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**Processes for maximizing VHP refinery sugar yield to 99%--  
Theory and Practices**

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**Introduction**

Dr Chou Technologies, Inc. was founded in 2001 with a goal to simplify refining process to save resources for next generation. The goal envisions a VHP refinery with a) NO need for GAC process and No need for Ion exchange system for decolorization, b) NO need for silo for sugar conditioning, and most importantly c) NO production of molasses. The former a) and b) have been easily implemented with proper selection of processes and engineering design. The above c) is the primary subject for discussion.

There are many VHP refineries annexed to raw sugar mills/factories with 2/3/4 “molasses/syrup” recycled back to the mill for further processing to recover additional sugar during the crop seasons. However, during the “off crop” season, continued boiling to 4/5/6 molasses is very inefficient due to extreme high viscosity resulting in low sugar yield and high energy cost.

This paper presents four alternative processes to quantum jump sugar yield from conventional VHP refinery sugar yield of 96.5/97.5% to a new high of 98.5/99.5% by introduction of value added sugar products via fully utilization of “final molasses”. The processes include: a) Micro-crystallization process--a continuous process, b) Areado process—a batch process, c) Muscavado process—a batch process, All above three processes have been in plant operation for many years, and, d) spray drying system.

In fact, a VHP refinery with one of the above processes fully implemented would produce no molasses and would have significant reduction in energy consumption and quantum jump in sugar yield. This can be achieved by transformation of molasses into value added products

The process of drying sugar via the “transformation” of a highly concentrated sugar syrup has been known for many years. The sugars produced in this manner are found to consist of aggregates of microcrystalline sugars with various physical and functional properties which offer considerable commercial opportunities and advantages.

In the early 1950's Amstar Corporation initiated an R & D program to study the nature of the process and to develop an efficient and flexible process to manufacture micro-crystalline specialty sugar for the food industry. This program resulted in a series of U. S. Patents and the installation of the world's first highly automated continuous commercial process in the early 1960's. Further details in the theoretical aspect of the process are described in the United States Patents 3,194,682, 3,365,331 and 3,642,535.

## THEORY

### Fundamental of the transformation process

- 1) Conditions of spontaneous nucleation
- 2) Rate of nucleation
- 3) Numbers of nuclei formed
- 4) Rate of crystal growth
- 5) Thermal balances

A process operation for tailor-made products requires proper control of the rate of the nucleation, number of nuclei formed, rate of crystal growth, and thermal balances during the "phases" change stage.

The rate of nucleation in a perfectly pure sugar solution can be approximated by the general equation of reaction Kinetics: (1)

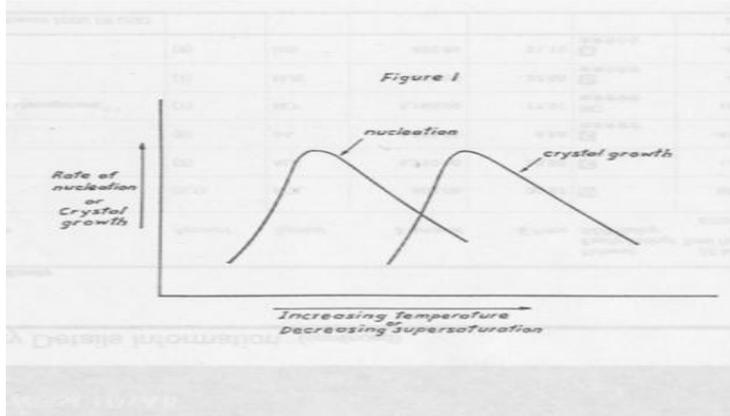
$$\frac{dN}{dt} \cdot \frac{1}{V} = K \cdot C^N$$

the term  $\frac{dN}{dt} \cdot \frac{1}{V}$  represents the number of nuclei formed per unit time and per unit volume.  $C$  is the degree of super-saturation expressed in concentration units.  $N$  is the reaction order. The rate of nucleation also depends on the impurities and/or ingredients added, the degree of agitation and the presence of seed crystals.

