

THE OCCURRENCE OF INVERSION

By A. VAN HENGEL

Summary

It is pointed out that there is no reason to lime excessively in order to maintain the pH of clarified juice at higher than 7.0 for fear of inversion. It is suggested that inversion will occur more likely at other places.

Introduction

When, some years ago, I had the privilege of attending a conference of the Queensland Society of Sugar Cane Technologists, I was quite impressed by the long and constructive discussions about subjects, which were not always altogether new. I remember two occasions on which authors presented papers merely for the purpose of initiating exchange of views.

In this paper, I would like to do the same, i.e. to bring to your attention a subject that is in no way new to any of us, but about which there exists a certain amount of confusion, in the hope that this will lead to discussion.

Rate of Inversion

The rate of inversion of sucrose solutions was investigated by Stadler and the results of his work are available in graphs and tables.¹ They are commonly accepted and used and although there is some doubt whether the tables are accurate in high brix regions, there is no reliable evidence on which their practical application should be condemned.

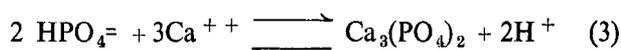
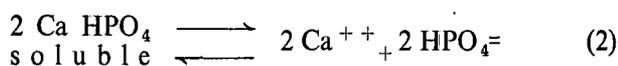
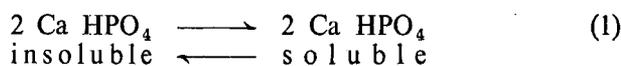
Table 1 shows a form of the Stadler tables, which is easiest for our purpose.

If we assume that our normal laboratory control methods will not enable us to detect the occurrence of inversion if the rate is less than 0.1 per cent per hour, then the conditions of temperature and pH associated with the inversion rates shown above the stepped line in Table 1 should be regarded as safe.

Inversion in Clarifier

As the retention in the clarifiers is three hours or more, whilst the average retention of the mud is even twice as long, and the temperature is at or just below boiling point, the clarifiers are normally regarded as the danger-spots. For that reason, it is standard practice to lime the mixed juice to such a pH that the overflowing clarified juice has a pH of 7.0 or slightly higher, whilst some factories allow the juice to have a pH of 7.4. It is often regarded dangerous if the pH of the juice is between 6.8 and 7.0. However, even if the juice left the clarifiers at that pH, it was not kept

at that low pH during its full residence time. The juice entered with a pH of at least 7.6 and the pH decreased only gradually with the transition of secondary into tertiary phosphate²:



The reaction of equation (1) is very slow, as it is assumed that the CaHPO_4 crystals are covered with a gelatinous layer of $\text{Ca}_3(\text{PO}_4)_2$.

The residence time of the mud in the clarifier is much longer but as the mud is only 30 per cent of the total amount of juice handled, the possible damage will be proportionally less.

A glance at Table 1 shows immediately that a pH of 6.8 even during many hours at 100°C. will not lead to a detectable rate of inversion. Or, in other words, inversion will not normally occur in a clarifier and the choice of pH, therefore, need not be guided by fear of inversion.

On the contrary, once it is accepted that the pH of clarified juice between 6.8 and 7.0 is harmless from the point of view of inversion, the advantages of that low pH are obvious:

- (1) Less lime consumption.
- (2) Less destruction of reducing sugars into coloured, acideous, products.
- (3) Smaller mud volumes.
- (4) Lighter coloured juices.
- (5) Less viscous molasses.

Inversion During Evaporation

The temperature of the juice in the first vessel of the evaporator is rather high. However, the residence time is limited to approximately $\frac{1}{4}$ hour and inversion at 110°C., 6.8 pH during $\frac{1}{4}$ hour can be neglected. During evaporation, the pH will drop but so will the temperature and even if the syrup pH is 6.2, the temperature will be only 60°C. and a residence time of many hours will still be harmless. In all normal cases, and these cover a wide range, there will be no serious inversion during evaporation.

