserts that the approximate number of crystals added as seed in a Plaistow granulated skipping is $3.10^{8}$, whereas the approximate number of crystals in the finished skipping is $2.10^{11}$; these figures would indicate the presence of 667 additional crystals for every crystal initially put in as seed. At another point in his discussion, Lyle ${ }^{5}$ states, in referring to the liquor, that "as it is now in a state of perpetual shock owing to the grain in it, false grain will appear". On the other hand, more recent work in Florida on continuous vacuum pan operation shows calculations which imply that no new centres of crystallization appear during the process over and above those initially put in as seed. It may be stated in passing that one of the objectives of this work will be directed towards the examination of this important and controversial state of affairs.

In this mathematical model of the crystallization process an initially hot, supersaturated solution of sucrose with no solid phase present will begin to crystallize at time $t=0$ upon the addition of seed crystals, and the course of the process will thenceforth be followed with the passage of time. The system may also be subjected to external temperature control, including no cooling whatsoever (isothermal crystallization) or to any arbitrary continuous law of temperature reduction as might be externally imposed upon it. Results of simple linear laws of cooling are first presented, followed by the results of subjecting the system to the time-optimal law of temperature control.

For the mass time-rate of growth of one crystal we may write

$$
\begin{equation*}
\frac{d p}{d t}=V . s .\left(\mathrm{g} \cdot \min ^{-1}\right) \tag{1}
\end{equation*}
$$

All calculations based on the fundamental working equation (1) must in every case first refer to the mass growth of each individual crystal present in the system, and must then subsequently consider by addition the joint effect of their individual mass growths on the overall composition of the liquid phase, which in turn determines (together with temperature) the value of the "driving force" $\triangle F M S$ exerted by the solution upon the whole collection of growing crystals. This is due to the fact that growth changes the surface area-to-mass ratio of crystals in keeping with the requirements of the "twothirds" law. For crystals identical in size and shape and growing uniformly, the joint effect of their growth upon the composition of the liquid phase is found at any time by simple multiplication by their number. For the more complex case of an initial size distribution of seed crystals, the above mentioned addition by groups of size categories is imperative.

The application of equation (1) with due consideration of all crystals present allows the calculation, given the initial condition at time $t=0$, of the total mass time-rate of crystallization

$$
\begin{equation*}
\frac{d P}{d t}=f[P(t), \theta(t)] \quad t>0 \tag{2}
\end{equation*}
$$

in which $\mathbf{P}=$ total mass of sugar crystallized at time $\mathbf{t}$ (not including the mass of crystals added as seed). This simple ordinary non-linear differential equation was integrated by the so-called Euler-Heun iterative method described by Lapidus ${ }^{6}$.

## Velocity of crystallization and massecuite circulation: the practical problem as reflected by theory

The practical experience of decades as collected together in the literature concurs in emphasizing the extraordinary importance of good massecuite circulation for rapid and efficient vacuum pan operation. This is repeatedly attested in several chapters of a well recognized and long standing work written by a staff of selected scientists and technical workers connected with cane sugar production; the editor, Pieter Honig, sums up this state of affairs as follows ${ }^{7}$ : "All the discussions on the different types of pans bring up the point of circulation in pans. The ideal of a vacuum pan is that all the conditions of the massecuite in process of crystallization be as uniform as possible as to density, crystal content, supersaturation and temperature, and to have these conditions uniform throughout the whole pan content". Summing up his experience in the refining industry, Lyle ${ }^{8}$ holds that "Good, quick circulation is of the greatest possible importance. We cannot say that there is any limit to the speed at which crystals of sucrose will grow except the limit imposed by circulation. Whenever we have done anything that improves circulation we have always found that faster crystallization takes place".

The above consensus finds strong support in the results of relevant researches in the field of physico-chemical hydrodynamics. Thus, in a lucid presentation and critique of the problems relating to chemical and physicochemical transformations that take place on certain surfaces in contact with liquid media, Levich ${ }^{9}$ has contributed an explanation in which he emphasizes the extraordinary importance to be attached to the effect of hydrodynamic factors upon the kinetics of such transformations. His analysis of such mass transfer processes shows that the relationship between the convective (kinematic, fluid in motion with respect to the surface) and the molecular or purely diffusional (static, fluid at rest with respect to the surface) transfer of matter is intimately connected with
(a) the properties of the fluid as quantified by the dimensionless diffusional Prandtl number $\operatorname{Pr}=v / D$ (also known as the Schmidt number),
(b) the dimensionless Reynolds number $R e=U_{0} L / V$ and
(c) the dimensionless Peclet number $P_{e}=U_{0} L / D$ in which
$v=$ kinematic viscosity of the solution,
4 "Technology for sugar refinery workers", (Chapman \& Hall, London). 1957, p. 248.
5 Idem: ibid., p. 249.
6 "Digital computation for chemical engineers", (Ediciones R). 1969.

7 "Principles of sugar technology", Ed. P. Honig (Elsevier, Amsterdam). 1959.
8 "Technology for sugar refinery workers" (Chapman \& Hall, London) 1959, p. 250.
9 "Physico-chemical hydrodynamics", (Prentice Hall, London) 1962, Chapter II.

$$
\left.\begin{array}{rl}
D= & \text { molecular diffusion coefficient or diffusivity } \\
\text { of the dissolved matter in the solution, }
\end{array}\right\}
$$

With regard to the diffusional Prandtl number, data and calculations presented by Levich show that, for gases, the diffusivity $D$ and the kinematic viscosity $v$ are of the same order of magnitude, and hence $\operatorname{Pr} \doteq 1$, whereas for common liquids such as water the kinematic viscosity $v \doteq 10^{-2} . \mathrm{sec}^{-1}$ ) and the diffusivity of ions and molecules in aqueous solutions are of the order of $\mathrm{D} \doteq 10^{-5}\left(\mathrm{~cm}^{2} . \mathrm{sec}^{-1}\right)$ (and for macromolecules $\mathrm{D} \doteq 10^{-6}$ ). Therefore, for water and comparable liquids $\operatorname{Pr} \doteq 10^{3}$. Also, as the kinematic viscosity of the liquid increases, the diffusivity decreases in accordance with the approximate relation $\mathrm{D} \doteq$ constant/v. Thus, the Prandtl number increases approximately as the square of the kinematic viscosity. In viscous fluids the Prandtl number attains values of $10^{6}$ or greater. A high value of the Prandtl number signifies that, even at very low velocities, the transfer of matter by convection predominates over molecular diffusion. Molecular diffusion of dissolved matter in liquids is so slow that convective transfer of matter plays in general a more important role than in gases.

After these introductory general remarks concerning the properties of the solution (kinematic viscosity and diffusivity) relevant to the problem at hand, Levich goes on to examine the steady-state dimensionless differential equation of mass transfer together with the pertinent boundary conditions and shows that near the surface a thin layer of solution must exist in which the concentration changes rapidly. The derivatives of concentration with respect to distance are in this region very large, with the result that the terms of the mass transfer equation representing molecular diffusion become comparable to the terms representing convective transfer, despite the small value of the diffusion coefficient. Thus, the liquid may nominally be divided into two regions: the first, a region of nearly constant concentration far from the reaction surface, as a consequence of convective transfer; the second, a region of rapidly changing concentration in the close vicinity of the surface; this region represents a thin liquid layer which is analogous to Prandtl's momentum boundary layer. Just as the effect of viscosity is significant in the momentum boundary layer, molecular diffusion must be taken into account in the thin layer adjacent to the reaction surface, which is therefore termed the diffusional boundary layer. The relationship between the convective transfer and the molecular diffusion of matter from the solution to a surface in contact with it is described by a single parameter: the dimensionless Peclet number. When the Peclet number is small the concentration distribution is determined largely by molecular diffusion; at sufficiently small Pe , mass transfer by convection is negligibly small. From the definition of the Peclet number it follows that such a situation occurs (for a given $D$ ) at sufficiently low velocities and in regions of small dimensions. Conversely, when the Peclet number is large, the concentration distribution is determined essentially by convective transfer and molecular diffusion can be neglected. However, as stated above, stress must be laid upon the fact that this argument does not apply to a region in the liquid where a sharp change of concentration occurs; here both types of mass transfer

## The kinetics of sugar crystallization by cooling

mechanisms, by convection and by molecular diffusion, may become comparable and significant.

On the basis of these conclusions, Levich examines next the problem of transfer of matter dissolved in a liquid to a surface in contact with it for the special case of a solid particle descending freely through the solution under the action of gravity, that is to say, in a context relevant to the problem of the growth of sugar crystals as affected by circulation. His analysis assumes that the particle is sufficiently small that the corresponding Reynolds number $R e=U a / v$ (where $U$ is the rate of descent and $a$ is the diameter of the particle) is small compared with unity; under these conditions the liquid around the particle is in creeping flow. The problem of the velocity distribution for creeping flow near a spherical particle was solved by Stokes. The velocity of the liquid changes gradually and smoothly with distance from the particle and there is no hydrodynamic boundary layer near the surface. Nevertheless, a diffusional boundary layer does exist near the particle surface. The motion of the particle with respect to the solution has the effect of greatly thinning out the region through which molecular diffusion of the solute to the particle surface must occur, or equivalently, of bringing up the bulk concentration of the solution, by a convective mechanism, closer to the particle surface, so that a sharp change of concentration over a small distance is established with a correspondingly large concentration gradient. This change in concentration occurs across a very thin diffusional layer, in spite of the fact that the liquid velocity changes gradually. It thus happens that the greatly reduced distance across which mass transfer by molecular diffusion from the solution to the particle surface must finally take place acts to compensate for the small value of the diffusion coefficient $D$, with a resulting increase in the rate of mass transfer by comparison with the rate under static conditions.

The role of circulation in accelerating crystal growth is further emphasized by comparison of the final expressions derived by Levich for the problem at hand. When the particle is so small that the Peclet number becomes negligible ( $P_{e} \rightarrow 0$, practically no motion of the particle relative to the solution), the total flow of matter to the surface of the particle is given by

$$
\begin{equation*}
I=4 \pi D a c_{0} \tag{3}
\end{equation*}
$$

in which $c_{0}=$ effective concentration difference between the bulk of the solution and the particle surface (molecules. $\mathrm{cm}^{-3}$ ), and $a=$ particle radius (cm).
For particles large enough that their motion relative to the solution becomes indeed significant ( $P e \gg 1$ ), the total flow of matter to the surface is given by

$$
\begin{equation*}
I=7.98 c_{0} D^{2 / 3} U^{1 / 3} a^{4 / 3} \tag{4}
\end{equation*}
$$

The solution of the problem is then generalized by means of the interpolation formula

$$
\begin{equation*}
I=4 \pi D a c_{0}\left(1+0.64 P e^{1 / 3}\right) \tag{5}
\end{equation*}
$$

which becomes equation (3) as $P e \rightarrow O$ and equation $(4)$ as $P e \geqslant 1$.
(Our calculations for the case of a sugar crystal $100 \mu \mathrm{~m}$ in diameter descending freely under the action of gravity through a $60^{\circ} \mathrm{Bx}$ sucrose solution at $70^{\circ} \mathrm{C}$ show that $R e=3.4 \times 10^{-3}$ and $\operatorname{Pr}=1.3 \times 10^{7}$. This

## The kinetics of sugar crystallization by cooling

value of the Prandtl diffusional number is so large that, in spite of the very small value of the Reynolds number, the product $P e=R e \times P r=4.4 \times 10^{4}$ is very large compared with unity. It thus comes out that the condition $R e \ll 1$ and the condition $P e \gg 1$ may both be simultaneously fulfilled for a wide range of practical situations on account of the very large value of the diffusional Prandtl number for concentrated solutions such as are of significance in industrial sugar crystallization practice.)

The foregoing theory throws light upon the reason behind a feature of experimental results which is not immediately evident: the fact that the specific superficial mass velocity of crystallization of sucrose has proved to be independent of the size of the crystals themselves. This result follows directly if the velocity of descent $U$ of a sufficiently large particle falling freely through a solution under the action of gravity as governed by Stokes law is substituted in equation (4); the size of the particle is found to cancel out. This important theoretical deduction serves to support our expectation that the results of measurements of velocity of crystallization such as those of Kukharenko, which were performed with rather large crystals generally exceeding commercial sizes, will be found to apply satisfactorily in the case of smaller crystals if adequate circulation is assured.

## Results of numerical integrations

Equation (2) was integrated numerically for the case of isothermal crystallization (no cooling whatever) and various linear cooling rates, for an initial condition of the system defined by
temperature $=70.0^{\circ} \mathrm{C}$;
composition of the solution: $10,000 \mathrm{~kg}$ of sucrose
dissolved in 2281.5 kg of water, which corresponds to 438.3 kg sucrose $/ 100 \mathrm{~kg}$ of water;
supersaturation coefficient $=1.35$;
Brix $=81.42^{\circ}$;
$F M S_{\text {Ssat }}=0.187482$;
$\triangle F M S=0.041511$;
number of seed crystals added $=4.29 \times 10^{9}$;
size of seed crystals $=100 \mu \mathrm{~m}=1 \times 10^{-2} \mathrm{~cm}$;
mass of seed crystals $=6.812 \mathrm{~kg}$.
For isothermal crystallization the computation was extended to a total crystallization time of 180 minutes. For the linear cooling rates the computation was extended until a final temperature of $40.0^{\circ} \mathrm{C}$ was reached.

Fig. 1 shows calculated values of mass of sucrose crystallized as functions of time for isothermal crystallization and for various cooling rates in ${ }^{\circ} \mathrm{C} . \mathrm{min}^{-1}$ Curve A , corresponding to a cooling rate of $1 / 10^{\circ} \mathrm{C} \cdot \mathrm{min}^{-1}$ is not shown in its entirety; for this cooling rate the limit temperature of $40.0^{\circ} \mathrm{C}$ was reached at $t=300 \mathrm{~min}$, at which time 4422.3 kg of sucrose had crystallized. The horizontal dashed line labelled "condition of exhaustion" at $P=4667.1 \mathrm{~kg}$ corresponds to the mass of sucrose that would crystallize out of the solution as defined if it were held at $40.0^{\circ} \mathrm{C}$ and allowed sufficient time to reach the equilibrium condition of saturation (exhaustion at 46.671\% yield of crystals). The fact that curves B, C and D (as well as the section of curve A) extend to the points labelled $40^{\circ} \mathrm{C}$ and no further does not mean, of course, that the process of crystallization comes to a stop then and there. Since those "end-points" are all located below the condition of exhaustion, the corresponding solutions are still supersaturated, so that crystallization would continue isothermally (and very slowly) if the temperature were maintained at $40.0^{\circ} \mathrm{C}$ until the condition of saturated equilibrium at this temperature were reached.


Fig. 1. P vs. time for isothermal crystallization and for linear cooling laws. Lines drawn for $t<15 \mathrm{~min}$ are approximated as straight lines. Actually they are inflected curves criss-crossing each other

It is noteworthy that a theoretical yield of crystals slightly greater than $40 \%$ is predicted by this model at a cooling rate of $1 / 3^{\circ} \mathrm{C} . \mathrm{min}^{-1}$, that is, in 90 minutes cooling time from $70^{\circ} \mathrm{C}$ to $40^{\circ} \mathrm{C}$ (Curve C). Since this result for yield of crystals and lapse of time shows good order of agreement with industrial experience in vacuum pan crystallization in the refining industry, it would seem that this theoretical model, taken in conjunction with Kukharenko's velocity data, constitutes a fair initial approximation to the physico-mathematical description of the sugar crystallization process in general, deserving continuing effort and inquiry.

Determination of the time-optimal cooling law by means of dynamic programming. Experimental test results
As was to be expected and is shown in Fig. 1, the course of the crystallization process proves to be very sensitive to the law of cooling imposed upon it. In view of this, it was decided to apply to one case study the method of dynamic programming in order to determine the cooling law that would minimize the time required to move the system from a given initial condition at time $\boldsymbol{t}=\mathbf{0}$ to a given final condition as to temperature and crystal yield. The initial condition adopted was the same as previously reported in this article. The desired final condition was defined as follows:
temperature $=40.0^{\circ} \mathrm{C}$
mass of sugar crystallized $=4000 \mathrm{~kg}$ ( $40 \%$ crystal yield) number of crystals $=4.29 \times 10^{9}$.

Fig. 2 shows the calculated course of the crystallization process when the system was subjected to the time-optimal cooling law thus found, as shown in Fig. 3. Values of the corresponding size of crystals and directing force $\triangle F M S$ as function of time appear in Fig. 4.

A laboratory test of this theory was made using specially developed equipment capable of imparting excellent circulation and mixing to the system as it gradually changed from its initial condition as a hot liquid solution while subjected to the time-optimal cooling law until its final condition as a cooler, dense and rather stiff massecuite was attained; the mass of seed crystals added was adjusted in proportion to equipment capacity. A crystal yield of $42.3 \%$ (dry basis, average of two runs) at the final temperature of $40.0^{\circ} \mathrm{C}$ after a period of 56 minutes counted from seeding time was obtained. Inspection of the crop of crystals showed remarkable uniformity as to size with no evidence of conglomeration or false grain. More detailed theory and test results of work now in progress will be presented in forthcoming articles.

## Acknowledgements

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Fig. 3. $\theta$ vs. time for time-optimal cooling law



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La cinétique de la cristallisation du sucre par refroidissement

La théorie de la cristallisation du sucre par refroidissement est analysée et l'application d'un modèle mathématique, basé sur les données de Kukharenko, pour l'établissement des conditions optimales de refroidessement (en termes de temps) est décrite. Les résultats préliminaires obtenus avec un équipement de laboratoire sont rapportés brièvement.

