

AN ATTEMPT TO RELATE FLOC FORMATION WITH SOME COMPONENTS OF NATAL VERY HIGH POL SUGARS

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Abstract

Very High Pol (VHP) sugars, delivered for export by the manufacturing mills to South African Sugar Terminals, were tested for flocculation properties. The results were subsequently compared with the colour, gum and nitrogenous matter content of the sugars in question. The resulting comparative study shows that there is correlation in sugars made by the individual mills, but no general significant correlation could be found.

Introduction

Traces of impurities in the refined sugars react in the low pH environment of carbonated beverages to form floc with high area to volume ratio. No simple explanation has been given for the relatively high proportion of inorganic components (mainly silica) in the floc. Pre or post precipitation processes may be involved^{1, 2, 3, 4}.

Many refiners are of the opinion that the floc-forming behaviour of the refined product may be influenced by the nature and origin of the raw sugar used for its manufacture⁵.

It was generally considered in the past that no useful results could be obtained by examining the floc-forming properties of raw sugars, as the impurities responsible for floc generation were present in a very much higher concentration and would, therefore, always produce very heavy floc under test conditions.

This investigation was initiated by a casual test to check the correctness of the above contention and consequent surprising results which indicated that a fairly wide range of floc-formation properties could be detected in VHP sugars originating from the various supplying mills.

It was felt therefore that, in view of the observed differences in floc formation, it would be of interest to examine, on a comparative basis, the consignments of bulk sugar delivered to the Terminal during the season. Up to 15 sugar mills supply raw sugar to the Terminal for export, and routinely composited weekly samples are available for testing.

It was further noted as a general observation, that the higher the sugar colour, the higher was its tendency to form floc. However when colour readings of the tested sugars were compared with the floc results, no obvious relations could be detected. It was subsequently decided that nitrogenous material should be determined, in the hope that it would provide further clues (some of the colour could be due to protein material)^{8, 11}.

A similar argument applied to gum determination. Gums as determined in sugar probably include some of the polysaccharides^{9, 13} which coacervate with proteins to form floc^{10, 12}. It was hoped that, when the values of these components were examined graphically against the time of the season, some pattern would emerge which might assist in the study of floc occurrence.

Test Procedure

Season's weekly composited samples of 15 mills delivering bulk raw sugar to SA Sugar Terminals were affinated with

a saturated sugar solution at ambient temperature, to remove non-crystalline coating, and examined for the following :

- (a) colour
- (b) gums
- (c) floc formation.
- (d) "protein" content.

The comparative results were examined by plotting the results for each mill on a monthly basis. The nests of graphical curves for each component were examined for trends, correlations and patterns (Figures 5, 6, 7 and 8).

Test Methods

(ab) Starch, colour and gum determinations were carried out according to the procedures listed in the Laboratory Manual for SA Sugar Factories¹⁴.

(c) Floc formation tests were carried out as per standard (ten day) Coca Cola Test, using distilled water. Comparisons were made on daily observations of each sample. Arbitrary units of (10 - number of days taken to form floc) were recorded for each sample¹⁵.

(d) "Protein" determination : For the purpose of this investigation it was decided to determine the total macromolecular organic nitrogen in VHP sugars, and to recalculate it to crude protein value. It was hoped that perhaps this rough measure of "protein" content might provide a clue when examined in context with other components. The above procedure was carried out as follows :

- (i) 50 g sugar portions were dissolved in 150 ml distilled water and dialysed for 11 hours in a slowly-agitated and continuously flushed aqueous medium, to remove sugars and other low molecular-weight matter.
- (ii) The resulting concentrate (collected in a cellulose tube with a cut-off of 10 000 to 12 000 molecular weight) was subjected to Kjeldahl digestion. Aliquots of the resulting solution were treated with excess of sodium hydroxide and the ammonia distilled off and titrated. Precautions were taken to test the reliability of the dialysis and subsequent quantitative analysis with known standards. The final results were converted to "standard protein content" by multiplying the ppm nitrogen results by 6,25^{16, 17, 18, 19}.

