CONTINUOUS SAMPLING AND ANALYSIS OF CLEAR JUICE, FILTRATE RETURN AND SYRUP AT DARNALL

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Abstract

Darnall sugar mill sometimes experiences unusual purity profiles from mixed juice through to syrup and a system was established whereby continuous sampling of clear juice, filtrate return and syrup could be undertaken, thus enabling the SMRI to investigate the authenticity of these profiles. The samples were analysed hourly and were also composited in a deep freeze at hourly intervals over eight hour periods. In contrast with the mill laboratory hourly catch samples, the SMRI continuous hourly and eight hourly samples gave good purity profiles from mixed juice through to syrup.

It was established that the analyses performed by the mill laboratory agreed well with those of the SMRI and poor sampling of filtrate return is probably the cause of the irregular purity profiles found by the mill laboratory.

The scatter of purity values obtained on the hourly samples compared with those obtained on the eight hourly composites was ± 0,60 units for clear juice and syrup, indicating that these products could be composited and analysed less frequently than is being done at present by the mill laboratory.

Introduction

The main anomaly found at Darnall concerning the purity profiles of products is that the purity of the filtrate return (F) is often found to be greater than the purity of the clear juice (CJ). The SMRI investigated this phenomenon and endeavoured to determine whether it was due to incorrect sampling and/or analysis. A system of continuous sampling of clear juice, filtrate return and syrup (S) was installed at the same points used by the factory, and an investigation was also carried out to determine the effect of compositing portions of the hourly continuous sample in the deep freeze for subsequent analysis after eight hour periods. In addition, comparisons of the SMRI results with those obtained by the factory and the Sugar Industry Central Board (SICB) laboratories were made. The SMRI results were used to establish the natural fluctuations in the individual products with time and, if found acceptable by the processing staff, to demonstrate the possibility of doing less frequent analyses on clear juice, filtrate return and syrup than is done at present.

Installation of Sampling Points

In order to have continuous samples of clear juice, filtrate return and syrup at the same points where the catch samples are taken, T-pieces were fitted so that the factory staff could sample in the normal way from one arm of the 'T', while continuous sampling was done on the other arm. The continuous sampling was done through solenoid valves with the open/close cycle time regulated to give approximately one litre of sample per hour. Since these streams are hot and the possibility of evaporation is high, the clear juice and filtrate return were passed through small cooling coils before entering the sampling receptacles which were closed and placed in waterbaths where cold water at about 15 to 20°C circulated. The syrup sampling system was modified so that the cooling coil was omitted and a known weight of water was placed in the receptacle before starting to collect the sample, thus eliminating the problem of the hot syrup crystallising in the cool container. Because the syrup was partially diluted, all that was necessary after the sampling period was over, was to add the amount of water required to make the one in four dilution needed for analysis.

Preparation and Analysis of Samples

Mercuric chloride preservative solution was added to the sample receptacles. Each hour, sub-samples from the receptacles were sealed into plastic sachets and also injected by means of a syringe into sealed blood transfusion bottles in which composite samples were accumulated over eight hour periods. These bottles were sealed to prevent any evaporation and condensation when the sachets and bottles were subsequently stored in a deep freeze. Sub-samples of mixed juice (MJ), which is sampled continuously and analysed on an hourly basis by the SICB, were prepared, composited and stored in the same way. All of the samples were analysed at the SMRI for pol and brix, while reducing sugars were determined on the 8-hour composite samples only.[1]

Results and Discussion

In order to establish the validity of preparing composites of the different products over eight hour periods, the average purity values for the hourly analyses of each product were compared with those obtained for the corresponding eight hourly composite. The results are given in Table 1, and show statistically no difference between the hourly means and the eight hourly composites at the 2,5% level of significance.

It could thus be accepted that the preparation and analysis of eight hour composites are allowed provided the procedure described above is followed. Perusal of the analysis figures of the eight hourly and hourly composites shows normal purity profiles from mixed juice through to syrup (see Table 1). Only the filtrate return showed a drop in purity compared with those of the mixed juice and clarified juices. Further support of the normal purity patterns found as shown by pol/brix analysis was also present in the reducing sugars/brix ratios of the different products (see Table 2). The drop in purity of filtrate return compared with that of clear juice is to be expected as factors such as pH, temperature of the wash water, quantity of wash water and bacterial activity could have a detrimental effect on the quality of the filtrate return.[2,3] Possible reasons for the apparent increase in the purity of filtrate return reported by the Darnall laboratory are not immediately clear. Checking the mill laboratory analyses against the SMRI analyses for the various products (see Table 3) showed that the laboratories agreed well, in spite of the fact that different methods of sampling were used. The most likely source of the error in the Darnall analysis is the sampling. It was noted that the outlet of the sampling point of the filtrate return was below the level of the pipe which carries the filtrate back to the tank and suspended solids containing a great deal of sand tended to collect in the sampling pipe. If this pipe is not thoroughly flushed with fresh filtrate prior to taking the sample, possible degradation of sugars may lead to inflated purities.