Other Possible Sources of Inversion

From the Stadler tables it appears that the pH of clarified juice may vary within a reasonably wide range without justifying concern for inversion occurring during its residence in the clarifier or during

Table 1
Percentage of Sucrose, Inverted in One Hour at Different Temperatures and pH's of the Solution (Stadler)

°C.	pH	4.6	4.8	5.0	5.2	5.4	5.6	5.8	6.0	6.2	6.4	6.6	6.8	7.0	7.2
50		0.025	0.016	0.010	0.0063	0.0040	0.0025	0.0016	0.0010	0.00063	0.00040	0.00025	0.00016	0.00010	0.00006
60		0.088	0.056	0.035	0.022	0.014	0.0088	0.0056	0.0035	0.0022	0.0014	0.00088	0.00056	0.00035	0.00022
70		0.26	0.18	0.11	0.070	0.044	0.026	0.018	0.011	0.0070	0.0044	0.0026	0.0018	0.0011	0.00070
80		0.83	0.52	0.33	0.21	0.13	0.083	0.052	0.033	0.021	0.013	0.0083	0.0052	0.0033	0.0021
90		2.22	1.41	0.89	0.56	0.35	0.22	0.14	0.089	0.056	0.035	0.022	0.014	0.0089	0.0056
95		3.49	2.21	1.39	0.88	0.55	0.35	0.22	0.14	0.088	0.055	0.035	0.022	0.014	0.0088
100		5.32	3.37	2.12	1.34	0.84	0.53	0.34	0.21	0.13	0.084	0.053	0.034	0.021	0.013
105			5.55	3.49	2.20	1.39	0.88	0.56	0.35	0.22	0.14	0.088	0.056	0.035	0.022
107				4.18	2.65	1.67	1.05	0.67	0.42	0.27	0.17	0.11	0.067	0.042	0.027
110				5.39	3.41	2.15	1.35	0.86	0.54	0.34	0.22	0.14	0.086	0.054	0.034
120					4.4	2.8	1.8	1.1	0.70	0.44	0.28	0.18	0.11		0.07

the evaporation process. Hence, chemical losses can be attributed only to a completely erratic control of pH and temperature during clarification or to abnormal conditions in the factory which tend to escape our attention. A few of the possible causes of inversion I would like to mention are:

(1) The action of enzymes (invertase or saccharase) in the scale-tanks, especially when they are oversized, can cause considerable inversion.³ The clarifier is often erroneously blamed for this inversion.

(2) Old heaters, with high pressure drop are sometimes used at too low flow rates. As the turbulence in the tubes will be insufficient to disrupt the film on the heating surface, the heat transmission coefficient will drop.⁴ This actually means that a layer of nearly stationary juice of 5.5-6.0 pH will be in contact for an appreciable period with a surface of 115°C. (steam of 10 p.s.i.).

(3) According to Davies,⁵ a retention time of juice in clarifiers less than 1.5 hours is short, 2-2.5 hours normal and more than 3 hours long. The capacity of the majority of clarification stations in Natal may be regarded as being very high in comparison with Davies's figures. Consequently, the mud retention time is proportionally longer. A very strict control on the performance of the filters is the only feasible way in which to maintain the mud in the clarifiers at the correct level thereby limiting the retention time.

(4) Sometimes sour bagacillo is added to inadequately designed heater-mixers without proper adjustment of the pH by means of a small continuous flow of milk of lime. This, of course, is different from liming to a pH of 8.5 in an attempt to improve the filtering of the mud.

(5) For the purpose of blowing up, some molasses tanks are fitted with steam coils (sometimes using live steam of 120 p.s.i. and 175°C.). It is easy to see what will happen in the viscous, stagnant layer of molasses in immediate contact with the steam pipe. Hence, blowing up, a necessity from point of view of pan boiling, should be done by direct injection of exhaust steam with a suitable injector. This will also provide a certain amount of agitation and the position of the injectors is therefore important.

In Appendix 1 a sketch of a very simple, cheap but satisfactory construction is given.

(6) Pans of bad design, specially if used for boiling C-masseccuite, have a very restricted circulation during the final concentration stages. A calandria at over 100°C. surrounded by practically immobile masseccuite of approximately 5.8 pH, will create optimum conditions for inversion. Admittedly this applies to a small part of the total amount of sucrose but these conditions exist during many hours.