## Die Kinetiken der Zuckerkristallisation durch Kühlung

Die Theorie der Füllmassekristallisation durch Kühlung wird analysiert und die Anwendung eines mathematischen Modells beschrieben, das auf den Geschwindigkeitsdaten

The kinetics of sugar crystallization by cooling
von Kucharenko für die Aufstellung optimaler Kühlungsbedingungen (Zeitangaben) basiert. Über vorläufige Ergebnisse, die mit einer Laborausrüstung erhalten wurden, wird kurz berichtet.

La cinética de la cristalización de azúcar por enfriamiento
Se analiza la teoria de la cristalización de masas cocidas por enfriamiento y se describe la aplicación de un modelo matemático basado en los datos de velocidad de Kukharenko para establecer las condiciones óptimas (en el tiempo) del enfriamiento. Se informa sumariamente de resultados preliminares obtenidos con un equipo de laboratorio.

## Polysaccharides in refined and raw sugar

# Determination in Hawaiian cane sugars by size exclusion chromatography 

By DONALD F. CHARLES

## Introduction

Refined cane sugar characteristically contains polysaccharides which precipitate from water solutions on adding alcohol. The resulting turbidity poses problems for distillers and blenders who produce cordial-type beverages which are high in both sugar and ethanol. The major polysaccharides are dextran, starch and indigenous sugar cane polysaccharide; the latter was reported by the Cane Sugar Refining Research Project group to be an arabinogalactan ${ }^{1}$.

At the Crockett refinery of C and H Sugar Company we have studied these substances over the years with a view to their removal. Complete removal has not been achieved, but we have been moderately successful in producing refined sugars tolerably low in polysaccharides by controlling the quality of raws melted, and taking advantage of the partial removal in various steps of the refining process.

In the summer of 1981 we started using size exclusion chromatography to study the molecular weight distribution of the alcohol-insolubles in our process streams. This report presents the major findings.

## Chromatographic system

The pump, refractive index (RI) detector and conductivity detector were described previously ${ }^{2}$. The columns were Brownlee Labs "Aquapore", packed with rigid 10 micron porous silica spheres to which were bonded hydrophilic glycerylpropylsilane groups. Most of the studies reported here were done with three columns in series, $\mathrm{OH}-300, \mathrm{OH}-500$, and $\mathrm{OH}-1000$. Eluent for most studies was water containing $0.01 \%$ calcium acetate and $0.02 \%$ sodium azide. Flow rate was 0.4 ml per minute. Samples were loaded with volumetric syringes into the Waters U6K injector.


## Generalized instructions for precipitating alcohol-insolubles

The quantity of sample taken is aimed to provide reasonable size peaks using the RI detector, and is usually around 50 grams for whole raw sugar and 200 grams for refined sugar. Water is added as calculated to give a standard concentration of $\mathbf{4 0}$ grams solids per 100 ml . To a given volume of this standard concentration is added twice the volume of absolute ethanol. After mixing, enough time is allowed for the precipitate to coalesce and stabilize; for refined sugars it may be desirable to add 1 ml of 1 N acid or alkali to encourage aggregation. The precipitate is collected on a Millipore pre-filter pad (AP2504200), and washed in the filter funnel with $80 \%$ alcohol to remove the bulk of the sucrose. The pad is dried to remove alcohol and water, put in a 100 ml beaker, and 4 ml of 0.25 N NaOH introduced with a volumetric pipette. The contents of the beaker are swirled several times during about an hour's time, then 2 ml of 0.5 N HCl are added, followed by swirling several times during ten minutes. Plain water slowly dissolves dextran, but experiments using the iodine test have shown that between 0.1 N and 0.25 N NaOH is required to extract amylopectin effectively. The extract obtained is in 6 ml of 0.17 N NaCl and is nearly neutral. It is filtered to remove particulates before injecting 200 microlitres into the liquid chromatograph. The iodine test for starch using 1 ml of extract is outlined below.

If RI peaks from the first chromatography run are too small for desired accuracy, the solution must be concentrated further. Starting with a dry volumetric syringe, a measured volume, say 2.0 ml of extract, is transferred to a 10 ml conical-bottom flask and taken to dryness on a rotary vacuum evaporator. 0.4 ml of eluent is added using a 0.5 ml syringe and swirled to dissolve over about 30 minutes. Injecting 300 microlitres of this concentrate increases the RI area by a factor of 7.5 .

## Raw sugars

Fig. 1 shows a typical chromatogram for raw sugar. Numbers on the horizontal scale are time in minutes between injection and RI recording. The dashed line is the conductivity trace, displaced to appear a quarter inch after the RI trace. There are three broad, overlapping

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RI peaks before the small sharp RI peak associated with conductivity emerges. The peaks are numbered from left to right in their order of elution. Raw sugars have been observed in which each of the three peaks was the largest.


The ionic content in the eluent acts to swamp out the weak charges on the column packing. This permits the low-molecular weight ionic constituents of the sample to enter the pores in the column packing and not emerge from the column until after the three major peaks of interest. Once the ionics emerge, the interpretation of the chromatogram is difficult. Sample ionics include calcium sulphate, precipitated by high alcohol concentration. Then the water injected in the sample causes dips in both RI and conductivity. Peaks which follow are due to residual sucrose and other low molecular weight constituents. Thus we are interested only in the peaks that emerge before the ionics.

## Reference dextrans

Fig. 2 shows the envelopes for three separate Pharmacia dextrans traced onto the same drawing to show relative positions. From left to right the nominal molecular weights (M.W.) are 2,000,000, 500,000 and 70,000. A plot of $\log$ M.W. vs. retention time makes a tolerably straight line from which one can estimate the M.W. of sample polysaccharides. It may be more than just coincidence that the three Pharmacia dextrans seem to correspond in position with the three peaks observed in raw sugars. Thus Peak 1 corresponds to about 2,000,000 M.W.; Peak 2 to the order of 500,000 M.W.


## Peak area estimation

The distributions for the three peaks run together. One can drop perpendiculars to the baseline at arbitrary retention times to separate the peaks and then, using a cut-and-weigh technique, measure peak areas. Areas are converted to concentrations using as reference standard a solution of the $2,000,000$ M.W. Pharmacia dextran.

## Refined sugar

C and H granulated sugar shows a high first peak suggesting a M.W. about $2,000,000$. Confectioner's AA is the trade name for a specially refined, large grained, sugar, boiled from the early light-coloured runnings off char; Peak 1 is present, though usually smaller than for granulated sugar.

## Dialysis of CON AA sugar

We took some CON AA sugar, returned from a distiller customer because of its high alcohol-turbidity; we dialysed a solution of this sugar against water and concentrated the retentate. Figure 3 shows a high Peak 1. There was also a small foot or preceding peak with associated slight conductivity rise. Injection of the flocculating agent used to facilitate clarification showed matching characteristics. The specification sheet for the flocculant listed its M.W. as $15,000,000$. Thus it appears one could measure the added polymer quantitatively from the area of this early peak. It also shows that the M.W. of the dextrans in refined sugar do not go much higher than $3,000,000$; there is no need to go to the $\mathrm{OH}-4000$ column.


Tailing from Peak 1
Figure 3 also illustrates the tailing from Peak 1 that is characteristic of refined sugars. To study the causes of tailing we injected a large amount of refined sugar alcohol-insolubles and collected fractions of eluent corresponding to peaks 1,2 and 3 . We reinjected the fractions; Peak 1 reinjected still gave significant tailing, Peak 2 fraction reinjected showed both Peak 2 and Peak 1, and Peak 3 fraction showed both Peak 3 and Peak 1. One concludes that the tailing has at least two causes. Part is due to small amounts of Peak 2 and Peak 3 materials present in the sample injected along with the large amount of Peak 1 material. Part of the tailing is due to retardation of some Peak 1 material, possibly by adsorption mechanisms.

## Fractional precipitation

Figure 4 illustrates the effect of a stepwise increase in alcohol concentration. We dissolved a Hawaiian raw sugar to the standard $34.8^{\circ}$ Brix ( 40 g per 100 ml ) and
added absolute ethyl alcohol to $44 \%$ by volume. Figure 4A is the chromatographic profile for the water extract of the precipitate. It shows primarily the high molecular weight Peak 1. To the filtrate from collection of the first precipitate, already $44 \%$ in alcohol, we added more alcohol up to $62 \%$ by volume. Figure 4B is the chromatogram for the thus-produced alcohol precipitate when redissolved in water, and shows a profile similar to Figure 1. Finally, Figure 4C shows the profile for the precipitate given on adjustment from 62 to $80 \%$ alcohol. Very little first peak remains to be precipitated but the proportions of Peaks 2 and 3 and the conducting Peak 4 are increased.


We can summarize by saying that, as the alcohol concentration increases, the M.W. of the precipitated constituents goes from high to low. The precipitation of significant amounts of material below $44 \%$ alcohol is consistent with the following quotation from the introduction to the CSR method for determination of dextran in raw sugar ${ }^{3}$ : " . . . dextran forms a haze
quantitively in $50 \%$ alcohol. Depending on the type of dextran, precipitation commences at an alcohol strength of about $35 \%$, and is usually complete at 42 to $45 \%$. Extending the concentration to $50 \%$ ensures that dextran has been quantitatively precipitated. Higher alcohol strengths, particularly beyond 60\%, encourage the precipitation of other polysaccharides, should they not have been removed by preliminary purification procedures."

Thus it appears likely that Peak 1 is primarily dextran. Precipitation at low levels of alcohol helps to explain the concerns of the cordial-makers. Turning the argument arourd, if the refiner's concern is to produce a sugar to meet the distiller's criteria, then $50 \%$ alcohol in a test procedure should generally be sufficient. However, the sugar proportion also is relevant. An increase in sugar appears to increase the potential for the precipitating effect of the alcohol. Probably the sugar ties up water by hydrogen bonding leaving less water to keep the dextran in solution.

## Refinery processing

Figure 5 shows a typical profile for raw sugar crystal washed free of its film. The first peak is most evident, while a small shoulder is due to the second peak material. Melt house affination liquor had a profile much like Figure 1. This illustrates how Peak 1 boils into the crystal.


Fig. 5
Removal by bone char and carbon is of interest. Clarifiers have very little effect, so the raw liquor onto char has a profile similar to Figure 5, which shows a small Peak 2 shoulder on Peak 1. The filling liquor for char filters, consisting of the darker liquor off char filters late in their cycles, also looks like Figure 5. (This filling liquor is also the running liquor for granular carbon filters.) Figure 6 is a profile for the effluent from granular carbon and shows that Peak 2 is reduced by comparison with Peak 1. The early runnings from freshly burned char also show profiles like Figure 6. Figure 7 is the profile of sweetwater obtained during sweetening-off of a char filter. This illustrates that Peak 2 material, adsorbed by the char during the liquor portion of the cycle, is released on dilution.

## Raw sugar factory studies

It is important to establish the source of the alcohol-
3 Proc. 13th Session ICUMSA, 1974, 363, (Subject 27, Appendix 1).

## Polysaccharides in refined and raw sugar



Fig. 6
insolubles in the raw sugar. We asked Hawaiian sugar factory personnel to obtain a set of samples from a milling train. We ran these by SEC. Peak 1 material was present in the first expressed juice and did not appear to increase during subsequent milling. Peaks 2 and 3 were also present in the first mill juice but appeared to increase through the milling process. This suggests that cane-cutting and handling before milling is crucial for the content of critical Peak 1 material.


## Amylopectin estimation

We have talked primarily about dextrans; however, tests for starch using iodine have suggested that 10 to $20 \%$ of the Peak 1 area for refined sugars was due to amylopectin. The starch analysis procedure outlined below is designed to use a 1 ml aliquot of the alcoholinsolubles extracted with sodium hydroxide as described above.

The following solutions are prepared: (A) potassium iodate, 2.14 g per litre and (B) potassium iodide, 30 g per litre. 100 ml of $A, 100 \mathrm{ml}$ of $B$, and 150 ml of water are mixed together and adjusted to pH 7 with NaOH to give reagent C .

Using a good quality spectrophotometer, the wavelength is set to 560 nm . (For double beam operation put water in the reference side 1 cm cell). With a 1 ml pipette 1 ml of sample is transferred into a clean, dry 1.5 ml cell with path length 1 cm . The absorbance scale range is set at 0 to 1 and absorbance adjusted to 0.1 or 0.2 . The absorbance (call it A) is read. After adding 1 drop of $85 \%$ phosphoric acid and mixing well, the absorbance (call it $B$ ) is read. $A-B$ will be a rough
measure of the pH -dependent "indicator effect". 25 microlitres of reagent $C$ is added with a volumetric syringe, mixed and the maximum absorbance reached within 10 minutes of mixing read (call it C ). $\mathrm{C}-\mathrm{B}$ is a measure of the sample absorbance. To establish correction for colour of the reagent on acidifying, the above is repeated using 1 ml of water instead of sample. $\mathrm{C}^{\prime}-\mathrm{B}^{\prime}$ will be a small correction to the value $\mathrm{C}-\mathrm{B}$. By running a standard containing 0.009 g per 100 ml of amylopectin, corrected sample absorbance may be converted to amylopectin concentration in g per 100 ml .

## Preparation of standard amylopectin

The moisture content of powdered amylopectin (AP) to be used as standard is determined. About 0.5 g of AP is weighed precisely into a 150 ml beaker, 50 ml of 0.25 N NaOH added and the whole stirred with a magnetic bar for several hours to allow time for the slow rate of dissolving. When fully dissolved 25 ml of 0.5 N HCl is added and the solution rinsed into a 200 ml volumetric flask and made to volume with NaCl solution ( 1 g per 100 ml ). The concentration of this solution A is calculated, correcting for moisture in the powdered AP; it should be near 0.225 g per 100 ml . Solution A is refrigerated and used as needed to prepare Solution B; 4 ml of $A$ is transferred with a volumetric pipette into a 100 ml volumetric flask and made to volume with NaCl solution ( 1 g per 100 ml ). Standard B should contain about 0.009 g AP per 100 ml . Sodium chloride solution, at 1 g per 100 ml , is near 0.17 N . This is used so that the salt concentration is the same in samples and standard. Absorbance of amylopectin-iodine complex increases slightly with increasing salt concentration.

## Summary

A system for size exclusion chromatography is described and applied to the study of alcohol-insoluble material. Three distinct distributions were found in Hawaiian raw sugars, with molecular weights peaking near $2,000,000,500,000$, and 70,000 . The highest molecular weight, Peak 1, presumed to be primarily dextran, was only partially removed by freshly-burned char. Peak 1 was strongly included in the refined sugar during boiling. It is the major contributor to turbidity of cordials; most of it precipitates at an alcohol concentration between 30 and $45 \%$. A test on alcohol precipitates in juices from a cane milling train suggests that Peak 1 is formed before the cane enters the mill, whereas Peaks 2 and 3 increase throughout cane handling and milling. An iodine test procedure suggests that amylopectin contributes about $10 \%$ to the area of Peak 1.

Polysaccharides dans le sucre raffiné et le sucre brut. Détermination dans des sucres de canne Hawaiiens par chromatographie d'exclusion stérique

Une méthode de chromatographie d'exclusion stérique est décrite et appliquée à l'étude de substances insolubles dans I'alcool. On a trouvé trois répartitions distinctes dans les sucres bruts hawaiiens avec des poids moléculaires atteignant prés de $2.000 .000,500.000$ et 70.000. La substance ayant le poids moléculaire le plus élevé, correspondant au premier pic, est supposée être principalement du dextrane et $n^{\prime}$ est que partiellement enlevée par du noir animal fraîchement régénéré. Elle est fortement retenue dans les inclusions du sucre raffiné, lors de la cuisson. C'est la cause principale de turbidité dans les spiritueux; la plus grande partie précipite à des teneurs en alcool comprise entre 30 et $45 \%$. Un essai de précipitation par I'alcool sur des jus provenant d'un train de moulins à cannes fait penser que
la substance du pic 1 est formée avant que la canne n'entre dans le moulin, tandis que les substances correspondant aux pics 2 et $\mathbf{3}$ augmentent au cours des manipulations et du broyage. Un test à l'iode suggère que I'amylopectine contribue pour environ $10 \%$ à l'aire du pic 1.

## Polysaccharide in Raffinade und Rohzucker. Bestimmung in hawaiischen Rohrzuckern durch Größenausschlußchromatographie

Ein Größenausschlußchromatographiesystem wird beschrieben und auf die Untersuchung alkohol-unlöslicher Substanzen angewandt. In hawaiischen Rohzuckern wurden drei unterschiedliche Substanzklassen mit Molekulargewichten von 2000 000, 500000 und 70000 gefunden. Die Substanzen mit dem höchsten Molekulargewicht, Peak 1, sind wahrscheinlich haupsächlich Dextrane und werden nur teilweise von frischgebrannter Knochenkohle adsorbiert. Peak 1 wurde auch stark während des Kochvorgangs in raffinierten Zucker eingeschlossen. Diese Substanzen sind die Hauptbestandteile der Trübung von Likören; das meiste davon fällt bei einem Alkoholgehalt von $30-45 \%$ aus. Eine Alkoholfällung in Säften von einem Mühlentandem legt nahe, daß Peak 1 gebildet wird, bevor das Rohr in die Mühle kommt. Ein Jod-Test legt nahe, daß Amylo-
pektin ungefähr 10\% der Fläche von Peak 1 ausmacht.
Polisacaridos en azúcar refinado y crudo. Determinación en azucares de caña de Hawaii por cromatografía de exclusión estérica.

