STARCH IN THE MANUFACTURE OF RAW SUGAR

By P. N. BOYES

Starch has formed the basis for a considerable amount of discussion and a number of interesting papers. It has been proved[1,2,3 4] that starch slows down the filtration of sugar liquor in the refining of sugar. It is claimed by Balch[5] that it exerts a considerable influence on the viscosity of massecuites and the data of Buchanan[1] suggest that it might double the viscosity of the low grade massecuites. Further it is well known that starch retards the rate of crystallisation of sucrose as witnessed by its use for this purpose in the confectionary trade. It is well known that it co-crystallises with sucrose and is to be found within the crystal. In cases where 3rd sugars are single-cured[6] and used as a footing for 1st and 2nd sugars large amounts of starch can be occluded. Finally the recent work of Bennett and Schmidt[7,8] has shown that starch—in particular potato starch added in viscous solution—acts as an aid to flocculation.

The question of where the starch exists in the cane is well illustrated by Balch[5] who shows that it is deposited principally in the nodes of cane stalks. It is usually located in one or more layers of cells immediately surrounding the fibro-vascular bundles as they emerge from the root band of each node. However, in some varieties Balch observed a certain amount of "diffused" starch where the starch was located throughout all tissues. Chiu[6] found that starch was distributed throughout the cane stalk for certain Formosan varieties including N:Co.310, but was much more concentrated in the tops and nodes.

Various authors[5,9] have shown that the starch content of juice extracted from different cane varieties is largely characteristic of the variety. In order to obtain more information on the quantity squeezed out from different N:Co. varieties normally crushed, a large number of tests were carried out on a milling tandem with the following results. (See Table I).

TABLE I

Starch in Juice from different N:Co. Varieties

Starch expressed in mgms per 1,000 gms juice

	CRUSHER JUICE				MIXED JUICE			
Variety	*Min.*	*Max.*	*Mean*	*Avg. Brix*	*Min.*	*Max.*	*Mean*	*Avg. Brix*
N:Co.310	245	625	450	21.2	300	655	432	16.6
N:Co.292	185	595	405	20.5	205	580	380	16.2
N:Co.339	250	535	355	20.4	220	535	335	16.4
N:Co.293	260	470	345	20.3	210	450	305	15.8
N:Co.376	160	375	290	19.9	220	415	300	15.9
N:Co.331	175	350	266	20.5	235	345	300	15.5
N:Co.334	165	320	240	19.9	210	290	260	15.6

The tests were carried out during October/November which is past the peak maturity period. By taking a number of samples from each variety it was hoped that variations due to ratoon, age, soil, rainfall, etc. would be smoothed out. The results indicated that there is a definite varietal trend but considerable fluctuations occurred within each variety. N:Co.310 showed an outstandingly high starch content while a new variety N:Co.334 was extremely low by comparison.

The considerable variation that occurred within each variety led the author to look at the origin of the various samples and this disclosed an interesting correlation with soil types.[10]

TABLE II

Influence of Soil Type on Starch Content

Starch expressed as mgms per 1,000 gms juice

Soil Type	*Average Starch Content of Mixed Juice*	*No. of Samples used in Average* 310	292	339	293	376	331	334
Alluvial	260	–	–	–	–	–	–	4
Table Mountain Sandstone ...	305	1	2	4	7	6	1	–
Recent Coastal Sand	340	1	3	–	1	1	5	–
Ecca Shale and Dolorite ...	355	5	–	4	–	5	1	–
TMS/Dwyka+Dwyka	510	4	2	–	–	–	–	–

It was observed that cane grown from a large inland area of sandstone gave consistently lower starch figures irrespective of variety. The correlation here is good but in other cases there is insufficient spread of different varieties for a particular soil type. It is also significant that some of the highest figures were obtained on Dwyka soil. It is quite possible that the water holding capacities and drought resistance of different soil types are controlling factors.

It has been suggested that heavy milling is the cause of large quantities of starch being squeezed into the mill juices. In order to investigate this problem more thoroughly a suitable milling tandem was selected. This tandem normally crushed at 70 TCH and consisted of two sets of knives before a 66″ × 35″ Krajewski Crusher with a Searby Shredder before the 1st Mill. There were 5 sets of mills with maceration fed onto the last. Imbibition from the 3rd mill was split evenly to feed the 1st and 2nd mills. The mixed juice to the scales therefore consisted of a combination of crusher, 1st mill and 2nd mill juices.

Throughout a period of two weeks samples of juice were taken from all mills and analysed for starch and brix. The weekly results were averaged and are presented in Table III.

