FLOCCULANT ASSESSMENT USING A PORTABLE BATCH SETTLING KIT

By G. R. E. LIONNET and A. B. RAVNÖ
Research and Development, Huletts Sugar Ltd., Mount Edgecombe

Abstract

The reproducibility of settling tests performed on a simple, portable laboratory settling kit has been evaluated statistically and found to be acceptable. A range of partially hydrolysed polyacrylamide flocculants, having differing degrees of hydrolysis, was tested and the optimum degree of hydrolysis for Mount Edgecombe juices was found to lie between 45 and 50%.

Introduction

The clarification step in raw sugar manufacture is designed to remove insoluble suspended impurities from the mixed juice. This suspended matter comprises primarily field soil, organic debris, denatured proteins and precipitated calcium phosphates.[1, 2] The range of particle sizes extends from those visible to the naked eye right down to those in the colloidal region. The smaller particles settle out extremely slowly and in fact may be kept in suspension by convection currents or Brownian motion.

The stability of these colloidal suspensions and their tendency to coagulate is to a large extent dependent upon the surface charge characteristics of the colloidal particles. The suspended matter in cane juice is predominantly of negative charge and it has been found that flocculation can be induced by a class of compounds known as anionic polyelectrolytes. Over the past 10-15 years the use of partially hydrolysed polyacrylamides has gained wide acceptance in the sugar industry.[1, 2, 3, 4, 5] A considerable amount of research has been done on the structure and mechanism of this floc formation.[2,3,4] It has been shown that optimum performance is highly dependent upon the correct matching of the flocculant properties (e.g. molecular weight and degree of hydrolysis) with the charge characteristics of the colloids present. Since the latter can vary widely with cane age, variety, district and time of year, it is to be expected that the optimum flocculant type will also change during the season.[1]

Over the past few years a consistent pattern of clarification difficulties has been experienced at Mount Edgecombe, particularly during dry periods in the second half of the season. The present study was undertaken in an attempt to monitor the seasonal variations in juice behaviour and to assess the relative effectiveness of a range of flocculants having differing degrees of hydrolysis.

Experimental

Batch settling tests

A portable settling kit designed by Fletcher & Stewart (F & S) was used for the laboratory settling tests. Tubes of 3,5 cm diameter with a settling depth of 40 cm were encased in an air bath kept at ± 98°C by small strip lamps. The kit was supplied with a small slow speed stirrer to apply shear to the mud layer, but unless otherwise stated all tests were done without the stirrer.

Throughout the season an average of four separate settling tests was undertaken daily using a sample of factory juice which had already been heated, flashed and limed. In each case the sample was split and settled simultaneously, one half with no flocculant and the other with the flocculant being tested. Readings of the respective mud levels were taken after 0,5/1,0/1,5/2,0/2,5/3,0/5,0 and 18,0 minutes. Unless specifically stated otherwise, all tests were conducted at a dosage rate of 2 ppm flocculant on limed juice.

At the outset a series of tests was undertaken in order to establish the experimental reproducibility of this batch settling technique and its ability to detect changes in juice behaviour. Details of these tests are given in the Appendix.

Analytical procedure

The initial settling rate (ISR) in cm/min was determined graphically from the slope of the initial linear portion of the settling curve of mud level against time.

The mud volume after 18 minutes (MV_{18}) was read off directly, whilst the final equilibrium mud volume (MV_{∞}) was obtained from the intercept on a plot of mud volume % vs $^1/t$, where t is time in minutes.

The sludge volume index (SVI) is a measure of the mud density and is defined as follows:

$$SVI = \frac{\text{mud volume @ 18 mins (m}\ell\text{)}}{\text{dry mud weight (gm)}}$$

Dry mud weights were obtained by filtering the tube contents after a settling test and drying the residue to constant weight at 105°C.

Juice turbidity was measured at a wavelength of 800 nm in a 1 cm cell using samples of the supernatant juice after 18 minutes.

Flocculant testing

The flocculants tested were limited to only two commercial brands, which had degrees of hydrolysis covering the range 20-65%. Details of the individual flocculant types are given in Table 1.

Separan AP273 was also included for reference purposes since this is the standard flocculant used by most South African factories.

TABLE 1
Details of flocculants tested

Flocculant type	Average molecular weight	Measured % hydrolysis
Superfloc—		
A130	6×10^6	47
A137	5×10^6	50
A150	5×10^6	65
Magnafloc—		
LT25	7×10^6	26
LT26	7×10^6	38
LT28	10×10^6	21
Separan—AP273	$\times 10^6$	40

The molecular weights listed are those which were quoted by the manufacturers. The nitrogen content of the individual flocculants was determined by the standard Kjeldahl method and used together with the known chemical structure to calculate the degree of hydrolysis.[3] Fairly large discrepancies were noted, on occasion, between the measured degree of hydrolysis and that claimed by the manufacturers.