Un sistema de cromatografía de exclusión estérica es describido y aplicado al estudio de materia insoluble en alcohol. Se han encontrado tres distintas distribuciones en los azucares crudos de Hawaii, con pesos moleculares que alcanzan casi $2,000,000,500,000$ y 70,000. La materia del pico 1, del P.M. más grande, que se supone estar principalmente dextrano, es separado no más que en parte por carbón animal, recientemente regenerado. Esta materia se incluye fuertemente en el azúcar refinado durante la cocción. Es el mayor contribuidor al turbidez de cordiales; el más grande parte se precipita a contenidos de alcohol entre 30 y $45 \%$. Un ensayo de precipitación por alcohol sobre jugos provenando de un tandém de molinos de caña sugere que la materia de Pico 1 se forma antes del entrado de la caña en la fábrica, mientras que Picos 2 y 3 se aumentan durante el manejo y molienda de la caña. Un test al iodo sugere que el amilopectino contribuye unos $10 \%$ al área de Pico 1.

# Continuous vacuum crystallization at Nantes refinery 

By JACQUES CUEL* and CLAUDE LONGUE EPEE**

## Introduction

The Nantes sugar refinery is located at the mouth of the river Loire, in the urban centre of the city of Nantes, 250 miles west of Paris. It is one of the $\mathbf{1 5}$ plants of the Béghin-Say Sugar Division, which is one of Western Europe's largest sugar manufacturing companies. It has a daily output of 450 tonnes and manufactures different types of products: several granulated sugars, tablets, specific sugars such as "La Perruche", candy sugar (fine or medium), "L'Antillaise", the "Vergeoise", liquid sugar, etc. . . for direct consumption as well as for industrial use.

At the end of 1978, the decision was taken to modernize this old plant, the construction of which dates from 1936. Owing to the high investment cost, this modernization was carried out in several stages.

By mid-1979, the boiling pans and crystallizers of the recovery house had been replaced and new Chambon machines for the manufacture of tablet sugar were installed.

At the same time, the first continuous vacuum pan for the white sugar first strike crystallization was set up experimentally. This Fives-Cail Babcock device, type M 320 (heating surface $324 \mathrm{~m}^{2}$, capacity $32 \mathrm{~m}^{3}$, massecuite output 28 tonnes $/ \mathrm{hr}$, fully automated) went on stream by the third quarter of the same year.

In a continuous boiling pan, operated in a sugar mill, the stirring of the massecuite is brought about by the incondensables in the steam. In the sugar refining process, these incondensables are almost non-existent and it is necessary to use around $600 \mathrm{~kg} / \mathrm{hr}$ of steam to stir the massecuite. However, this additional steam consumption is counterbalanced by the continuity of the processing.

Because of the high energy consumption, which stems from the wide range of products manufactured, the Company was led to re-consider the refining process as a whole, focusing on energy-saving. Solutions were sought, firstly through an increase in extraction yield with a continuous vacuum mixer, and secondly through the recycling of the boiling steam and the mechanical recompression of part of the steam.

The theoretical studies of vacuum crystallization (see calculations below) brought out the fact that continuous cooling by means of flash evaporation of the massecuite diluted in its own mother liquor leads to an increase in extraction yield, and therefore to a saving of heating steam for boiling significantly higher than with any other existing process or device which uses air or water as a cooling agent.

## The semi-experimental installation

By the beginning of 1980, the decision was taken to set up experimentally a vacuum mixer connected with a continuous boiling pan. As may be seen in the diagram (Fig. 1), the semi-experimental installation comprised:
(1) a $30 \mathrm{~m}^{3}$ boiling pan, fitted with a stirrer, operated automatically (V.P.) for the boiling of magma footing (actually, this equipment was also used for the second strike boiling),
(2) a $40 \mathrm{~m}^{3}$ isothermal mixer ( M ) to store the magma,
(3) a FCB M320 continuous vacuum pan for the first strike (CVP),
(4) a $20 \mathrm{~m}^{3}$ vacuum crystallizer (CVC) with two

[^3]

Fig. 1. Flow sheet of the 1st strike continuous boiling and crystallizing process at Nantes sugar refinery, 1981
compartments, connected to the high vacuum condenser (CHV) which gave over 28.7 in Hg ,
(5) a recycling circuit for the first strike low-grade syrup (LGS) suitably re-heated (H) and demulsified (D), and
(6) an adjustable flow massecuite pumping system (inlet and outlet of the vacuum mixer), while
(7) the continuous functioning of the installation was provided by a complete control and regulation system (regulation of pressure, vacuum, temperature, outflow, level, Brix, consistency).

## Operating principle

The magma footing $\left(88^{\circ} \mathrm{Bx}, 80^{\circ} \mathrm{C}, 30 \%\right.$ crystals of 0.25 mm aperture, $25 \%$ of the massecuite outflow of the continuous vacuum pan) is pumped into the continous vacuum pan which is itself fed with standard syrup SS1 $\left(70^{\circ} \mathrm{Bx} 70,75^{\circ} \mathrm{C}\right.$ ) made up of char liquor and first strike wash syrup.

The massecuite from the continuous vacuum pan $\left(90-91^{\circ} \mathrm{Bx}, 80-82^{\circ} \mathrm{C}\right.$ ) is pumped into the first compartment of the crystallizer inside which a regulated vacuum at $24.8-25.2 \mathrm{in} . \mathrm{Hg}$ is maintained. The auto-evaporation of the water from the hot products entering a chamber under vacuum creates a powerful flash at the intake of the massecuite. As a result, there is an increase in mother liquor supersaturation, a fast increase in grain size and an increase in the massecuite viscosity.

The dilution syrup, which is actually the mother liquor of this massecuite, previously re-heated to $75-80^{\circ} \mathrm{C}$ and demulsified, is fed continuously into the crystallizer. This re-heating - which can be carried out by means of hot condensate or exhaust steam - generates the flash evaporation of part of the water from the syrup in the
massecuite, which definitely improves mixing. The mother liquor outflow and its distribution to several intakes of the crystallizer are adjusted in order to maintain a constant stirring of the massecuite and high supersaturation of the mother liquor while providing an adequate viscosity of the massecuite.

The circulation inside the mixer is continuous. The massecuite remained 20 min in the first compartment, at around $65^{\circ} \mathrm{C}$. The vacuum inside the second compartment was higher (around 26.7 in Hg ). This difference of vacuum results in a permanent flow of massecuite from one compartment into the other. The massecuite temperature was $55^{\circ} \mathrm{C}$ in the second compartment where it remains for around 25 min .

Owing to the difference in vacuum between the two compartments, the level was higher in the second than in the first. At the outlet of the second compartment, the massecuite was pumped continuously into the old Werkspoor mixer (WrC) in order to achieve cooling and centrifugalled at around $43.45^{\circ} \mathrm{C}$.

## Results

We carried out several series of adjustment and efficiency trials in order to investigate various possibilities of the installation. Alterations and adjustments were made so as to improve the operating conditions and achieve a more reliable and efficient installation; these included:
modification of massecuite, syrup and vacuum circuits in order to suppress clogging and sugar removal and to achieve a homogeneous massecuite,
modifications or transformation of control and regulation circuits and appliances, to achieve a suitable automated functioning of the system,
adjustments of pressures, vacuums, temperatures, outflows, levels, Brix and consistency at various stages of the circuit in order to reach optimum operating conditions, and
adjustment of the installation to existing operating conditions (weekly 48 hr stop, insufficient storage facilities for the sugar produced, etc.)
Results of the trials obtained during November $2-6$ 1981 are indicated in Table I.
improved further by increasing the mixing capacity, so as to reach a higher vacuum.

## Description of the present installation

We are now able to control all the operating parameters of the vacuum crystallizer. The results of the trials also led us to set up a second vacuum crystallizer

| Table 1 |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Products | ${ }^{\circ}$ Brix | Temperature, ${ }^{\circ} \mathrm{C}$ | Weight of dry substance | Crystal weight \% dry substance | Extraction ratio* | M.A., mm | C.V., |
| S S 1 | 70.5 | 75 | 76.37 | - | - | - | - |
| Magma | 86.3 | 80 | 23.63 | - . | - | - | - |
| "Continuous" massecuite | 90.0 | 81 | 100.00 | 58.0 | 1 | 0.60 | 35.8 |
| Recycled low grade syrup | 73.0 | 75 | $\begin{gathered} \text { CVC } \\ 32.50 \\ W \\ 2.50 \end{gathered}$ | - | - | - | - |
| Massecuite outlet from C.V.C. | 88.1 | 54 | 132.50 | 58.2 | 1.33 | 0.62 | 33.1 |
| Massecuite outlet from Werkspoor | 87.4 | 43 | 135.00 | 63.4 | 1.11 | 0.64 | 31.3 |
| N.B. Extraction yields are calculated from conductimetric ash measurements. The extraction ratio is determined by the ratio mass of crystals out : mass of crystals in. |  |  |  |  |  |  |  |

We were able to draw the following preliminary conclusions:
(1) The processing under vacuum inside the compartments - within the limits of the formation of poor grain - led to an increase in crystallization yield of more than $35 \%$.
(2) The average grain aperture increased slightly; of course, this increase is not the same as the cube root of the ratio of the weights of crystals leaving and entering the vacuum mixer, because the finer grains are more numerous and have a faster increase in weight than the larger crystals. In fact, we have usually observed a decrease in C.V., often higher than 2 points, because there has been no fine grain formation during the vacuum mixing.
(3) Centrifugalling of the cool massecuite $\left(43-45^{\circ} \mathrm{C}\right)$ did not present any specific problem.
With a well decolorized mother liquor (colour lower than 100 ICUMSA units) the quality of the sugar produced remained quite satisfactory, with under 8 European points (standard for EEC 1 grade white sugar). Lowering of the quality of sugar might have been expected owing to the improvement in extraction (and consequently a lower purity of the mother liquor) as well as to the. low temperature of centrifugalling. This seems to be counterbalanced, however, by the fact that crystallization can be carried out without steam heating and therefore without colour formation.
(4) The cooling in the Werkspoor mixer still improves crystallization yield; in other words, the efficiency of the vacuum crystallization may be
which has been connected with the first, which is no longer partitioned into two compartments (Fig. 2). During the yearly stop of the refinery, in August 1982, a $30 \mathrm{~m}^{3}$ non-compartmented crystallizer was installed.

Instead of a two-compartment crystallizer, we thus have two connected vacuum crystallizers with a volume - and thus a duration of crystallization - more than doubled. Characteristics and costs are noted in Tables II and III.

| Table II. Main characteristics of the continuous vacuum <br> crystallizers |  |  |  |  |
| :--- | ---: | ---: | :---: | :---: |
| No. 1 |  |  |  | No. 2 |
| Overall length, mm | 7250 | 8050 |  |  |
| Inside length, mm | 5950 | 6100 |  |  |
| Overall height, mm | 3000 | 4650 |  |  |
| Inside diameter, mm | 2400 | 2900 |  |  |
| Thickness, mm | 10 | 10 |  |  |
| Total volume, $\mathrm{m}^{\mathbf{3}}$ | 25 | 37 |  |  |
| Live volume, $\mathrm{m}^{\mathbf{3}}$ | 26 | 20 |  |  |
| Weight empty, kg | 8300 | 12,900 |  |  |
| Gearmotor, kW | 2.2 | 4 |  |  |
| Stainless steel |  |  |  |  |

## Operating results

At the start-up of the installation, we needed just a few days to adjust the regulation parameters and optimize the operating conditions. We reached an extraction ratio we had never before obtained: $K=1.5$ and above, with a massecuite centrifugalled at around $40^{\circ} \mathrm{C}$. The Werkspoor crystallizer is not used any longer.

This outstanding result enabled us to limit the number of strikes to two as the volume of massecuite had been


Fig. 2. Flow sheet of the 1st strike continuous boiling and crystallizing process at Nantes sugar refinery, 1982

| Table III. Cost for the setting up of a vacuum mixing facility <br> in US $\$$ as at January 5,1983 |  |  |  |
| :--- | ---: | ---: | ---: |
|  | 1981 | 1982 | Total |
| Mixer only | 76,944 | 70,277 | 147,221 |
| Civil works | 6,222 | 37,361 | 104,583 |
| Piping | 24,305 | 47,083 | 71,388 |
| Electricity | 1,388 | 7,083 | 8,471 |
| Control equipment | 21,111 | 8,888 | 29,999 |
| Heat insulation | 5,000 | 13,750 | 18,750 |
| Adjustments | $\underline{7,916}$ | 1,527 | 9,443 |
| Total | 203,886 | 185,969 | $\mathbf{3 8 9 , 8 5 5}$ |

cut down by $20 \%$ when the 2 nd vacuum crystallizer started operating. As a consequence, the steam saving is quite significant. The purity of the low-grade syrup came down to around 97 .

Results obtained during September 20-24, 1982 are given in Table IV.

The quality of the sugar produced declined to 8.15 European points (maximum 8 points for EEC 1 grade white sugar). The high colour was due not only to the too low centrifugalling temperature but also to the poor quality of the raw sugar processed at that time. Our purification-decolorization facilities had not yet been modernized and, as a result, the syrup was processed at

| Table IV |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Products | ${ }^{\circ}$ Brix | Temperature, ${ }^{\circ} \mathrm{C}$ | Weight of dry substance | Crystal weight \% dry substance | Extraction ratio | M.A., mm | C.V., |
| SS 1 | 68.6 | 74 | 63.77 | - | - | - | - |
| Magma | 86.7 | 79 | 36.23 | - | - | - | - |
| "Continuous" massecuite | 90.0 | 82 | 100.00 | 54.4 | 1 | 0.54 | 33 |
| Recycled low grade syrup | 72.6 | 78 | 56.3 | - | - | - | - |
| Massecuite outlet from C.V.C. 1 | 88.0 | 61 | 121.7 | 56.8 | 1.27 | 0.59 | 32 |
| Massecuite outlet from C.V.C. 2 | 86.1 | 40 | 156.3 | 58.1 | 1.31 | 0.61 | 31 |
| Total extraction ratio $\mathrm{K}=1.27 \times 1.31=1.66$, so that the theoretical increase in extraction yield $=66 \%$. |  |  |  |  |  |  |  |

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more than 130 ICUMSA colour units. With an average quality syrup the quality of the sugar can be improved to 8 points. A slight re-heating of the massecuite before centrifugalling will be necessary.

We have also noted that, in some cases corresponding to a low Brix of massecuite at the outlet of the continous boiling, an extraction yield above $70 \%$ could be reached. There is an auto-regulation of the system as a lower extraction in the boiling is almost made up for by a better extraction in the vacuum mixer.

## Plant operation with relation to the production personnel

Once our engineers and technicians had completed the final adjustments, the installation worked entirely automatically, almost without supervision. The training of the operating personnel who attended the trials required a very short period of time as the functioning of the installation is very simple and the regulation system which we devised is highly reliable.

## Conclusion

The continuous vacuum crystallization process developed by our company for the first strike at Nantes Refinery met perfectly the objectives set according to theoretical calculations. A system of vacuum crystallization can be connected with a battery of discontinuous boiling pans without any problem, provided that the massecuite is stored into a buffer mixer. This buffer mixer should be isothermal in order not to lose the benefit of the stirring resulting from the flash evaporation of the product.

We think that further interesting applications will be possible, particularly for the other refinery strikes, in beet sugar factories as well as in cane sugar refineries, whether they are equipped with continuous or discontinuous pans. The simplicity of the equipment, the ease of operation, the improvement in uniformity of grain and, above all, the accompanying energy saving resulting from the decrease in the quantity of massecuite to be processed, makes it certain that this process will develop rapidly.

## Summary

The Béghin-Say company has recently developed a continous vacuum crystallization process, operated on the first strike massecuite at the Nantes refinery, which has brought about a $50 \%$ increase in crystallization yield while the quality of the sugar produced has been maintained and meets the standards for high quality EEC 1 grade white sugar.

## Cristallisation continue sous vide à la Raffinerie de Nantes

La Société Béghin-Say a récemment mis au point un procédé de cristallisation continue sous vide travaillant sur la masse cuite de ler jet, à la Raffinerie de Nantes, ce qui a amené un gain d'environ $50 \%$ en rendement de cristallisation. La qualité du sucre produit a continué à satisfaire aux normes sévères de qualité du sucre blanc du type C.E.E. No. 1.

## Kontinuierliche Vakuum-Kristallisation in der Raffinerie Nantes

Vor kurzem hat Béghin-Say einen kontinuierlichen Vakuum-Kristallisationsprozeß für Erstprodukt-Füllmassen entwickelt, der in der Raffinerie Nantes arbeitet und der eine ungefähr 50\%ige Erhöhung der Kristallausbeute ermöglicht hat, während die Qualität des erzeugten Zuckers weiter den hohen Qualitäts-Standards der EGQualität Nr. 1 für Weißzucker entspricht.

## Cristalización continua en vacuo a la refinería de Nantes

La Sociedad Béghin-Say ha desarrollado recientemente un proceso de cristalización contínua en vacuo trabajando sobre la masa cocida de la primera templa a la refineria de Nantes en Francia, que ha logrado un aumento de 50\% en rendimiento de cristalización. La calidad del azúcar producido ha mantenido satisfacer las normas severas de calidad de azúcar blanco del tipo CEE No. 1.

Belize government to purchase Tate $\&$ Lyle holdings ${ }^{1}$. - The Belize government has decided to start negotiations with Tate \& Lyle PLC to buy $85 \%$ of its holdings in the Belize sugar industry ${ }^{2}$. The decision to negotiate, taken after consideration of a UN evaluation of Tate $\&$ Lyle's proposals, was given close attention and a sub-committee named by the government is to prepare a plan for the terms and conditions of the transfer of shares.

