AN ATTEMPT TO INVESTIGATE CHANGES OF IMPURITY CONCENTRATION WITH THE DEPTH OF CRYSTAL MATRIX OF VHP SUGAR

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Abstract

Graded VHP sugar crystals were treated under controlled conditions, so as to progressively dissolve successive crystal layers. At each stage, a portion of the treated crystals was measured and analysed. The tabulated analyses indicate that to a large extent, the dissolution took place in a regular manner, that the impurities are mostly present in the outermost portion of the crystals and also that there is a minor but progressive quality improvement in respect of pol and colour, as the "peeling" action progresses.

Introduction

It is an established fact that VHP sugar contains various impurities such as colour forming substances, mineral salts and polysaccharides, within the crystal matrix.

This may be due to co-crystallisation with, and inclusion of various impurities present in the mother liquor during the boiling cycle.[1] It is reasonable to assume that in spite of the fact that crystallisation is one of the most effective purification procedures known, changes in impurity concentrations during the boiling of the liquor (i.e. their progressive increase) may produce change of concentration in the co-crystallised and included material, as the VHP sugar crystals are built up.[2,3] Thus a stepwise removal of the crystal material from the outside and analysis of the remaining solid, may show whether in fact a gradient in impurity concentration exists.

In order to test the above contention it was decided to dissolve the outer crystal layers in 5% linear-size fractions and to analyse portion of the remaining material, the latter serving as a starting substance for a fresh step. Five such dissolution cycles were planned for Test 1 and seven cycles for Test 2.

To increase the "detectability" of possible change, a dark coloured (with presumably high impurity content) VHP sugar was selected and sieved to isolate sugar crystals sized between 1,7 and 1,2 mm. It was expected that the uniformity of crystals would assist in the regular dissolution and removal of crystal layers.

The following steps were planned to implement this investigation:-

1. Collection and sieving of selected grain size fraction.
2. Mixing of the above to ensure uniformity.
3. Methanol washing to remove impurities from the surface of the crystals.
4. Affination at 20°C prior to the commencement of crystal layer removal.
5. Measurement of average crystal size, calculation of the concentration of the undersaturated sugar liquor to remove the required volume of crystal.
6. Dissolution of superficial VHP sugar crystal layers and analysis of the diminished crystals.

Experimental Procedure

Collection and Sieving

A sample of 25kg of dark coloured VHP sugar was collected from the storage silo and sieved using a 1,7mm and a 1,18mm mesh sieve to remove all the larger and finer crystals. All the retained sugar was well mixed and stored in an airtight container.

Methanol Wash and Affination

The graded sugar was then washed using methanol, in the same manner as for grain size analysis. The required analyses were carried out, after which the sugar was affinated in the usual manner and re-analysed. All analyses were done in duplicate.

Dissolution of the Calculated Portion of Outer Crystals

It was considered that if the VHP sugar crystals of similar size were exposed to the action of a slightly undersaturated refined sugar solution, i.e. conditions similar to affination, the resultant dissolution would remove the quantity of solute equal to the extent of undersaturation at a given temperature. Further, since the volume/area ratio of all crystals was similar, it was reasonable to assume that the dissolution would be uniform and would dissolve away a similar thickness of sugar from each crystal.

Crystal Size Measurements prior to the "peeling" steps

Tests were carried out to devise a reliable method of average crystal size measurement, to provide a basis for the necessary volume and weight calculations. As the crystals were already screened and a selected fraction retained, it was necessary to sample, examine and measure a given number of crystals to provide a reliable guide to the average crystal size. To calculate the volume, the length, width and height of the crystal must be known. When a sugar crystal is examined under a microscope, the length and width can be readily measured over a calibrated graticule but the height can only be obtained by rotating the crystal through a 90° angle. To establish a database for the minimum number of measurements necessary to give average representative size of crystals, the following tests were carried out:- separately sampled batches of crystals were measured with the following averaged results:-

No. of Crystals	Length (mm)	Width (mm)	Height (mm)
50	1,77	1,44	0,95
30	1,76	1,37	0,96
20	1,77	1,45	0,94

From the above results it was decided that averaged measurements of 50 crystal samples should provide the necessary information with the required reliability.