Results and discussion

Settling test procedure

The F & S portable batch settling kit, which was used throughout the present study, differs from that used by many other investigators,[6] in that the settling tubes are enclosed in a heated air bath instead of a more conventional water bath. This apparatus was recently rejected by Murray and Shephard on the grounds that the temperature difference between the juice and the surrounding air caused persistent convection currents and other spurious effects.[5] In a much earlier study, Schmidt reported that good, reproducible results were obtained even when using an unheated air bath,[7] although he did concede that it was advisable to minimise the temperature difference between the juice and the surrounding air.

This kit has also been used extensively by Whayman and his co-workers in Australia,[3, 4] who state specifically that convectional effects in the settling tubes were not observed.[3] During the present study no problems were encountered with convection currents provided that each settling tube was stoppered with a large bung as soon as it had been filled with juice.

Certain aspects of this study were undertaken in close collaboration with Murray and his co-workers at the Sugar Milling Research Institute (SMRI). In order to aid the comparison of future results it was decided to check the reprodicibility between the portable F & S kit and the more sophisticated SMRI settling apparatus, which is described elsewhere.[5] The results of these tests are shown in Table 2, which lists the actual % mud volumes against time in minutes, both tubes being filled to the same height.

TABLE 2

Comparison of F & S and SMRI settling kits

Time	Test 1		Test 2	
	SMRI	F & S	SMRI	F & S
0,0	100,0	100,0	100,0	100,0
0,5	87,0	90,0	72,0	76,0
1,0	77,0	76,0	40,0	40,5
1,5	64,0	65,5	27,0	27,5
2,0	54,0	54,0	24,5	24,5
2,5	46,0	47,5	23,0	22,5
3,0	42,0	43,5	21,5	20,5
5,0	34,0	35,5	18,0	17,5
18,0	23,0	24,5	14,0	12,5
ISR (cm/min)	9,7	9,3	23,2	21,3
SVI (m*l*/gm)	30	26	27	26

Statistically these results can be considered to display no significant differences.

A considerable amount of work was also done to assess the reproducibility of the settling procedure using the F & S kit as well as its ability to detect changes in juice settling characteristics. These results are presented in detail in the Appendix. It was concluded that both the reproducibility and sensitivity of the method were adequate.

Flocculant assessment

For some unknown reason Mount Edgecombe did not experience its traditional clarification difficulties during the 1975/76 season and if anything clear juice turbidities improved as the season progressed. The results from the batch settling tests were remarkably constant in most cases. No major seasonal trends were detected in flocculant performance and the flocculant efficiency ranking remained virtually unchanged throughout the season. In view of this all the individual results were grouped up and the seasonal averages are presented in Table 3.

TABLE 3

Flocculant performance

Flocculant	ISR (cm/min)	MV_{18} (%)	MV_{∞} (%)	Turbidity @ 800 nm	SVI (m*l*/gm)
Superfloc—					
A130	34,9	16,6	15,2	0,24	24
A137	29,9	18,0	15,5	0,26	25
A150	7,5	23,0	16,5	0,41	32
Magnafloc—					
LT25	14,0	20,9	16,6	0,40	26
LT26	12,6	20,6	16,8	0,38	29
LT28	5,1	26,8	17,0	0,66	37
Separan					
AP273	25,9	17,8	15,0	0,36	25
No flocculant	5,2	25,1	16,3	0,58	31

It is readily apparent that Superfloc A130 was consistently the most efficient flocculant tested, giving the best initial settling rate and juice clarity coupled with the lowest mud volumes. Both Superfloc A130 and A137 appear superior to Separan AP273 in the present study.

In line with previous studies,[3, 4, 5] the relative clarification efficiency, as expressed by initial settling rates and juice turbidities, has been plotted against the degree of hydrolysis for the various flocculants (see Figure 1).

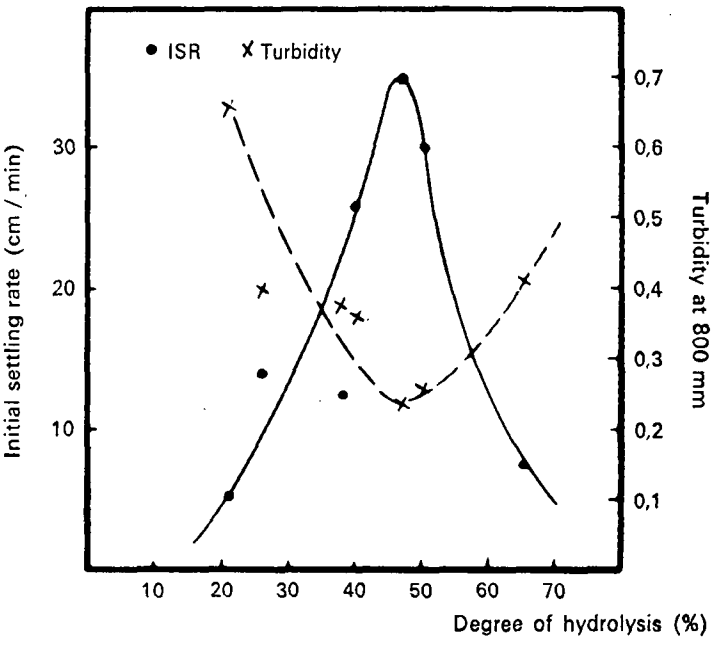

FIGURE 1 Flocculant efficiency.