Zaire sugar industry expansion ${ }^{3}$. - Sugar production at the Kiliba factory in Zaire is to be raised from 11,000 to 28,000 tonnes per annum. At the same time the cane area is to be extended from 2100 to 3000 hectares. The project will require the purchase of agricultural and factory equipment, improvement of the infrastructure and maintenance, as well as technical aid. Total costs are estimated at approx. $\$ 30$ million.

Indonesia HFS production ${ }^{4}$. - More than 20 companies have so far filed high fructose syrup project proposals in recent years, using cassava as a raw material, in order to meet the rapidly growing demand for sweeteners. One plant is already operating, a second is under construction and building of a third has started. Four plants have been licensed by the government, 12 have obtained provisional licences and three others are being evaluated. Indonesia's annual cassava output of around 13 million tonnes goes mainly for domestic consumption, but at present cassava supply is largely inadequate for large-scale agroindustry operations because farmers plant the low-priced crop only if it is unrealistic to grow alternative crops such as rice. Government officials are reported to back the HFS scheme as they say demand for sweeteners is rising rapidly. Indonesia hopes to achieve self-sufficiency in sugar with a balance of consumption and production at 2.16 million tonnes by 1985. Production in 1983 is estimated at 1.6 million tonnes against a consumption of just less than 2 million tonnes.

New cane sugar factories in Pakistan. - The Rs. 274 million (approx. $£ 14$ million) Faran Sugar Mills Ltd. sugar factory at Sheikh Bhirkio in Sind Province went into trial production on November 25, 1983. The 2000 t.c.d. plant, with provision for extension to 3000 t.c.d., makes plantation whites only, and was built in just 11 months by Karachi Shipyard \& Engineering Works Ltd., in collaboration with Fives-Cail Babcock. Teething troubles over, the factory is expected to crush 280,000 tonnes of cane in the current season, making about 26,600 tonnes of white sugar. The factory is owned and operated by the Bawany Group, in the private sector, who have another sugar factory in the same province. Ittefaq Sugar Mills Ltd., at Noorpur in the Sahiwal district of Punjab, was formally inaugurated by the President of Pakistan on December 29, 1983. It is the country's 38th sugar factory and was supplied by Ittefaq Foundries Ltd. in collaboration with Buckau-Wolf of Grevenbroich, Germany. The 4000 t.c.d. unit is the largest in the country and cost an estimated $\mathbf{5 5 0}$ million rupees (about $£ 28.5$ million). In a normal season, the factory is expected to make some 57,600 tonnes of white sugar. Pakistan's cane sugar factories now have a combined cane crushing capacity of 76,630 t.c.d. and the estimated white sugar production capacity is 983,024 tonnes per season. Beet sugar production capacity is 36,750 tonnes/year, giving a total capacity of $1,019,000$ tonnes/year.
Zanini distillery sales. - In 1983 three turn-key autonomous distilleries built by Zanini entered operation with a total capacity of 450,000 litres/day. Equipment was also supplied to a further 16 distilleries in Brazil, while the first distillery to be exported by the company was supplied to Cía. Nacional de Melazas of Guayaquil, Ecuador, for production of 30,000 litres/day of alcohol. The value of the contract was $\$ 800,000$.

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## Sugar Industry Technologists Inc

## 43rd Annual Meeting, 1984

The 1984 meeting of Sugar Industry Technologists will be held at the Westin Galleria Hotel in Houston, Texas, during May 6-9. Registration will take place on the first of these days as will meetings of the Executive Committee and Board of Directors, while a "mixer" will be held for members and guests in the evening. The technical meetings will commence on the following morning with an address of welcome by the President, Bruce Foster (St. Lawrence Sugar, Canada), followed by papers on "Management philosophy at Savannah sugar refinery" by George Fawcett, "Some incentives for improving raw sugar quality" by John Alexander and Bernard Ravnö, "Non-sucrose components of raw sugar and efficiency of press filtration" by James Deveraux and Margaret Clarke, and "Continuous centrifugal development for high-grade massecuites" by Christina Goodacre, Henry Bristow and Bob Connor.
The 43 rd Annual Meeting of S.I.T. will then be held and a new Board of Directors elected for 1984-85. After lunch, Ted Stephenson (Atlantic Sugar, Canada) will chair a symposium on the pros and cons of different adsorbent systems, with contributions by Dennis Martin (Tate \& Lyle) on resins, L. G. Sansaricq (Amstar) on bone char, Lyle Zemanek (C \& H Sugar) on "Canesorb" and John McManus (Atlantic Sugar) on carbon.

On the following morning the program includes a paper on "Energy conservation" by Chung Chi Chou, "Energy savings in the new Tirlemont refinery" by

Marcel Braeckman, "Programmable control at the C \& H sugar refinery" by Dennis Mosher, "The application of SCADA (Supervisory Control and Data Acquisition) for process control at Imperial Sugar" by Brian Harrison and Joe Ruzicka and "Thames refinery fibre optic system for computer control of process" by John Fitzpatrick.

After lunch, "The sugar refining process at SSA ArIöv, Sweden (rebuilt 1981)" will be described by Gert Akesson and Kaj Lilja, while the remaining papers include "TALO clarification at Godchaux-Henderson" by Jim Burt and Calvin Rousse, "New resin decolorization station at Finnsugar's Porkkala refinery" by Leif Ramm-Schmidt and Goran Hyoky, "Sugar and alcohol technology - quality and uses in Brazil" by Dietrich Quast, Manoel Sobral, Jr., and Julio Borges, and "Looking back at sugar technology" by Mark Hertzberg.

The President will close the Technical Session at the end of the day, but the Banquet and presentation of awards relating to the 1983 meetings will take place in the evening. On the following day, there will be a tour of Imperial Sugar Company in the morning followed by a Texan Barbeque hosted by Imperial. Members should register for the meeting no later than April 26, at a cost of $\$ 150$ ( $\$ 100$ for accompanying spouses); registration forms may be obtained from Sugar Industry Technologists Inc., P.O. Box 1407, Oak Harbor, WA 98277, U.S.A.

UK beet sugar production ${ }^{1}$. - A British Sugar spokesman said at the end of January that the UK sugar crop amounted to 1,060,000 tonnes in the 1983/84 campaign, against the 1982/83 record. of 1.4 million tonnes, white value.

Japan refinery companies liquidation ${ }^{2}$. - Dai-Nippon Sugar Manufacturing Co. and Meiji Sugar Manufacturing Co. have said they would go into voluntary liquidation in mid-February and transfer all employees and business rights to new companies to be established by Mitsubishi Corporation, the principal shareholder of the two debt-ridden sugar refining companies.

Beet sugar project for Maxico ${ }^{3}$. - British companies are expected to win contracts for joint ventures worth more than $\$ 600$ million in the State of Sonora in Mexico. Among the prospective projects is one by Tate \& Lyle to establish a beet sugar project on the coast of the state.

New US HFS facilities ${ }^{4}$. - Cargil Inc. is building a corn wetmilling plant in Eddyville, lowa, to be completed in early 1985. Projected HFS production capacity is 300,000 short tons a year, dry basis. The Minnesota Corn Processors plant in Marshall, MN, began to operate in July 1983 and is being expanded to produce about 250,000 tons of HFS per year. Ultrasonics Inc. plans to construct a small corn wet-milling facility in Holtville, California, to produce both HFS and alcohol.

US beet sugar production, 1983/84 ${ }^{5}$. - McKeany-Flavell Co. Inc., forecast US beet sugar output in the 1983/84 campaign at $2,771,300$ short tons, raw value, against $2,717,800$ tons in 1982/836. They have now reported that growing conditions during September and October for many areas of the country were less than ideal and have now amended the estimate to a total of $\mathbf{2 , 6 1 8 , 8 2 5}$ short tons, or less than the previous campaign.

Refining in the USSR ${ }^{7}$. - With the increase in cane raw sugar refining in the USSR - in the current vear white sugar from this source will constitute $30 \%$ of the total national white sugar output - the industry has also increased its refining efficiency from a recovery of $\mathbf{9 2 . 7 8 \%}$ on weight of sugar in 1970 to $95.36 \%$ in 1983. A large number of sugar factories in the Soviet Union, including many in the eastern republics, process cane raw sugar as well as beets at an average daily refining capacity of 159 tonnes of raw sugar.

Pakistan fuel alcohol project ${ }^{8}$. - The Minister of Oil and Natural Resources has stated that he has a project for the development of industrial alcohol. A feasibility study of alcohol production from molasses and its use as fuel for trucks has already been drafted and calls for an investment of 300-400 million rupees ( $\mathbf{\$ 2 2 5 - 3 0 0}$ million).
Sierra Leone sugar output, $1982 / 83^{9}$. - The Magbasa sugar factory near Magburaka in the northern province is reported to have produced 6600 tonnes of granulated sugar against a claimed present capacity of only $\mathbf{6 0 0 0}$ tonnes, although it is believed to be designed to produce 20,000 tonnes/year ultimately. The Chinese who built the plant have just signed a further contract with the goverment to manage the operation; this suggests that the training program instituted by the Chinese to give Sierra Leoneans the opportunity to run the factory is not yet complete. Sugar from Magbasa, it is claimed, now meets about one-third of domestic demand which thus must be running at about 20,000 tonnes per year. Per caput consumption is thus about $6 \mathrm{~kg} / \mathrm{year}$. With fast population growth (2.5\%) and an economy that can ill afford imports, the prospects for rising consumption levels seem bleak even though latent demand could, if satisfied, easily double per caput consumption. Even when Magbasa produces at its designed capacity it will not be able to satisfy domestic demand completely since this seems set inexorably to reach at least 25,000 tonnes per year by 1990 unless consumption is further stifled by higher prices.
Sweden campaign results, $1983{ }^{10}$. - In the 1983 campaign which ended on December 12, the seven sugar factories of Sockerbolaget processed 831,616 tonnes of beet to give 224,516 tonnes of white sugar, $\mathbf{5 3 , 6 3 3}$ tonnes of raw sugar and 76,389 tonnes of molasses.
${ }_{1} 1$ Public Ledger, February 4, 1984.
${ }_{2}$ F. O. Licht, International 'Sugar Rpt., 1984, 116, 98.
3 Westway Newsletter, 1983, (122), 11.
4 USDA Sugar \& Sweetener Outlook \& Situation Rpt., Dec. 1983, 8.
5 MF Sweetener News, December 19, 1983.
6 I.S. J., 1983, 85, 352.
7 Sakhar. Prom., 1984, (2), 2-5.
8 Westway Newsletter, 1983, (122), 11.
9 F. O. Licht, International Sugar Rpt., 1984, 116, 15-16.
10 Zuckerind., 1984, 109, 88.

## British Society of Sugar Cane Technologists

The 1984 Annual General Meeting of the B.S.S.C.T. will be held at the Royal Commonwealth Society, Northumberland Avenue, London W.C.2., on Monday April 16, commencing at 11 a.m. After luncheon, the Spring Technical Meeting will be held, commencing at 2 p.m., the subject being "Information services in Britain". Applications for membership should be sent to the Honorary Secretary-Treasurer, Mr. W. N. L. Davies, c/o Tate \& Lyle Agribusiness, 45 Homesdale Road, Bromley, Kent BR2 9TE.

Pakistan sugar quality program ${ }^{1}$.-Government policy in Pakistan is to increase production of export quality white sugar and to improve the quality of the sugar cane crop. Manufacture of an internationally accepted quality of white sugar would give a better return to cane growers and sugar producers while the planners are also trying to improve factory efficiency. The plan provides for loans and assistance to growers for agricultural inputs and purchase of tractors and implements, and it is proposed to restrict the milling season to 160 days, from the first week in November to mid-April, to achieve maximum recovery. The planners have suggested measures to increase cane yield including emphasis on September planting instead of February. Efforts are to be made to secure export markets in Iran and the Gulf states for the surplus, calculated at 278,000 tonnes.

Bulk sugar terminal for Queensland ${ }^{2}$. - The Queensland Sugar Board plans to build and operate a $\$ 33.7$ million bulk raw sugar terminal in Brisbane. The terminal is intended to be fully operational by June 1985 with a first year export of 180,000 tonnes. It will have a storage capacity of $\mathbf{6 0 , 0 0 0}$ tonnes.

Fiji sugar production, 1982/83 ${ }^{3}$. - Output of raw sugar in Fiji in 1982/83 fell to 276,000 tonnes from the record 487,000 tonnes produced in the previous season. The 1983/84 crop is not expected to be so low because the drought which affected the recent crop came to an end in October.

Belgium sugar production, 1983/84 ${ }^{4}$. - Belgian sugar production in 1983/84 reached 790,000 tonnes, white value, up from earlier estimates but down, as expected, from the $1,130,000$ tonnes of the previous campaign. Despite late sowing, followed by drought and frost affecting a few hundred hectares of beet in December, the 1983/84 campaign thus finished very favourably. Average yield is estimated at 51 tonnes/ha and average sugar content 15.84\%.

GEPLACEA role in the world sugar economy ${ }^{5}$. - In an article prepared by the Secretariat of the Group of Latin American and Caribbean Sugar Exporting Countries (GEPLACEA), a number of conclusions were drawn with respect to the development of the Group and its position in the world sugar economy in recent years. The Group's share of world production has declined owing to a lower growth rate, while its share of consumption has increased, and that of exports has declined. Brazil, Cuba, Mexico and the Philippines represent more than $70 \%$ of the Group's production, and Brazil and Mexico account for more than 50\% of the Group's consumption. Cuba provides more than $50 \%$ of the Group's exports and Mexico and Venezuela more than $90 \%$ of net imports. The USA and USSR are important customers for the Group.

Zimbabwe sugar industry economics ${ }^{6}$. - The retail price of sugar was increased by $26.5 \%$ from January 3, which should bring the producers, Hippo Valley Estates Ltd. and Triangle Sugar Estates Ltd., to a break-even point and give the refiners ZSR a financial base for expansion. The estates will benefit most from the increase because the raw sugar price has not been increased since 1978 and they had been losing money on domestic sales as well as exports. Soaring demand and ZSR's inability to finance the expansion of its refineries led both the estates to re-commission idle refinery sections in their factories; each will produce 15,000 tonnes of white sugar in 1984, and maximum output will be 50,000 tonnes a year from each refinery.

New Yugoslavian sugar factory ${ }^{7}$. - A new sugar factory, supplied by Poland, has been put into operation at Padinska Skela, near Belgrade, with a daily slicing capacity of 6000 tonnes. The factory is designed to avoid environmental damage and processing is fully automated.

UK sugar imports and exports ${ }^{8}$


Venezuela sugar industry ${ }^{9}$. - Total output of refined sugar in Venezuela during 1982/83 accounted for only $57 \%$ of the country's domestic requirements with the shortfall covered by imports, including 200,000 tonnes from Brazil and the remainder purchased on the world market. Venezuela has not covered its own needs since 1972, when it was able to export 144,000 tonnes of sugar, and has had to rely on imports to meet requirements. The root of the problem was that the Venezuelan government had kept sugar prices too low for too long. With the exception of two factories - Rio Yaracuy and Rio Turbio all the factories were privately owned; however, as money was lost and they fell into debt the factories were taken over by the State. At present there are only four factories in private hands with eleven others owned by the Corporación Venezolana de Fomento (CVF) and an additional two operating as mixed capital ventures.

PERSONAL NOTES
At the October 1983 general meeting of the Association of Official Analytical Chemists, Dr. Margaret A. Clarke, Director of Sugar Processing Research Inc., New Orleans, was appointed General Referee for Sugars and Sugar Products.

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## CAME SUGAR MANUFACTURE

Use of digital simulation for the evaluation of evaporator schemes. L. Hernández C. CubaAzúcar, 1982, (Oct.Dec.), 51-54 (Spanish). - A simulation program has been developed for comparison of evaporation schemes and examples are given of some of the results obtained with its use.

Exhaustion of Louisiana final molasses. M. Matic and C. Wong. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 4952. - Weekly composite samples of final molasses were obtained from 20 sugar factories in Louisiana, one at the beginning of the 1980 crop and one at the end. They were subjected to analysis for sucrose, reducing sugars, ash, Brix and true purity. A drop in the reducing sugars: ash ratio from about 2 to about 1 during the season was observed, with a corresponding increase of 4-6 in molasses purity. Target purities calculated by the formula of Keller and by the SMRI (South Africa) formula showed that the latter indicated a higher recovery was possible than did the former. Samples were sent to SMRI for examination and it was confirmed that they were not completely exhausted, so that there is scope in the Louisiana factories for additional sugar recovery.

High test molasses: a possible solution to the crisis of the Puerto Rico sugar and rum industry. G. Samuels. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 53-58. - Production of rum has increased markedly in Puerto Rico and now meets $85 \%$ of the US market's needs. At the same time the sugar industry has declined and cannot provide sufficient blackstrap molasses for the rum producers who therefore have to import molasses, which leaves them vulnerable to interruptions in supply. It is suggested that the sugar factories produce high-test molasses instead of sugar; this would provide the raw material for rum manufacture while the factories would not have the expense of manufacturing sugar unless they choose to do so during periods of high prices. The bagasse fibre would provide fuel for both factory and distillery operations.

Boiler water treatment technology "state of the art": practical solutions to common problems. J. J. Opelka. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 59-65. - A survey is presented on the conditioning of boiler water by external and internal treatment for reduction of hardness, dissolved oxygen, etc., as well as condensate treatment to prevent corrosion in piping. Means of overcoming a number of problems are discussed: these include chemical feed control, blow-downs, and boiler cleaning and preservation while out of use.