The rate of nuclei formation increases very rapidly with increases in the degree of super-saturation or decreasing temperature within certain ranges for each. The rate drops sharply at a lower temperature due to an increase in viscosity.

The rate of crystal growth in terms of weight increase per unit surface,  $\frac{dM}{dt} - \frac{1}{V}$ , is also proportional to absolute super-saturation, or with decreasing temperature; it then drops off, again due to the low diffusion rate at a higher viscosity. The peak maximum in

the nucleation curve usually takes place at a lower temperature than that for the crystal growth curve as shown in Figure 1.



1) Nielsen, A.E. Kinetics of Precipitation, Pergamon Press, Oxford (1966)

It should be noted that at a given degree of super-saturation, both the rate of nucleation and the rate of crystallization increase with increasing temperature.

For a given saturated sugar solution, the faster the rate of nucleation, the greater the number of nuclei formed, and the smaller the size of the final sugar crystal. Von Weimarn's equation relates the final crystal diameter,  $d$ , to the degree of super-saturation,  $C$ , as follows:

$$1/d = K-C/C_o$$

where  $K$  is a constant and  $C_o$  is the saturation solubility at a given temperature.

Production of specialty sugars with various functionality are achieved partly via control of the "retention time" of the process conditions at the peak maximum in both nucleation and crystal growth curves.

Another important design and operational variable for the process is the control of heat balance between the heat of crystallization and the latent heat of water evaporation during the transformation of liquid syrup to a solid sugar. The heat of Crystallization at the elevated temperature can be estimated via conventional thermo-dynamic treatment of the heat of "reaction" at different temperatures. The latent heat of water evaporation can be derived from the boiling point date of a highly concentrated syrup at various pressures.

### **Commercial practices**

In a typical stand alone VHP sugar refinery, the sucrose goes to molasses averaged around 2% which is sold at a price much lower than that of recovered sucrose.

Based on the material balance, the syrup purities after #4 and #5 boiling would range from 87.83 to 92.68 respectively. The present Molassugar process would convert and recover all the sucrose in the mix of #4 and #5 syrup into value added products. The ratio of the mix would depend on the value added products desired/preferred. There would be NO molasses produced.

For a refinery with annual production of 200,000 tons sugar the additional revenue would be:  $200,000 \times 2\% \times \$750/\text{ton} = \$3 \text{ millions USD}$  using the price of \$750/ton for the value added products

## Refinery Sucrose Yield

					Products 97%
Conventional refinery	Sucrose in 100%	Refinery			Molasses 2%
					Sucrose loss 1%

## Refinery Sucrose Yield

Convert molasses into value added products

					Products 99%
Improved refinery	Sucrose in 100%	Refinery			Sucrose loss 1%

## Material balance of a VHP refinery 1

750 ICU Raw Sugar		Material and color Balance			
100 lbs 99.5 purity		O C M refinery 4-Apr-10			
Phosphatation	100 lbs 99.5 purity 415 ICU				
Filter press with carbon	100 lbs 99.5 purity 200 ICU				
Evaporator optional	100 lbs 99.5 purity 200 ICU				
White Boiling #1	100.00 lbs 99.50 purity				
Centrifuge #1	54% → 46.00 lbs 98.94 purity	54.00 lbs 99.92 purity	54.62%	% of total solids	
White Boiling #2	455 ICU				
Centrifuge #2	52% → 46.00 lbs 98.94 purity	23.92 lbs 99.90 purity	24.19%	% of total solids	
White Boiling #3	995 ICU				
Centrifuge #3	50% → 22.00 lbs 97.84 purity	11.04 lbs 99.88 purity	11.17%	% of total solids	
White Boiling #4	2100 ICU				