TABLE III

Starch Content of Individual Mills

Starch expressed as mgms per 1,000 gms juice

	W/E. 20.9.59		W/E. 27.9.59	
	Starch	*Brix*	*Starch*	*Brix*
Crusher	301	20.29	376	22.04
1st Mill	423	16.25	557	17.95
2nd Mill	244	12.52	254	13.10
3rd Mill	191	9.55	196	9.90
4th Mill	118	4.93	114	5.60
5th Mill	72	2.34	61	2.87
Mixed Juice	374	15.71	381	17.05

Using the weekly figures for the tandem for the respective weeks concerned it was possible to calculate the tons of juice being squeezed out at each set of rollers. It was then a simple matter using the starch figures given in Table III to calculate the amount of starch contained in the juice. The results of this calculation are given in Table IV and for simplicity the starch figures are given in lbs. per week.

TABLE IV

Weekly Starch Balance

Starch expressed in lbs./week

		W/E 20.9.59	W/E 27.9.59
CRUSHER	Juice from Crusher	2092	2237
1ST. MILL	Maceration to 1st Mill	908	897
	Juice from 1st Mill	3475	4891
2ND. MILL	Maceration to 2nd Mill	908	897
	Juice from 2nd Mill	1496	1623
3RD. MILL	Maceration to 3rd Mill	1018	926
	Juice from 3rd Mill	1817	1794
4TH. MILL	Maceration to 4th Mill	545	432
	Juice from 4th Mill	1018	926
5TH. MILL	Maceration to 5th Mill	Nil	Nil
	Juice from 5th Mill	545	432
MIXED JUICE	Determined from Mixed Juice Analysis	7924	8047

This balance can be checked by comparing the cumulative starch figures for crusher, 1st mill and 2nd mill which together constitute the mixed juice with the figure obtained separately for mixed juice. Thus using the figures given in Table IV it will be seen that the cumulative figures for the two weeks are 7,063 and 8,751 pounds compared with the separately determined figures of 7,924 and 8,047 pounds for mixed juice. The accuracy is considered sufficient for broad conclusions to be drawn and by subtracting the starch content in the juice from a mill from the starch in the maceration to the mill it is possible to calculate the actual quantity of starch squeezed into the juice at that mill. This has been done and the results are given in Table V below.

TABLE V

Individual Extraction of Starch at Each Mill

Starch expressed in lbs./week

	W/E. 20.9.59	W/E. 27.9.59
Crusher	2092	2237
1st Mill	2567	3994
2nd Mill	588	726
3rd Mill	799	868
4th Mill	473	494
5th Mill	545	432

It will be seen that in each case the crusher and 1st mill together extract about 70 per cent of the total starch. The extraction for the 1st mill is higher than the crusher. All subsequent mills extract at a reasonably constant figure. It is therefore clear that the cane preparation before the first two units has resulted in the squeezing out of considerable quantities of starch. The inference that heavy milling facilitates the squeezing out of starch granules is therefore given a quantitative basis.

Consideration will now be given to the development and installation of a method for removing starch from juice.

Certain factories operating in the same rural area showed widely different starch contents in their raw sugars. Investigation showed that the starch contents in the mixed juice of three factories under revue were similar yet the sugars contained the following starch contents. (See Table VI).

TABLE VI

Starch Content of Raw Sugars

Mgms Starch per 1,000 gms sugar

	Factory A	*Factory B*	*Factory C*
May	—	370	780
June	265	250	640
July	345	350	680
August...	290	335	780
September	370	530	680

Clearly A and B were eliminating more starch in their process than C and subsequent investigation gave the following results (Table VII). The analyses were carried out on snatch samples only and results are therefore only indicative of trends.

TABLE VII

Starch Content in Juice Streams

Mgms per 1,000 ccs juice

	FACTORY A		*FACTORY B*		*FACTORY C*	
	Starch	*Brix*	*Starch*	*Brix*	*Starch*	*Brix*
Mixed Juice	450	13.82	420	14.84	360	16.20
Juice Ex. 1 Primary Tanks ...	165	13.56	225	15.36	*No Tanks*	
Juice Ex. 2 Primary Tanks ...	143	14.27	235	15.56	*No Tanks*	
Clear Juice	130	13.22	185	14.88	310	15.00
Per cent Elimation...	71		52		14	

The elimination of starch was taking place in the primary tanks of factories A and B where the procedure was to heat raw juice to 160-180°F and then allow this juice to remain for 7-20 minutes. Factory A, which is a sulphitation mill, appeared to have better control and consequently gave better results than factory B, a defecation mill. In the case of factory C raw juice was partially limed and then heated to 215°F in 2-4 minutes. It is interesting to observe therefore that the two mills were unknowingly making use of enzyme action in starch removal.[11,12]

