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**“Pros and Cons” of various decolorization processes for production of refined sugar**

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12<sup>th</sup> edition of “Cane Sugar handbook” and 1<sup>st</sup> edition of “Handbook of Sugar refining”

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**Abstract**

Over the last half a century, many industries have advanced/updated their processes to meet the challenges and opportunities of the 21<sup>st</sup> century, and yet the conventional processes are still the “norm” in the sugar industry.

With increasing energy cost and pressing environmental issues related to refined sugar production, it is imperative that sugar technologists undertake a critical re-evaluation of 1950’s/1960’s decolorization technologies with respect to their effectiveness, Namely: 1) mechanical decolorization -- affination process, 2) primary decolorization processes— carbonation or phosphatation, 3) secondary decolorization processes – bone char system, granular activated carbon process, ion exchange resin system, and powdered activated carbon process, and finally 4) crystallization process.

In this paper, the advantages and disadvantages of each decolorization process is reviewed. The incompatibility and serious problems associate with combined use of phosphatation process with the “single massecuite back boiling system”, a common design of newer refineries in developing countries, are discussed in details. The redundancy and ineffectiveness in color removal using both phosphatation with decolorant and Ion exchange process are also presented.

Recommendations to stream line and simplified the sugar refining using the best available process are presented in hope for sugar refining industry to survive and prosper in the 21<sup>st</sup> century.

**(I) Function of “refining”**

Sugar refining involves purification of sugar to meet food safety requirements and customers’ demand. There are six groups of non-sugar: suspended matters, color, ash, macromolecules (turbidity), microbes, and heavy metals. Food safety requirements would mandate that microbes and heavy metals in the final sugar products must meet food grade quality. Turbidity, mostly consists of macromolecules such as polysaccharides, dextrans, starch, waxes, gum, need to be removed for customers’ quality control. Microbes, turbidity and suspended matters can be removed by carbonation and phosphatation during the processes of decolorization and finally by tight filtration via filter presses with diatomaceous earth (DE). Color and ash removal are optional, depending on customers

requirements. For example brown sugar is a refined product but the color and ash are many times that of refined white sugar.

## **(II) Selection of Decolorization Processes**

To achieve the desired degree of decolorization, The decolorization process chosen should be designed to give a fine (pan feed) liquor color of 250 maximum and an average of 200 color (ICUMSA method at 420 nm) from an input color of 1000 ICU. In North America, a decolorization scheme generally includes a primary decolorization process such as carbonation or phosphatation, followed by a secondary decolorization process such as bone char, granular activated carbon, or ion exchange process. However, more often than not, due to inefficiency in its operation many refiners would use more than one secondary decolorization processes, such as ion exchange process followed by granular activated carbon.

## **(III) Primary decolorization process: Carbonation vs. Phosphatation**

### **Phosphatation**

Phosphatation process has the following characteristics:

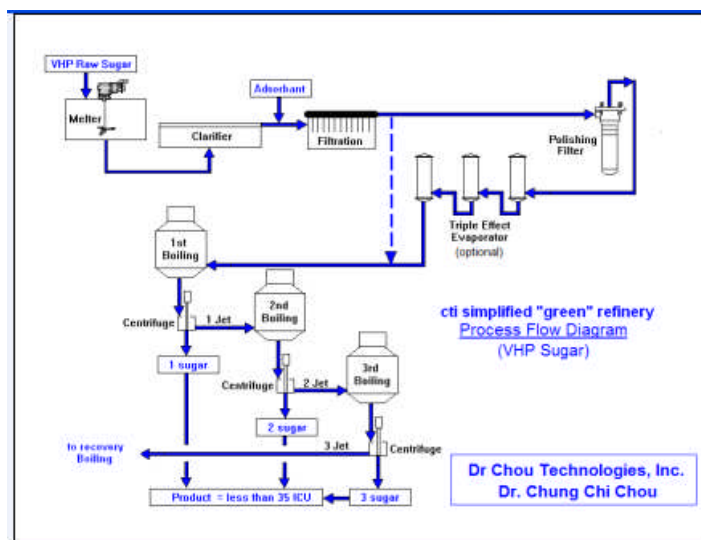
- a) Higher sucrose loss due to low pH and three stage scum dewsweetening system
- b) Higher energy consumption due to process instability and scum desweetening
- c) Lower initial capital cost.
- d) More flexible for capacity expansion
- e) Lower degree of color removal without decolorant.
- f) The best available refining process for VHP raw sugar consist only Phosphatation and press filtration with addition of up to 0.3% carbon adsorbent. No carbonation, no ion exchange resin, and no granular activated carbon is needed.**
- g) Phosphatation process is incompatible with Single Masecuite boiling system.**
- h) Use of both phosphation with decolorant and Ion exchange process are redundant, ineffective, and therefore costly for color removal.**