(7) The effect of temperature on pH is not yet fully known. Payne⁶ recommends that pH should be measured at process temperature and compared with buffers also at process temperature. As suitable glass electrodes appear to be available at present, there should be little doubt as to what method of measurement to follow.

Conclusion

From the above, it appears that the danger of inversion in a defecation-raw sugar factory lies not so much in the chemical processes involved, but in the technological way in which those processes are conducted. In particular, attention should be focussed on high residence vessels and the heating of viscous sugar liquors.

Bibliography

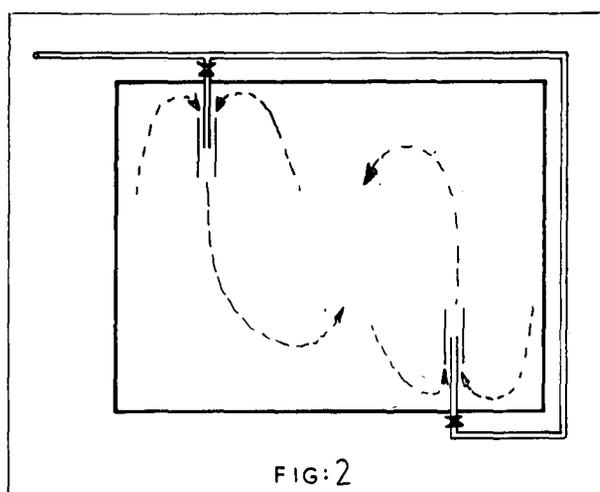
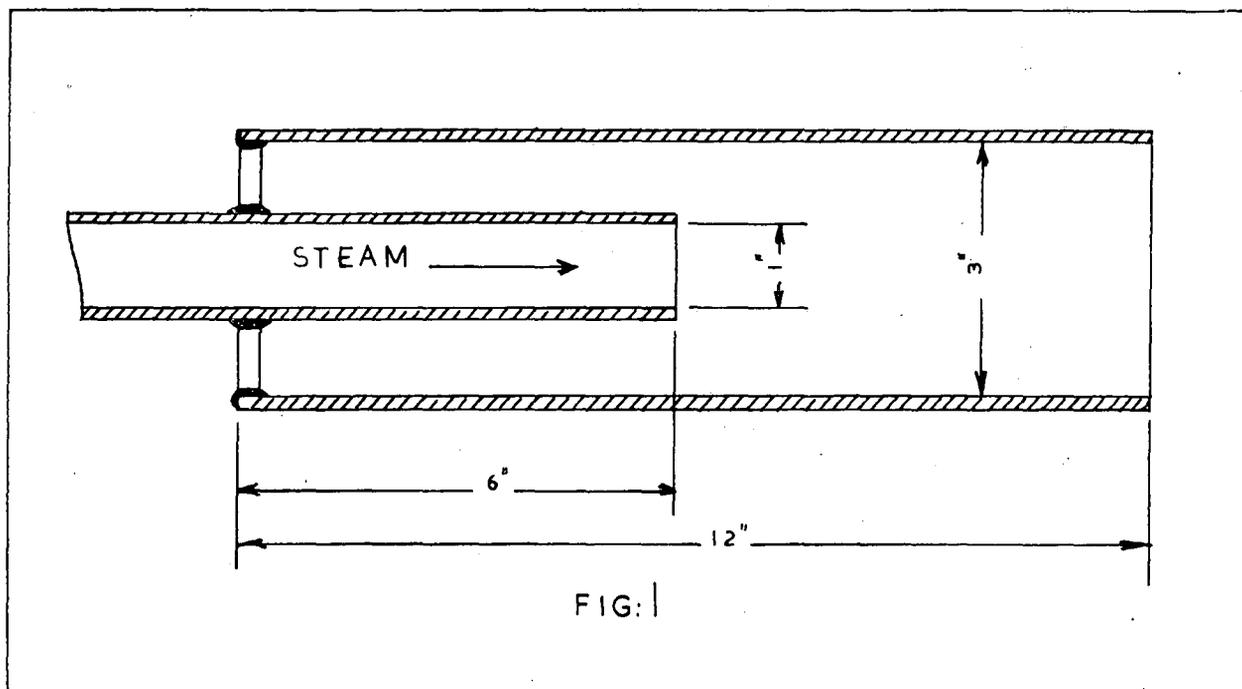
- ¹Honig, P., "Principles of Sugar Technology", Vol. I, 1st Ed., p. 589, Elsevier, London, 1953.
²Honig, P., *Ibid.*, p. 510.
³Brujin, J., "Sucrose Decomposition During The Milling of Cane", S.M.R.I. Quarterly Bulletin, No. 12, 1959, 28.
⁴Hugot, E., "Handbook of Cane Sugar Engineering", Revised Edition, p. 314, Elsevier, London, 1960.
⁵Honig, P., "Principles of Sugar Technology", Vol. I, 1st Ed., p. 314, Elsevier, London, 1953.
⁶Honig, P., *Ibid.*, 466.

