

SOME NOTES ON THE NEW MELT CLARIFICATION AT HULETT'S S.A. REFINERIES

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Previous to March 1959, Hulett's S.A. Refineries employed various modifications of the phospho-defecation process coupled with paper-pulp and occasionally kieselguhr filtration, followed by single charring of liquors. In March 1959, flue gas carbonatation alar Tate and Lyle was introduced. It is difficult to make an accurate comparison of the new and old processes as many of the properties now used to measure clarification efficiency were not in vogue prior to July 1959. However, there is no doubt that both the clarity of the liquors and the colour of the refined sugar produced by the carbonatation process are vastly superior to those produced immediately prior to its introduction.

Briefly, the clarification process as practised at present is as follows:

After being weighed by a Servo-Balans the raws to be melted are cold mingled with 85-92 purity affination syrup in a scroll agitator giving a retention time of about 10 minutes at 50 t.p.h. melt. The mingled sugar is spun on 10 E.D. 48×30-inch Watson Laidlaw machines with maximum speed of 960 r.p.m. The washed sugar is melted with Johnson sweetwater to an automatically controlled brix. The liquor is again brixed by a polishing brixer before being fed to carbonatation.

Carbonatation is conducted in 3 saturators in series, giving a retention of some 90 minutes with a melt rate of 50 t.p.h. All the milk of lime (Density 1.085) is added to "A" saturator and control of lime and liquor is effected by means of variable speed lime and liquor wheels. Lime per cent solids has varied between 0.75 and 1.5 during the season and has averaged 1.21.

Gassing-out is achieved with scrubbed flue gas. The flue gas runs at a steady 12 per cent CO₂ concentration leaving the boilers but is reduced to 10 per cent after having passed the scrubbing units which consist of a Musgrave aerodynamic dust collector followed by a water scrubbing tower and finally a soda tower. The main object of the gas-scrubbing is to remove corrosive sulphur dioxide. About 80 per cent gassing out is achieved in "A" saturator corresponding to a pH of approximately 10.5. In "B" saturator gassing-out by a further 15 per cent causes the pH to fall to around about 9.6. The gassing-out in "C" saturator finally reduces the pH to 9.

The filter-station consists of 9 Auto filters (1,260 sq. ft each) with 4 Johnson plate and frame presses

(1,500 sq. ft. each) for sweetening-off. The clear filtered liquor (63° Brix) is passed over bone char twice. It first comes into contact with char which has already been used for partly decolourising liquor before coming into contact with freshly burnt char.

As in most sugar refineries the efficiency of the clarification process at H.U.L.S.A.R. is measured indirectly by the removal or non-removal of the impurities rather than gain in purity as such. The fates of the following classes of impurity are used as a yardstick to gauge the thoroughness of clarification:

- (a) All types of coloured bodies.
- (b) Suspended and colloidal impurities.
- (c) Both organic and inorganic anions.
- (d) Cations.
- (e) Reducing sugars.

The concentration of coloured bodies in sugar liquors is measured by the attenuation index at 420 millimicrons using a Beckman model DU. spectrophotometer. In the case of turbid liquors such as carbonatation supply liquor, a double filtration over kieselguhr is made prior to colour measurement.

The concentration of suspended and colloidal impurities is still judged visually in the plant by examination of liquors for brilliance. Although A*c 720 readings are made on many liquors their interpretation as turbidity is often suspect and thus no A*c 720 figures are shown in Table I.

It has been customary to regard either gravimetric or conductimetric ash as giving a suitable measure of the total inorganic matter in liquors. Although conductimetric ash is still regarded as a very rapid and desirable guide as to what is happening in respect of ionised matter, the use of the determination of "total cations" would appear to have many advantages especially where a higher degree of accuracy is required. It is less time consuming and requires less skill than a gravimetric ash determination and by expressing the results in terms of micro-equivalents a very simple balance of ionic constituents results. The determination is carried out in a manner similar to that described by Domingues.¹ If the sulphate and chloride determinations are made on the same liquor an approximate measure of the organic acids may be obtained by difference, since other anions do not normally occur in refinery liquors to any extent. Ca+Mg and sulphate have been determined by titrations with

E.D.T.A. solution and chloride has been determined by conductimetric titration with silver nitrate.