Results

Initially the analyses were plotted for each mill (Table 1), but no significant correlation could be established. Examination of a typical graph e.g. NB (Figure 1) indicates that, at some periods during the season, a rough visual correlation appears to exist between gums, colour and protein. This rough relationship can also be traced to a greater or lesser extent in sugars from other mills. Floc curve, however, does not appear to have pronounced configuration similarity to other components.

Major alterations in VHP sugar impurities shown on the graph, may indicate seasonal changes in the incoming sugar cane.

TABLE 1

Analyses of affinated V.H.P. sugar received from individual mills during 1978/9 season

| 1978 | | | | | | |
|-------------|-------|-------|-------|-------|-------|-------|
| Mill UF | July | Aug. | Sept. | Oct. | Nov. | Dec. |
| Colour . . | 0,04 | 0,06 | 0,05 | 0,07 | 0,08 | 0,09 |
| Gums . . | 1 000 | 1 425 | 1 050 | 1 400 | 1 350 | 1 450 |
| Floc . . | 5 | 5 | 5 | 5 | 6 | 6 |
| Protein . . | 116 | 73 | 86 | 96 | 67 | 41 |

| Mill FX | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,10 | 0,07 | 0,08 | 0,10 | 0,10 | 0,10 |
| Gums . . | 925 | 850 | 800 | 950 | 775 | 800 |
| Floc . . | 6 | 6 | 7 | 6 | 6 | 5 |
| Protein . . | 127 | 75 | 110 | 157 | 71 | 58 |

| Mill SZ | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,05 | 0,05 | 0,06 | 0,10 | 0,07 | 0,10 |
| Gums . . | 575 | 525 | 750 | 900 | 850 | 875 |
| Floc . . | 7 | 5 | 6 | 8 | 7 | 7 |
| Protein . . | 73 | 75 | 135 | 128 | 90 | 75 |

| Mill AK | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,10 | 0,12 | 0,12 | 0,12 | 0,13 | 0,16 |
| Gums . . | 750 | 800 | 750 | 825 | 775 | 850 |
| Floc . . | 6 | 5 | 7 | 7 | 7 | 6 |
| Protein . . | 132 | 128 | 145 | 158 | 131 | 78 |

| Mill GD | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,06 | 0,08 | 0,10 | 0,13 | 0,12 | 0,10 |
| Gums . . | 825 | 875 | 800 | 925 | 950 | 775 |
| Floc . . | 3 | 5 | 6 | 7 | 7 | 6 |
| Protein . . | 104 | 138 | 140 | 184 | 96 | 72 |

| Mill EN | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|-------|------|------|
| Colour . . | 0,10 | 0,10 | 0,11 | 0,10 | 0,12 | 0,11 |
| Gums . . | 650 | 700 | 700 | 1 075 | 775 | 875 |
| Floc . . | 4 | 4 | 5 | 5 | 5 | 5 |
| Protein . . | 82 | 112 | 150 | 119 | 96 | 69 |

Mill DL

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,05 | 0,06 | 0,07 | 0,09 | 0,08 | 0,09 |
| Gums . . | 700 | 650 | 675 | 825 | 700 | 675 |
| Floc . . | 7 | 5 | 7 | 7 | 7 | 6 |
| Protein . . | 94 | 110 | 137 | 152 | 85 | 69 |

Mill UC

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,08 | 0,10 | 0,11 | 0,13 | 0,10 | — |
| Gums . . | 650 | 550 | 700 | 900 | 900 | — |
| Floc . . | 4 | 4 | 6 | 6 | 5 | — |
| Protein . . | 132 | 130 | 171 | 225 | 125 | 106 |

Mill NB

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|-------|-------|-------|
| Colour . . | 0,07 | 0,08 | 0,09 | 0,14 | 0,12 | 0,12 |
| Gums . . | 650 | 625 | 800 | 1 075 | 1 175 | 1 375 |
| Floc . . | 2 | 3 | 5 | 6 | 5 | 5 |
| Protein . . | 116 | 131 | 195 | 158 | 125 | 131 |

