

# WHITE CANE SUGAR PRODUCTION

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## **Abstract**

The options and prospects for the direct production of white sugar in a cane sugar mill are reviewed. Plantation white sugar has been made for many years in some countries but cannot meet the requirements for refined sugars. These low colour sugars are now a popular quality raw sugar for the increasing number of destination refineries. Attainment of refined sugar standards needs the application of some or all of the new separation technologies such as membrane separation, the use of adsorbents, and chromatographic separation. Only one of the new processes being proposed is close to commercialization, namely the WSM process developed by Tongaat Hulett Sugar. A new process for the production of white sugar in a cane sugar mill developed at the Audubon Sugar Institute is described in some detail. It does not require membrane separation and involves adsorption of colour and other impurities using granular activated carbon and ion exchange resins. Chemical regeneration of the carbon is utilized, which enhances the attractiveness of the process. Preliminary information is given on a possible full-scale installation in a raw sugar mill.

## **Introduction**

Some significant trends are evident in the world sugar market. In the 1980s the proportion of white sugar traded on the world markets, both beet and cane showed a clear increase relative to the raw sugar (*Clarke* 1986). More recently, the trend has changed, since a number of new large refineries have been built and white sugar exports from producing countries, particularly from the EU, have reduced. Another trend evident is the swing to VHP or better raw sugar supply to refineries, and in particular an increase in deliveries of very low colour raws exemplified by Brazilian “cristal”.

*Todd* (1997) has shown that the average cost of production of refining in attached refineries is about 55 % of the cost in stand-alone refineries. In order to compete, autonomous refineries have to be larger to achieve economies of scale and lower unit production costs. A further trend evident in the US has been the increase in vertical integration from field to white sugar output. All beet sugar factories in the US are now owned by the growers, and many large raw cane sugar producers have invested in refining, both white-end refineries and in existing or new refinery capacity.

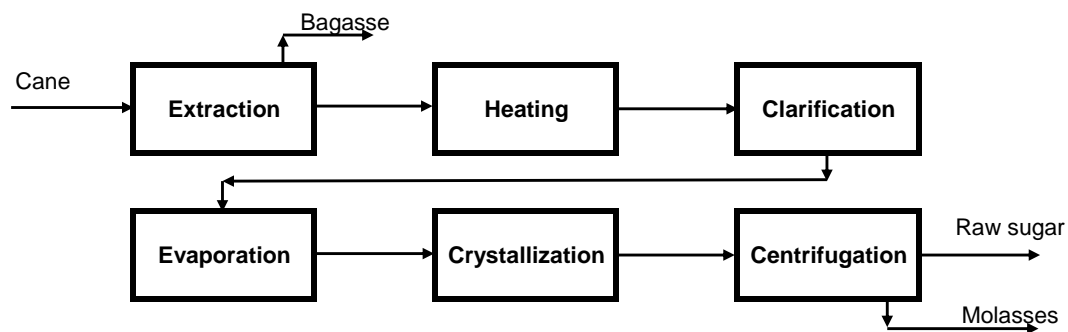
The first new stand-alone refinery to be build for 25 years was constructed in 1990 in Dubai. Since that time, over a dozen new refineries have been built and existing refineries have been expanded. Currently a number of new refineries are being built or are planned in areas such as Bangladesh, Egypt, Syria, India, Louisiana and Israel among others.

The relationship between white and raw exports has reversed, with raw sales now increasing relative to whites. Freight differentials, between the costs of shipping raw sugar in bulk and bagged white sugar, are partially responsible, particularly with larger vessels now shipping bulk raw sugar. In many countries too the tariff protection for import of white sugar is higher than for raw sugar.

Another factor promoting the development of new destination refineries is the considerably enhanced raw sugar quality available. Sugar quality has a major impact on refining costs and an interesting example of a refinery working together with a raw supplier was reported by *Hikmat and Oliveira* (2006). Improvements in sugar quality to achieve a polarization of over 99.6 °Z resulted. This study validated their statement: “Reducing the impurities in raw sugar is a governing factor in improving performance of the sugar refinery and can be achieved easily and economically in the sugar mill”. This effort has resulted in the Dubai refinery being among the lowest cost refiners in the world.