Calculation for crystal size reduction by dissolution of 5% linear size

Using the above described techniques, the following average measurements were obtained for sugar crystals which were previously alcohol-washed and affinated i.e. ready for the first "peel" (Test 2).

Length	1,71mm
Width	1,38mm
Height	0,92mm

It is required that 5% (by size) of the outer crystal be dissolved.

If the original volume = 1,71 × 1,38 × 0,93 = 2,1946mm³ then to convert to weight, multiply by SG of sucrose 2,1946 × 1.587 = 3.4828mg . . .(1)

Reducing crystal sizes by 5%:1,6245 × 1,311 × 0,8835 = 1,8816mm³

similarly 1,8816 × 1.587 = 2.9861mg . .(2)

therefore (1)−(2) = 0.4967mg

= 14.26%

It is therefore necessary to remove 14,26% by weight, from a batch of VHP sugar, by washing it with an undersaturated sugar solution, and if 1 kg batches of VHP sugar are used, 142,6 weight of solid must be removed. The solubility of sugar = 199,4g/100g at 20°C.[4]

Therefore, to "peel" 1 kg of VHP crystals to the required size, 71,4g of water at 20°C were added to the saturated sugar solution. Both the sugar and undersaturated sugar solution were conditiond to 20°C prior to washing. The latter was carried out by slow tumbling on the rotary mixer for 30 min. Subsequently the solids were separated by centrifuging and air drying.

The sizes of crystals were measured after each dissolution and the necessary calculations done to establish what concentration of the sugar solution was required for the following "peel" (Table 1).

TABLE 1

Average crystal measurements after each dissolution

Dissolutions	Crystal measurements (mm)		
	length	width	height
1	1,71	1,38	0,92
2	1,66	1,35	0,88
3	1,67	1,31	0,84
4	1,66	1,27	0,80
5	1,56	1,24	0,82
6	1,53	1,18	0,79

Two separate tests were carried out on separately selected sugars. The results obtained had a similar pattern and are shown in Table 2 and Table 3.

TABLE 2

Analytical results of Test 1

Sample	POL	R S %	Dextran ppm	Icumsa colour		Starch ppm	Cond. Ash %	Gums ppm	P_2O_5 ppm		Grain size analysis					Sieve Analysis					
																Retained on sieve %					
			Haze method	420 nm	560 nm				Tot.	Inor	SGS	on 1000 microns %	thru' 600 microns %	Coeff. of Variance	Mean Aperture	1,70 mm	1,18 mm	1000 microns	600 microns	355 microns	PAN
Original	99,16	0,18	28	2611	0,48	156	0,19	1585	147	111	–	–	–	–	–	–	–	–	–	–	–
MeOH washed	99,49	0,09	30	2125	0,40	156	0,14	1511	127	102	1,329	97,55	0,47	27,2	1,72	0,94	86,98	9,64	1,98	0,12	0,35
Affinated	99,48	0,10	24	1833	0,34	155	0,13	1432	131	103	1,181	84,85	2,17	29,4	1,41	1,48	69,89	13,48	12,99	2,02	0,15
1st peel	99,52	0,09	20	1613	0,27	131	0,12	1295	118	97	1,080	76,30	3,56	28,7	1,23	0,56	55,57	20,17	20,15	3,45	0,11
2nd peel	99,57	0,09	16	1548	0,26	129	0,10	1178	114	88	1,044	73,74	4,05	28,8	1,18	0,84	49,45	23,95	22,22	3,70	0,36
3rd peel	99,55	0,08	19	1438	0,27	123	0,10	1148	107	83	0,962	63,08	5,18	27,6	1,08	0,18	35,30	27,60	31,74	4,90	0,28
4th peel	99,58	0,08	14	1416	0,25	129	0,09	1170	101	88	0,911	55,37	6,27	27,0	1,01	0,21	26,40	28,77	38,37	5,96	0,31
5th peel	99,60	0,08	10	1410	0,25	124	0,08	1128	98	85	0,798	42,36	11,33	27,4	0,891	0,04	11,45	30,87	46,31	9,53	1,80