Design and aeration requirements for cane wash ponds. Y. K. Cho and D. F. Day. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 66-69. - Environment protection regulations have obliged Louisiana sugar manufacturers to recycle their cane wash water but the fermentation occurring has resulted in acid formation and subsequent corrosion of the wash table. Acid formation is attributed
to incomplete oxidation of the organic matter and can thus be countered by adequate aeration and retention time, and calculated values are tabulated for calculation of requirements in a specific case.

Total energy. V. Baillet. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 70-71. - All but one of the Louisiana sugar factories has low-pressure boilers suited to the steam engines in use when they were installed. Steam turbines have replaced the engines but their efficiency is low under the steam conditions available, while power demand has grown to meet air and water pollution control requirements, thus creating an imbalance met by purchase of electricity from public supplies or use of gas as supplementary fuel, both of these increasing continually in cost. A series of recommendations is made for increasing boiler performance and efficiency with the aim of eliminating the need for gas and bought power.

A practical method of evaluating polymer flocculants in factory operation. J. C. P. Chen, J. S. Rauh and P. R. Arellano. J. Amer. Soc. Sugar Cane Tech., 1983, 2, $72-$ 77. - Assessment of the effectiveness of polymer clarification aids in many sugar factories is on a basis of acceptable clarity of juice and mud level; these are not necessarily the best achievable and the dosage may not necessarily be the optimum. Experience is quoted of testing with a kit devised by the Sugar Research Institute in Queensland and made by Fletcher and Stewart Ltd. in England; use of this from time to time to examine the benefits of existing practice, and testing of variations in procedure and material is recommended.

Acid wash ponds (causes and solutions). D. F. Day. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 78-81. - The extent of pollution load and sugar loss at Cora Texas sugar factory in Louisiana was measured and found to be $2.5 \%$ sugar on cane, producing an effluent of 3500 ppm BOD. The pH profile along the wash pond was examined and found to rise in the vicinity of the aerators; however, aeration was insufficient to give complete oxidation of the pollutants and gave rise to acid water for recycling, which led to wash table corrosion. At St. James factory, however, there was no problem in spite of an identical aeration system; this is attributed to the volume of the pond which is double that of the one at Cora Texas and allows a retention time of 15 hours.

Taking maximum advantage of hydraulics when automating a sugar mill operation. E. Alfonso. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 88 (Abstract only). - The history of hydraulics is briefly surveyed, with a description of several applications and information on the choice of hydraulics for these applications. Also included is a description of blending with pneumatic, mechanical and electrical loops.

Boiling techniques and ranking parameters in the evaluation of surfactants. Part I. Boiling characteristics and ranking parameters. J. C. P. Chen, J. S. Rauh, B. A. Smith and R. V. Romo. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 88 (Abstract only). - In order to compare surfaceactive formulations, a uniform sugar boiling scheme has been designed to keep all operating aspects constant so that the only variable remaining for comparison is the surfactant. A pilot vacuum pan was constructed in late 1979, with the capacity to boil a full strike of 5.1 US gallons ( 19.3 litres) of massecuite. The pan was equipped with a massecuite agitator, a proof stick and monitors for both supersaturation and massecuite consistency.

Two series of boilings have been carried out: one, which used $B$-molasses from Texas, collected at mid-season in 1979, which represented normal-quality material for $C$-strike boiling, and a second which used $B$-molasses from Florida, collected during the latter part of the 1979 season and containing high levels of dextran and aconitic acid. The criteria used for ranking of 31 surfactant formulations included boiling time, solids recovery and sugar quality, the last graded according to grain size (MA) and uniformity (CV), colour and ash.

Milling quality of four sugar cane varieties processed with trash. B. L. Legendre. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 89 (Abstract only). - Good milling quality, essential for the overall acceptance of a new variety by the sugar industry, is carefully evaluated before a variety is released. However, during the 1980 harvest season, reports from several factories cited CP 70-321 released in 1978, for poor "millability" and lower than expected sugar recovery. Previous studies under controlled conditions had indicated that CP $70-321$ had above-average milling quality. Fibre content and normal juice extraction are the main factors used to evaluate milling quality, while another factor, the varietal correction factor (VCF), is used in the calculation of recoverable sugar per tonne of cane. The milling quality of CP $70-321$ cane, containing $13.6 \%$ trash, was compared with that of commercial varieties CP 65-357, CP 70-330 and N:Co 310 with $14.1 \%, 14.8 \%$ and $19.9 \%$ trash, respectively, in four tests; two were conducted with plant cane and two with first ratoons. Trash in these tests included all leafy tissue still attached to the stalks after the tops had been removed above 10 cm above the stalk apex. The results showed that CP 70-321 had the lowest fibre content ( $13.6 \%$ ), the highest normal juice extraction ( $76.3 \%$ ) and highest VCF ( 0.943 ). The other varieties had similar fibre content ranging from $14.7 \%$ to $15.1 \%$, normal juice extraction in the range $73.3 \%$ to $74.2 \%$, and VCF ranging from 0.899 to 0.912 . The absolute milling quality of a variety cannot be judged unless the effects of trash are controlled; however, the overall ranking of varieties in regard to milling quality remains essentially the same with or without trash.

Combustion system for firing pulverized bagasse. G. G. Tauzin, D. Maples and G. L. Harper. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 89 (Abstract only). - A combustion system was developed for firing of pulverized bagasse. The system consists of a research furnace, combustion air system, flue gas system, furnace system and pulverized burner system. The combustion system was designed for multi-fuel firing for subsequent studies on alternative fuels. A chamber efficiency equation was developed for the combustion system. This equation gives the fraction of the fuel's heating value used in raising the product gases to the exit temperature. When adapted to moist bagasse, this equation will provide an analysis for the combustion of bagasse based on variable levels of moisture content. Preliminary firing of pulverized bagasse provided a self-sustaining flame with $40 \%$ excess air. This full-suspension type burn was produced in an axial swirl burner. The only secondary fuel used was a natural gas pilot that was left on during the bagasse firing. Different modifications to this burner, as well as different types of burner, will be tested in future firings. Size and moisture content will also be a variable in future tests. Initial testing, conducted with very dry 60 -mesh bagasse, is presented.

Evaluation of the differential conductimetric transducer for the crystal content in massecuite. F. Pantuso N. Centro Azúcar, 1982, 9, (1), 7-18 (Spanish). - Measurement of the crystal content in sugar solutions allows maintenance of a proper relationship between the crystal surface area and the dissolved sucrose so as to avoid the formation of false grain. Experimental results are reported on a test made with the title transducer at Central Espartaco, as well as the analytical method used to determine the variation of the resistance $\mathbf{R}_{\mathrm{t}}$ as a function of the crystal content. It is calculated that the relative error of the measurement is $1.48 \%$.

Technological process scheme for the purification of sugar cane juices. L. Gómez G. and R. González R. Centro Azúcar, 1982, 9, (1), 19-33 (Spanish). - A review with 31 references is given of part of the literature on separation of impurities by screening, settling or hydrocyclone treatment, followed by liming, heating, settling and filtration.

Variables and equations of state for the sugar crystallization process. F. Herrera F. Centro Azúcar, 1982, 9, (1), 79-85 (Spanish). - From other mathematical models and from operating conditions, appropriate variables of state are determined and used to derive a dynamic model and the equations of state which describe analytically the operations of a vacuum pan.

The quality of raw material and its influence on the different stages of the purification process simulated on a laboratory scale. L. Gómez R., A. P. Nikolaiev, P. M. Fabregat and A. M. Alonso. Centro Azúcar, 1982, 9, (1), 87-102 (Spanish). - Analyses were made of juice Brix, pol, reducing sugars, sucrose, purity and pH during the various stages of clarification of juice obtained from clean cane and cane with $8 \%$ and $10 \%$ of foreign matter (tops, earth and leaves). The results are tabulated and relationships between different characteristics examined. It is concluded that the content of foreign matter considerably affects the pol and reducing sugars in cane juice and that there is a correlation between sucrose content and pH . There is no appreciable variation in sucrose content and pH or cane quality if the cane is crushed fresh, i.e. within 24 hours of cutting. The content of foreign matter in the cane affected the clarity of supernatant juice in the settling process, although the settling rate tended to increase, possibly as a consequence of the presence of earth. With delay in milling, whether of clean cane or containing foreign matter, the sucrose content fell and the reducing sugars rose. The purity of clarified juice fell with increase in the content of foreign matter.

Heat transfer coefficients for natural-circulation evaporators. S. Y. Guo, E. T. White and P. G. Wright. Sugar J., 1983, 45, (14), 5-9. - See I.S.J., 1983, 85, 372.

Studies on the colour of sulphured syrup and sugar crystal. M. Prasad and S. K. Upadhyay. Sharkara, 1978, 17, (1-4), 7-12. - The optical densities of samples of sulphitation syrup from nine Indian sugar factories and the colour content (ICUMSA units) of samples of crystal sugar from the same factories were determined and tabulated. The results are discussed. Regular determination of syrup optical density is suggested; a value of 0.17 is considered the maximum at which the final sugar colour should be satisfactory.

## beti sucar WANUFACTURE

Stone and trash separation and trash processing as dry fodder. K. Hansen and N. Kolbye. Zuckerind., 1983, 108, 623-625, 627 (German). - Descriptions are given of a beet conveyor in the form of a studded rubber belt for removal of soil, and a twin-drum trash separator; both pieces of equipment were developed by SN Engineering A/S, a wholly-owned subsidiary of Sukkerfabriken Nykobing Lmt., all of whose newly designed plant is first operated for at least one campaign in their sugar factory. Details are also given of a system for removal of flume water pulp and separation of beet tails and pieces from trash and stones; the trash may be pressed and used as fodder, while the press water is preferably concentrated. Results are given of a scheme in which the trash was denatured with steam, transferred to two presses in series where the dry solids content was increased to $27 \%$ while the press water was concentrated to $40 \%$ dry solids in a two-stage evaporator using pan vapour, after which it was added to the trash sent to the pulp dryer.

Treatment of beet, beet pieces, trash and flume water. G. Gerlach. Zuckerind., 1983, 108, 627-630 (German). Illustrated descriptions are given of a Bammann \& Schreiber system and of the individual items of equipment embodied in it for stone separation, trash separation and treatment of this and flume water solids in a continuous push-type centrifuge acting as a pre-drying stage (raising the dry solids content from 11\% to at least $20 \%$ during two campaigns of continual testing). A perforated filter belt is used to remove solids from flume water; the solids, including beet pieces and tails, are separated on an endless belt with a lower run separated from an upper run and the latter vibrated vigorously. A classifier for removal of soil from dry beet is also described.

Screw press for beet pieces. R. Baur. Zuckerind., 1983, 108, 630-632 (German). - Details are given of a screw press manufactured by Grau Feinwerktechnik GmbH \& Co. which in tests at Wabern sugar factory in 1982 raised the dry solids content of a mixture of trash and beet pieces and tails from 12.1-21.6\% to 23-28\% at an hourly throughput averaging 7 tonnes (maximum 16 tonnes). The lowest input solids content given above occurred when no preliminary dewatering was used.

Beet tails and trash treatment. J. Söltzer. Zuckerind., 1983, 108, 632-634 (German). - Descriptions and diagrams are given of Köllmann + Gruhn equipment for separation of beet pieces and tails and green trash and their treatment, and a sccheme is shown that incorporates the individual plant.

Use of Rhewum magnetic screens in the sugar industry. W. Blachetta. Zuckerind., 1983, 108, 653-655 (German). The head of Rheinische Werkzeug- und Maschinenfabrik GmbH describes the salient features of Rhewum magnetic
screens and the various types available as well as explaining the theoretical basis on which they operate.

Tests on an automated station of modified FiLS filters for 2nd carbonatation juice at Sambor sugar factory. M. I. Zhenchuk et al. Sakhar. Prom., 1983, (7), 24-26 (Russian). - Details are given of the 2 nd carbonatation juice filter-thickener station at Sambor. Modifications included means of recycling the initial fractions of turbid filtrate, back-flow cleaning of the filter cloths, omission of complete emptying of the filters and increase in the distance between frames and in the diameter of the juice withdrawal tubes. Tests conducted on an automatic pilot plant are reported. During the trial periods, 1st carbonatation juice filtration properties fluctuated widely, with a filtration coefficient in the range $3-20$ and a settling rate of $1.5 \cdot 3 \mathrm{~cm} \cdot \mathrm{~min}^{-1}$ in the first 5 minutes. The solids phase in the filtered 2 nd carbonatation juice ranged from 0.007 to $0.058 \mathrm{~g} / \mathrm{litre}$, the colour from 12.8 to $14.4^{\circ} \mathrm{St}$ and total lime content from 0.11 to $0.30 \% \mathrm{CaO}$ at an average filtration rate of $14 \mathrm{I} \cdot \mathrm{m}^{-2} \cdot \mathrm{~min}^{-1}$ and a total cycle time of 110 minutes.

Effect of the design of the calandria of a vacuum pan on its throughput. V. T. Garyazha and A. V. Karpenko. Sakhar. Prom., 1983, (7), 26-29 (Russian). - Tests are reported with an experimental batch vacuum pan having interchangeable calandrias. In one form of calandria (A) the seven boiling tubes had a conventional diameter of 102 mm and a wall thickness of 3.5 mm , giving a total heating surface of $4.51 \mathrm{~m}^{2}$, while the other calandria (B) contained 13 tubes of 76 mm dia and with walls 3 mm thick, giving a total heating surface of $6.08 \mathrm{~m}^{2}$. The total boiling tube volumetric capacity was the same in both cases. Results showed that with calandria B the water evaporation rate was $38 \%$ greater than with calandria A, while the daily massecuite throughput was 13.8 compared with 10.9 tonnes; a $12 \%$ reduction in colour formation with calandria B was due to the shorter boiling time and to a thinner layer of over-heated massecuite in the tubes. However, there was some reduction in the average crystal size with calandria B and this was ascribed to lack of improvement in the hydrodynamics to balance the shorter time available for crystal growth, although this could be solved by increasing the length of the tubes.

Method and equipment for electrode cleaning in pH control of juices, syrups and other sugar factory intermediate products. Yu. V. Goryainov, V. V. Radchenko and K. A. Varfolomeeva. Sakhar. Prom., 1983, (7), $29-$ 32 (Russian). - Means used to clean electrodes in various countries are surveyed, and the advantages and disadvantages of mechanical, chemical and ultrasonic cleaning indicated. Chemical cleaning is preferred by the authors for a number of reasons which are stated, and an automatic cleaning system for electrodes used to measure carbonatation juice pH is briefly described.

Alteration to a scheme for automatic greasing of an A1-PDS-20 diffuser. V. I. Shevchenko and A. E. Tkachenko. Sakhar. Prom., 1983, (7), 36-37 (Russian). A proposed automatic control scheme for lubrication of the scroll bearings and discharge mechanism of the title diffuser is briefly described with the aid of a circuit diagram.

Effect of cossette liming on dewatering of beet pulp. J. M. Randall, W. Camirand and E. M. Zaragosa. J. Amer. Soc. Sugar Beet Tech., 1982, 21, 221-234. - Experiments are reported in which the effect of liming of cossettes on pulp pressing and drying was evaluated. Two methods
of liming were used, viz. mixing of the cossettes with powdered lime, and dipping of the cossettes in a thin slurry of lime in thin juice. In both cases, treatment was followed by a short equilibration period before diffusion. Results showed that treatment contributed to significant reductions in energy consumption in both pressing and drying, although emphasis was on pressing, since this is already less energy-intensive than drying and would thus react more favourably to cossettes treatment.

A comparison of forced ventilation and natural convection as means of cooling sugar beet storage piles in several geographic locations. R. E. Wyse and R. M. Holdredge. J. Amer. Soc. Sugar Beet Tech., 1982, 21, 235-246. - The effects of free convection and forced ventilation cooling of commercial beet piles in various regions of the USA were compared by means of a computer simulation model and showed that considerable benefit in terms of sucrose losses may be derived from forced ventilation at those locations having variable or mild autumn temperatures at harvest.

Results of tests on viscose-polypropylene bags for sugar packaging. V. F. Evfimenko, I. D. Stepchuk, S. A. Brenman and V. M. Pavlov. Sakhar. Prom., 1983, (8), 27-29 (Russian). - The advantages of viscose-polypropylene bags are discussed and comparison is made with natural fibre bags. The economic benefit of using the plastic bags for beet sugar is indicated.

Vapour compression in the sugar industry. K. E. Austmeyer. Zuckerind., 1983, 108, 715-728 (German). The thermodynamic principles of heat pump application to vapour compression as a means of fuel economy in a sugar factory are explained; two variants are described, viz. the absorption heat pump in which compression is effected by means of a heat absorber, and the heat transformer in which some of the waste heat is brought to a higher temperature and used for heating purposes. In both cases, replacement of the conventional throttle valve with a turbine permits electricity to be produced from waste heat. In examination of the potential application of vapour compressors, attention is focused on treatment of pan vapours. Sample arrangements and the required drive capacity under different operating conditions are demonstrated, and the economics of vapour compression and the amount of energy supplied by the different systems indicated. It is stressed that the results must be regarded only as rough guidelines. The point is made that, in a sugar factory operating along conventional lines, a noticeable reduction in power consumption can be achieved if the same enthusiasm is applied to control of the electricity distribution system as to heat flow control. A major problem lies in the limited time a sugar factory operates each year, since this restricts the time available for amortization of the investment required for vapour compression. For this reason, such investment is justified in the case of a factory of high energy efficiency only when there is a significant rise in the price of fuel.