The threats to the refiners are a reduction in the white premium, a new higher sustained level of energy costs and the prospect of direct white sugar production technology developed to a feasible stage (e.g. *Fechter et al.* 2001; *Rossiter et al.* 2002; *Kochergin et al.* 2001; *Oliverio and Boscariorl* 2006; *Rein et al.* 2006).

The raw sugar produced in a cane sugar mill is normally sent to a refinery, to be converted from a light brown raw sugar with a purity of around 98.5 % to an almost pure (>99.9 %) white sugar for sale to consumers. A flow diagram showing the unit operations in a raw sugar mill is shown in Figure 1.



**Figure 1:** Raw sugar mill flow sheet

The refining process incorporates colour removal steps, filtration and crystallization. The latter is a particularly efficient purification process, but is expensive in terms of energy and equipment.

Mill white sugar has been produced at raw sugar mills for many years without refining or recrystallization. This has generally involved the use of juice and/or syrup sulphitation, producing a sugar with a colour of around 100 to 200 IU. Nowadays standards for white sugar are considerably higher, requiring a colour below 45 IU for EU No 2 or even 22.5 IU for grade 1 white sugar.

More recently many attempts have been made to produce white sugar directly in a raw sugar mill without melting (dissolving) and re-crystallizing through the use of new and innovative separation technologies. The prize is the significant additional value of the product, with a white premium consistently over \$60 /tonne (*Todd 1997*), as well as lower energy costs, because the mill uses sugarcane bagasse as a fuel.

## **Plantation white sugar**

### ***Indian sulphitation process***

Juice sulphitation has a remarkable decolourizing effect in some industries, particularly India, where it is widely practised. Sulphur is burnt in a sulphur burner under carefully controlled conditions to produce SO<sub>2</sub> while avoiding sublimation and the production of SO<sub>3</sub>. Contact of the gas with juice pre-heated to about 70 °C takes place in a sulphitation tower or a series of tanks. The precipitate formed due to the addition of lime and SO<sub>2</sub> separates in juice clarification. This process is generally used together with syrup sulphitation in flotation clarification. Sulphur consumption is 250 to 500 mg sulphur/kg cane if only juice sulphitation is practiced. If both juice and syrup are sulphited, usage can be as high as 900 mg/kg cane. The advantages of sulphitation are:

- The mud settles more rapidly (hence improved capacity of clarifiers).
- The massecuites are less viscous and boil faster.
- There is a marked improvement in the colour of the sugar – there appears to be a significantly reduced transfer of colour from the mother liquor into the crystal.
- There is a slight gain in the capacity of centrifugals.

However there are also some disadvantages:

- There are heavier fouling deposits in heaters. These may largely be avoided by sulphiting hot, but this involves an increase in the heating surface required.
- An increase in chemical and maintenance costs (in sulphitation tanks, circulation pump, sulphur, corrosion of vessels and pipes, etc.).
- Heavier use of lime in the process and in the recirculating cooling water.
- The residual sulphur in the sugar may exceed the specification demanded by some customers.

### ***Brazilian process***

Juice sulphitation is common using sulphitation towers or juice ejectors, using 150 to 300 mg sulphur/kg cane. However it is more common to see syrup clarification carried out using phosphatation, with the addition of phosphoric acid at 50 to 150 mg/kg sugar, and lime. This helps to get the SO<sub>2</sub> content of the sugar down below the 10 mg/kg specification.

The Brazilian production of low colour sugar is materially assisted by the simultaneous production of ethanol. This enables to mill to choose the higher purity streams (e.g. primary juice) to be used for sugar production and the lower purity streams go to ethanol production; in addition there is no recycle of low purity streams back to sugar production. In addition, a lower pH profile in the mill can be employed to minimize colour formation; inversion of sucrose is not an issue since the reducing sugars formed are still utilized in ethanol fermentation.

### ***Blanco Directo sugar***

Tate and Lyle introduced the Blanco Directo process, which involves filtrate flotation clarification and syrup clarification, together with phosphoric acid and lime addition, very similar to the refinery phosphatation process (*Bennett and Ross 1988*). A comparison by *Trott (1988)* of the qualities of refined sugar with direct mill white sugars is shown in Table 1.

