

CLARITY & CLARIBILITY

By G. C. DYMOND

The clarity of a juice is measured by the Kopke or Luximeter methods. Both are simple and comparative.

The evaluation of clarity in cane sugar manufacture and the factors which influence it, are both complex and varied.

P. Honig discusses some of these factors and the wax complex, which he calls "lipids" in a paper presented at the 1953 I.S.S.C.T. Congress¹. Axtell and Keller at the same Congress², showed that, "the removal of the wax complex by solvent treatment of the raw juice substantially improves the claribility of the juice by increasing the purity, the settling rate and decreasing the mud volume."

In the past clarity was considered important in Natal in the making of plantation white sugars and to assist in filtration of the muds in plate and frame presses. To achieve this up to 3 gms. of sulphur dioxide were used per litre of juice, but with the advent of the Oliver Campbell filter and diminution

of the quantity of mill whites produced, the tendency has been to make what appears an obvious monetary saving by reducing chemicals and leaving the refinery to deal with the raw sugars so produced.

As a result the clarity of the juice has generally deteriorated wherever chemicals have been cut.

In order to throw some light on this complicated problem a series of experiments was carried out, with the following results:

1. The Effect of Pre-Heating Raw Juice in Simple Defecation

A composite sample of cold mixed juice was treated with 0.3 gms. of phosphoric paste, a standard procedure in all these experiments. One 2 litre sample was limed to 8 pH at atmospheric temperature, boiled and settled. The rest were pre-heated at temperatures ranging from 40°C to 90°C before treatment. The results were positive and illuminating.

Mixed Juice Temperatures	Atmospheric Temperature	40°C	50°C	60°C	70°C	80°C	90°C
Luximeter reading:							
Clarified juice	62	58	56	54	52	46	42
pH Clarified juice	6.7	7.1	7.0	7.4	7.5	7.6	7.6
Repeat different sample	7.3	7.45	7.45	7.55	7.7	7.75	7.85

A repeat experiment using acid sulphitation gave the same decrease in clarity through pre-heating. The raw juice was sulphited to 2.8 pH and then limed to 8 pH before boiling and settling.

	Atmospheric Temperature	Pre-Heat 40°C	65°C
Luximeter reading:			
Clarified juice	80	74	74

2. Chemicals

The quantity of lime required to bring a juice to a given pH varies considerably with the nature of the juice. Thus in simple defecation the amount of lime used to bring a juice to 8 pH was found to vary from 1.13 to 2.17 lbs. CaO per ton of cane and in acid sulphitation comparative tests varied from 3.35 to 5.65 lbs. CaO per ton of cane. In each case the lower purity juices 82.1—84.6° required less lime than juices at 87.0°.

This may be accounted for by the fact that the lower purity juices contained approximately 200 mgs.

per litre more magnesia than the high purity juices.

In sulpho-defecation the amount of lime required varies with the quantity of the lime first added. Thus at 9.5 pH the quantity in one sample was 1.56 lbs. CaO per ton of cane and at 11.0 pH it was 4.45 lbs. per ton of cane.

3. Lime Salts

Natural lime salts in mixed juice at Darnall range from 250 to 400 mgs. per litre. A much wider range exists with magnesium salts. The minimum found was 46 mgs. in juice from flat cane at Felixton and the maximum was 844 mgs. at Darnall.

Pre-heating has the following effect on the lime and magnesia salts in clarified juice. Thus:

	Mixed Juice	Atmospheric Temperature	Acid Sulphitation 40°C	65°C
CaO+MgO mgs. per litre ...	1,130	1,210	1,200	1,170
CaO mgs. per litre	504	616	672	728
MgO mgs. per litre	626	594	528	442

These experiments show that pre-heating raw juices prior to simple defecation or acid sulphitation, causes a progressive decrease in clarity, increasing lime salts and decreasing magnesia.

The effect of increasing the final pH from 7.5, to 8.0, 8.5 and 9.0 also caused a progressive increase in lime salts and a reduction in magnesia.

In general, cold simple defecation gave lower lime salts than any other method. An average of 5 runs gave the following comparison with acid sulphitation:

	Mgs. per Litre	
	CaO	MgO
Mixed juice	287	765
Simple defecation	470	624
Acid sulphitation	624	594

The following series were carried out before renewed interest in simple defecation arose:

	Mgs. per Litre	
	CaO	MgO
Mixed juice	385	718
Acid sulphitation	662	531
APC process	663	377
General factory average	700	566
Sulpho-defecation	812	466

The maximum mgs. CaO found was 938 in a sulpho-defecated juice pre-heated to 65°C and pre-limed to 10 pH, and the lowest was 320 mg. in a simple defecation different sample. In the factory the maximum CaO found was 882 mgs. per litre,

and the maximum 588 mgs. magnesia was 486 maximum and 186 minimum.