All reducing sugar determinations have been made using the Lane and Eynon method as attempts to introduce the more rapid T.T.C. method have not yet produced the degree of reproducibility desired.

Table I shows the average monthly analysis of materials through clarification over seven months. A general increase in reducing sugars is noticeable from November when the refinery started drawing on stored sugars. Table II shows the reducing sugar "destroyed" by carbonatation and the percentage colour and Ca+Mg removed by carbonatation. The percentage of colour and Ca+Mg (based on filtered liquor) remaining after double charring are also shown.

Although only seven sets of figures are available the possibility of an inverse relationship between reducing sugars destroyed and percentage colour removed and percentage Ca+Mg removed appears not unlikely.

The drop in efficiency of char in removing both Ca+Mg and colour can probably be attributed to the fact that a relatively large amount of new char was added to the stock in the July period. Apparently regeneration of char as practised at H.U.L.S.A.R. is unable to restore char to the level of its original efficiency.

In an attempt to measure the amounts of organic acids throughout the clarification process composite daily samples were analysed over the period 11th-16th February. Both the washed raw sugar and the carbonatation supply liquor were filtered over kieselguhr at 50° Brix before analysis to prevent the interference of insoluble salts which would be removed in practise by filtration in any case.

The decrease in total cations shown in Table III is due almost entirely to removal of calcium and magnesium as can be seen by the almost constant level held by cations other than Ca+Mg. The sulphate ion is rather efficiently recovered by carbonatation and is still further reduced by char. Even

the initial laboratory kieselguhr filtration of the carbonatation supply liquor at 50° Brix was able to remove considerable sulphate. The increase in chloride content from washed raw sugar to carbonatation supply liquor is undoubtedly due to the water and Johnson sweet water used for melting.

The increase in organic acids from washed raw sugar to carbonatation supply is also apparently due to organic acids in the Johnson sweetwater. The increase across carbonatation and first charring is probably due to the destruction of reducing sugars already mentioned. Organic acids appear to be the main anion after carbonatation and even after charring the organic acid content is still nearly double that of the original washed raw sugar. This increase in organic acids not only represents a loss of sugars but is also no doubt responsible for the less efficient removal of impurities over char. The mechanism of formation of organic acids during carbonatation and first charring is not known. However, previous laboratory tests carried out by the author at the Sugar Milling Research Institute were able to demonstrate that manganese acts as a catalyst in the oxidation of sugars to acids, by atmospheric oxygen. Even calcium saccharate solutions containing small quantities of manganese ions produced considerable quantities of organic calcium precipitates when oxygen was passed through the solution at 40°C. Since the lime used for carbonatation contains considerable amounts of manganese (0.5-2 per cent) it seems not unlikely that one of the sources of organic acids may be the oxidation of sugars (sucrose included) by the oxygen contained in flue-gases.

Acknowledgement

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Reference

¹ L. P. Domingues: Determination of Total Cations in Commercial Sugars. Proc. of the Fourth Session of Bone Char, 1955.