	<b>Refined</b>	<b>Blanco Directo</b>	<b>Mill white</b>
Polarization	99.9	99.8	99.6
Colour (IU)	10-80	100-200	200-250
Turbidity (ICUMSA)	10-30	20-50	100-500
Ash (g/100g)	0.01-0.04	0.05	0.1
Invert sugar (g/100g)	0.01-0.04	0.02	0.1
SO <sub>2</sub> (mg/kg)	-	1-5	20-50

**Table 1.** Comparison of white sugar specifications

The benefits of syrup clarification are widely realized and it is becoming a more popular process, not only in Brazil, particularly when a higher quality sugar is required for subsequent refining. It can remove about 85 % of the turbidity, gives a small ash and colour improvement and reduces massecuite viscosity. Its effect on sugar quality is generally greater than the improvements measured in syrup quality suggest (*Steindl and Doherty 2005*), largely because high molecular mass impurities are removed. Filtrate clarification is still successfully used in some mills, but has been found to be difficult to operate in others and has been abandoned where tried in South African mills.

### **Options for white sugar production in a raw mill**

#### ***The Dedini DRD process***

Dedini have taken the Blanco Directo approach a stage further by introducing ion exchange. The process includes juice sulphitation, syrup clarification/phosphatation, deep bed filtration and then three ion exchange stages for softening (demineralization), decolorization and polish decolorizing (*Oliverio et al. 2007*). This process has been run as a pilot plant in Brazilian mills for two years. It is claimed that on average 52 % decolorization is achieved with ion exchange giving a syrup of 4200 IU, regenerating the columns with NaCl and NaOH. This syrup boiled in a small pan gave sugar meeting EU No. 2 colour specifications, indicating a very low colour transfer, intermediate between that obtained in the beet and cane industries.

Dedini are promoting this process for new installations or as a retrofit into an existing sugar mill.

### ***Membrane separation***

Developments in membrane technology have been such that they can cope with the demanding requirements in a sugar mill, namely the ability to handle high temperatures and varying suspended solids characteristics including abrasive particles, while still achieving good separations at high flux rates and reasonable cost. Ceramic, stainless steel/ceramic and polymeric membranes are now available which can meet these requirements.

Although great strides have been made in terms of both cost and technology, the use of membranes for decolourization of juice is limited. It is not feasible to apply membranes to raw juice, but they have been tried in many situations using clarified cane juice. This has to be done at high temperature around 90 °C to ensure that no microbiological loss or fouling occurs. *Steindl* (2001) surveyed reported results which show an average colour removal from clear juice of 14 % using ultrafiltration, based on different membrane molecular weight cutoff values down to 50 kDa. There is some evidence to suggest that the improvement in colour of sugar may be slightly higher than the reduction in juice colour implies. Nevertheless it does not appear that membranes alone can enable the direct production of white sugar. In addition, the recovery of the sugar remaining in the retentate is costly.

No improvement in recovery of sugar results because there is only a marginal improvement in purity after membrane filtration (*Kochergin et al.* 2000). It appears that in future membranes may only find application in raw sugar mills in combination with other processes which add value.

The new Applexion Process (NAP) was developed in Hawaii, using membrane separation and decolorization (*Kwok* 1996). This process operated for some time in a raw sugar mill in Hawaii but has been discontinued. It also does not have the ability to remove ash to achieve refined sugar standards.

### ***ARi Chromatographic separation technology***

Chromatographic separation has been used for many years to recover sugar from beet molasses and is a well-developed process. It has not found much use in the cane industry, because of the lower sucrose content, the suspended solids content and the high calcium content in cane molasses which interferes with the process. The advantage of chromatographic separation lies in the fact that a high purity sucrose stream can be isolated, from which refined sugar can be produced, but in addition other products of higher value can be recovered.

Direct white sugar production involves either the removal of the sugar from the juice or the removal of colour and ash from the juice. This process developed by ARi (Amalgamated Research Inc.), which involves the use of chromatographic separation, is an example of the former. It is preceded by membrane filtration.

