

SOME FACTORS AFFECTING THE MEASUREMENT OF COLOUR OF WHITE SUGAR

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Abstract

A selection of refined sugars from all South African refineries was analysed for pH, SO₂, attenuation indices at 420 nm and 720 nm, colour index and ICUMSA colour, and the relationships between these were analysed statistically. From these results it was established that sugar solutions vary in pH from refinery to refinery, that the pH has a marked effect on the colour measurements and that if measurements are made at pH 7 a good correlation between colour indices and ICUMSA colour is found.

Introduction

An important step in the refining of sugar is the removal of colour, and the consumer has come to associate this absence of colour with the purity of the product. Until 1970 there were several ICUMSA methods for determining colour on both raw and refined sugars in which an adjustment of pH to 7,0 ± 0,2 was made on raw sugars but none on white. Since 1978, however, ICUMSA Method 4 — one of the two remaining recommended methods — includes the adjustment of pH to 7,0. The necessity of standardising the pH at which colour measurements are made was stated by Carpenter and Deitz³ who showed that colourants of sugar are highly sensitive to variations in the region of pH 7 where a variation of 0,1 pH results in a 5% error (See Figure 1).

Equipment and Apparatus Used

A Beckman Expandomatic pH meter with combination electrodes and fitted with a magnetic stirrer was used for all pH determinations. A Zeiss PM 4 Spectrophotometer and 50 mm cuvettes were used in the measurement of optical density at 420 nm and 720 nm for colour, while 10 mm cuvettes on the same instrument at 560 nm were used for SO₂ determinations by the rosaniline method⁴. Brix measurements were made on an Itago bench refractometer.

Solutions for the determination of ICUMSA colour⁷ were filtered through Schleicher and Schull membrane filters with a pore size of 0,45 μm. All other apparatus was standard laboratory glassware.

Procedure

Thirty-one samples of refined sugar, consisting mainly of weekly composites taken during the month of November 1979 were received from refineries throughout the Republic.

These were analysed for SO₂ by the rosaniline method⁴ and then for attenuation at 420 nm⁵ and 720 nm⁶. At the same time the pH of the solutions was measured and, finally, the colour index⁶ was calculated. Following this, the ICUMSA colour⁷ was determined, and this complete set of results was considered to be at 'natural' pH. The above colour measurements were then repeated after adjusting the pH to 7,00 ± 0,05 with 0,5 M sodium hydroxide or 0,05 M hydrochloric acid.

Attenuation indices a*c at different wavelengths were calculated using the following formulae :

$$(a) \ a^*c \ 420 \text{ nm} = \frac{\text{Optical density} \times 10}{\text{conc of solids (g/cm}^3\text{)} \times \text{cell length (mm)}}$$

$$(b) \ a^*c \ 720 \text{ nm} = \frac{\text{Optical density} \times 10}{\text{conc of solids (g/cm}^3\text{)} \times \text{cell length (mm)}}$$

$$(c) \ \text{Colour Index} = a^*c \ 420 \text{ nm} - 2 \ a^*c \ 720 \text{ nm.}$$

$$(d) \ \text{ICUMSA Colour (Method 4)} = \frac{\text{Optical density (420 nm)} \times 10 \times 1000}{\text{conc of solids (g/cm}^3\text{)} \times \text{cell length (mm)}}$$

The method of Dubuorg, Saunier and Devilliers⁸ was then used to determine the pH of the sugar itself. The definition of sugar pH is 'the pH of the water which is unchanged after dissolving the sugar in it'. A range of three unbuffered water solutions was prepared by adding a few drops of acid or alkali to distilled water to give pH values of about 6,0; 7,0 and 8,0. From these 50° brix solutions were prepared and the pH of the three water samples and each sugar solution was measured. The water pH was plotted along the x-axis and the sugar solution pH along the y-axis and the point where the curve cut the bisector gave the pH of the sugar.

Results and Discussion

Results at natural pH and at pH 7 are listed in Table 1. These were analysed statistically on a multi-linear programme on the Institute's computer and the regression analyses are given in Tables 2 and 3.