## Material balance of a VHP refinery 2

11.04 lbs 95.91 purity		99.5 ICU		Material and color balance continued	
White Boiling #4	2100 ICU				
11.04 lbs 95.91 purity		48%		5.30 lbs 99.86 purity	#DIV/0! % of total solids
Centrifuge #4				262.5 ICU	
5.74 lbs 92.68 purity					
White Boiling #5	4250 ICU				
5.74 lbs 92.68 purity		46%		2.64 lbs 99.84 purity	#DIV/0! % of total solids
Centrifuge #5				640 ICU	
3.10 lbs 87.83 purity					
White Boiling #6					
3.10 lbs 87.83 purity		42%		1.30 lbs 99.81 purity	#DIV/0! % of total solids
Centrifuge #6					
1.80 lbs 81.39 purity					
White Boiling #7					
1.80 lbs 81.39 purity		32%		0.67 lbs 99.78 purity	#DIV/0! % of total solids
Centrifuge #7					
1.13 lbs 73.60 purity					
Molasses					
1.13 lbs 73.60 purity					

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## Conversion of molasses into value added products--Molassugar

- 1) Conventional boiling to extract sucrose from molasses is not economical due to high viscosity
- 2) The whole massecuite of molasses transformed into Molassugar via spontaneous crystallization
- 3) The molassugar consists of aggregate of microcrystalline, Flavor ingredients can be added for profit enhancement
- 4) No molasses is produce in the refinery

## Major problem to Convert/transform molasses into "Molassugar"-value added products

Require cultural change

Resistant from sale group to sell the product

Need visionary management to make it happen

## Examples of benefits of cultural change

**We have converted three sulfitation plants to produce refined raw sugar with practically no capital cost and with reduced operating cost & improved environment. Results:**

**Replacing sulfitated white sugar (color: 150 to 200 ICU) with New refined raw sugar color: 1500 to 2000 ICU**

No problem to sell the new products--refined raw sugar

### PROCESS DESCRIPTION

The essence of the process has been described in patents assigned to Amstar Corporation by the inventors. A patent issued July 13, 1965, to Tippens and Cohen describes the process for making fondant-size crystals of sucrose agglomerates with the flavor of brown sugar. The Miller-Cohen patent, issued January 23, 1968 provides further details of the transformation process.

## Operating requirements for Molasses conversion

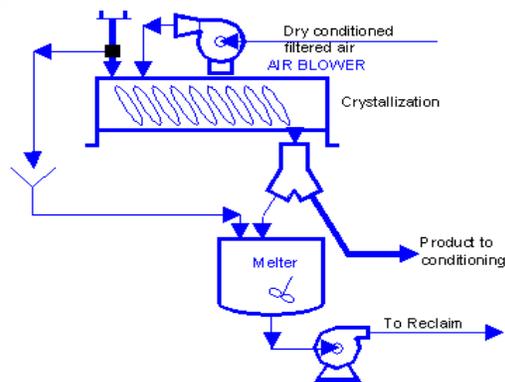
- 1) Massecuite purity minimum: 88
- 2) % invert maximum in massecuite: 7%
- 3) Massecuite temperature over 245 °F for high heat of crystallization
- 4) Massecuite brix minimum 94 brix
- 5) Agitation to create surface area for evaporation
- 6) Evaporated vapor must be carried away via vacuum or air

## Four industrial processes for conversion of molasses

- 1) Micro crystallization—continuous process
- 2) Areado process
- 3) Muscavado process
- 4) Spray drying process

### 1) Micro crystallization process

#### 1) Micro crystallization—continuous process



The evaporator is operated at a temperature in the range 120 – 130 °C with or without vacuum, to produce concentrated syrup having a solids content of 91 to 97% by weight. Concentrated syrup flows from the evaporator to the vapor-liquid separator where the vapor is removed by a barometric condenser and the syrup is fed to the nucleator/crystallizer.

The retention time of sugar in this unit ranges from 10 seconds to 120 seconds depending on the products desired.

Air is introduced into the nucleator/crystallizer to remove vapor released by the crystallization of material therein due to the heat of crystallization. The air flow rate ranges from 9 to 40 cubic feet per minute per pound of sugar product per minute. If desired, a dual temperature air system can be used during the nucleation and/or crystallization phases to further maximize the process flexibility and efficiency.