Thermocompression at Zuckerfabrik + Raffinerie Aarberg AG: history, development and outlook. H. R. Brunner. Zuckerind., 1983, 108, 729-736 (German). Historical factors that led to the introduction of mechanical vapour compression at Aarberg sugar factory/ refinery in Switzerland in 1946 are explained, and details given of the technical background and performance of the compressor units for both the evaporator and pan stations; a major advantage of the initial scheme
was the autonomous operation of these two process stations, and particularly the low vapour temperature ( $<100^{\circ} \mathrm{C}$ ) which ensured that no juice colour increase occurred in evaporation. The disadvantage of having to use reduced steam direct from the boiler house for juice heating was substantially decreased when a pre-evaporator and steam-jet ejectors (acting as heat pumps) were added in 1972 in order to raise the quantity of heat required for an increased slice of 4500 tonnes/day; in 1975, a down-flow concentrator heated with vapour from the pre-evaporator was used for standard liquor treatment and thus lightened the load on the pan station compressor. With further expansion of the daily slice to 5500 tonnes/day, a second pre-evaporator was installed and thermal vapour compression introduced. For the future, a new energy plan has been conceived which would involve a quintuple-effect evaporator with twostage compression of the 2nd effect vapours; the scheme would provide enough steam for optimum processing of 8000 tonnes of beet per day.

Vapour compression in a raw sugar factory. Initial experiences with mechanical vapour compression at the Appeldorn sugar factory of Pfeifer \& Langen. H. Weidner. Zuckerind., 1983, 108, 736-742 (German). - The various schemes considered for Appledorn factory as a means of reducing energy consumption included mechanical compression of pan vapours and compression of the vapours from a quadruple- or quintuple-effect evaporator. The choice fell on compression of evaporator vapours (for reasons which are discussed); initial operation involved a quintuple-effect evaporator with preheater but without compressor, but at maximum beet processing of 5100 tonnes/day the 4 th effect proved to have an inadequate heat surface area, and 4 -stage operation with compression of 1 a and 1 b vapours was adopted for the remainder of the campaign. Comparison of the two schemes showed no significant differences in invert content or sugar degradation, while juice colour increased by a smaller extent when vapour compression was used. A Quentin unit was installed simultaneously with the vapour compressor, while better use was made of the heat from condensate and pan vapours. Advantages of each measure are indicated, and a total reduction of 4.11\% on beet shown between the 1980/81 and 1981/82 campaigns (before and after the changes). The economics are also discussed.

The single-stage radial compressor. U. Jacobsen. Zuckerind., 1983, 108, 742.746 (German). - Details are given of the Borsig GmbH GKS 900 radial compressor as installed at Appeldorn sugar factory (see preceding abstract). Potential applications and limitations of singlestage compressors are discussed, and a two-stage arrangement for a pan station is briefly described. Details are given of essential and recommended spare parts to be kept by a sugar factory so as to avoid standstill of a compressor, and the source of noise emission from a single-stage compressor are discussed.

Discussion on "Heat pumps in the sugar industry". P. Valentin. Zuckerind., 1983, 108, 746-748 (German). The question of suggested schemes, including heat pump application, is discussed against the background of investigations conducted by Süddeutsche Zucker-AG. The author is of the opinion that any expanded heat schemes, e.g. using gas turbines, involving pulp drying should be viewed with scepticism, and considers that use
of coal, possibly combined with other fuels, is justified if power generation is increased. The use of heat pumps in the evaporation station should be avoided because of unpredictability of electricity prices and because of the added costs of thermal separation processes, both factors making the heat pump an economically very risky means of utilizing waste heat. On the other hand, direct utilization of waste heat is to be encouraged.

Examples of utilization of pocket calculators in the sugar factory. M. Farinella and P. Parisi. Ind. Sacc. Ital., 1983, 76, 65-68 (Italian). - The applications discussed for a portable programmable calculator are the combustion of methane in a steam generator and the control of a quintuple-effect evaporator. The equations used in both cases are recorded and the program for the first, which provides a calculation of the loss in sensible heat as a function of the combustion conditions, is presented. The second program, illustrated by a block diagram, with inputs of the juice and steam feed temperatures, juice temperature and Brix in each body, etc., allows calculation of steam consumption, water evaporated, vapour withdrawal and thick juice and condensate quantities.

An automated system for process control. J. Radek and Z. Hotovy. Listy Cukr., 1983, 99, 177-179 (Czech). The basics of an automatic process control system in a sugar factory are explained, and the scheme introduced at Melnik factory for pan boiling control is outlined; this is a conventional three-tier system centred on a Soviet SM 3101 computer acting as data bank and overall strategic controller, with a JPR 12 R computer as local controller and the usual slave units.

Remote data transfer. J. Panec and K. Dobroruka. Listy Cukr., 1983, 99, 179-181 (Czech). - The role played by remote data transfer within an overall computerized control system is discussed, and the three basic types of data transfer used within the sugar industry are explained: data collection and distribution, the question-and-answer system, and information passed between computers. The advantages and disadvantages of each system are indicated.

Complex processing of information on preparation for and the course of a sugar beet campaign. K. Dobroruka, Z. Pochyly and B. Charamza. Listy Cukr., 1983, 99, 181-184 (Czech). - Details are given of a Czechoslovakian sub-system for beet campaign management, which involves supply of required quantities of fuel and limestone, etc. to the sugar factories (based on slicing capacity and predicted energy consumption) before the start of the campaign, and provision of daily, 5 - and 10 -day reports compiled from laboratory and factory data during the campaign.

A stock control sub-system. P. Rogner. Listy Cukr., 1983, 99, 186-189 (Czech). - A central stock control sub-system designed for use by the Czechoslovakian sugar industry is outlined.

Processing of the Basic Means sub-system. M. Hlavnickova. Listy Cukr., 1983, 99, 189-191 (Czech). - The title sub-system is a centralized scheme for stock control in the Czechoslovakian sugar industry based on economic data fed to a RC 3600 mini-computer in Prague. The scheme is to be expanded to include technical data.

Investigation of the rheological properties of a juicecossettes mixture. L. V. Zotkina, V. M. Lysyanskii and A. I. Fel'dman. Izv. Vuzov, Pishch. Tekh., 1983, (3) 118 (Russian). - In an investigation of the rheology of the juice cossettes mixture in a diffuser as a contribution to the design of a transport system, it was found that the bulk viscosity of the mixture was a function of particle size, diffuser specific load, displacement velocity and the physical properties of the particles. Mixtures containing fresh, unscalded cossettes and a sugar solution of low concentration may behave as dilatant and pseudo-plastic, non-Newtonian liquids; mixtures of well-scalded cossettes and low-concentration sugar solution act only as pseudoplastic, non-Newtonian liquids. In the temperature range $20-85^{\circ} \mathrm{C}$ and sucrose concentration in the dispersed medium of $\mathbf{0 - 1 5 \%}$, bulk viscosity was unaffected by the factors investigated; this was attributed to the fact that change in viscosity occurring between the particles in the film of sugar solution as a result of change in temperature and concentration had no essential effect on the coefficient of friction. Hence, change in the viscosity of the dispersed medium had no evident effect on the bulk viscosity of the mixture.

Effect of pressed pulp production on sugar factory operation. J. Le Blanc. Sucr. Franc., 1983, 124, 295-296 (French). - Aspects of beet pulp pressing are discussed in relation to their effect on sugar factory operation, including recycling of press water and the need to allow for an ultimate increase in the amount of waste water for treatment and disposal. In order to recover as much heat as possible from the condensate replaced by the press water, the thermal balance has to be adjusted. For optimum pressing, the temperature in diffusion should be reduced, but this increases the risk of greater bacterial infection; a lower pH also favours the texture of the pulp and hence its pressability, but a pH reduction imposes constraints in the form of stainless steel construction for tanks and ancillary equipment. The use of additives, e.g. Ca or Al sulphate, to improve pulp pressing increases the costs of treatment, while the $\mathrm{Ca}^{++}$and $\mathrm{Al}^{+++}$cations are mostly replaced by $\mathrm{Na}^{+}$and $\mathrm{K}^{+}$which will either pass into juice and be largely removed by purification or will pass to molasses where they will be accompanied by four times their weight of sugar. Double pressing increases the pulp solids and is viable where the water from the second pressing can be recycled.

A complete disinfection process within the sugar beet processing technology. I. Toth-Zsiga. Cukoripar, 1983, 36, 95-97 (Hungarian). - The occurrence of microorganisms in the beet sugar factory, means of determining them and methods of inhibiting their activity and reducing their numbers are surveyed, and a short bibliography of works by the author on the subject is appended.

Characterization of the DC diffuser performance by mode determination. J. Gerse and F. Toth. Cukoripar, 1983, 36, 97-104 (Hungarian). - Determination of the major diffusion parameters revealed $65-70 \%$ scatter in the values obtained at different times. The "typical" operating conditions were determined by evaluation of the frequency distribution of given parameters, and their order of importance thus established. It was found that changes in temperature and level needed to be measured more precisely, and that it would be preferable to synchronize the rotary speed of the scrolls in this DDS-type diffuser with throughput at any given time. Suggestions are made regarding prevention of clogging at high and low levels.


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Descriptive literature upon request

# STARCH BASED SWETEMERS 

Isosyrup production in the laboratory and factory. L. Ludvig. Szeszipar, 1982, 29, (1), 26-27 (Hungarian); through Food Sci. Tech. Abs., 1982, 14, (12), 12 L 820. After discussing various laboratory experiments on the production of high fructose corn syrup from starch the necessary conditions of large-scale production of starch from maize and HFCS from the starch are outlined.

Properties and uses of isomerized liquid sugars. S. Magarra. New Food Ind., 1981, 23, (10), 6-12 (Japanese); through Food Sci. Tech. Abs., 1983, 15, (3), 3 L 222. - This discussion-type article outlines manufacture of isomerized liquid sugars with medium and high fructose contents, and tabulates the composition of glucosefructose and fructose-glucose liquid sugars as laid down by Japanese Agricultural Standards, and of commercial mixtures (Nyufruct range) containing 35, 41 and 55\% fructose, and mixtures containing $20 \%$ sucrose. Sweetening power, osmotic pressure, crystallization, colour and water absorption of the liquid sugar mixtures are considered. Their use in soft drinks, bakery products and frozen desserts is discussed, with tabulated formulations.

Isomerization of glucose to fructose in the presence of borate. L. D. Bobrovnik, G. A. Lezenko and O. P. Nazarova. Izv. Vuzov, Pishch. Tekh., 1983, (2), 41-44 (Russian). - A study was made of glucose isomerization to fructose on AV-17-2p anion exchange resin in the presence of sodium tetraborate. In the 17 series of tests, each of 5 experiments, the ratio of borax to glucose was varied as well as the initial pH , temperature and reaction time; results are given in the form of graphs. Maximum fructose yield of $\mathbf{4 0 - 4 2 \%}$ was obtained at a pH of approx. 11 , a temperature of $80-90^{\circ} \mathrm{C}$, use of $\mathbf{2 \%}$ borax on glucose Brix and a reaction time of about 40 minutes. Amberlite 93 was similar to AV-17-2p resin in its effect on the reaction, both removing a substantial quantity of colouring matter from the end-product. Thus, sodium tetraborate proved an effective catalyst in small quantity provided heating was not prolonged.

The use of starch products in the manufacture of food concentrates. O. E. Pavlovskaya and N. N. Tregubov. Sakhar. Prom., 1983, (6), 40-43 (Russian). - A survey is presented of the use of starch products, including corn syrups, in the food industries of a number of countries.

Selection of temperature conditions for manufacture of granulated glucose. V. V. Ananskikh, N. G. Gulyuk and N. D. Lukin. Sakhar. Prom., 1983, (6), 46-48 (Russian). The title product, containing particles 2.3 mm in size, is produced by spraying syrup onto a bed of "priming" granules and then drying. Although moisture removal is greater at higher temperatures, the major constraint is the low temperature at which the granules fuse together. Investigations carried out to find suitable temperature
conditions are described and a diagram presented for establishing the optimum temperature of air entering the dryer/crystallizer.

Ways of improving glucose crystallization processes. N. G. Gulyuk, N. D. Lukin, L. S. Khvorova and V. V. Ananskikh. Sakhar. Prom., 1983, (7), 43-45 (Russian). A single-stage process for crystallization of syrups having a glucose equivalent (GE) of $94-95 \%$ is described and its advantages considered. While it is suitable for production of medicinal glucose in anhydrous form, a two-stage scheme is better for syrups of $97-98 \%$ GE obtained enzymatically. Under normal conditions, crystallization is complicated by the presence of two tautomeric $\alpha$ and $\beta$-forms of glucose. A study of the properties of the granulated product has revealed different forms, since mutarotation cannot take place in just the few minutes required for crystallization.

Glucose-fructose syrup - a new sugar substitute. T. A. Ladur and T. S. Puchkova. Sakhar. Prom., 1983, (7), 53-54 (Russian). - The manufacturing process is outlined for a product of $71 \%$ dry solids content which contains $52 \%$ glucose, $42 \%$ fructose, up to $6 \%$ higher sugars, $0.1 \%$ ash and a colour not exceeding 0.05 units of optical density.

Improving the technology for purification of glucose syrups. N. G. Gulyak, E. K. Sidorova, I. P. Dubinskaya and N. N. Nyunina. Sakhar. Prom., 1983, (7), 56-57 (Russian). - The use of granular carbon for the treatment of glucose syrup is discussed.

The utilization of washings and spent active carbon in syrup manufacture. Yu. N. Zarudnev and Z. S. Konieva. Sakhar. Prom., 1983, (7), 58-60 (Russian). - The use of washings to sweeten-off filter-cake in place of barometric water reduced losses of dry solids and obviated the need for dilution of syrups before evaporation. Use of spent carbon as a supplementary filter medium allowed savings in perlite whilst having no adverse effect on the finished product.

Manufacture, use and nutritional aspects of $90 \%$ highfructose corn sweeteners. L. S. Young and J. E. Long. Proc. 2nd Int. Flavour Conf., 1981, 195-210; through S.I.A., 1983, 45, Abs. 83-949. - The manufacture of 90\% HFS is outlined and its properties are described, with emphasis on its advantages compared with sucrose and glucose syrups. Carbohydrate profiles and relative sweetnesses of various nutritive sweeteners are tabulated. Food products in which HFS can be used are described. Nutrition issues regarding $90 \%$ HFS are discussed, with regard to general metabolism of fructose, hypoglycaemia, diabetes, weight control, dental caries, alcohol metabolism and athletic performance.

First crystalline fructose plant in US. C. E. Morris. Food Eng., 1981, 53, (11), 70-71; through S.I.A., 1983, 45, Abs. 83-1231. - The American Xyrofin turn-key plant in Illinois, claimed to be the first plant to produce pure crystalline fructose (PCF) from corn syrup, is reported. The microprocessor-controlled process is based on conversion of corn syrup ( $92-94 \%$ dextrose content) into $42 \%$ HFS, refining and crystallization into PCF; all stages of production, from the raw material to bagging and quarantine of the product (to ensure quality) are discussed, and environmental controls are outlined. Plant capacity is 10,000 tons of PCF/year; major markets for the PCF include bakery and confectionery industries and dietetic-food processors.

# LABCRATORY STUDIES 

interest in sugars analysis is the use of a disposable silica column, adapted for carbohydrate separation, in a radial compression device.

Rapid analyses of lactic acid, an indicator of sugar cane deterioration, and aconitic acid, an indicator of sugar cane maturity, by high performance liquid chromatography. M. A. Clarke and M. A. Brannan. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 88 (Abstract only). - Lactic acid is a product of the bacterial degradation of sucrose and therefore constitutes an indicator of freeze or other injury to sugar cane. It can be analysed rapidly by HPLC using a carbohydrate analysis column. Because lactic
acid can be analysed on the carbohydrate column this analysis is also important for testing for total fermentable sugars for ethanol production. Lactic acid is often present in fermentation media and can be misinterpreted as a sugar, which gives falsely high assessments of sugar levels. Aconitic acid is an indicator of sugar cane maturity since levels decrease as the cane matures. The analysis of aconitic acid by reverse-phase HPLC offers a rapid alternative to the chemical procedure.

Potassium and sugar recovery. M. A. Clarke and E. J. Roberts. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 89 (Abstract only). - The fact that high concentrations of K and Na in syrups lower the recovery of crystalline sugar from those syrups is well known. The decrease in sugar recovery is explained in the light of change in solubility of sucrose in the presence of $K$ and/or Na , and of complex formation between sucrose and various inorganic ions. The effect of varying Ca and Mg concentration on sugar recovery is discussed. The impact of the composition of total ash is considered.

Study of a calcium electrode in cane juices by the sample addition method. Comparison with the titration method using EDTA. H. Manso J., R. Vuervo F. and A. Rodriguez A. CubaAzúcar, 1981, (Oct.-Dec.), 7-11 (Spanish). - The two title methods were compared and the former found to be convenient, with a mean relative difference from the second not higher than $5 \%$ for mixed juice and $9 \%$ for clarified juice.

Chemical method for retarding the deterioration of sugar industry products. II. Syrup components and alkaline treatment. J. A. Urrutia, E. L. Ramos and L. R. Orozco. CubaAzücar, 1982, (Jan.-March), 11-16 (Spanish). The first part of the report dealt with the retardation of deterioration by alkali treatment of syrups and, on the basis of the results obtained, work has continued on the effects of pH and the amino-acids content of the syrup. Both of these affect the parameters defining susceptibility to deterioration but only pH affects reducing sugars and NaOH requirement. The alkaline treatment causes little colour formation and the colorants produced are easily eliminated with carbon. Reducing sugars are destroyed to the extent of $1.5-12 \%$, with formation of saccharinic acids, while $\mathbf{2 0 - 6 0 \%}$ of the amino-acids are eliminated.