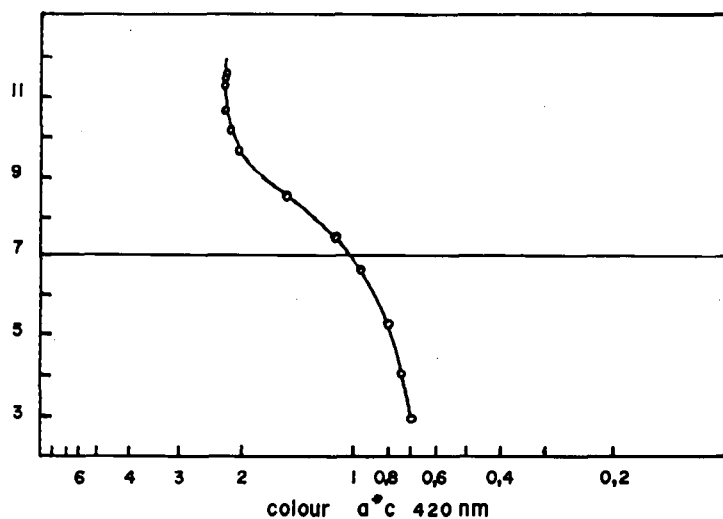


FIGURE 1 Effect of pH on colour at 420 nm.

In addition, South African refineries use a variety of processes for colour removal, viz. sulphitation, carbonation/sulphitation, Talafloc and ion-exchange, and the visual appearances of the sugars produced by these processes differ. The presence of sulphite in the end product was therefore investigated and its relationship with the colour and turbidity established. Since the pH of the sugar solution was found to be other than neutral, the pH of the sugar itself would also affect the pH of the solution. It was deemed necessary, therefore, to investigate (a) the effect of the pH of 50° brix solution, (b) the pH of the sugar and (c) sulphite content on each of colour, turbidity, colour index, and ICUMSA Method 4.

TABLE 1
Analyses conducted at natural pH and pH 7,0

Sample	Natural pH							pH = 7,0			
	pH Sugar sol.	pH Sugar	SO ₂ ppm	1 000 × a*c 420 nm	1 000 × a*c 720 nm	Colour Index	ICUMSA Colour	1 000 × a*c 420 nm	1 000 × a*c 720 nm	Colour Index	ICUMSA Colour
1	7,75	7,86	22	157	37	83	87	100	9	82	70
2	7,93	7,86	32	121	11	99	89	93	9	75	66
3	8,00	8,02	16	125	12	101	90	93	8	77	67
4	7,37	7,49	0	119	19	80	62	102	18	66	58
5	7,41	7,53	0	140	33	74	51	137	31	75	47
6	7,27	7,44	0	136	29	78	53	122	27	68	47
7	7,28	7,44	0	137	28	81	55	121	26	69	52
8	7,15	7,39	7	55	1	53	46	49	1	47	46
9	7,10	7,32	6	54	1	52	41	50	1	48	46
10	7,39	7,55	3	67	1	65	52	62	4	54	48
11	8,04	7,95	9	119	6	107	65	71	3	65	54
12	7,27	7,40	6	66	3	60	53	59	3	53	50
13	6,95	7,25	0	65	1	63	50	69	5	59	51
14	6,93	7,27	0	55	2	51	42	55	2	51	40
15	6,89	7,25	0	52	1	50	44	55	2	51	41
16	6,80	7,55	0	67	4	59	55	68	5	58	53
17	6,87	7,25	0	70	7	56	56	71	6	56	49
18	6,68	7,03	1	90	9	72	68	92	10	72	62
19	7,10	7,32	1	115	11	93	87	112	13	86	71
20	6,84	7,24	1	96	9	78	78	97	9	79	65
21	6,59	6,94	1	96	8	80	72	104	8	88	74
22	6,79	7,22	1	102	10	82	72	100	10	80	65
23	6,60	7,00	3	73	9	55	56	80	8	64	58
24	7,38	7,63	1	97	9	79	70	91	12	67	61
25	6,70	7,14	2	60	4	52	51	66	5	56	54
26	6,61	6,97	1,5	115	11	93	91	134	12	110	93
27	6,30	6,82	3	28	1	25	25	33	0	33	24
28	6,00	6,55	6	42	4	35	36	51	3	45	41
29	6,20	6,78	6	48	5	39	38	58	4	50	41
30	6,42	6,94	7	57	6	45	32	57	4	49	49
31	6,10	6,66	5	51	5	41	55	64	6	52	50