Appendix I

A simple, but efficient and silent injector can be made from a 1 in. pipe with a 3 in. pipe fitted concentrically, as shown in Fig. 1. The pipes are kept apart with spacers.

A fairly good stirring can be obtained by placing two injectors in a tank as shown in Fig. 2.

APPENDIX 1



Mr. Rault, in the chair, said that the author had explained that mixed juice should not be limed to maintain the pH of the clarified juice higher than 7.0 pH, for fear of inversion. Dr. Honig had pointed out the danger of losses due to keeping molasses one or two hours too long. Losses which should be watched carefully were those which were not measured, but which occurred in the milling plant especially during the hot weather.

When we tried to trace inversion by keeping account of reducing sugars we could not be sure of our results, because these sugars decomposed rapidly as did other products formed from them.

Mr. Antonowitz commented on inversion which could take place at the filters by adding sour bagasse. From personal observation he had found that trying to correct for drop in pH by the addition of lime resulted in a greater drop in pH of the syrup. Often the mud had insufficient heating to keep it sterile, and adding extra lime did not decrease the rate of inversion, but actually increased it. There were two alternative remedies, firstly to see that the temperature did not drop below, say, 90°C, and secondly to add chloride of lime instead of lime.

Mr. Fourmond thought we normally destroyed about 20 per cent of the reducing sugar during our liming process. A balance sheet of reducing sugars showed that there was an increase of these thereafter.

Mr. van Hengel said that by liming heavily we did not prevent much inversion. If a juice limed to say, 7.8 pH, gave rise to a clear juice of 6.8 pH, heavier liming would make no difference. The main source of inversion he had found in a factory was the blowing-up of molasses of, say, 5.8 pH, by means of a steam coil, instead of blowing in steam. There was a rather

static layer of molasses in constant contact with the very hot steam coil, reaching a temperature of over 100°C, and this caused considerable inversion in this layer. The sketch in the paper showed a method which was cheap to install, obviated the undesirable condition he had mentioned, and which also achieved mixing.

Mr. Phipson asked if it would not be advisable to store molasses cold and heat it just prior to drawing it into the pan by admitting steam into the pipe line, and controlling the temperature by means of a suitable thermometer.

Mr. van Hengel said that in many factories molasses was stored cold and then heated in a supply tank on the pan floor.

Mr. Rabe related that he had conducted some experiments at Umzimkulu in which the liming had been adjusted so that the syrup pH came down to 6.2, and during that period no increase in reducing sugars had been found.

Mr. Rault asked if there was not more chance of inversion in factories which were refining, as the pure liquors were not well buffered.

Mr. van Hengel said that the remelting of sugars was one of the most common causes of inversion. In a non-buffered solution the chances of inversion were, of course, great.

Mr. Rault stated that the paper showed that raw sugar factories which had high undetermined losses should not look for them in what would be termed chemical losses, but rather, mechanical losses should be sought. A balance sheet of brix as well as sucrose, would in such cases, show brix losses larger than sucrose losses.