TABLE I

AVERAGE MONTHLY ANALYSIS OF MATERIALS THROUGH CLARIFICATION

	<i>Raw Melt</i>	<i>Affinated Sugar</i>	<i>Carbonation Supply</i>	<i>Filtered Liquor</i>	<i>Single Charred Liquor</i>	<i>Double Charred Liquor</i>	<i>Refined Sugar</i>
AUGUST 1959							
Reducing Sugars per cent on Solids...	0.26	0.11	.09	0.06	.06	.06	0.002
Ash ...	0.43	0.25	0.27	0.18	0.13	0.12	0.007
Ca+Mg (Micro equiv./g solids) ...	—	—	17.6	9.7	2.2	0.6	0.09
A*c 420...	—	—	2420	1380	61.7	27.8	17
pH ...	—	—	7.8	8.9	8.2	8.6	—
SEPTEMBER 1959							
Reducing Sugars per cent on Solids...	0.24	0.10	0.10	0.07	0.06	0.06	0.001
Ash per cent on Solids ...	0.42	0.23	0.27	0.19	0.15	0.14	0.011
Ca+Mg (Micro equiv./g solids) ...	—	—	17.8	10.0	3.5	1.6	0.18
A*c 420...	—	—	2368	1371	65.4	30.0	21.2
pH ...	—	—	8.0	9.1	8.2	8.4	—
OCTOBER 1959							
Reducing Sugar per cent on Solids ...	0.28	0.11	0.09	0.07	0.06	0.06	0.09
Ash per cent on Solids ...	0.46	0.25	0.25	0.20	0.15	0.13	0.10
Ca+Mg (Micro equiv./g Solids) ...	—	—	21.3	11.2	5.1	2.1	0.19
A*c 420...	—	—	3089	1465	81.5	39.2	22.3
pH ...	—	—	8.5	9.0	8.1	8.3	—
NOVEMBER 1959							
Reducing Sugars per cent on Solids...	0.39	0.16	0.095	0.08	0.06	0.06	0.002
Ash per cent on Solids ...	0.44	0.23	0.28	0.24	0.21	0.18	0.009
Ca+Mg (Micro Equiv./g Solids) ...	—	—	26.0	16.2	10.8	6.2	0.41
A*c 420...	—	—	3433	1672	122.7	62.3	23
pH ...	—	—	8.4	9.0	7.8	8.0	—
DECEMBER 1959							
Reducing Sugars per cent on Solids...	0.32	0.13	0.16	0.12	0.10	0.08	—
Ash per cent on Solids ...	0.36	0.19	0.26	0.20	0.16	0.15	0.008
Ca+Mg (Micro Equiv./g solids) ...	—	—	22.3	15.4	9.3	5.6	0.29
A*c 420...	—	—	2642	1545	114	51.2	21.0
pH ...	—	—	7.9	8.9	7.9	7.9	—
JANUARY 1960							
Reducing Sugars per cent on Solids...	0.42	0.17	0.23	0.18	0.165	0.12	0.001
Ash per cent on Solids ...	0.39	0.215	0.28	0.22	0.19	0.13	0.009
Ca+Mg (Micro equiv./g solids) ...	—	16.2	21.4	15.5	11.8	7.6	0.60
A*c 420...	—	—	2428	1622	152	71.5	28
pH ...	—	—	7.2	8.8	7.6	7.8	—
FEBRUARY 1960							
Reducing Sugars per cent on Solids...	0.54	0.14	0.18	0.13	0.12	0.09	.002
Ash per cent on Solids ...	0.42	0.185	0.24	0.18	0.15	0.135	.008
Ca+Mg (Micro equiv./g solids) ...	—	22.5	18.4	11.8	7.2	4.2	0.32
A*c 420...	—	—	2025	1308	112	55	25
pH ...	—	—	7.0	8.9	7.8	8.1	—