TABLE 3

Analytical results of Test 2

Sample	POL	R S %	Dextran ppm	Icumsa colour		Starch ppm	Cond. Ash %	Gums ppm	P_2O_5 ppm		Grain size analysis					Sieve Analysis					
																Retained on sieve %					
			Haze method	420 nm	560 nm				Tot.	Inor	SGS	on 1000 microns %	thru' 600 microns %	Coeff. of Variance	Mean Aperture	1,70 mm	1,18 mm	1000 microns	600 microns	355 microns	PAN
Original	99,14	0,15	42	2211	0,40	155	0,17	1419	100	78	1,40	99,91	0,02	27,8	2,284	1,48	94,81	3,62	0,08	0,02	0,01
MeOH washed	99,43	0,10	44	1846	0,35	151	0,15	1331	97	78	1,37	99,59	0,12	25,6	1,768	0,63	90,32	9,00	0,30	0,02	0,10
Affinated (normal)	99,39	0,10	36	1651	0,31	130	0,14	1279	92	74	1,30	99,48	1,01	29,05	1,674	0,92	83,91	10,15	4,02	0,88	0,13
1st peel	99,38	0,10	19	1570	0,30	127	0,15	1333	93	65	1,21	91,99	0,50	22,0	1,262	0,17	61,85	29,98	7,52	0,40	0,10
2nd peel	99,36	0,08	18	1556	0,29	126	0,13	1211	92	65	1,18	89,49	0,81	21,7	1,181	0,18	49,96	39,36	9,71	0,70	0,10
3rd peel	99,40	0,09	17	1444	0,27	124	0,13	1144	80	61	1,07	80,98	1,56	21,8	1,093	0,16	35,14	45,69	17,47	1,34	0,22
4th peel	99,38	0,08	19	1380	0,26	125	0,12	1157	80	62	0,99	68,77	2,05	21,1	1,030	0,08	24,00	44,69	29,18	1,78	0,28
5th peel	99,37	0,08	18	1392	0,27	126	0,12	1091	82	64	0,93	57,73	2,22	20,3	0,992	0,06	17,05	40,63	40,05	1,90	0,32
6th peel	99,39	0,08	17	1386	0,26	128	0,13	1069	81	65	0,89	46,16	2,17	19,2	0,961	0,03	11,36	34,77	51,67	1,97	0,20
7th peel	99,41	0,07	18	1316	0,25	125	0,12	1050	81	65	0,88	42,67	1,86	18,6	0,958	0,15	10,09	32,43	55,46	1,73	0,14