TABLE 2
Regression analyses of various colour measurements against the influence of pH of solution, pH of sugar and the SO₂ content of the sugar

	pH soln	pH sugar	SO ₂ cont
Colour 420 nm	r = 0,68 (**)	r = 0,64 (**)	r = 0,27 (ns)
Turbidity 720 nm	r = 0,43 (*)	r = 0,40 (*)	r = 0,15 (ns)
Colour Index	r = 0,74 (**)	r = 0,70 (**)	r = 0,30 (ns)
ICUMSA Colour, 420 nm	r = 0,51 (**)	r = 0,49 (**)	r = 0,38 (*)

** = Highly significant
* = Significant
ns = Not significant

Because of the high correlation between the pH of the sugar solution and that of the sugar itself (r = 0,95), their individual influence on colour in general is very similar. This is indicated by the different correlation factors given in Table

2. The pronounced influence of pH on true colour in solution is shown by the highly significant correlation factors for all colour measurements, but pH has only a minor affect on turbidity measurements.

Although a slightly significant relationship was found between SO₂ and pH, there is no relationship between SO₂ and colour and the relationship found between SO₂ and pH is coincidental. This could be due to the different origins of the sugars and an investigation into sugars from the same source but with different concentrations of sulphite may give a definite correlation.

From the results in Table 3 it can be seen that turbidity has a highly significant influence on colour measurements, both at natural pH and at pH = 7,0, while ICUMSA colour, where turbidity is supposedly removed by filtration, showed virtually no relationship. Colour indices at natural pH show a highly significant relationship with ICUMSA colour at

TABLE 3
Regression analyses of various colour measurements at natural pH and at pH = 7,0

	Colour 420 nm	Turbidity 720 nm	Colour Index	ICUMSA Colour 420 nm	pH solution
Colour 420 nm	0	r = 0,86 (**)	r = 0,87 (**)	r = 0,73 (**)	r = 0,68 (**)
Turbidity 720 nm	r = 0,86 (**)	0	r = 0,47 (**)	r = 0,38 (*)	r = 0,43 (*)
Colour Index	r = 0,87 (**)	r = 0,51 (**)	0	r = 0,86 (**)	r = 0,74 (**)
ICUMSA Colour 420 nm ..	r = 0,69 (**)	r = 0,26 (ns)	r = 0,93 (**)	0	r = 0,51 (**)

← pH = 7 →

** = Highly significant * = Significant ns = Not significant.

↑ Natural pH ↓

natural pH and this correlation is further improved if both are measured at pH = 7,0. Although this correlation is good, the actual values of colour index and ICUMSA units are not the same but differ significantly as is shown by a simple t-test (table $t_{95\%, 30} = 2,04$ and the calculated $t = 8,39$).

In order to overcome the time consuming adjustment of pH to 7,0 with dilute hydrochloric acid and sodium hydroxide solutions, a pH 7,0 buffer (the Sørensen - sodium/potassium phosphate) was used to dissolve the sugars. Sugars of both high (8,02) and low (6,00) pH read 6,7 and when a mixture of 20% buffer to water solution was used, the resulting solutions read 6,5. It seems therefore that the use of a buffer solution to dissolve the sugars does not automatically give a solution of pH = 7,0.

Conclusions

- (a) In the past it has been suggested that the colour index, where the attenuation at 420 nm is compensated for turbidity, is closely related to the ICUMSA colour. This was confirmed by the results obtained in the present investigation but there is an even better correlation at pH = 7,0 than at natural pH.
- (b) It is recommended that the existing method for the determination of colour in white sugar described in the

SASTA Laboratory Manual for South African Sugar Factories⁵ be amended to include the adjustment of pH to 7,0.

- (c) SO₂ has little effect on the colour of white sugar, probably due to the fact that a wide variation in origin of the sugars existed.

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