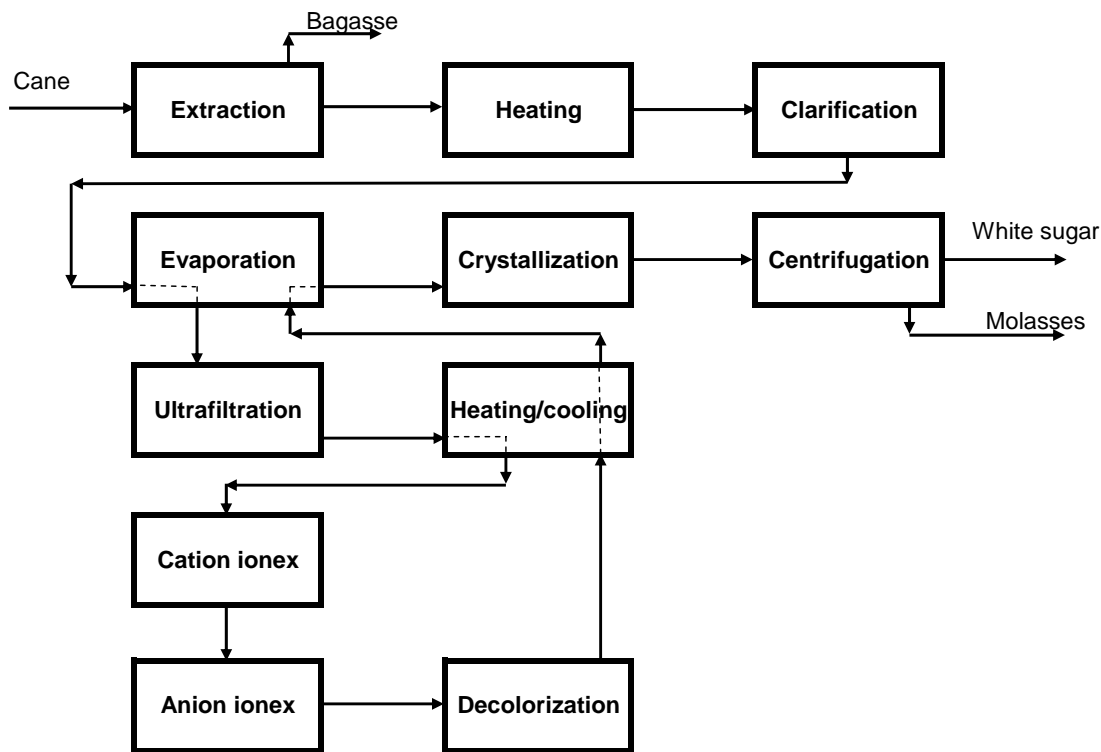
Use in a cane sugar mill requires the juice to be softened before chromatography can be applied. This is an expensive process, but would have the added advantage of eliminating evaporator scaling. The cost of the process could however be reduced by the use of fractal distributors (*Kearney et al.* 2001). Until recently, it was thought that softening would have to be preceded by membrane separation; however trials have shown that possibly this may not be necessary.



The process does have some other significant advantages, namely (Rossiter et al. 2002):

- an increase in sugar yield
- a large improvement in sugar quality
- the production of a high grade molasses for by-product use
- the removal of species that foul the evaporators
- higher heat transfer coefficients in pans and evaporators

The process is represented schematically in Figure 3. The juice is concentrated to 25 Brix because the maximum dissolved solids flux is achieved on membrane separation at this concentration. Cooling of the juice is necessary before cation exchange treatment because significant inversion could otherwise occur at the low pH values experienced in cation exchange. Some heat recovery is achieved in reheating the treated stream. The ion exchange processes are carried out in a simulated moving bed system, with 36 cells for each of the three ion exchange steps (Jensen 2007), which reduces the resin inventory, regeneration chemical usage and water consumption. Disposal of the membrane retentate becomes a problem if the process is not operated as a slip stream in a mill.



**Figure 3.** The WSM process for direct white sugar production incorporated in a raw sugar mill.

The process has been running as a slip stream process at Felixton in South Africa for the last 3 years, designed to process 15 % of the mill's clarified juice through the WSM process. During the 2006 season over 5000 t white sugar with an average colour of 37 IU and meeting all EU No. 2 specifications was produced. Various problems largely associated with peripheral equipment have been experienced and the process is not yet in commercial production.

The process is characterized by high chemical requirements for ion exchange demineralization. The use of suitable regenerants can produce effluents rich in potassium and nitrogen, which could overcome a disposal problem if used to fertilize the fields.

### **Development of a new direct white sugar production process**

The objective of the work done at Audubon Sugar Institute was to investigate options for producing white sugar directly without the use of ultrafiltration. Mill clarified juice was treated with a combination of granular activated carbon (GAC) column decolourization and ion exchange columns to remove ash and additional colour. The use of oxidants in a combination with the treatment was also investigated.

The process investigated was expected to achieve the same advantages as the WSM process given above. In addition it eliminates the need to clean first effect evaporators.