Lab experiments revealed that gelatinisation of starch took place at 160°F (70°C) but enzyme was de-activated at 180°F (80°C). Further, sucrose destruction being dependent on time, temperature and pH, it was essential to strike a practical balance between starch removal and these factors. It was found that raw juice at 5.2 pH heated to 170°F (70°C) for 10 minutes lost 0.3 per cent sucrose. Liming the cold juice to 7.6 pH eliminated sucrose loss but no starch removal was observed. The process adopted was therefore to add Oliver filtrate to the raw juice and in this way increase the pH to 5.8-6.0 thereby reducing the sucrose loss to a figure that varied from zero to 0.17 per cent for 15 minutes heating at 170°F. In this way the starch removal obtained was 50-60 per cent.

The final process therefore consisted of mixing Oliver filtrate with raw juice thereby increasing alkalinity and also allowing reaction between enzymes and the gelatinised starch in the filtrate. The juice was then heated to 160-180°F and allowed to settle in tanks for 8-12 minutes. The primary lime was added after the settling tanks.

The subsequent investigation at factory C on a factory scale was divided into a study of starch removal and sucrose removal. The results of starch removal are given in Tables VIII and IX. It will be seen that it took 10 days before the system had been cleared to establish the true picture. Subsequent data are given of daily raws and it will be seen that the results are comparable with factories A and B. The result has been a reduction in starch content of sugar of 50-60 per cent.

Sucrose losses were studied by collecting composite samples of juice before the primary heaters and juice emerging from the settling tanks. The apparent sucrose was determined in each case because results were purely comparative. The glucose ratio was also used as a guide to destruction. It will be observed from the figures given in Table X that a small amount of evaporation takes place in passage through the settlers and during sampling. The average sucrose figures have therefore to be corrected for this small difference in brix and average out at 12.58 per cent before and 12.56 per cent after the settlers. It could be argued that this small difference has resulted in a loss of sucrose of 0.16 per cent but the analytical method cannot be considered so sensitive. The glucose ratio gives an average figure of 4.77 before and 4.64 after, indicating a small loss in glucose.

In connection with sucrose loss it is of interest to note that factory B employing the defecation process had the second highest boiling house recovery in the industry (90.9 per cent). It would therefore be interesting to know how much the small loss of sucrose possibly occurring in the process is compensated for by increased recovery in the boiling house in the light of the known influence of starch.

To date the investigation has been purely to establish a process and get it working in a satisfactory manner. The factory operation occurred during the last four weeks of crushing and liquidation of stock masked any possible differences in recovery. Certain impressions were gained with regard to settling, sugar colour and boiling, but these aspects would have to be studied carefully over an extended period.

Analytical Method for Starch Determination

The analytical technique employed in all the starch tests given above is that of Balch[5] and Alexander.[9]

Acknowledgements

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TABLE VIII

Starch Readings during Settling Down Period of Starch Removal Process

Date	*Mixed Juice*	*Clear Juice*	*A Sugar*	*B Sugar*	*Comments*
27.11.59	—	148	—	—	Start up of process.
28.11.59	—	135	560	—	
30.11.59	595	75	335	750	
1.12.59	415	223	420	540	Juice heaters off 7 hours for
	—	—	450	700	cleaning.
2.12.59	—	155	575	375	
4.12.59	340	155	330	620	
7.12.59	235	143	320	425	Treated juice now considered to be right through process.
Average readings before process	360	310	710	—	

Starch in juices expressed as mgms litre juice.
Starch in sugars expressed as mgms 1,000 gms sugar.
Results are for snatch samples only.

TABLE IX

Daily Starch Content of Raw Sugar during normal running of Process

	Date	*Starch ppm*
Monday	7.12.59	250
Tuesday	8.12.59	350
Wednesday	9.12.59	350
Thursday	10.12.59	300
Friday	11.12.59	385
Saturday	12.12.59	290
Sunday	13.12.59	275
Monday	14.12.59	290
Tuesday	15.12.59	325
Wednesday	16.12.59	400
Thursday	17.12.59	385
Friday	18.12.59	275
Average reading before process		710

Results are for daily composited samples.

