

TOWARDS A NEW PROCESS TO PRODUCE WHITE SUGAR DIRECTLY FROM CANE JUICE

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Abstract

The options for producing white sugar directly in a raw mill are reviewed. Two broad approaches are considered, the first being to extract a purified sucrose stream from impure sugar juice and the second is selectively to remove the impurities from the impure juice leaving a pure sucrose solution. In the first category, chromatographic separation and crystallisation are considered and, in the second, membrane separation, ion exchange and decolourisation are reviewed. Possible processes are summarised and, where these have been practically demonstrated, this has been highlighted.

Keywords: demineralisation, chromatography, direct white sugar, membrane, separation, ion-exchange

Introduction

The production of a high grade white sugar directly from sugar cane juice has challenged the cane sugar industry since its inception. The only procedure currently used to make white sugar that meets the requirements of industrial users is the remelting of a raw intermediate product, and then re-crystallization. An optional purification of the melt prior to the final crystallisation is usual and the intensity or number of operations dependent on the quality of the raw sugar processed. While this conventional process suffers from apparent inefficiencies, it is robust and reliable and has enabled the mass production of a very high quality product by relatively unsophisticated operators.

In recent years there has been an upsurge in interest in applications of “new” technology to the sugar industry (Bekker *et al.*, 2001; Kearney, 1996; Kochergin, 1998; Kochergin *et al.*, 2000; Kochergin, 2002; Paanen, 1996; Rossiter, 1999; Thompson, 1994b; Saska, 1995; Godshall, 1999). This paper explores some of the options available to the industry to produce direct white sugar and propose a process to achieve this.

To compete with the conventional process of purification by recrystallisation, a replacement process needs to meet the following requirements:

- The product quality must not be compromised.
- The unit operations must be proven to be robust and reliable.
- Recoveries must be as high as, or better than, current.
- Any by-products should be as valuable, or more so, than current by products.

It is also obvious that it must be more cost effective than the current processes. This aspect will not be discussed further since the commercial aspects are strongly dependent on the local industry conditions, and would detract from technical issues.

It is instructive to consider the typical composition of cane juice and compare this with the requirements of white sugar. This is presented in Table 1, where it should be noted that the specifications for white sugar are a simplification of the actual EEC Grade 2 requirement.

What this means for the development of a new process is that it must remove significant amounts of suspended solids, colour, ash and invert from the raw juice, down to very low levels. This is a demanding task for a single step process. There are various unit operations, which will work, to remove some or all of these impurities to the required level.

Table 1. Comparison of raw juice and EEC2 sugar specifications.

Component	Juice	EEC 2 Sugar (simplified from Kirikiridis, 1992)
Brix (%)	12%	99.94%
Suspended Solids (%)	0,25%	0%
Ash (sulphated) (% on Brix)	3,5%	0,0018%
Sucrose Purity (%)	85%	99.7%
Invert (% on Brix)	5%	0.04%
Colour (ICUMSA)	>20 000	< 35
Organic non sugars (% on Brix)	6%	Related to purity

Several investigations into possible processes to produce white sugar have been published (Clarke, 1996; Godshall, 1999; Kearney, 1996; Kwok *et al.*, 1999; Kwok, 1996; Monclin, 1996; Saska, 1995; Fechter, 2001; Lancrenon, 1988; Honig, 1953; Dymond, 1948) and Kochergin (2002) has reviewed the more recent of these. Although much time and energy have been spent on these production schemes or the unit operations involved, to date none has been successfully implemented on an industrial scale. The traditional method of crystallisation remains a relatively cheap, simple and effective purification technique for sugar.

In principle, all processes published to date take one of two approaches to separate the impurities from sucrose:

1. Extract the sugar from the ash and other impurities. Here there are two methods; the first is crystallisation, which is already the standard procedure. The second is ion exclusion chromatography, which appears to be a well-established technology (Kearney, 1996; Thompson, 1994; Saska, 1995; Kochergin *et al.*, 2000).
2. Remove the impurities from the sugar. This approach involves targeting each impurity, or group of impurities, separately. There are a number of separation processes which could be used, each removing one or several impurities.

These two options will be considered in turn.