Activated carbon is commonly used for decolourization in sugar refineries, but has not been applied in treating cane juice. Because of the low viscosity of cane sugar relative to refinery liquor, higher flow rates are possible. The carbon columns are also capable of handling some turbidity in the juice without serious fouling.

Oxidants have been used to obtain additional decolourization in refining when circumstances demand it. They destroy colorants by attacking susceptible functional groups without attacking sucrose and have the advantage that they have no adverse environmental effects.

*Bento* (2004) reported that hydrogen peroxide added before resins has a number of beneficial effects. Conjugated double bonds are broken and carboxylic acids are formed. This decreases the molecular weight of the colorants and increases their propensity to be removed by ion exchange resins. Dosage rates of 500 mg/kg DS resulted in longer cycles and higher rates of colour removal. As a consequence, chemical and effluent disposal costs are reduced.

### ***Initial laboratory investigations***

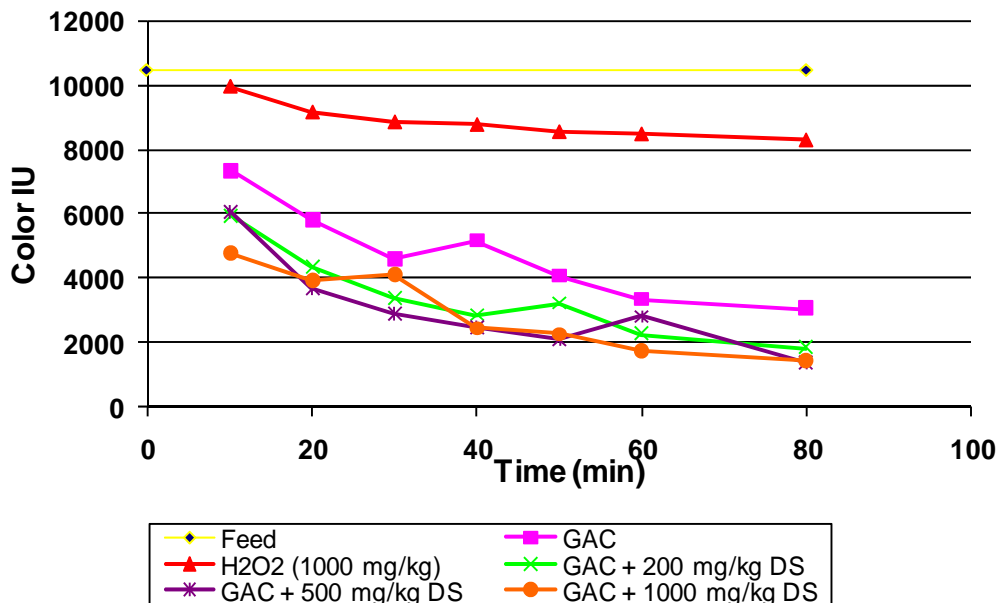
Pure sucrose is colourless, but may appear coloured because of the inclusion of small amounts of coloured material in the sugar. Colour is the generic term used to cover a wide range of components which contribute to the colour of sugar. Most of these compounds are complex and not easy to quantify and colour is measured as the total effect of all colorants on light absorbance at 420 nm. This is not altogether satisfactory, because different colorant components behave differently in the various processing and decolourizing operations.

Work done on modelling the removal of colour in the resin columns of the WSM process has been reported by *Broadhurst* and *Rein* (2003). Attempts were made to characterize colorants on the basis of their molecular mass, using GPC measurements. Suitable deconvolution of the retention time peaks was used to yield quantitative data on various molecular mass pseudo-components (*Broadhurst* and *Rein* 2003a).

This work was done on ultrafiltered juice samples. Subsequent work was aimed at modifications to the process to obviate the use of membranes, because the cost of membrane separation militates against the economic viability of such processes, in spite of recent advances in the technology.

Granular activated carbon (GAC) was substituted as an initial juice treatment step. It is less prone to fouling than resins, and is a useful decolourizing process in its own right. A high juice flow rate through the GAC column of 15 BV/h was used. It was still necessary to cool the juice after the carbon columns to below 10 °C to prevent inversion in the ion exchange columns. A comparison of the juice colour obtained using the carbon column and ultrafiltration prior to ion exchange showed that using GAC, a juice colour of 2000 ICUMSA units (IU) was possible even after repeated regenerations, about half of that using membrane separation before ion exchange (Ellis 2004). Juice feed colour averaged about 12 000 IU. Regeneration of the carbon was undertaken in a laboratory furnace at 600 °C.