Table X—STUDY OF SUCROSE AND REDUCING SUGARS IN STARCH REMOVAL

DATE	*Days without cleaning digest tanks*	*RAW JUICE + OLIVER FILTRATE TO HEATERS*						*JUICE AFTER HEATING TO 170°F AND DIGESTING 8-12 MINUTES*						*COMMENTS*
		Brix	*Suc.*	*Pty.*	*Gluc.*	*Gluc. Ratio*	*pH*	*Brix.*	*Suc.*	*Pty.*	*Gluc.*	*Gluc. Ratio*	*pH*	
Friday 11.30 a.m. 27.11.59	0	15.50	13.24	85.4	0.49	3.69	—	15.40	13.14	85.3	0.46	3.52	—	Started process.
Saturday ,, 28.11.59	1	15.37	13.29	86.4	—	—	—	15.27	13.20	86.4	—	—	—	
Monday ,, 30.11.59	3	15.22	13.06	85.8	0.56	4.31	—	14.80	12.60	85.1	0.51	4.30	—	
Tuesday ,, 3.12.59	–	—	—	—	—	—	—	—	—	—	—	—	—	Cleaned tanks.
Thursday ,, 4.12.59	1	15.32	13.47	87.9	0.49	3.66	—	15.34	13.47	87.8	0.50	3.69	—	
	–	14.51	12.42	85.6	0.50	4.02	—	14.57	12.65	86.2	0.50	3.90	—	
	–	14.89	12.73	85.5	0.42	3.03	—	15.05	12.98	86.3	0.38	3.42	—	
Tuesday ,, 8.12.59	0	14.34	12.25	85.4	0.47	3.84	—	14.47	12.27	84.8	0.47	3.83	—	Cleaned tanks.
	–	14.04	12.13	86.4	0.77	6.35	—	14.61	12.42	84.9	0.79	6.37	6.0	
	–	14.24	12.27	86.2	0.51	4.16	—	14.64	12.57	85.9	0.50	3.98	5.9	
Wednesday ,, 9.12.59	1	14.24	12.00	84.3	0.51	4.25	—	14.64	12.32	84.2	0.49	3.98	6.0	
Thursday ,, 10.12.59	2	14.21	12.28	86.4	0.66	5.37	—	14.71	12.61	85.7	0.67	5.31	5.2	
	–	14.77	12.74	86.2	0.81	6.36	—	15.14	13.04	86.1	0.80	6.13	5.8	
Saturday ,, 12.12.59	4	14.74	12.62	85.6	0.64	4.34	—	14.84	12.74	85.8	0.65	4.38	5.7	
Monday ,, 14.12.59	6	14.64	12.42	84.8	0.66	5.31	5.5	15.04	12.67	84.2	0.63	4.97	5.8	Tanks dirty—not taken
	–	15.27	13.04	85.4	0.61	4.68	5.5	15.74	13.27	84.3	0.62	3.94	5.7	in average.
Tuesday ,, 15.12.59	0	14.74	12.64	85.7	0.76	6.01	—	15.11	13.04	86.3	0.78	5.98	—	Cleaned tanks.
Thursday ,, 17.12.59	2	14.36	12.05	83.9	0.74	6.14	6.0	14.59	12.25	84.0	0.74	6.04	6.1	
	–	14.79	12.12	81.9	0.61	5.03	5.9	15.09	12.15	80.5	0.60	4.94	5.8	
Averages	–	14.70	12.58	—	0.60	4.77	—	14.89	12.72	—	0.59	4.64	5.8	
Corrected for brix	–	—	—	—	—	—	—	—	12.56	—	—	—	—	

The President, Mr. Bentley (in the Chair) stated it was obvious that a great deal of work had been done by the author on this subject.

Dr. Douwes-Dekker congratulated Mr. Boyes on using the Australian process of Nicholson & Horsley. He asked if the author had found here, as they did in Australia, that starch content and phosphate content of mixed juice were related. Mr. Boyes had discussed the effect of heavy milling. He asked what was meant by this and if the extraction was increased from 92 to say 94 would the starch content thereby be increased? Studying the figures shown in Table VII he had noticed that in factory C starch removal had been 14 per cent only. Factory C, applying the defecation process, one might be tempted to conclude that the normal defecation process was not capable of removing more than 14 per cent of the starch. The S.M.R.I., when conducting a similar investigation at two factories, had however found a much higher figure. He now wanted to know, in view of the fact that the paper states "The analyses were carried out on snatch samples only and results are therefore only indicative of trends" what accuracy Mr. Boyes attached to the figure of 14 per cent? In Table VIII he noticed that the average amount of starch in the B sugar was only 710 parts per million while similarly in Table IX the average before the process was applied still remained at 710, although this figure apparently now applied to all the sugar.