New criteria for the study of buffer capacities in sugar products. H. Manso J., E. Cuervo F. and A. Rodriguez A. CubaAzúcar, 1982, (Jan.-March), 17-22 (Spanish). Buffer capacity of syrups was assessed between the initial pH and 8 and the effects of non-sugars determined using systems with the same Brix but different purity and vice-versa. The buffer capacity of systems with identical Brix decreases linearly with purity but is also dependent on the nature of the impurities, while for systems of the same purity, the buffer capacity fell

Recent advances in carbohydrate analysis by high performance liquid chromatography. M. A. Clarke. J. Amer. Soc. Sugar Cane Tech., 1983, 2, 88 (Abstract on/y). - High performance liquid chromatography has been used successfully for some years in the analysis of sugars in cane juice, process liquors and syrups, raw sugars and molasses. Recent developments in the analysis of sugars, other carbohydrates and other compounds of concern in sugar processing are discussed. Of particular

Trace components in sugars. I. Quantitative determination of amino-acids by gas chromatography. S. Saito, T. Miki, H. Ito and M. Kamoda. Proc. Research Soc. Japan Sugar Refineries Tech., 1983, (32), 1-8 (Japanese). The authors have developed a rapid and accurate GLC method for determining amino-acids in sugars whereby the amino-acids were absorbed on Dowex 50W-x8 cation exchange resin in $\mathrm{H}^{+}$form and eluted with $1.0 \mathrm{~N} \mathrm{NH}_{4} \mathrm{OH}$. The eluate was concentrated to dryness and the residue esterified in iso-butanol solution containing HCl . The iso-butyl esters were trifluoroacetylated using trifluoroacetic anydride in dichloromethane, and then concentrated under reduced pressure at $23^{\circ} \mathrm{C}$. A $2.0 \mu$ litre portion of the concentrate was subjected to GLC analysis. Results are tabulated for individual amino-acids found quantitatively in raw sugars from eight countries, in cane juice and molasses as well as refinery molasses, and in various kinds of refined sugar.

Elimination of colour in affination syrup by ultrafiltration and decolorization of the ultrafiltrate with adsorbents. S. Kishihara, S. Fujii and M. Komoto. Proc. Research Soc. Japan Sugar Refineries Tech., 1983, (32), 87-92 (Japanese). - Bio-Engineering G-01T and G-05T membranes and Amicon PM-10, XM-50 and XM-100 membranes were used in investigations of refinery liquor decolorization by ultrafiltration. A Bio-Engineering MC-2 test cell of $10.7 \mathrm{~cm}^{2}$ effective membrane area and $60 \mathrm{~cm}^{3}$ volume, equipped with a stirrer, was used at 2 or 4 atm feed pressure, $30^{\circ} \mathrm{C}$ or $60^{\circ} \mathrm{C}$ and 1500 rpm . Flux was calculated on the basis of the time required for the outflow of the first 25 ml of permeate from 50 ml of feed, while colour was expressed as absorbance of a $1-\mathrm{cm}$ layer of solution at 420 nm . Results showed that the $\mathrm{pM}-10$ membrane gave the best performance for affination syrup in terms of decolorization and sugar loss ( $80 \%$ and $6 \%$, respectively). This membrane was also tested on other refinery materials, including raw sugar liquor, earbonatation liquor, final molasses and refined sugar. While decolorization was on the high side, flux was too low at $70^{\circ} \mathrm{C}$ and $65^{\circ}$ Brix for practical purposes. In batch ultrafiltration or diafiltration of $10^{\circ} \mathrm{Bx}$ affination syrup through the PM-10 membrane, $90 \%$ of the sugar was recovered, having a residual colour content of $30 \%$ and $25 \%$, respectively. The permeate from syrup passed through the PM -10 membrane was more easily decolorized than was the retentate and the original affination syrup.
linearly with Brix. For high Brix syrups, a correction factor is necessary in determining buffer capacity and this is obtained by measurement at two dilutions.

Adjustment of an equation for sugar crystal growth. Solubility point. M. Wong Q. CubaAzúcar, 1982, (Jan.March), 58-63 (Spanish). - Crystal growth studies have always met with the problem that calculation of supersaturation depends on the value taken for the concentration of the saturated solution, and different values have been reported in the literature for these. To avoid the problem, a growth rate equation has been adjusted in terms of concentration, $V=k\left(C-C_{o}\right)^{g}$, where $g$ is a factor depending on the growth mechanism. This is rearranged to $C=k^{\prime} V^{1} / g+C_{o}$ and experimental data were used for $C$ and $V$, using different empirical values for $1 / \mathrm{g}$ until a linear graph was produced, having an intercept of $\mathrm{C}_{0}$, the saturation concentration. The values of solubility so obtained at different temperatures are compared with those reported in the literature; they are close to those of Grut between $20^{\circ}$ and $60^{\circ} \mathrm{C}$ and to those of Charles and Wise \& Nicholson at $70^{\circ}-90^{\circ} \mathrm{C}$.

Determination of the size of sugar crystals in massecuite and their distribution. J. Lodos F., I. Diaz and J. Delgado C. CubaAzúcar, 1982, (April-June), 3-7 (Spanish). Limitations of the various existing methods for measuring crystal size and size distribution are explained and a new method described which is considered to be more accurate, simpler and faster, while requiring less equipment. The method involves addition to 10 parts of massecuite of $3-5$ parts of a compatible and preferably transparent surfactant; after mixing, a thin layer is placed on a Petri dish and the crystals, which become visible, are projected by means of a microfilm reader for counting. The surfactant preferred is a Czech prodduct, Slovapon.

Isolation of polysaccharides present in molasses. J. Hormaza M., L. Ramos E. and A. Leon G. CubaAzúcar, 1982, (April-June), 17-21 (Spanish). - A method has been developed for separation of polysaccharides from molasses and in three cases was found to isolate between 92 and $98 \%$ of that present, contaminated with 2.4-3\% of protein. A $40-\mathrm{g}$ sample of molasses is diluted with 200 ml of distilled water, left to stand for one hour and centrifuged. The polysaccharides are precipitated from the supernatant by addition of $800 \mathrm{ml} 95^{\circ}$ alcohol and separated by a second centrifuging. They are purified by dissolving with 2 g of sucrose in 100 ml of water, recentrifuging and eliminating salts from the supernatant using a 1 -litre Sephadex G- 50 column. Elution with a 2:5 v/v mixture of pyridine and acetic acid yields two fractions, one containing low M.W. compounds and the other the polysaccharides. The latter is evaporated to dryness and dissolved with sucrose ( 2.5 g for 0.5 g of polysaccharide, in 50 ml of water). Proteins are removed by extraction with 10 ml of chloroform and 2 ml of butanol, washing the extract with water and combining the aqueous solutions. A solution is made in 0.1 borax buffer ( 10 ml per 200 mg of polysaccharides), $5 \%$ cetyl trimethyl ammonium bromide solution added $(15 \mathrm{ml})$ and the solution applied to a Sephadex G-50 column ( 100 ml ). This is eluted with a borax solution ( $3.81 \mathrm{~g} / \mathrm{litre}$ ) until no anthrone reaction is found, after which the polysaccharides are eluted with 0.1 N NaCl .

Determination of the thermal conductivity coefficient of sugar solutions. F. L. Falcón. CubaAzúcar, 1982, (AprilJune), 46-49 (Spanish). - A "zone fusion with temperature gradient" technique was applied to a saturated

## Laboratory studies

solution of sucrose at $60^{\circ} \mathrm{C}$, using a temperature gradient of $75^{\circ} \mathrm{C} / \mathrm{cm}$ and a zone width of $430 \mu \mathrm{~m}$; the value obtained for the coefficient was $1.29 \times 10^{-3} \mathrm{cal}^{2} \mathrm{~cm}^{-1}$. $\mathrm{sec}^{-1} .{ }^{\circ} \mathrm{C}^{-1}$. This is close to the value for solid sucrose, which is attributed to the high concentration of the solution.

Sterilization of Leuconostoc mesenteroides with ultrasonic radiation. O. Rodrigez C., J. Lodos F. and L. Cruz G. CubaAzúcar, 1982, (April-June), 50-55 (Spanish). Preparations containing $10^{5}$ cells per ml of $L$. mesenteroides were subjected to ultrasonic vibrations between 20 and 100 W for treatment times of $15-350 \mathrm{sec}$. There was always an induction period of $15-60$ seconds but thereafter the numbers were reduced to $10 \%$ in 17-77 seconds, depending on the power applied. The results are sufficient to envisage the use of ultrasound as a means of sterilization in the sugar industry.

Determination of the colour of sugar solutions. E. Ramos. CubaAzúcar, 1982, (Oct.-Dec.), 21-28 (Spanish). - A method has been developed withich is claimed to give the same results as the ICUMSA method but which is quicker, cheaper to employ and is more readily adopted in a sugar factory laboratory. A solution of the sugar is made ( $10^{\circ} \mathrm{Bx}$ for raw sugar, $50^{\circ} \mathrm{Bx}$ for a refined sugar) and filtered through a layer of asbestos (prepared by dispersing $\mathrm{d}^{2} / 28$ grams of long-fibre asbestos in water and placing this on the surface of a filter-paper in a Buchner funnel of diameter $\mathbf{d ~ c m}$ ), ensuring that the layer remains unbroken. The first 25 ml of filtrate is discarded and the remainder adjusted to $\mathrm{pH} 7 \pm 0.1$ and the attenuation measured, relative to distilled water, using a photocolorimeter with a mercury lamp and blue filter of $440 \pm 10 \mathrm{~nm}$; the cell length is chosen to give an instrument reading between $20 \%$ and $80 \%$ transmittance.

Action of ultrasonic radiation on Leuconostoc mesenteroides present in cane juice. J. Lodos F., O. Rodriguez C. and L. Cruz G. CubaAzúcar, 1982, (Oct.Dec.), 29-34 (Spanish). - Previous work ${ }^{1}$ has been extended to samples of cane juice. They were subjected to ultrasonic radiation at 25 kHz and powers of $50-300 \mathrm{~W}$ using a continuous flow device and adjusting the flow to give different retention times. The results are indicated in graph form; at the lowest power and a flow of $0.34 \mathrm{ml} /$ sec, reduction to $10 \%$ of the micro-organisms required 2.95 seconds while at 300 W and a flow of $1.47 \mathrm{ml} / \mathrm{sec}$, only 0.68 seconds were required.

A method for determining the dry solids content in highly coloured sugar solutions. A. I. Gromkovskii, N. S. Ostroukhov, N. V. Petrenko and O. V. Vorob'eva. Sakhar. Prom., 1983, (7), 42-44 (Russian). - While dilution of molasses with water for measurement of refractometric dry solids leads to inaccuracies because of the increase in the degree of hydration of some of the non-sugars and/or because of dilatation (when the dry solids content exceeds 66\%) or contraction (when it is below $40 \%$ ), dilution with pure sugar solution or remelt liquor was shown to avoid these phenomena. Either diluent was suitable and there was no difference in results between 1:1 and 1:2 dilution. However, a critical factor was the concentration of the sugar solution, which should not be less than 60\%. Under these circumstances, results were comparable to direct refractometric measurement without dilution when the colour was not excessive.

[^6]Study of active carbon production by pyrolysis of hydrolytic lignin from sugar cane bagasse. C. J. Triana F., R. González O. and R. Bradshaw C. CubaAzúcar, 1982, (July-Sept.), 25-29 (Spanish). - Hydrolytic lignin obtained from bagasse was pyrolysed in the presence of acid, basic and neutral catalysts, including phosphoric acid, zinc chloride, ammonium carbonate, calcium chloride, ammonium hydroxide, sodium hydroxide, etc., and the carbons produced treated to remove traces of catalyst, tars, acids, phenols, etc., dried and ground to provide uniform particle sizes. They were then tested for decolorizing raw sugar solutions, with satisfactory results.

Prospects of bagasse drying as a system. A. Arrascaeta R., D. Clerch A. and T. Llanes O. CubaAzúcar, 1982, (JulySept.), 42-49 (Spanish). - A combined bagasse dryer is under construction at ICINAZ for production of dried and screened bagasse particles suited to new methods of handling, storage and combustion which may improve boiler combustion efficiency to allow saving of bagasse for use in other applications.

Effect of the application of organic wastes from the sugar industry on cane growth. C. C. Wang, H. Y. Liao and P. Y. Song. Taiwan Sugar, 1983, 30, 44-52. - Field experiments showed that cane yield was increased by addition of a 1:1:0.3 blend by weight of vinasse, filtercake and bagasse pith to the soil. Significant increases also occurred in soil pH , available P and extractable K . It was estimated that $\mathrm{N}, \mathrm{P}_{2} \mathrm{O}_{5}$ and $\mathrm{K}_{2} \mathrm{O}$ use could be reduced by up to 50,10 and $100 \mathrm{~kg} / \mathrm{ha}$ per crop by application of 40 tonnes/ha of twe mixed organic wastes. Reducing the rate of chemical $\mathbf{N}$ improved the available sugar content in the cane. Trials showed that direct use of sludge cake at $10-40$ tonnes/ha improved the cane yield by $11-18 \%$ over the control plot. However, the electrical conductivity of the soil rose with application between 10 and 90 tonnes/ha of sludge cake and it is suggested that no more than 50 tonnes/ha should be applied. Disposal of the wastes by application to the soil reduces pollution and recycles plant nutrients. Sludge from a pulp factory usually contains undesirable chemicals which may be toxic to cane and protection of the soil from adverse environmental impact on application of industrial wastes is the first priority.

New molasses sugar recovery plant at Amino GmbH, Frellstedt. Anon. Zuckerind., 1983, 108, 636-638 (German). - Background information is given on the activities of Amino GmbH where a Finnsugar ion exclusion plant has been installed for recovery of sugar, betaine and specific amino-acids from molasses. Each of the three fractions eluted is concentrated by evaporation and the sugar fraction (of about $92 \%$ purity) decolorized and demineralized with resins, treated with granular carbon and concentrated by evaporation. With introduction of the new process, production of glutamate had to be discontinued on technical grounds, although aminoacid recovery has been greatly expanded, to make significant inroads into the world market for L-leucine and L-iso-leucine.

Determination of the matrix which characterizes the system of semi-chemical pulp production. E. González S., A. Ribot E. and V. González R. Centro Azúcar, 1982, 9, (1), 63-78 (Spanish). - A simple method has been devised to obtain a mathematical model of the production process by the multiplication of matrices which represent the individual stages in the manufacture of semi-chemical bagasse pulp.


## UNITED STATES

Utilizing the waste hekt content of condensate and/or vapour produced in sugar manufacture. H. Huber, of Schifferstadt, and H. Schiweck, of Worms, Germany. $4,290,818$. October 31, 1979; September 22, 1981. Thin juice is (treated to hydrolyse completely its glutamine and asparagine content and) cooled and flashevaporated (in one or more stages) to produce a cooler and more concentrated thin juice. This is then heated in two stages using first condensate and/or vapour from elsewhere and, second, the initial uncooled thin juice, thereby cooling the latter. The reheated juice is subjected to a second carbonatation process to produce an initial second thin juice which is sent to a multipleeffect evaporator and concentrated. Vapour is bled from the last effect, compressed and returned to an earlier effect.

Production of fructose and fructose-based syrups. L. Degen, P. Branduzzi, R. Olivieri and N. Cimini, of Rome, Italy, assrs. Snamprogetti S.p.A. 4,291,123. June 8, 1978; September 22, 1981. - Fructose and syrups containing fructose and glucose are obtained by bringing a glucose solution into contact with a Streptomyces sp. micro-organism NRRL 11,120 or NRRL 11,121, or with an enzyme derived from it.

Sucrose ester of 2-methoxy-3,6-dichlorobenzoic acid. G. F. Luteri, of Mount Prospect, IL, USA, assr. Velsicol Chemical Corporation. 4,291,158. January 28, 1980; September 22, 1981. - The title ester (prepared by mixing of sucrose and 2 -methoxy-3,6-dichlorobenzoyl chloride in pyridine, reacting at $75^{\circ} \mathrm{C}$ for 2 hours, then at $100^{\circ} \mathrm{C}$ for 2 hours, followed by cooling and stirring, stripping of the pyridine, dissolving in ethyl acetate, washing with dilute $\mathrm{HCl}, 5 \% \mathrm{NaCl}$ solution and water, drying and solvent removal) is useful for application to cane (at e.g. $0.1-10 \mathrm{lb} / \mathrm{acre}, \mathbf{2 - 1 0}$ weeks before harvest) in order to increase its sugar content.

Bagasse cellulose extraction. A. Regnault, J. P. Sachetto, H. Tournier, T. Hamm and J. M. Armanet, assrs. Battelle Memorial Institute, of Carouge, Switzerland. 4,292,089. October 4, 1979; September 29, 1981.

Lignocellulosic material, e.g. bagasse, in hopper 305 is delivered by feeder 307 and port 321 into the sealed auxiliary drum IA which leads into the main drum DR to which it is connected via seals 322 . Both drums are rotated independently. Within drum IA are a scroll 324 and axial partitions 340 and concentrated hydrochloric acid is provided from container 304 through valve 306, pipe 308 and spray system 323, whereby the bagasse is thoroughly impregnated with acid in the helical channel 325. Within the main drum are three compartments formed by the end wall 326 and partitions 327-329,
the central apertures 331-333 having increasing diameters. The impregnated bagasse and acid fall into the first compartment to form a reaction mass 303a, and overflow into the next two chambers in turn to form masses 303b and 303c.