TABLE II

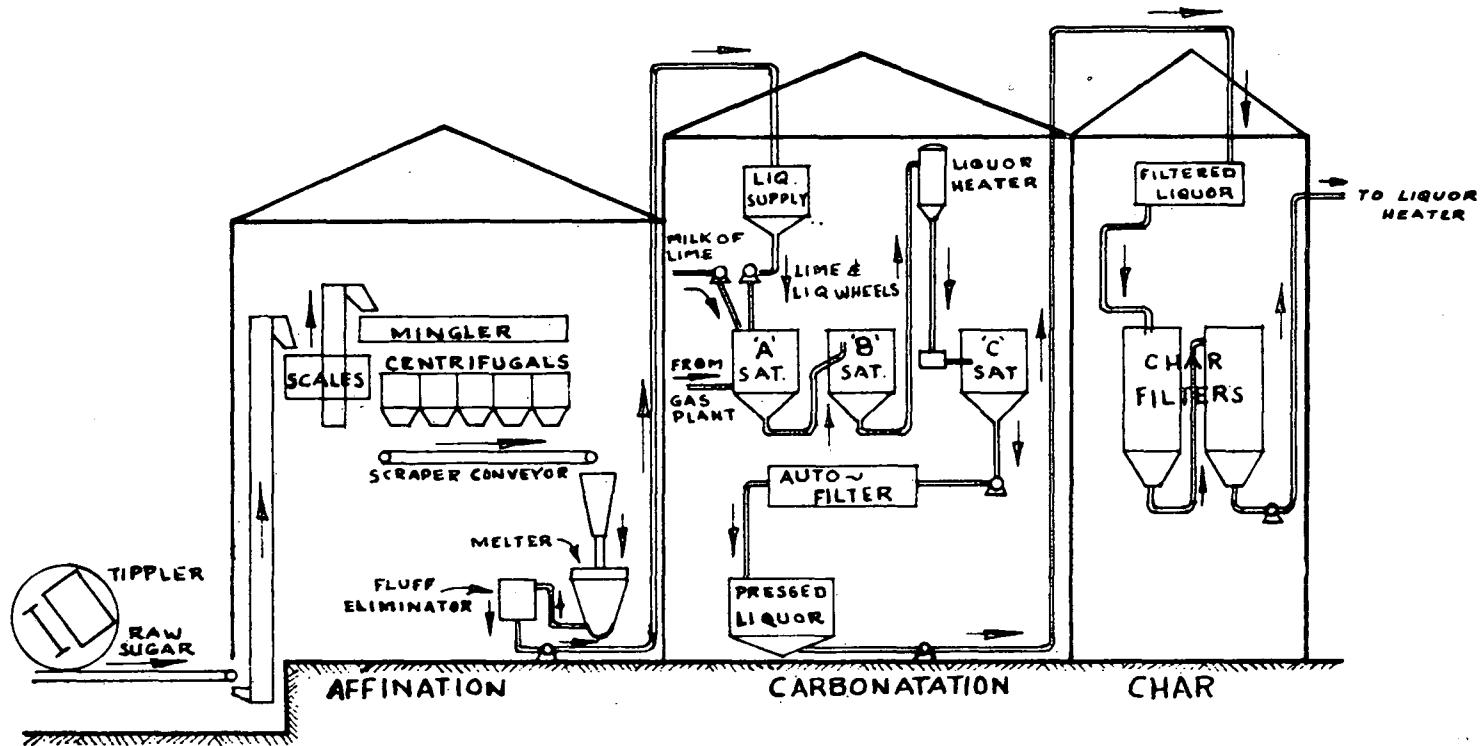
<i>MONTH</i>	<i>Reducing Sugar destroyed by Carbonatation per cent Solids</i>	<i>Per cent Colour removed by Carbonatation</i>	<i>Per cent Ca+Mg removed by Carbonatation</i>	<i>Ca+Mg remaining after Double Charring per cent on Filtered Liquor</i>	<i>Colour remaining after Double Charring per cent on Filtered Liquor</i>
August	0.03	43	45	6	2.0
September	0.03	43	44	16	2.2
October	0.02	53	47	19	2.7
November	0.015	51	38	38	3.7
December	0.04	41.5	31	36	3.3
January	0.05	33	28	49	4.4
February	0.05	35	36	37	4.2

TABLE III

**CONCENTRATION OF SOME IMPURITIES IN MICRO-EQUIVALENTS
PER GRAM SOLIDS THROUGH CLARIFICATION IN PERIOD
11th-16th FEBRUARY 1960**

	<i>Washed Raw Sugar</i>	<i>Liquor to Carbonatation</i>	<i>Liquor from Carbonatation</i>	<i>Single Charred Liquor</i>	<i>Double Charred Liquor</i>	<i>Johnson Sweetwater</i>
Total Cations	25.05	23.8	19.85	18.8	15.9	99.6
Ca+Mg	15.65	15.4	11.55	8.1	5.0	81.5
Cations other than Ca+Mg ...	9.4	8.4	8.3	10.7	10.9	18.1
Sulphate	15.4	12.0	3.9	1.5	2.1	32
Chloride	2.8	4.1	4.1	4.1	4.5	11.7
Phosphate... ..	1.4	1.0	—	—	—	—
Organic acids by Difference ...	5.5	6.7	11.9	13.2	9.3	55.9

For discussion on this paper see page 125



III

— CLARIFICATION PROCESS FLOW DIAGRAM —