The effect of adding hydrogen peroxide was investigated for this process in conjunction with carbon treatment. The results of some laboratory batch tests are shown in Figure 4. This indicated that additional colour removal could be achieved when peroxide treatment was combined with GAC decolourization. The tests showed that little colour was removed by peroxide on its own.



**Figure 4.** Batch tests on colour removed from juice by combinations of carbon and peroxide treatment at 80 °C.

It was found that the modified process using a carbon column could be substantially improved by the addition of hydrogen peroxide to the juice before the carbon columns. Because the colour of the juice is much higher than the colour in refinery streams, a dosage rate of 1000 mg/kg DS was used ahead of the carbon columns. A reaction time of 30 minutes at a temperature of 85 °C was used.

Another important aspect of the resin performance is the deashing ability of the resin. The combination of carbon and peroxide also leads to a far superior overall ash removal.

**Factory trials.**

Trials have been run over three seasons in Louisiana sugar mills. More detail is given elsewhere (Rein et al. 2006). The first trial investigated only the performance of GAC in treating clarified juice, pre-treated with hydrogen peroxide over a 10 week period. The carbon was chemically regenerated once/week, using a combination of washes of HCl, NaOH, ethanol and a special additive (Bento 2006). On average the juice colour was reduced from 12 000 to about 3 000 IU.

The system proved to be fairly robust, handling periodic entrainment from the clarifier, but in spite of this still achieving substantial decolourization. The first step in regeneration involved backflow at a high rate to remove entrapped mud from the column.

Ash removal was minimal, as expected. The pH generally dropped across the column, as the acid wash appeared to take out the magnesite in the carbon which would otherwise maintain a neutral outlet pH. Variations in temperature over the test period appeared to have little effect. A more important factor was the mud carry-over from the clarifier. On two occasions after heavy rains, the column had to be back-washed with water to restore the required juice flow rate.

A second trial was conducted at St. James mill during the 2005 season. Some changes were made to the test program. Apart from eliminating experimental problems experienced the previous year, the trial was modified to assess a different ion exchange treatment. Instead of employing strong acid cation and weak base anion columns after the carbon, as was practiced in the laboratory trials, a mixed bed of softening and decolourizing resin was employed. This mixture did not change the pH of the juice, and so dispensed with the need for cooling the juice to a low temperature to avoid inversion.

A small GAC guard column was used to protect the second column from suspended solids carried over with the juice. Before the second column, juice was contacted with hydrogen peroxide. After passage through the GAC columns, juice was treated with cationic and anionic resins in a mixed bed column, to remove hardness in solution and to reduce juice colour. The installation worked continuously for two months with clarified juice at a flow rate of 1 BV/h (on total carbon). Average data are shown in Table 1. A reduction of 81.5 % in juice colour was obtained over the test period (75.6 % on carbon and 24.1 % on resin).

**Table 1:** Composition of clarified juice before and after treatment

	<b>Brix</b>	<b>Colour (IU)</b>	<b>Turbidity (NTU)</b>	<b>pH</b>	<b>Ash (g/100 g DS)</b>
Clarified juice	14.3	9 070	212	7.7	3.5
Juice after 1 <sup>st</sup> GAC column	14.3	6 870	191	5.9	3.7
Juice after 2 <sup>nd</sup> GAC column	14.1	2 220	187	5.6	3.9
Juice after ion exchange	13.9	1 680	165	6.0	4.0
Reduction (%)		81.5	22.2		

A turbidity decrease of 22.2 % (Table 1) and hardness (calcium and magnesium) reduction of 88.5 % was observed (Table 2). An increase in lactic acid through the columns was also registered at times, because it was not possible to keep the temperatures consistently above 80 °C in the pilot plant.