These results indicate that the optimum degree of hydrolysis for Mount Edgecombe juice lies between 45 and 50%, which is considerably higher than the range of 22-33% given by Whayman *et al* for the Australian industry.[3, 4] This is not entirely unexpected since it has been established that the higher

the negative zeta potential of a juice, the greater the anionicity (or degree of hydrolysis) of the optimum flocculant.[3] Murray and Shephard have already found that the zeta potential of typical Mount Edgecombe juices attains higher negative values than those quoted for Australia.[5]

Conclusions

It has been established that the reproducibility and sensitivity of the portable F & S laboratory settling kit is adequate for assessing juice settling characteristics and the effect of flocculants.

For a flocculant dosage rate of 2 ppm on limed juice, the optimum degree of hydrolysis for Mount Edgecombe juices is between 45 and 50%. However further work is required to assess the effect of various levels of flocculant addition and any seasonal variations which may arise in flocculant performance.

Acknowledgements

The authors are indebted to Messrs D. Falconer, R. Scott and P. Wagener, all of Huletts R & D, for the many hours of hard labour which went into obtaining the data, to the SASA Experiment Station for doing the flocculant Kjeldahl analyses, and to the Board of Directors of Huletts Sugar Ltd for permission to publish this paper.

REFERENCES

1. Hale, D. J. and Whayman, E. (1967). "AP273 and other settling aids", Proc. QSSCT **34**, 301-306.
2. Bennett, M. C. (1975). "Flocculation technology in sugar manufacture". Proc. Societe de Technologie Agricole et Sucriere de Maurice, 7th Congress; In press.
3. Crees, O. L., Hale, D. J., Whayman, E. and Willersdorf, A. L. (1973). "Flocculants in raw sugar production". Proc QSSCT **40**, 239-250.
4. Whayman, E. and Crees, O. L. (1974). "Mechanistic studies on cane mud flocculation". ISSCT Proc **15**, 1175-1182.
5. Murray, J. P. and Shephard, G. S. (1975). "Some aspects of flocculant use in clarification and filtration". SASTA Proc **49**, 53-58.
6. Burgess, I. G., Beardmore, R. H., Fortescue, G. E. and Davis, C. W. (1962). "Development and application of a laboratory clarification test". Proc ISSCT **11**, 920-928.
7. Schmidt, N. O. (1953). "The settling of defecated sugar juices". Proc ISSCT **8**, 729-742.

APPENDIX

Reproducibility of settling test data

In order to test the internal reproducibility of the method, a number of sets of settling tests were performed in quintuplicate using 2 ppm of Separan AP273. Each individual settling was analysed and the average results of four such groups of tests are shown in Table 4 together with their relevant 95% confidence limits.

TABLE 4

Reproducibility of settling test data

Test No.	ISR (cm/min)	MV_{18} (%)	MV_{∞} (%)	SVI (mℓ/gm)
1	25,4 ± 1,1	15,0 ± 1,2	12,5 ± 0,6	21,0 ± 0,7
2	14,4 ± 0,9	16,6 ± 1,4	13,1 ± 0,8	29,3 ± 0,9
3	15,2 ± 1,5	16,2 ± 1,0	12,7 ± 1,0	31,6 ± 1,0
4	12,2 ± 0,6	16,5 ± 0,9	12,4 ± 1,3	30,5 ± 0,2

Statistically these results indicate that there is a 95% chance that a single settling test will yield results which will not differ by more than about 7% from the mean value for a full set of five replicates. This shows very good reproducibility for the method and all further work was based on single settling tests. These were usually conducted in pairs, one with and the other without flocculant.

Sensitivity of the method

If a batch settling test is to be used to monitor juice behaviour then the variability between replicates on the same sample must be less than the hour-to-hour or day-to-day variations for the parameters under observation. This can readily be tested by means of the Analysis of Variance technique. This type of statistical treatment was applied to:

(*a*) the results listed in Table 4, where each of the individual test sets was obtained at 90 minute intervals on the same day.

(*b*) the weekly averages for the limed juice tests without flocculant addition over a 15 week period (*Note:* Between 10 and 15 individual reference samples of limed juice were analysed each week).

These results have shown, with 99% certainty, firstly that the hour-to-hour variations in ISR and SVI may be ascribed to real changes in juice behaviour and secondly that the week-to-week variations are significantly greater than those occurring from day-to-day within any one week. Hence it was concluded that the batch settling technique is sufficiently sensitive to monitor changes in juice settling characteristics.