Table 1

Comparison of hourly mean and composite purities over eight hours

Run No.	Product							
	Mixed Juice		Filtrate Return		Clear Juice		Syrup	
	Mean	Composite	Mean	Composite	Mean	Composite	Mean	Composite
1	83,15	83,29	82,11	81,88	83,81	83,83	84,13	84,23
2	83,64	83,84	83,49	82,93	84,68	84,59	85,07	84,81
3	83,62	83,53	82,86	83,67	84,16	84,55	84,88	85,15
4	84,50	84,22	84,45	83,68	84,64	84,66	85,33	85,16
5	85,75	85,95	85,90	85,78	86,35	86,35	86,53	86,52
Average	84,13	84,17	83,76	83,59	84,71	84,80	85,19	85,17

Mixed Juice : $t_{0,05;5} = -0,34 < 2,78$ = Critical Value
Clear Juice : $t_{0,05;5} = 0,64 < 2,78$ = Critical Value
Filtrate Return : $t_{0,05;5} = -0,99 < 2,78$ = Critical Value
Syrup : $t_{0,05;5} = 0,15 < 2,78$ = Critical Value

Table 2

Reducing sugars/brix ratios over eight hours

Run No.	Product			
	Mixed juice	Filtrate return	Clear juice	Syrup
1	6,72	5,31	5,51	5,44
2	6,51	5,09	5,11	5,45
3	4,40	4,08	4,13	4,32
Average	5,88	4,83	4,92	5,07

Table 3

Comparative averages of SMRI and Darnall analyses

Product	Number of Analyses	SMRI			Darnall		
		Pol	Brix	Purity	Pol	Brix	Purity
Clear Juice	44	9,58	11,34	84,48	9,61	11,39	84,37
Filtrate Return	34	6,50	7,73	84,09	6,61	7,80	84,74
Syrup	7	56,57	66,60	84,94	56,42	66,58	84,74

Clear Juice : $t_{0,05;43} = 1,87 < 2,02$ = Critical Value
Filtrate Return : $t_{0,05;33} = 0,66 < 2,04$ = Critical Value
Syrup : $t_{0,05;6} = 0,04 < 2,45$ = Critical Value

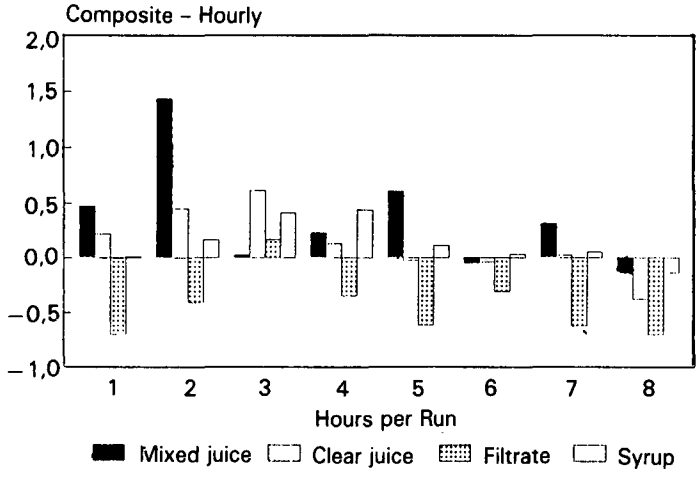

FIGURE 1 Average purity differences over eight hours.

When the individual hourly composite purities of the products are compared with the purities obtained from the eight hourly composites (see Figure 1), it can be seen that the deviation of the hourly purity from the eight hour composite purity for clear juice and syrup is ± 0,60 units. This indicates that a reduced frequency of analysis could be adopted for those products, ie an analysis on the eight hour composite only. However this will depend on the requirements of the processing staff. The varying nature of the mixed juice and filtrate return, in terms of quality, would not warrant a frequency of less than the existing once per hour analysis.

Conclusions

During this investigation no apparent destruction of sucrose from mixed juice through to syrup was observed. This was indicated by purity as well as by reducing sugars/brix ratio measurements. The pol and brix analyses performed by the Darnall mill laboratory agreed well with results obtained by the SMRI for the same products, and it is suggested that the high purities of filtrate return observed by the Darnall laboratory could be the result of faulty sampling.

Comparison of the hourly sample purities with the eight-hourly composite purities indicates that the frequency of analysis of clear juice and syrup can be reduced to once per shift. This, however, will depend on whether a deviation of ± 0,60 units of purity for clear juice and syrup is acceptable for process control. Because of the inherent variation in the composition of mixed juice and filtrate return, a reduced frequency of analysis cannot be considered for the latter products.

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