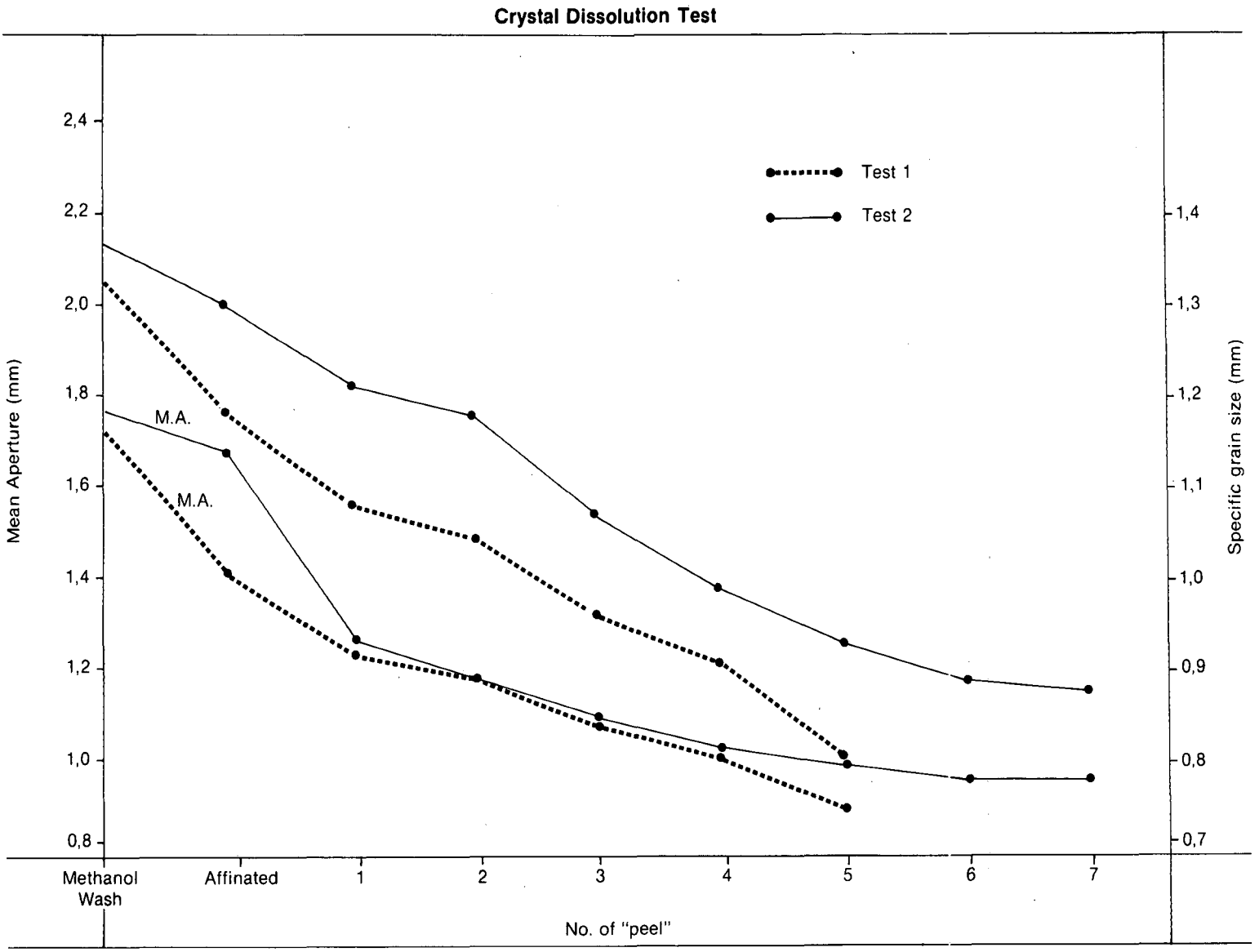

FIGURE 1 Specific grain-size plotted against tests carried out.

Results

(1) Figure 1 shows a fairly uniform change of specific grain size with the progressive crystal reduction

(2) From the retained on 1000μ and passed through 600μ results, size reduction was regular between 1,7 and 0,6mm with small increments to the last stage.

(3) Interesting facts were that:-
- the methanol wash removed a major portion of impurities,
- the affinating process had very little effect compared with the methanol wash.
- small but consistent increase in pol was noted toward the centre of the crystal and this may reflect the relatively low impurity/sucrose ratio in the early stages of each boiling.

(4) Reducing sugars are removed to almost a constant level with the methanol wash.

(5) Colour is sharply reduced by affination and decreases at a slower rate as the centre of crystal is approached.

(6) The above pattern is repeated in the results of the gum tests.

(7) The starch content is not affected after the first "peel".

(8) Ash, and P_2O_5 concentrations decrease steadily towards the inner structure of the crystal.

Summary and Conclusions

The alcohol wash and/or affination appear to have had a significant purifying effect on the VHP sugar crystals used. The pol and colour are most prominently changed.

As the above step is possibly comparable to the steam/water rinse during centrifugal separation of crystals from the mother liquor at the manufacturing mill, the results given may provide some guide to the quality change during this stage.

Acknowledgments

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