As previously discussed, the process stability of phosphatation is known to be “temperamental”, particularly when scum desweetening process is used. The result is both high physical and chemical sucrose loss, and high sediment in refined sugar if no filter press is used for clarified liquor. In addition, when there is a process upset, the operating variable available to resolve the problems is limited. It is preferred to use dewatering belt filter to desweetening the scum after one stage scum clarification

**(A) Phosphatation followed by press filtration has the least capital and operating cost for production of refined sugar from VHP raw sugar (Sit 2009 paper). Here is the summary:**

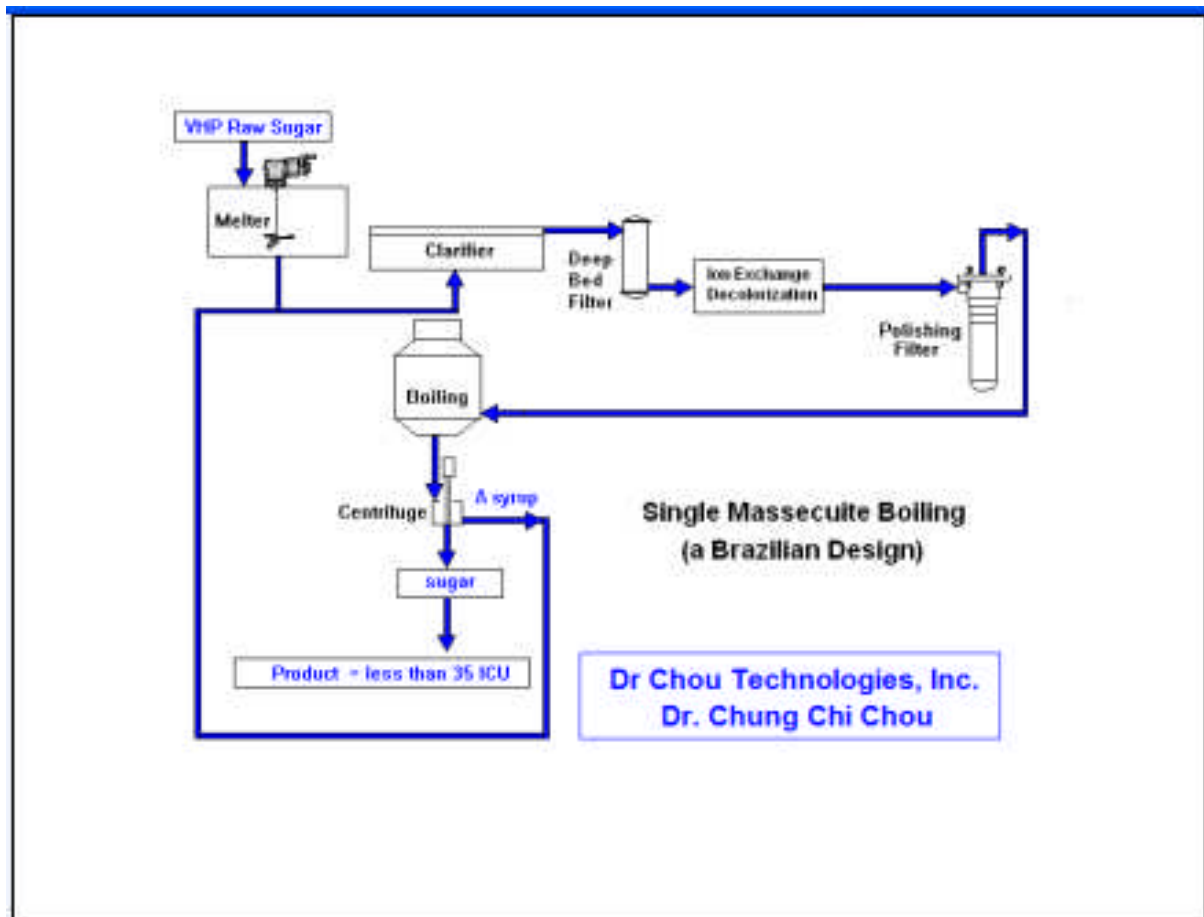
- (1) The color of VHP raw sugar entering the refinery should not exceed 1000 ICU.
- (2) If color is less than 600 ICU, It is optional to use phosphatation process. Only press filtration with decolorizing processing aids is needed.
- (3) If color exceeds 600 ICU, selective decolorization aids will be needed at the phosphatation clarifier depending the VHP raw sugar color level.
- (4) Baggasse is the only fuel needed for a mill with an attached refinery to produce refined sugar.
- (5) There is an estimated saving of 0.8 ton steam per ton of refined sugar produced in a refinery attached to a sugar mill as compared an autonomous refinery.
- (6) No sulfitation, ion exchange decolorization process, carbonation, and Granular carbon system are needed in a simplified refinery resulting in considerable savings in capital investment, and both operating & environmental cost
- (7) An attached simplified refinery is ideal for countries where the crop (cane grinding) season is over eight month a year.
- (8) Capital cost of an attached simplified refinery is much less than that of an autonomous simplified refinery.
- (9) The refining cost for processing aids ranges from US\$11.5 to \$20 per ton of raw sugar processed depending on raw sugar color input.
- (10) % sugar yield and % sucrose recovery are estimated to be 97.57% and 98.16% respectively, assuming a sucrose loss of 0.9% and molasses purity of 65. .
- (11) Sugar product color (combined R1, R2 and R3 sugar) of less than 40 ICU
- (12) Ability to produce value added sugar based products meeting US food grade quality standard.