Mr. Boyes said he had no phosphate figures which could be correlated with the individual tests shown in the paper here. By heavy milling he meant heavy cane preparation such as two sets of knives and a Searby Shredder. With regard to the removal of starch it is known that juice contains a certain amount of starch in the form of granules. It is also known that juice contains enzymes which are capable of breaking down the starch particles. Unfortunately in its granular form enzymes are unable to attack starch effectively. Sufficient temperature is required whereupon these granules swell and burst, enabling the enzymes to come into better contact with the starch. The temperature at which this takes place is about 70°C. It is therefore the first pre-requisite that at some stage of the process a temperature of 70°C is reached. On the other hand the enzymes themselves are inactivated above a certain temperature. This temperature, has been determined to be about 80°C. Therefore, taking the case of Factory C, a period is reached during the heating by the juice heaters where a temperature of 70°C is reached but very rapidly after that a temperature of 100°C is also reached. In other words the time factor plays its part in that the enzymes have not got sufficient time to do their work. There is also a second great important feature about Factory C, i.e. it was adopting the process of adding some of its lime to the cold juice. This heavy liming has also been found to have the effect of de-activating the enzyme. These two factors combined have resulted in Factory C not achieving a very high removal. Now he could not talk about Factory A because he was not familiar with the process, but he could speak about Factory B. In this case when the process of defecation was started the procedure adopted by the process manager there was to heat his juices to between 160-180°F. He also had available three tanks. He decided instead of by-passing these tanks to make use of them as surge tanks, which he was still doing to this day. He therefore had the ideal conditions for removing a certain amount of starch and, as Dr. Douwes-Decker pointed out, he obtained a 42 per cent removal. He said he subsequently understood from Dr. Douwes-Dekker's remarks that the S.M.R.I. carried out an investigation whereby they made fuller use of the tank capacity giving about 20 minutes standing, whereas in fact the factory manager had only been using about 7, and thereby managed to increase the removal figures to something like 60 per cent. However this long period of residence would result in a loss of sucrose by inversion.

Dr. Douwes-Dekker explained that the S.M.R.I. had conducted two investigations, one at Z.S.M. and the other at Melville (Factory B). At Z.S.M. weekly periods of normal work were alternated with weeks where the juice was heated to 70-75°C and kept at that temperature for 20-25 minutes, in open tanks. Starch was determined in hourly-taken samples of mixed and clarified juice, care being taken that after sampling, starch decomposition was interrupted by pouring the sample into absolute alcohol. Composite daily samples of sugar were also tested for starch. It was found that under "normal" conditions, approximately 43 per cent of the starch in mixed juice was removed in the clarification process, which figure increased to approximately 71 per cent when the Nicholson-Horsley process was applied. Simultaneously the starch content of the raw sugar dropped from approximately 417 to 240 ppm., i.e., by 42 per cent. In the tests at Melville the retention time of the juice heated to 70-75°C was only 10-14 minutes. Here it was found that in two periods of one week that when the factory was working "normally", 48.5 and 55.3 per cent respectively of the starch in mixed juice was removed during clarification, whilst in the week that the juice was retained at 70-75°C, the removal was 65 per cent. Simultaneously, the starch content of the sugar dropped by 21 per cent. The lesser effect of the Nicholson-Horsley process at Melville was thought to be due to the shorter retention time. The interesting point was, however, that whilst the S.M.R.I. investigations indicated that normal defecation was capable of removing

45-55 per cent of the starch present in mixed juice, Mr. Boyes had found only 14 per cent at factory C. It would seem that further work on the question was desirable.

Mr. Boyes said Dr. Douwes-Dekker mentioned the fact that the samples were snatch samples only. He thought that in determining starch it was probably advisable to stick to snatch sampling rather than try and collect composite samples over a long period. The results were actually determined from a great number of analyses and not just the result of a couple of determinations. From the results given for different varieties and over a short space of time there was a variation in mixed juice between 175 parts per million up to a maximum of 625 parts per million, so he agreed with Dr. Dekker that figures of removal based on mixed juice and clear juice are rather difficult to call accurate. At Factory A it was quite possible that during the testing of mixed juice, the starch fluctuated very considerably and when compared with the sample of clear juice taken 3 to 4 hours later the comparison would not be strictly true. In comparing removals he had tried to use sugar figures to indicate removal. Referring to Dr. Dekker's fourth question, Mr. Boyes said he was afraid the Table VIII was in error. The figure of 710 was not meant to indicate starch in B sugar. It was meant to indicate starch in total sugars. This was the S.M.R.I. figure and the same figure appeared in Table IX.

Mr. Alexander said that in a number of tests done at Darnall in 1955 when sulphitation was being used, the percentage of the removal of starch was about 17 per cent. Quite a number of samples contributed to obtaining this average.

Mr. A. C. Barnes said that the varieties from which these figures were obtained were more closely related to wild varieties than those grown in the West Indies where no process difficulties caused by starch were experienced.