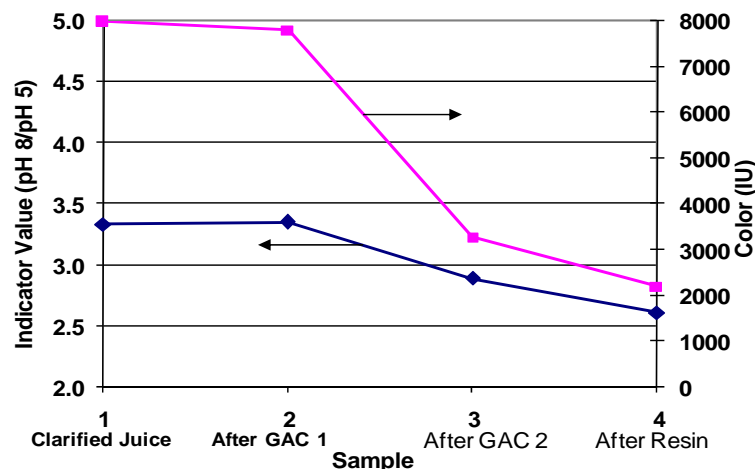
**Table 2.** Composition of juice

	<b>Apparent purity (%)</b>	<b>Ca + Mg content (mg/kg DS)</b>
Clarified juice	87.4	4240
Juice after 1 <sup>st</sup> GAC column	88.6	4460
Juice after 2nd GAC column	88.2	4350
Juice after resin column	89.0	489
Reduction		88.5 %

Resin cycles varied between three and four days. After each cycle, the resins were washed with condensate water followed by regeneration with NaCl and NaOH. The performance of the ion exchange resins was affected by contamination of the “condensate” stream with low quality water, which adversely affected regeneration. Nonetheless it appears that this option will not reduce the ash content of juice sufficiently to be able to achieve EU No 2 sugar ash specifications; an ash reduction of at least 75 % is necessary.

After each five day cycle, carbon was washed and regenerated chemically. Colour loading was high, well over 200 t DS·IU/L resin per cycle.

Some analyses done on four samples during one day of the trial gave the values of colour and Indicator Value shown in Figure 5. In this case the Indicator Value is the ratio of colour measurements at 8 and 5 pH instead of the usual values of 9 and 4 pH. This shows a steady decrease in Indicator Value, suggesting that a substantial part of the natural colorants (phenolics and flavanoids) is removed. *Bento* (2006) evaluated the propensity of other juice colorant types to be adsorbed on GAC.