**The process flow diagram of the simplified refinery is shown below:**



## B) Phosphatation process is incompatible with Single Masecuite boiling system

The boiling system is shown below:



The process only has R1 boiling and recycle all the R1 syrup back to the melter mixing with the melted VHP raw sugar and send it to the clarifier again. Most newer refineries then send the clarified liquor to the deep bed filters to remove any suspended solid over 5 microns, and then to ion exchange resin for final decolorization and crystallization. No filter presses are used

This **Single Masecuite in-boiling system**, as designed by a Brazilian vendor, recycled R1 syrup back to the clarifier for 60 cycles before sending the syrup to the recovery house. The process has many and serious disadvantages namely:

1. Contaminations of sugar products, particularly insoluble, due to repeated recycling "R1" syrup and impurities back to the clarification process.
2. High steam consumption

3. Unwarranted high chemical consumption for clarification (Phosphoric acid, lime, flocculant and decolorant)
4. Excessive sucrose loss and low sugar recovery due to prolonged/repeated boiling of recycled "R1" syrup.
5. Excessive sugar washing in centrifugals to achieve the desired refined sugar color and to remove residual flocculant to facilitate sugar drying in the granulator.
6. Significant loss of production capacity due to high viscous refined liquor resulting from repeated boiling of returned "R1" syrup and excessive usage of flocculant. In addition, prolonged boiling time in the vacuum pans for both refined and recovery strikes due to high viscous refined liquor also destroy more sucrose and create more color for the refining process to remove.
- 7) Flocculant dosage significantly exceeds US legal limit due to the need to put in additional flocculant into recycled "R1" syrup for clarification.

**C) Use of both phosphatation with decolorant and ion exchange process are redundant, ineffective and therefore costly for color removal due to the fact that both processes remove the same group of colorants as shown below:**

Anion IER

a) Strongly Basic (SBA)  $R-CH_2(CH_3)_3N^+OH^-$  (type 1)

b) Quaternary amine

Weakly Basic (WBA)  $R-CH_2(CH_3)_2N^+OH^-$

Tertiary amine

Decolorants (color precipitant) is in a chemical family of polyamine/quaternary ammonium salt

***Carbonation is technically the best choice for decolorization***

a) Percent color removal

Carbonation removes about 55-60% of color. Phosphatation removes only about 25-30% of color at a dosage of 250 ppm  $P_2O_5$ . This is because that similar type of colorants was removed in the raw sugar mill clarification process, which is basically a phosphatation process at a minimum dosage of 300 ppm  $P_2O_5$ .

It is known that use of Decolorant (Quaternary Ammonium salt, a color precipitant) in conjunction with phosphatation would improve the decolorization level to that of carbonation. However, the use of Decolorant will reduce the degree of decolorization by the ion exchange resin and, therefore, less cost effective. The reason is that both Decolorant (Quaternary ammonium salts) and ion exchange resin remove similar type of anionic colorants. If combination of phosphatation with color precipitant is selected, it is suggested Ion exchange resin in  $OH^-$  and  $H^+$  form respectively be considered for both

color and ash removal. Carbonation is more compatible with ion exchange system regarding decolorization efficiency.

#### b) Destruction of Invert

Carbonation, because of high pH in the first A saturator, eliminates most invert from sugar liquor. Phosphatation process not only does not destroy invert, it actually creates more invert, because of low pH.

Invert, at high temperature, is converted to organic acids which lower the pH and destroy sucrose, creating more invert. It is a vicious cycle.