Extracting a pure sugar stream from the impure juice

Crystallisation

Crystallisation is a well-established technique for purifying and separating materials and has proved itself in the sugar industry to be robust and reliable. Drawbacks are:

- It requires energy each time the sucrose is redissolved, recrystallised, centrifuged and washed. Traditionally energy has not been a problem for the cane sugar industry but more recently trends towards greater energy efficiency have been noted.
- Each recrystallisation also requires a large investment in hardware (pans; centrifuges etc.) as well as increased complexity involved with operating the extra equipment.
- Each recrystallisation is very effective at improving the quality of the sucrose product, and does so by large “jumps” in quality; typically of about 95%. The process is therefore not easily adjusted to raise the quality by small or incremental amounts. To overcome this, partial or selective purification steps may be employed during the refining process (e.g. carbon or ion exchange).

Configurations to produce white sugar based on crystallisation are well established and will not be considered further.

Chromatography (ion exclusion)

Industrial chromatography has developed significantly in recent years into a mature and robust technology (Kearney *et al.*, 1995; Pynnonen, 1996; Kochergin, 2002). It is currently established in the beet industry for the recovery of sugar from final beet molasses, and appears to be an attractive, apparently “off the shelf”, technology. There are however three technical drawbacks to its implementation to produce a pure sucrose stream:

- Water is used as the transport medium and the product sugar stream is produced in a diluted form, even when the technology is optimised to reduce dilution (Kochergin, 2000).
- For chromatographic separation to be feasible the process stream needs to be substantially free of divalent ions (i.e. calcium and magnesium) (Thompson, 1994b).
- The stream must be free of suspended material which will foul the resins (Kochergin, 2000).

Cane juice contains a significant concentration of divalent ions and would need to undergo some sort of softening process to make it suitable for ion exclusion chromatography. The most developed softening process is ion exchange, replacing the divalent ions with sodium or, more elegantly, potassium (which is present in juice in significant quantities) (Thompson, 1994a). The quantity of divalent cations in molasses is essentially the same as that found in syrup or clarified juice. Therefore it is sensible to perform ion exchange softening as far as possible towards the front-end juice stream (e.g. clear juice) and obtain the benefit of softened juice on the evaporators (Thompson, 1994a; Shore *et al.*, 1988; Davis *et al.*, 1997a). It should be noted however that softening will not completely eliminate scale but will reduce it (Davis *et al.*, 1997b). Chemical softening using sodium carbonate is used in some instances to reduce the calcium content of carbonated beet juice (van der Poel *et al.*, 1998) but this may not be practical on a large scale on raw juice.

To prevent the ion exchange resins from becoming fouled, a filtration step is necessary before the softening step. How “tight” a filtration is necessary is not clear but it would seem from the work of Amalgamated Research Inc. (ARi) that microfiltration or ultrafiltration would be necessary on cane juices/syrups (Kearney *et al.*, 1995; Kochergin *et al.*, 2000). To optimise the “brix flux” over the membrane process some pre-concentration of juice is expedient. This has been presented previously (Fechter *et al.*, 2001; Kochergin, 2002). Thus the process so far would consist of pre-concentration, ultrafiltration, softening and then chromatographic separation. The choice is then whether to carry out chromatographic separation of the sugar at the front end (clear juice end) or at the back end (final molasses after crystallisation) of the process. As noted by Kochergin *et al.* (2000) the size of the unit required is about the same if performed on juices or on (diluted) molasses streams (or anywhere in between). Clearly there are process benefits to removing impurities as soon as possible in the process. Arguments similar to that used to optimise brix flux through membranes can be used to minimise the equipment required but maximise throughput (Kochergin, 2000). If chromatography is used towards the front end, e.g. syrup, then the process developed is similar to that described by Kearny (1996) (the ARi process). If chromatography is used at the end (molasses) the process resembles the NAP process (Herve, 1995; Kwok, 1996). This is not as attractive since a direct white sugar is not produced, and the full benefit of processing a stream purified by chromatographic separation is not realised.

A possible process based on chromatographic separation would be as follows:

- Normal juice clarification
- Concentration
- Ultrafiltration
- Softening
- Further concentration
- Chromatographic separation
- Re-concentration of the diluted sugar streams
- Sugar boiling.

Such a process has been developed and patented by Amalgamated Research Institute (ARi). The ARi process offers the possibility of a clean juice stream from which a white sugar can be produced according to a process of ultrafiltration, softening, ion exclusion, and sugar boiling. The nature of the ion exclusion chromatography is such that the requirement of more sugar recovery is achieved and the possibility of a low ash fermentable sugar stream exists. The process is therefore technically attractive, robust, and has proven reliable in other industries. However, the increased amount of equipment required is significant. For ion exclusion about 30 times more resin is required than that for ion exchange demineralisation, and the evaporator capacity has to be increased to deal with the dilute streams produced (about 3 times increase in area even with the improved ARi technologies (Fechter *et al.*, 2001)).