Hydrogen chloride gas is delivered from container 313 through a 3 -way valve 314 and into bubble tubes $316 \mathrm{a}, 316 \mathrm{~b}$ and 316 c where it provides agitation for the reaction masses. The acid and hydrogen chloride dissolves the cellulose from the bagasse in a continuous manner, the finer undissolved particles rising to the surface of the masses whilst the larger particles remain near the bottom of the compartments. Agitation is consequently provided by fitting of vanes $310 \mathrm{a}, 310 \mathrm{~b}$ and 310 c which rotate with the drum. Surplus HCl gas is withdrawn from the stationary end housing 338 of the main drum, linked to it by way of seals 337; the gas is passed through a compressor 318 and pipe 319 to be recycled to the drum DR. The bagasse lignin residue and acid solution of cellulose overflow into the bottom of housing 338 and are withdrawn through valved pipe 341 into container 342.

Anaerobic fermenter-decanter for sugar factory waste water treatment. J. P. Lescure, of Mons-en-Baroeul, France, assr. Syndicat National des Fabricants de Sucre de France. 4,293,412. January 22, 1980; October 6, 1981.

A tank 1, excavated in the ground, is in the form of a truncated pyramid with a square base. A fluid-tight skin 2 is fixed at the periphery by burial in trenches and weighted with masonry. Half-way up the sloping sides are apertures $4,4^{\prime}$ to which the skin conforms; in these are placed concrete beams 4a, 4'a to form a frame on which are fixed piles or anchoring points 11, 11' for a submerged flexible cover 10 . This is connected to a collecting bell 12 which partly emerges from the water and has in its centre a motor 13 for a rotary agitator with a vertical shaft 14 ending near the bottom of the tank with a helical impeller 15 . The bell 12 is supported by tripod 17 and connected to the periphery by platform 20 which also supports a tube 21 for evacuation of fermentation gases which come from the annular chamber 18 within bell 12. The platform also supports channel 22 for removal of purified water.

The raw effluent is brought by heater 24 to about $35^{\circ} \mathrm{C}$ and introduced through pipe 23 which discharges through its end 23a near the bottom centre of the tank. This temperature favours the activity of the anaerobic

[^7]will be horizontal sections not receiving wash water, while if it is too far away the sprays will overlap, resulting
 in furrows where sugar has been dissolved. To avoid these extremes, the element 30 is provided to sense the position of the sugar surface (and hence the wall thickness); it is mounted on a shaft 29 carried by support 32 and can be switched from a rest to an active position.

The latter is selected when the basket is
fermentation micro-organisms which convert organic matter in the effluent to methane; the purified effluent passes upwards around the periphery of cover 10 and is withdrawn through channel 22, whilst the methane gas collects under cover 10 and rises to the collecting bell to be removed through tube 21. The $\mathbf{p H}$ of the fermentation may be controlled by addition of waste lime, while nutrients such as nitrogen and phosphate may be added to maintain required proportions.

Drying co-mingled carbohydrate solution and recycled product by dielectric heating. P. L. Veltman, of Severna Park, MD, USA. 4,294,624. March 14, 1980; October 13, 1981. - A solution of (1 part of) carbohydrate (fructose, glucose, sucrose, total sugars from cane or beet, or cane or beet molasses) is mingled with (5-20 parts of) recycled dry product to form a particulate mixture dryable in a dielectrically heated zone (and having a water content $\varangle 4 \%$ by weight). The mixture is dried (in a fluidized bed, under reduced pressure) at a temperature below its melting point (in a gas stream) (and cooled), to provide a stable, solid, substantially anhydrous product, part of which is (adjusted to a particle size of $50-500 \mu \mathrm{~m}$ and) recycled and the remainder recovered.

Centrifugal. C. Delfosse, of Hellemes, France, assr. Fives-Cail Babcock. 4,297,210. July 31, 1980; October 27, 1981.

The position of the wash-water manifold 38 within the batch centrifugal depends for proper washing on the thickness of the wall of sugar; if it is too close there

empty and, as the centrifugal is charged and the wall of sugar builds up, the element is moved inwardly until the wall thickness reaches a predetermined value. The element is then switched to its rest position and the feed to the centrifugal is cut off. The wall thickness has then reached the correct value, corresponding to the fixed position of the manifold 38, for proper washing of the sugar.
Production of a surfactant containing sucrose esters. H. R. Galleymore, K. James, H. F. Jones, C. L. Bhardwaj and J. S. Plant, assrs. Talres Development (N.A.) N.V., of Curacao, Dutch Antilles. 4,298,730. July 31, 1980; November 3, 1981. - See UK Patent Application 2,065,634 ${ }^{1}$.
Preferential separation of fructose from glucose. K. Venkatasubramanian, S. M. Jain and A. J. Giuffrida, assrs. The Hubinger Co. and lonics Inc. 4,299,677. November 3, 1980; November 10, 1981. - A mixture of the two sugars [containing a mediating cation of an alkaline earth metal ( $\mathrm{Ca}, \mathrm{Ba}$ or Mg )] is (1) passed through a first feed chamber of an electro-osmosis cell comprising at least two chambers defined between ion exchange membranes having alternately high and low permeability coefficients with respect to each other, and (2) subjected to a direct current passed transversely through the membranes and chambers, whereby the fructose passes from the feed chamber through the high permeability coefficient membrane into the adjacent chamber, there being substantially retained, after which glucose and fructose-enriched fractions are recovered from the separate chambers.

Continuous system for separation of crystals from massecuite. J. C. V. Ducasse, of Martinez, CA, USA. 4,303,522. November 21, 1979; December 1, 1981.
The circular separator is supported on a frame by means of pads and has a stationary top casing 18 with a flange 19 with an inner peripheral groove 20 . The stationary bottom casing 21 has a top flange 22 with a corresponding groove 23; the flanges are held together by bolts 24 tightly gripping a PTFE seal 25 which has a downwardly sloping inner face 26 . The casing 18 has a conical upper portion 27 with a lower cover plate 29 from which extends upwardly a cylindrical plate 31 with an upper flange and two concentric cylindrical plates 35, 36 spaced apart and extending downwards. The lower peripheries of these have PTFE seals ending in a common horizontal plane 38 and having slanted surfaces. Through the outer ring plate 33 is a connexion to a duct 17 leading to a source of hot air 16, and ports 40,41 and 42. These ports are connected to external sources of water and steam, which are admitted to internal pipes $43,44,45$, which have downwardly directed nozzles between plates 34 and 35 .
1/.S.J., 1984, 86, 27.


Also between these plates are an inverted Y -shaped scraper 47, the bottom tips of which lie in plane 38, and plates 50 and 51, the former adjustable vertically and the latter fixed at a suitable height above plane 38. They form chambers 52 (connected to a feed duct from a massecuite tank) and 53 (connected to a source of vacuum via a cyclone separator). The lower conical portion of the bottom casing 21 is connected to the suction side of a pump while the upper cylindrical portion 56 is connected by port 57 to a vacuum source. Also within casing 21 is a vertical cylindrical ring 58 held by stiffeners 59, with its top horizontal and at a distance below plane 38 .


The flange on plate 31 supports a system of bearings and seals through which passes the shaft 61 , driven from above by a variable-speed motor; at the lower end of
layer the height of which is governed by the plate 50. The vacuum builds up in the casing 21 and sucks molasses through the filter plate. It collects in the casing and a level control starts a pump for its evacuation when sufficient has been separated. The separated crystals are washed with water and steam as the layer passes underneath the spray nozzles and dried by hot air admitted from source 16 through duct 17 into chamber 65 . Part of this hot air is drawn into casing 21 but the remainder is drawn out by blower 8, the duct to which is located in side-wall 33 just before the scraper 47; dried crystals lifted by the scraper are entrained in the air flow and separated by the cyclone 10 from which they are recovered.

Bagasse process and product. G. E. Campbell, of Baton Rouge, LA, USA. 4,304,361. November 19, 1979; December 8, 1981. - Bagasse is converted to a peat moss-like material, useful as a planting or potting soil, by screening to remove short fibres $\frac{1}{4}-\frac{3}{8}$ inches long, feeding to a hammer mill to reduce longer fibres to a shorter length, removing new $\frac{1}{4}-\frac{3}{8}$ inch fibres as they are generated in the hammer mill, and combining all these short fibres.

Continuous acid hydrolysis and saccharification of bagasse. A. Regnault, J. P. Sachetto, H. Tournier, T. Hamm and J. M. Armanet, assrs. Battelle Memorial Institute, of Carouge-Geneva, Switzerland. 4,304,608. September 3, 1980; December 8, 1981.
Lignocellulosic material, e.g. bagasse, is delivered from hopper 24 by a feeder conveyor 25 to the entry in the stationary end wall of a rotary drum reactor 1 , from which it is sealed, as is the other end housing. Hydrochloric acid of $40 \%$ concentration is admitted from container 28 by way of control unit 27 , valve 21 and pipe 20 and is sprayed on the fresh bagasse. The drum has an internal scroll and paddles so that the bagasse is alternately lifted out and mixed with the acid in the impregnation zone (I) of the drum. It then passes into the hydrolysis zone $(\mathrm{H})$ where it is held for sufficient time for the cellulosic material to be dissolved and hydrolysed, only the lignin the shaft is a circular screen assembly 64 , the top of which lies in plane 38, forming an internal annular chamber 65. Assembly 64 comprises a disc 66 with a central hub 67 and an upper surface with concentric grooves 69 in the bottom of which a number of holes are drilled to connect chamber 65 with the casing 21. At the outside periphery is a top slanted surface 71 bearing snugly against the sloping surface 26 of seal 25. Below the plate is an annular ring 72 which carries a number of rollers 73 which bear on the top 59 of ring 58. On top of disc 66 is an annular filter plate 76 held between the inner flanges 77, 78 and outer flanges 79,80 by fasteners 81, the upper flanges 77 and 79 having slanted surfaces corresponding to those of the seals attached to rings 36 and 35 . When the motor operates, the shaft 61 and filter rotate within the housing in the direction of arrow 86. Massecuite is admitted to chamber 52 on top of the filter surface and forms a


## Patents

being undissolved. The scroll in zone H causes the material to pass along it and to enter the collecting pipe 16 as a suspension of lignin in an acid solution of sugars which passes to buffer tank 29.

From this it is sent by pump 30 along pipe 31 to a valve 32 which either recycles it to the buffer tank, sends it along pipe 33 to zone I or to the dryer 35 . This is supplied with hot air from generator 39 through pipe 37 and valve 38 and produces a dry mixture of lignin and solid sugars and a vapour which is sent along pipe 43 to control unit 27. Here the HCl content is recovered and sent for re-use as the hydrolysis agent, while by-products such as water, acetic and formic acids, etc., are separated (SP). The dried mixture of sugars and lignin is discharged into the container 42.

Preparation of sucrose monoesters. H. F. Jones, of Reading, England, assr. Talres Development (N.A.) N.V. 4,306,062. May 27, 1980; December 15, 1981. - See UK Patent Application 2,052,492 ${ }^{2}$.

Preparing cellulose. J. B. Thompson, of Cumberland, MD, USA. 4,307,121. November 26, 1979; December 22, 1981. - A particulate, relatively non-ligneous cellulose material (beet pulp) [which has been extracted with a
fat solvent (a lower alcohol) to remove lipids], in a slurry state in water with an oxidizing agent, is heated for a time and at a temperature sufficient to solubilize most of the non-cellulose material present, after which the cellulose pulp is separated, redispersed in water to give a second slurry and this treated with an oxidizing agent (chlorine gas), heated to boiling to eliminate any excess of the latter and to solubilize any non-cellulose remaining. A base material ( $\mathrm{NaOH}, \mathrm{KOH}$ or $\mathrm{Na}_{2}^{2} \mathrm{CO}_{3}$ ) is added to the slurry which is then (bleached with further oxidizing agent,) cooked to digest any further non-cellulose material present and the pulp separated as substantially pure short-fibre cellulose suitable for human consumption (which is dried, ground and classified to yield a powdered cellulose).

Crystallizer. H. Garcia, of Mexico City, Mexico.4,308,236. September 11, 1979; December 29, 1981.

The vertical crystallizer comprises a cylinder 11 with an upper section 12 and below this a processing section 13. In the latter are located a series of horizontal screens 14, 15 with peripheral and central openings respectively, while at the bottom is a discharge valve 21. The screens divide the processing section into subsections 16, 17 and 18 within which are heat exchange elements 23 mounted on a central shaft 24 which passes through the screens. The shaft is supported on frame 25 at the top of the crystallizer and this is mounted on the pistons 26 resting on the bridge 27.

Heat exchange fluid from one of the fixed nozzles 31 passes through a flexible hose 30 to connexion 28 at the top of the shaft, passes down internal pipe 33 to the bottom and then follows a labyrinth path from the centre to the outside of the lowest heat exchange element, passes upwards and outwards to the periphery of the next element and then back to its centre then up to the next and so on, eventually returning up pipe 32 to

connexion 29 and through the other flexible hose 30 to the other fixed nozzle 31. The hydraulic pistons are operated and reciprocate, causing the shaft to rise and fall. It carries with it an agitating vane 22 as well as the heat exchange elements. The massecuite path in the processing section 13 is horizontal from the periphery to the centre in subsection 16, then outwards in subsection 17, then inwards again in subsection 18. The heat exchanger fluid path is thus in direct counter current to massecuite flow, for highest efficiency.

Fructose production. R. S. Leiser, of Decatur, IL, USA, assr. A. E. Staley Mfg. Co. 4,310,628. March 14, 1980; January 12, 1982. - Increased fructose yields are obtained when using a fixed bed of immobilized glucose isomerase enzyme obtained from a Bacillus sp. (B. coagulans) and exhibiting an enhanced rate of isomerizing glucose to fructose, by conducting the reaction in the presence of $\mathrm{Co}^{++}$ions and at a temperature greater than about $60^{\circ} \mathrm{C}$, [at $55^{\circ}-61^{\circ} \mathrm{C}\left(58^{\circ}-61^{\circ} \mathrm{C}\right)$ ] and at pH 7.0-7.5 with a continuous feed of syrup [of 50-55\% dry solids including at least $90 \%$ ( $95 \%$ ) on dry solids of monosaccharide (and at least $0.002 \mathrm{M} \mathrm{Mg}^{++}$)] to compensate the continuous withdrawal of isomerized syrup. The process is continued for at least 2000 hours ( $>3000 \mathrm{hr}$ ) and ended when enzyme activity is reduced to $<20 \%(<15 \%)$ of its optimum level. The pH is within 7.0-7.5 for at least $90 \%$ of the operational time and above 8.0 or below 6.5 for $>5 \%$.

Beet diffuser. G. F. M. Duchateau, C. H. J. Pinet and P. X. Hanot, assrs. Raffinerie Tirlemontoise S.A., of Brussels, Belgium. 4,311,673. September 17, 1980; January 19, 1982. - See UK Patent Application 2,059,798 ${ }^{3}$.

[^8]
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[^0]:    ${ }_{2}^{1}$ Public Ledger's Commodity Week, February 4, 1981.
    ${ }_{3}^{2}$ Sugar Report, 1984, (1686), 23.
    ${ }_{4}^{3}$ International Sugar Rpt., 1984, 116, 81-87.
    4 World Sugar J., 1984, 6, (7), 4-5, 11-18.

[^1]:    ${ }^{1}$ Data quoted in "Principles of sugar technology", Ed. P. Honig, (Elsevier, Amsterdam). 1959.
    2 I.S.J., 1960, 62, 126-131.
    ${ }^{3}$ Aust. J. Chem., 1967, 20, 1087-1095.

[^2]:    1 Roberts et al.: I.S.J., 1976, 78, 163-165.
    ${ }_{2}$ Charles: ibid., 1981, 83, 169-172.

[^3]:    Paper presented to Sugar Industry Technologists, 1983.

    * Vice President Operations - Sugar Division.
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[^4]:    ${ }^{1}$ World Sugar J., 1983, 6, (6), 31.
    2 See I.S.J., 1984, 96, 72.
    ${ }^{3}$ F. O. Licht, International Sugar Rpt., 1983, 115, 630.
    4 Westway Newsletter, 1983, (122), 10.

[^5]:    ${ }^{1}$ S. African Sugar J., 1983, 67, 452.
    2 Sugar J., 1983, 46, (7), 28.
    3 World Sugar J., 1984, 6, (7), 31.
    4 F. O. Licht, International Sugar Rpt., 1984, 116, 30.
    5 World Sugar J., 1984, 6, (7), 31.
    6 F. O. Licht, International Sugar Rpt., 1984, 116, 56.
    7 Zuckerind, 1984, 109, 86.
    8 C. Czarnikow Ltd., Sugar Review, 1984, (1690), 43, 44.
    9 World Sugar J., 1984, 6, (7), 35.

[^6]:    ${ }^{1}$ See Rodriguez et al., this page.

[^7]:    Copies of specifications of United Kingdom patents can be obtained on application to The Patent Office Sale Branch, Block C, Station Square House, St. Mary Cray, Orpington, Kent, England (price $£ 1.75$ each). United States patent specifications are obtainable from: The Commissioner of Patents, Washington, D.C., USA 20231 (price 50 cents each).

[^8]:    2 I.S.J., 1983, 85, 318.
    3 ibid., 1984, 86, 26.