In beet sugar production, practically all invert is destroyed in its carbonation process. The carbonated beet liquor going to pan boiling has a pH of above 8.5. Under this condition, no invert is formed by inversion of sucrose in the subsequent processes. This is the reason that invert content in beet molasses is less than one (1) %, as compared to 10% to 25% of invert in cane molasses depending on the type of decolorization process used in refineries.

#### c) Removal of ash

Carbonation removes up to 20% ash mostly sulfate, which is a culprit of scaling in both evaporators and vacuum pans. Phosphatation does not remove ash, and, therefore, has more scaling problem, unless ion exchange resin in  $\text{OH}^-$  and  $\text{H}^+$  form as discussed in the previous section.

#### d) Turbidity removal

The clarity of carbonated liquor is superior to that of phosphatation liquor due to the fact that carbonated liquor is pressed filtered using carbonated cake (created in situ) as filtering media. In addition, phosphatation is known to have “secondary floc” which would give precipitate after sand filters, unless press filters are used.

#### e) Quality of sugar product

Sugar produced by carbonation with press filtration is much more “sparkling” and has much less sediment in the final products. Sugar produced by phosphatation is of lesser quality due to a) secondary floc and b) some particulates are not removed by sand filter used in phosphatation process. Sand filter is known to remove only particles over 5 micron size.

#### f) Buffer capacity/sucrose loss

Carbonated liquor maintains a much higher buffer capacity (pH of 7.8-8.2) as compared to that of phosphate liquor at pH of 6.8 to 7.1. Higher buffer capacity also mean that

liquor will remain at higher pH as sugar boiling proceeded to 2<sup>nd</sup> or 3<sup>rd</sup> strike boiling, resulting in a much lower chemical sucrose loss.

g) Stability of process

Carbonation is known to have excellent process stability. In addition, process upset due to poor raw sugar quality, particularly high starch content, can be resolved by adjusting operating conditions, such as brix, temperature, filter aid level and press cycle, without reducing plant through-put.

The process stability of phosphatation is known to be “temperamental”, particularly when scum desweetening process is used. It is preferred to use dewatering press to desweetening the scum. The results are both high physical and chemical sucrose loss, and high turbidity. In addition, when there is a process upset, the operating variable available to resolve the problems is limited.

h) Capital cost

It goes without saying that carbonation will have higher capital cost.

i) Disposal and Environmental issues

Disposal of carbonate cake will be expensive due to the fact that carbonation will have 10 to 15 times more in volume of solid cake to be disposed of than scum from phosphatation. Therefore, disposal of carbonate cake may become an environmental issue in the future.

j) Additional benefit of the carbonation

The following additional benefits are expected from carbonation processes:

- 1) Products contamination by pan scale due to sulfates will be practically eliminated.
- 2) Acid washing of vacuum pan scale will be avoided. This will give vacuum pans longer service life and higher productive cycle.
- 3) The operation of a carbonation process generally is more consistent than other decolorization process, resulting in a steadier and/or higher melt rate (refinery capacity).

#### **(IV) Secondary Decolorization Process : Granular Carbon versus Ion Exchange**

##### **Granular Activated Carbon**

For a 1000 ton per day capacity refinery, in a granular carbon configuration, 8 columns 10'd x 42'h with column carrying 2,400 cubic feet of granular carbon and a carbon regeneration kiln with many peripheral equipment are required. In an ion exchange decolorization scheme, only 3 columns 8'd x 24' h are required. The more equipment necessary to run the operation, the more manning and maintenance are expected, resulting in higher refining operating costs.

Granular Activated Carbon (G.A.C.) is technically preferred as the adsorbent for removing color after carbonation. G.A.C. will also remove the odor in liquor, if any. An after burner for the stack gasses is required to eliminate odor emission problems from the furnace's regeneration of G.A.C. A GAC system is known to be capital intensive as compared to an ion exchange system.

One disadvantage of the granular carbon system is the high sucrose loss associated with the process; sucrose loss probably averages about 0.04% on an operating day and as much as 0.4% during weekend shutdown periods. To minimize the sucrose loss due to inversion, magnesite is added to the system during the revivification of G.A.C. to maintain the PH of treated sugar liquor.

### **Ion Exchange resin process**

Two problems with resin regeneration are the disposal of the high colored chemical effluent from the regeneration process and the disposal of the spent beads after their useful life. Recent innovation, nano membrane filtration for treating the high colored effluent, has reduced the quantity sodium chloride that must be disposed of outside the refinery. However, it is increasingly difficult to dispose of the remaining dark color nano membrane waste.

Some refiners have had product contamination that is organoleptic sensed (odor) when resins are used. The pre-washing process of make-up resins must be monitored and steps taken to insure that some of these resin beads are not exposed to elevated liquor temperatures. The operating and capital costs of the ion exchange process are about half and one third respectively of that of the granular carbon system.