Impurity removal from the sugar stream

Traditional

The second approach starts with examining ways of removing the ash, colour, and invert from the juice. The most obvious processes are the traditional methods based on carbonation or sulphitation. These could also be considered colour-based removal methods since colour is generally used to measure their success.

Originally the Natal industry produced direct “white” sugar via a double carbonation process similar to that used by the beet industry (Campbell, 1925; Rault, 1946). Entumeni used sulphitation and phosphatation (Carter, 1954) to produce a mill white. The process appears to have served the industry well and was only abandoned in the 1960’s; with some discussion of its failings given by Rault (1960). Reasons for its demise include:

- Difficulties with achieving suitable absorption of CO₂ into the juice. The pH value determines the rate of CO₂ absorption, being significantly reduced at pH values approaching neutral levels. (van der Poel *et al.*, 1998). However at higher pH values problems with invert arise as colour formation becomes a problem. A similar problem arises with absorption of sulphur dioxide, which is also strongly dependent on pH value.
- Filtration problems. Filtration problems arose apparently as a result of the many impurities (gums, phosphates and starch) acting as crystal habit modifiers for calcium carbonate.
- The high cost of transporting lime. Although some attempts at lime burning are mentioned it seems that consumption was high and the cost became excessive, especially if sulphitation was used.

The beet industry has reported designs of saturators to deal with absorption at constant pH values (van der Poel *et al.*, 1998; Mathlouthi, 2000). While this could improve matters, by reducing invert destruction, the presence of crystal habit modifying impurities will still present a problem for cane juice. Nevertheless it seems that carbonation has not been revisited seriously by the cane industry.

At this point it becomes clear why a process used with success in beet appears not to be viable in the cane industry. Chou (2001) has listed the main differences between cane juices and beet juices and highlighted the difficulties of transferring any beet process to cane. Essentially, for cane juice invert sugars are higher, impurity levels are greater and apparently more intractable than for beet juice. Ash removal also remains a problem with carbonatation and the beet industry (where carbonatation is used) goes to great lengths to reduce alkalinity.

There are many well-established unit operations specifically targeted at colour (including carbonatation) currently in use in the sugar industry. Davis (2001) has reviewed these colour removal techniques and highlights many successful methods. Most commonly, colour removal involves an absorption mechanism such as carbonatation or phosphatation and or an ion exchange process to remove colour. Ion exchange is commonly used in refining operations where a strong base anion resin in the chloride form is usually used and is regenerated with hot brine. The bulk of the colour load is removed in the raw house and thus the refinery resin encounters a low colour load. Under such conditions the resin has a life of about 1 year. Kirkiridis (1992) has described the operation of the ion exchange plant at Hulett's refinery. Hubbard *et al.* (1996) and MacDonald *et al.* (1996) have shown the improved efficiencies associated with using an ISEP unit (a simulated moving bed) for ion exchange colour removal.

Sometimes pre-treatment with flocculant or oxidising agents to modify the coloured compounds is used to optimise removal. Davis' review (2001) is geared towards refining, but where bulk colour removal is required on raw juice these methods do not achieve the required levels of colour removal, and rather produce a "mill white" product. Clarke (1996) and Godshall (1999) have reviewed such production schemes using carbonatation and/or sulphitation. They note that sulphitation is preferred over carbonatation but that product quality does not meet EEC 2 specifications. Ash and colour levels in particular are not achieved.

A new concept in colour removal is the use of special absorbent resins. These are not well established at this stage in the sugar industry but have been incorporated into the "ABC" Process described by Monclin (1996). The ABC process uses ultrafiltration followed by an absorbant resin to produce a direct white sugar. This and other published schemes are reviewed by Kochergin (2000) and Godshall (1999). Since the absorption resins have not been established to be robust or proven at this stage; processes based on this approach will not be considered further.

Softening

Ash in sugar can be removed effectively to reasonably low levels by softening (using ion exchange) before crystallisation and a calculation showing this, based on the work of Lionnet (1998), is given in Appendix A. This is used in beet factories and in some refineries where ash removal is a problem (van der Poel *et al.*, 1998). Such a process does not appear to remove any colour and would probably require pre-filtration to protect the resin. Furthermore, the high sodium levels adversely affect the exhaustion (Thompson, 1994b), and to recover more sugar chromatographic separation would be necessary. Thus a softening type process would still require recrystallisation to remove colour or an absorption or ion exchange step. An alternate proposed by Kwok *et al.* (1999) is to use nano-filtration to remove colour and other impurities to a level where suitable sugar can be produced. A process based on softening would thus involve pre concentration, ultra or micro filtration, ion exchange softening, crystallisation, remelting and possible chromatographic recovery of sugar from the molasses stream. Bekker *et al.*, (2001) have included a decolourisation stage. Flowsheets based on this approach have been presented by Herve (1995). The softening approach thus does not produce a first white sugar directly. Encouragingly the work done in this area suggests that invert is effectively removed by crystallisation and special attention is not necessary to deal with this impurity.