The sugar product in aggregate form, leaving the nucleator/crystallizer with a moisture content ranged from 0.5% to 2.5%, is fed to the dryer for further drying if needed. The resulting dried aggregates are then supplied to a cooler where they are reduced in temperature to about room temperature. The sugar aggregates are then milled and screened to meet product screen specifications.

In general, the physical characteristics and functionality of desired transformed products not only determine the selection of operating conditions, but also affect the design and therefore, the capital and operating costs of the process.



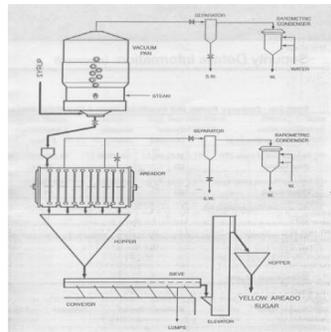


## 2) Areado process

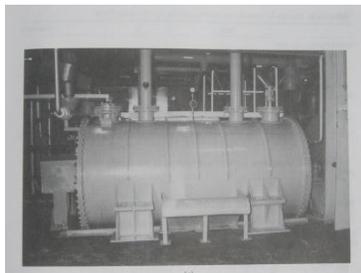
In this system a mixture of liquor and syrup with purity of 98.5 to 99.5 and brix of about 68 to 72 was boiled in a standard vacuum pan and concentrated to a brix around 92 of massecuite. Before discharging into a vacuum mixers called Areadoes for transformation, the massecuite is heated to 105 to 110 degree centigrade to increase the heat of crystallization (for example: The heat of sucrose crystallization is 57 J/g at 60 °C and 107 J/g at 90 °C) which is needed for evaporation of water to induce spontaneous crystallization and produce Areadoes brown/yellow sugar. The vacuum mixer is also fitted with condenser to remove the water evaporated.

The Areado sugar is then dried to a moisture of 2.8 to 3.4 % for sale

## Areado process



## Areado of RAR refinery



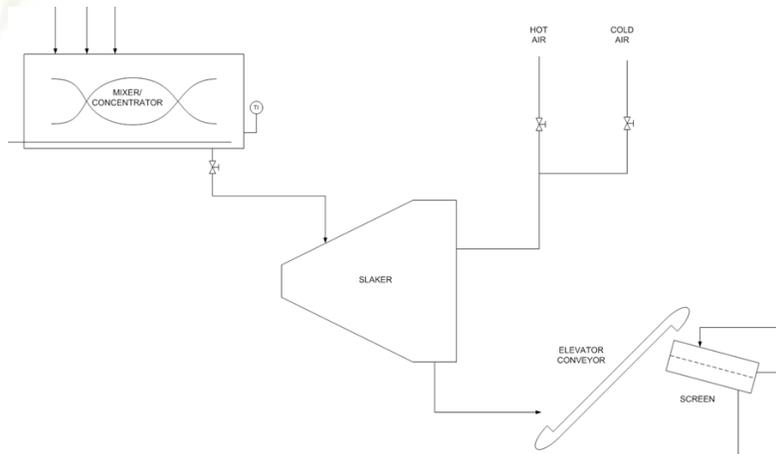
## 3) Muscavado process

Muscavado sugar is produced using a mixture of various process streams at a brix between 78 to 80 and purity of 89 to 92. The mixture is sent to a cooker to heat up and concentrate to about 92 to 94 brix and then drop in to a rotating slaker for mixing with no air addition. When the sugar is transformed, first hot air at 60-65°C is then introduced into the slaker followed by cold air.

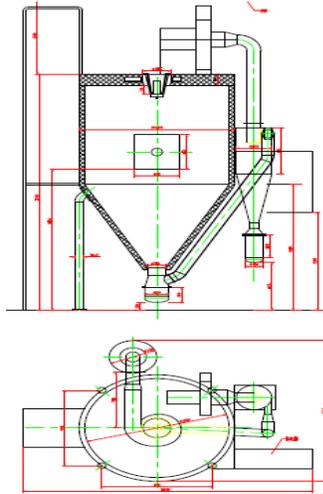
The muscavado sugar is formed while tumbling inside the slaker. This is then dropped by rotating the slaker Counter Clockwise to empty the slaker. The Muscavado sugar will drop into a conveyor which will feed a shaker screen. The balls of sugar is separated from the Muscavado sugar and sent into a crusher where the balls are crushed and the sugar is returned to the screen.