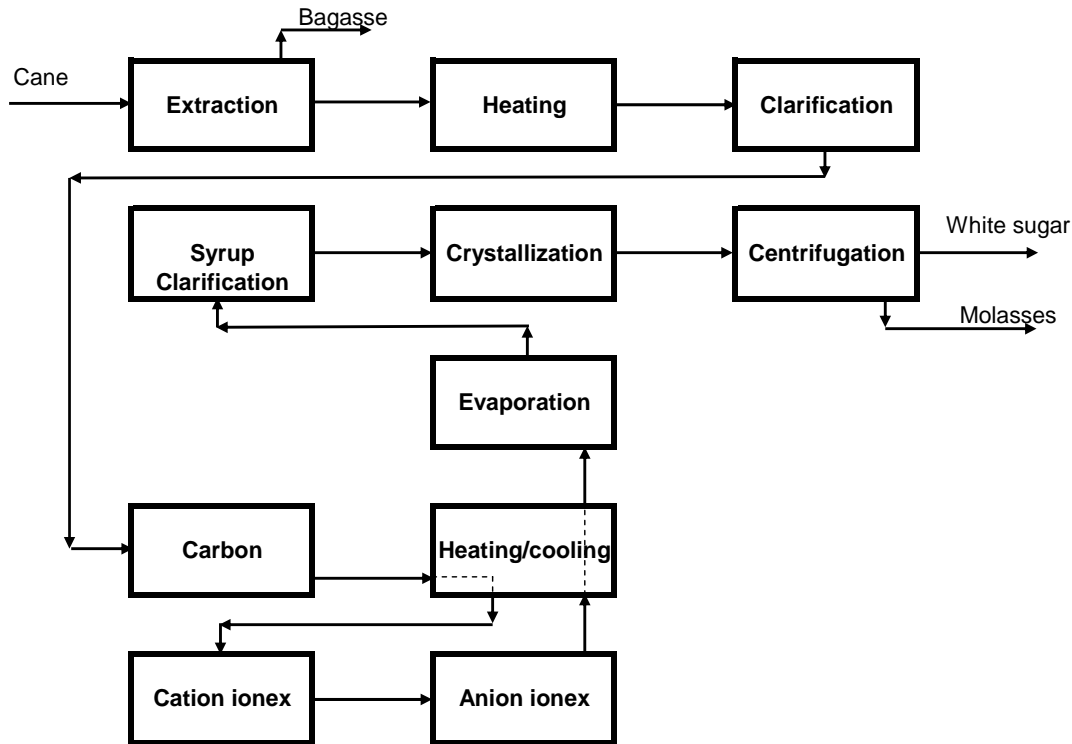


**Figure 5.** Measurements of colour and Indicator Value

A third trial was run at Raceland mill in 2006, with demineralization following the initial GAC treatment. The juice was refrigerated before resin demineralization. The trial was seriously affected by equipment problems and the plant was unable to run the full process continually for long enough to establish some of the required design parameters required. However it did appear that more than 50 % decolourization could be achieved routinely with GAC and 80 % decolourization over the resin columns when they were properly regenerated. The trial also showed that the resin performance was not affected by resin fouling. Again the short season proved to be a problem in getting pilot plant equipment to work reliably in a short period of time.

**Application to a raw sugar mill.**

The flow sheet for a raw sugar mill using this process is shown in Figure 6. Both the process and the regeneration are the subjects of patents (*Rein et al. 2007; Bento et al. 2008*). A syrup clarifier is incorporated to ensure that the suspended solids content is reduced sufficiently for the high quality sugar required. It goes without saying that juice clarification must be well-controlled to ensure that the columns are not subject to carry-over from the juice clarifiers at any time.



**Figure 6.** Alternative process for direct white sugar manufacture in a raw sugar mill.

A number of options exist for the configuration of the GAC system. Instead of a guard column, a pulsed bed GAC system would on a regular basis discharge the carbon layer which could get fouled by carry-over from the clarifiers. A substantial advantage of the chemical regeneration system however is the ability to configure the GAC system in the form of a simulated moving bed. The advantages, as with ion exchange, could be substantial (*Hubbard and Dalgleish 1996*), in particular reducing the cost of regeneration and reducing the amount of sweet water produced.

Chemical regeneration of GAC seems viable, at least over a period of 10 weeks. Beyond this time it may be necessary to introduce thermal regeneration of part of the GAC to keep the process operating efficiently. Transport of carbon to an outside facility would be the best option if transport costs allow it. In thermal regeneration, something between 4 % - 10 % of the GAC is lost in the regeneration process. Thus after 10 regenerations a significant amount has already been lost in the process of thermal regeneration. This is avoided in chemical regeneration.

The use of ethanol in regenerating GAC is particularly suitable in a sugar mill which also produces ethanol, with the following advantages:

- The spent regenerant can be sent back to the distillation plant for a total recovery of the ethanol.
- A periodic ethanol wash also improves the decolourization capacity of the SAC column (*Broadhurst and Rein 2003*), which could be recovered.
- Inversion to reducing sugars is not important, eliminating the need for refrigeration before ion exchange

The cost of regenerants for the process presented here is estimated to be roughly \$0.50 for a colour loading of 1 t DS·IU/L of carbon per cycle. In the Louisiana context, this translates to 0.40 US cents/kg sugar for regenerating GAC. The cost of regenerating the ion exchange columns is 1.67 US cents /kg sugar, giving a total of about 2 US cents /kg. This can be reduced to < 1.50 US cents with some reuse of the HCl and NaOH.

The disposal of ion exchange effluent is a problem or perhaps an opportunity depending on the particular mill's circumstances, as in the case of the WSM process. The inorganic material removed is that present in the cane, and is particularly high in potassium, which has considerable fertilizer value. Regeneration of the WBA columns with ammonium hydroxide can significantly boost the fertilizer value of this regenerant (*Rossiter et al. 2002*). The use of the regenerant as a fertilizer could be the best solution if it could be integrated into the combined operation of growing and milling without any detrimental effects on the environment.

Additional power and steam requirements are expected to be modest, in contrast to processes involving membranes which incur high power costs.

## Conclusions

Considerable attention is being given to the direct production of white sugar in a cane sugar mill. Improvements in separation technologies are making some approaches look viable and will probably be used to produce better quality sugar if not sugar of refined quality. Combined with the advantages of higher sugar recovery, better molasses quality and the elimination of evaporator scaling, prospects for direct white sugar production are good. The step from small scale proving of a process to full scale viability is a difficult one to take and further work is still necessary to establish optimal design criteria and financial viability.

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