Membranes

Membrane technology has made great strides in recent years both in terms of cost reductions and technology. In principle a series of membranes can be used to isolate a reasonably pure sugar stream from juice (Saska, 1995; Kochergin, 1998). Saska (2000) has proposed such a process using only ultrafiltration and nano filtration to produce juice suitable to crystallise white refined sugar. This is an appealing process in that no chemical use is required, but it needs to be remembered that chemical cleaning of membranes is necessary. Flux rates for nano filtration appear to be very low, 28-35 l/(m².h) are recommended as average design figures (Saska, 2000) making equipment required for the colour removal stage large. To achieve reasonable recoveries some diafiltration (retentate is diluted with water and re-filtered to recover more sugar) will probably be necessary, increasing the quantity of equipment required. The energy requirement would also be significant. Although the technology is reasonably well established in other industries, colour removal from sugar juice is a new application. Concentration adjustments to optimise the “brix flux” are also probably necessary.

Demineralisation

Ion exchange demineralisation is a well developed and mature technology (Kim, 1997) used with success in the Beet and Corn Syrup industries (van der Poel *et al.*, 1998). Clearly this approach may be used to reduce ash levels in juice (and hence in the subsequent sugar made) to suitable levels. Work by Dymond *et al.* (1948) carried out at Darnall and work done in Hawaii (Honig, 1953; Shore *et al.*, 1988) and more recently confirmed in laboratory studies by Saska (1995) showed that demineralisation not only removed ash but also could remove substantial quantities of colour at the same time. In all cases a direct white sugar was claimed possible, but it seems that this was not of EEC2 quality. Experimentation was not followed up, presumably because of resin fouling problems since there was no suitably tight filtration step ahead of the resins. It also appeared that again invert was not a problem, being satisfactorily removed during crystallisation, washing and curing.

It has been established that cation resins in the H⁺ form can invert sucrose rapidly (Khan *et al.*, 1996), and to reduce this, cooling of the feed stream to below 20C is required (Honig, 1953; Shore *et al.*, 1988). Demineralisation technology has been implemented in the Beet industry with apparent success (Shore *et al.*, 1988) and demineralisation is widely used in the corn syrup industry, and can be considered to be a mature technology.

Demineralisation involves removal of all the ash, replacing the cations with H⁺ ions and the anions with OH⁻ ions. Instead of swapping one ash species for another as described above in the softening process, the ash is replaced by water. Since the quantity of ash in molasses or syrup is the same as that present in clear juice, syrup or any intermediate sugar stream, the quantity of resin required to remove all the ash from the process remains essentially the same. Clearly it is advantageous to perform the ion exchange operation as early in the boiling house as possible to achieve the maximum benefit. As already noted, filtration is a necessary pre-treatment for ion exchange and this unit operation would still be required. An optimum “brix flux” would again need to be considered and suggest that the UF and IX steps are best placed after some evaporation has taken place. Thus the full benefit of reduced scaling in evaporators is not seen in the 1st effect evaporators.

The conceptual process based on demineralisation is thus as follows:

- Normal juice clarification
- Concentration
- Ultrafiltration
- Cooling
- Demineralisation/ decolourisation
- Further concentration
- Sugar boiling to produce a white sugar.

The process as outlined now offers the following possible benefits:

- Demineralisation offers the possibility of a low ash sugar.
- Demineralisation combined with decolourisation allows the production of a white sugar.
- Increased sugar recovery as a result of lower ash (Dymond, 1948; Kochergin *et al.*, 2000).
- UF ensures that turbidity specs are satisfied.
- A low ash molasses, suited to being a fermentation feedstock, or chromatographic recovery is achieved (Fechter *et al.*, 2001).
- Invert levels satisfy EEC 2 Specifications.
- The process is relatively easily integrated into an existing mill (Rossiter *et al.*, 2002). While some changes to the boiling scheme are required, the proposed process requires the installation of new unit operations (Ultra filtration, and ion exchange) at some point between clear juice and syrup.
- Regeneration with suitable acids and alkalis allow possible schemes for the recovery of potassium or the production of a fertiliser feedstock well balanced to the needs of the cane plant (Fechter *et al.*, 2001; Honig, 1953).