In all cases simple defecation gave the lowest clarity, the lowest Luximeter reading being 30 and the highest 58. Other processes ranged from 60 to 80.

Wax Complex or Lipids

The wax complex content of mixed juice ranged from 1.5 to 2.5 gms. per litre at Darnall. Honig states that "the percentage of hard wax recovered in the milling process via the extracted juice, decreases when the pre-treatment of cane in the milling process by knives and shredders is intensified."²

A sample containing 2.03 gms. of wax per litre was boiled, filtered and washed with *cold* water over Kieselguhr under vacuum. No wax was lost. Another sample containing 1.33 gms. wax per litre was treated in the same way but washed with hot water. No wax was lost. This shows that an efficient absorbent medium such as Kieselguhr will retain the wax complex whether partly emulsified or not.

On applying simple defecation to the first sample, with no pre-heating, 37.6 per cent. of the total wax was lost. The top scum after boiling, which is voluminous in this process, contained 0.39 gms. of wax per litre and the mud 0.89 gms., a total of 1.28 gms.; 0.75 gms. were therefore lost. In other words, the mud only absorbed 43.4 per cent. of the total wax originally present in the mixed juice.

Wax retention varies with the raw material, time of heating, agitation, reaction, the absorbing medium or the method of clarification used.

The effect of the clarification process used is shown in the following series:

	Mixed Juice	A.P.C. Process		Acid Sulphitation			Sulpho-Defecation
		1st ppt.	2nd ppt.	Atmospheric Temperature	40°C		
					65°C		
Gms. dry cake per litre	3.98	10.88	10.78	10.22	10.00	8.74	
Wax percentage dry cake	38.04	27.5	1.2	10.4	5.1	5.9	9.2
Gms. wax per litre	1.52	1.50	0.07	1.12	0.52	0.59	0.80
		1.57					

In the foregoing series the A.P.C. process retained all the wax with cold washing. In practice hot washing and mixing of the two cakes might cause some loss. This point was not determined. Cold acid sulphitation came second with 73.6 per cent. retention and the others ranged from 34 to 54 per cent.

In simple defecation nearly all the wax may be recovered by raising to the boil quickly, removing the top by skimming and incorporating it with mud at a low temperature. In the A.P.C. process there is no scum as most of the wax is precipitated in the cold first precipitate.

General

The foregoing observations were made from a large number of experiments carried out during the 1954 crop.

Dr. Honig says, "A better understanding of the behaviour of waxy material in sugar manufacture can only be obtained, if we differentiate between the various types of lipids and not classify them as one group. This system of classifying lipids as a uniform single group leads to more confusion than real understanding, just as is the case when explaining differences in the behaviour of juices by a general term such as "non-sugars" without a proper analysis of what is actually to be understood and what kind of reaction we are specifically considering."

In theory this is undoubtedly true but in practice the variations are so great and unpredictable that no practical application would be possible, were this knowledge available.

It remains therefore to use a process of clarification which will cover all known extremes and not revert to primitive methods which are subject to all the variables common to raw material in this country.

REFERENCES

¹The Lipids in Cane-sugar Manufacture by Pieter Honig. I.S.S.C.T. Proceedings, Page 710, 1953.

²Effect of Wax Removal on Claribility of Cane Juice by W. R. Axtell and Arthur G. Keller. I.S.S.C.T. Proceedings, Page 719, 1953.

Mr. Dymond, in reply to Mr. Rault, said that the higher figures obtained with the luximeter indicated the better clarity. Water gave a reading of 100.

Dr. Douwes Dekker said he did not want the audience to gain the impression that the clarity of a clarified juice was connected with the claribility of the mixed juice (from which the clarified juice was prepared) as defined by Axtel and Keller. In their

paper dealing with the removal of wax by solvent extraction on the claribility of mixed juice it is said: "Claribility of the juice is a concept developed to permit expression of clarification efficiency in mathematical rather than descriptive terms. It can be considered as being a function of the available sucrose, the settling rate and the mud volume."