The schematic system diagram is shown below to depict the process flow.

# Muscavado process



## Spray drying process



## Spray drier



### PRODUCT CHARACTERISTICS

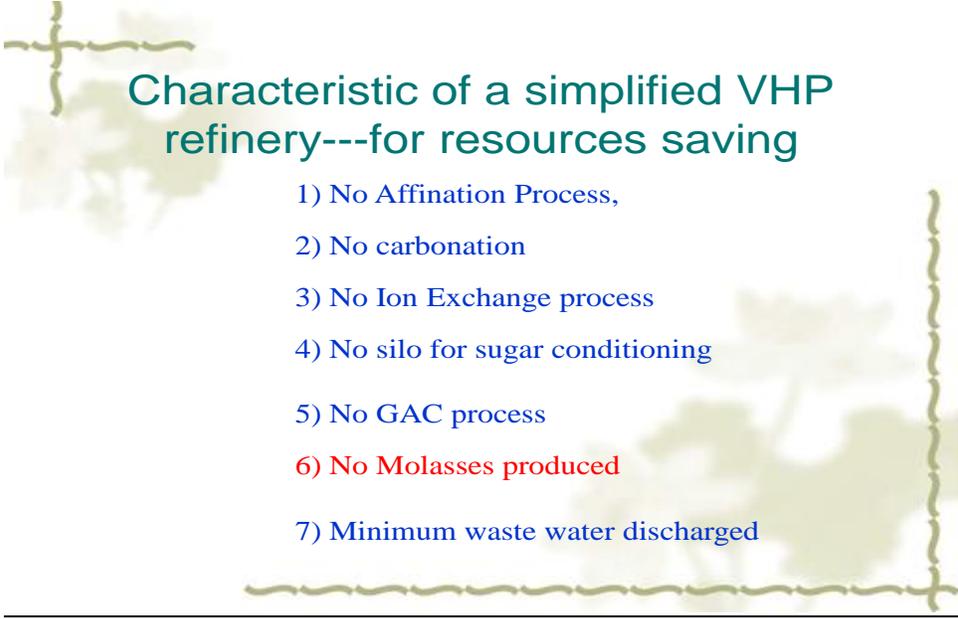
These products, in the form of aggregates, with crystals size ranging from 20 to 100 micron as formed in the processes are bound to one another partly by the moisture remaining in the product and partly by non-sucrose.

## Characteristics of Molassugars

<u>Characteristics</u>	<u>Areador</u>	<u>Muscovado Sugar</u>	<u>Transformed sugar Microcrystallization</u>
Purity	96.5 to 97.5	90 to 92	94 to 96
% moisture	2.5%	1.2 to 1.5	<0.9
Color	3000	8,000 to 10,000	1,500 to 2,000

## Pro & Con of Molassugar process

<u>Characteristics</u>	<u>Areador</u>	<u>Muscovado Sugar</u>	<u>Microcrystallization</u>
Pro	high yield per batch	1) high yield over all 2) purity just right	Semi continuous Semi automated
Con	1) high capital cost, 2) low capacity- utilization 3) purity too high	1) low capacity/unit	1) High in both capital/ operating cost 2) low yield 3) purity too high



## Characteristic of a simplified VHP refinery---for resources saving

- 1) No Affination Process,
  - 2) No carbonation
  - 3) No Ion Exchange process
  - 4) No silo for sugar conditioning
  - 5) No GAC process
  - 6) No Molasses produced
  - 7) Minimum waste water discharged
- 

### **Conclusion**

In this presentation, we have demonstrated three commercially viable processes for transformation of various sugar process streams, including but not limited to, “final molasses”, directly into solid sugar and sugar-containing value added products which have gained wide acceptance by the trade.