Practical experiences with the processes presented

Of the processes discussed above the chromatographic approach has been experimentally tested on a pilot scale and some results reported by Kochergin *et al.* (2000). This work has confirmed the technical feasibility of the process but also the problems associated with excessive resin inventory and dilution effects. Chromatography is extensively used in the beet industry to recover sugar from final molasses and can be considered a reliable and robust technology (Godshall, 1999).

A process based on softening, and involving an industrial scale installation of both membranes and ion exchange softening, has been reported by Kwok (1996). It would appear that the membrane installation was effective at achieving the design criteria but the process did not produce direct white sugar. The installation in Hawaii is no longer in operation. Pilot plant investigations based on the NAP, including full scale costs, have been reported by Bekker *et al.* (2001).

A demineralisation-based process has been described by Fechter *et al.* (2001), based on extensive pilot plant scale work involving membranes, ion exchange and trial sugar boilings. Rossiter *et al.* (2002) have reported integration of the demineralisation process into an existing mill demonstrating that the process is relatively easily "added on" to an existing sugar mill. Experience with deashing of high-test molasses on an industrial scale, using an ISEP unit at Hulett's Refinery has been documented by Ahmed *et al.* (2001). The filtration and demineralisation processes used in the demineralisation-based process are similar to those used by African Products at Klipriver locally and at many other corn sugar producers around the world. Thus the unit operations proposed are well-established technology.

Conclusions

From a theoretical examination it is apparent that:

1. It is technically feasible to make a white (refined quality) sugar "directly" from cane juice (i.e. in a single mill without a backend refinery).
2. More than one approach is possible.
3. Overall recovery benefits are possible.
4. High quality molasses and other useful by-products are possible.

Unit operations available are summarised in Table 2, which is based on a similar summary by Kochergin (2002). Most of these operations have been well established in similar applications and tested to varying degrees by the sugar industry.

Table 2. Impurity removal summary (based on Kochergin, 2002).

Process	Membranes	Softening	Demineralisation	Chromatography	Absorbent resins	Crystallisation
Colour	Negligible (UF) Yes (NF)	No	Yes	Yes	Yes	Yes
Ash	No	Partial	Yes	Yes	No	Yes
Invert	No	No	No	Yes	No	Yes
Recovery	Yes	No	Yes	Yes	No	No
High quality molasses	No	No	Yes	Yes	No	No
Useful by-products	No	No	Yes	Yes	No	No

In all possible flowsheets requiring a crystalline product the crystallisation will be adequate to remove invert and can be used as a final purification stage (Invert removal in a single crystallisation is sufficient to meet EEC2 standards). Table 2 shows that several permutations of unit operations offer the possibility of success. Of these, two are most attractive from a technical and “proven” technology point of view, and meet the requirements listed in the introduction to this paper. These are the process based on chromatographic separation and that based on demineralisation. Since chromatography results in large resin inventories and significant dilution, a process based on ion exchange demineralisation appears to be the most promising at this stage. Ultimately capital and operating costs will determine economic feasibility, and this is too dependent on specific circumstances to be considered here.

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APPENDIX A

The effect of softening on ash in sugar can be estimated from the work of Lionnet (1998). The transfer of impurity to crystal is given by:

$$X_i = a F_i^b$$

Where:

X = Concentration of the impurity in the crystal (kg/kg)

a = constant

F = Concentration in the feed (mg/kg)

For Calcium, b has a value of 1,0

For K (presumably the same as Na) $b = 0,6$

Thus, if one were to soften a juice with Ca and exchange for Na or K, then the ratio of ash in Crystal made from softening juice to unsoftened juice can be estimated as follows:

$$\frac{X_K}{X_{Ca}} = \frac{a F_K^{0,6}}{a F_{Ca}}$$

and $F_{Ca} = x \text{ mg/kg}$ then

$$F_K = \frac{2x}{40} \times 39 \approx 2x \text{ mg/kg}$$

Then

$$\frac{X_K}{X_{Ca}} = \frac{(2x)^{0,6}}{x} = \frac{1,52}{x^{0,4}}$$

So for a typical stream of liquor with $x = 3500 \text{ mg/kg Ca}$.

The $X_K/X_{Ca} = 0,06$ or 6%, i.e. there is a 94% ash reduction in the crystal product.

Obviously, the lower the Ca concentration the less dramatic the effect.