Hence the claribility is closely connected with plant capacity. In the paper by Axtel and Keller, the following data for claribility of the mixed juice and clarity of the clarified juice are given in respect of a series of tests in which the proportion of solvent to juice were increased.

Raw juice	...	—	1	2	3	4
Clarity, clarified juice	...	56	43.7	35.5	36.2	35.5
Claribility, raw juice	...	103.2	128.8	116.9	337	339

Underneath raw juice we find the data for clarity and claribility obtained in the tests of the untreated raw juice, under 1, 2, 3 and 4 the data pertaining to juice treated with trichloroethylene in increasing quantities. It appears that solvent extraction did not improve the clarity of the clarified juice (on the contrary, the clarity was worse), but the claribility increased considerably, in particular when larger quantities of solvent were used.

He referred Mr. du Toit and Mr. Rault to the I.S.S.C.T. Proceedings for the details of the paper on claribility. He said further, that the explanation given by Mr. Dymond to account for the smaller quantity of lime required to clarify juices of lower purity, i.e. the higher magnesium content, was interesting, but would have to be confirmed by further tests.

He also said that the better removal of magnesium salts by the A.P.C. process, as found by Mr. Dymond, was contrary to results obtained by the S.M.R.I. Moreover the superiority of the A.P.C. process in this respect was difficult to explain theoretically. To remove magnesium, a high alkalinity was required, as in the first carbonation. He also objected to Kieselguhr being termed "an efficient absorbent medium." Kieselguhr was an excellent filter aid, but its absorptive capacity was insignificant.

Mr. Rault said that the February issue of *Sugar* had an interesting article on the effects of cold versus hot liming at a Puerto Rican central.

The authors claimed that when dealing with juices of high P_2O_5 content (600 to 1,000 p.p.m.), hot liming gave superior clarity and rise in purity. The conclusion was also that a high pH after clarification was easier reached by hot liming and this method was a means of saving chemicals.

Farnell working on West Indian juices had found hot liming eliminated a larger proportion of nitro-geneous impurities than cold liming.

With carbonation in South Africa it was found that juices carbonated hot at 70°C., in spite of the danger of glucose destruction, gave a better filtration rate than if carbonated at 55°C., and subsequently heated to 70°C. before filtration.

The composition of the ash before and after carbonation, although showing a practically total elimination of magnesia, could not explain the enormous rise in purity experienced at Mt. Edgecombe. Organic impurities removal was of a greater magnitude than the demineralisation effect, made somewhat ineffective by the substitution and increase of lime salts.

Dr. Douwes Dekker agreed that the removal of the organic non-sugars was most important and this led to a higher purity being obtained, but the very good removal of magnesia was also a factor. He said that the higher pH attained with hot liming was explained by coagulation of proteins by heating and before liming.

Mr. W. H. Walsh asked for Mr. Dymond's definition of claribility.

Mr. Dymond said that he had understood that claribility meant the ease with which a juice could be clarified, together with the factors which caused bad clarification. However he now accepted Dr. Douwes Dekker's more precise definition.

Dr. van der Pol said that he had done a considerable amount of laboratory work on clarification and his experience was that laboratory results alone were not a true indication of what would happen in the factory. Exact duplication of a factory process in the laboratory was impossible and the effect of

the return of filtrates as well as the effect of the hold-up time in the subsider cannot be assessed accurately in the laboratory. He said that laboratory tests, while being a useful adjunct, could not completely cover what was required under practical working conditions. In Australia, for example, prediction on the method of liming to be used for optimum clarification results was considered impossible. Often a season was started with cold liming and as the season progressed it was found necessary to change to the hot liming process for better results. The next season might show the reverse. He remarked that such experiences were really not surprising, in view of the great variability of juice properties which were encountered throughout the season and even from hour to hour. He was of the opinion that clarity of the clarified juice was no criterion of a well clarified juice and related the experience of one factory manager in Natal who had obtained a record recovery during a season when clarity was at its worst.

Mr. Dymond said that he was limited to laboratory tests because he had no plant. As far as simple defecation was concerned, he thought that difficulties might occur later on, especially in the refinery.

Mr. Antonowitz said that experience at Umfolosi had shown that pre-heating of the juice before liming paid dividends as far as clarification was concerned. He said that the sugars obtained from hot liming were superior to those obtained during the period they used cold liming.

Mr. Dymond pointed out that there were a number of factors involved in clarification. Hot and cold liming were not the only ones to be considered. In the same way sulpho-defecation was a loose term for a process in which the clarification varied considerably with the amounts of lime and sulphur used.