Mill IL

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|-------|-------|-------|
| Colour . . | 0,06 | 0,08 | 0,09 | 0,14 | 0,11 | 0,11 |
| Gums . . | 550 | 600 | 750 | 1 150 | 1 050 | 1 275 |
| Floc . . | 2 | 3 | 5 | 7 | 7 | 6 |
| Protein . . | 75 | 104 | 138 | 153 | 98 | 81 |

Mill GH

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|-------|-------|-------|
| Colour . . | 0,09 | 0,12 | 0,11 | 0,15 | 0,13 | 0,16 |
| Gums . . | 750 | 750 | 825 | 1 000 | 1 000 | 1 125 |
| Floc . . | 7 | 5 | 7 | 8 | 7 | 8 |
| Protein . . | 143 | 135 | 181 | 181 | 119 | 106 |

Mill UK

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|-------|------|
| Colour . . | 0,09 | 0,12 | 0,11 | 0,11 | 0,12 | 0,14 |
| Gums . . | 725 | 725 | 925 | 925 | 1 050 | 950 |
| Floc . . | 6 | 5 | 6 | 6 | 7 | 6 |
| Protein . . | 142 | 184 | 266 | 175 | 197 | 125 |

Mill TS

| | July | Aug. | Sept. | Oct. | Nov. | Dec. |
|-------------|------|------|-------|------|------|------|
| Colour . . | 0,11 | 0,16 | — | 0,17 | 0,14 | 0,14 |
| Gums . . | 850 | 850 | — | 945 | 775 | 975 |
| Floc . . | 8 | 7 | — | 8 | 8 | 7 |
| Protein . . | 170 | 231 | — | 260 | 166 | 142 |

On NB graph, this pattern is noticeable in September and mid-October 1978. If a similar graph for UC (Figure 2) is examined, it is noted that, although NB and UC mills are situated in close proximity and presumably receive cane from neighbouring areas, the patterns do not coincide and the only symmetrical alignment is observed in August 1978.

Similar non-related patterns were noted in VHP sugars received from other mills. With the exception of UF (Figure 3) it appears that high colours are generally associated with high gum content, but not necessarily with high protein concentration. This indicates that the coloured material may not

be of nitrogenous nature. The second exception is IL (Figure 4) where, surprisingly, there is a rough correlation between all the investigated components throughout the season.

When separate components for all mills are plotted together, a rough seasonal pattern emerges, but again there is no apparent interrelationship between the components.

Multiple linear regression analysis with the aid of ICL 2903 computer 20 indicated that :

- (1) "The floc values are positively influenced by the amount of proteins and/or gums. The exact values of the correlation co-efficients are rather uncertain."

- (2) The time-of-season effect was only significant at the 1% level for the 1978 results, indicating a peak around the beginning of November.
- (3) The mills tend to have their own particular levels of floc values which can be explained only partly by the other constituents in their respective sugars (perhaps due to differences in process, equipment, cane type and climate).

Further consideration was given to the report¹ that more than 60% of the mass of floc has been found to be SiO₂. This is a significant proportion of the floc and, regardless of the mechanism of its inclusion, the proportion of SiO₂ in the beverage must obviously be taken into consideration.

At this stage it may be pertinent to consider the fact that about 85% of the carbonated acid beverage consists of water, which is usually obtained from town mains. If the silica content of such water varies significantly, it may in turn affect the floc behaviour of the beverage made at that time, regardless of the properties of sugar used for its manufacture.

A study of analysis of domestic water in Durban⁶ reveals that SiO₂ content of the main supply (Nagle water) varied from a minimum of 4 ppm in March 1978 to 18 ppm in October 1978. The lesser source of supply (Shongweni water) shows SiO₂ variation of minimum 1 ppm in January 1978 to maximum 10 ppm in December 1978⁷.

FIGURE 1
NB 1978

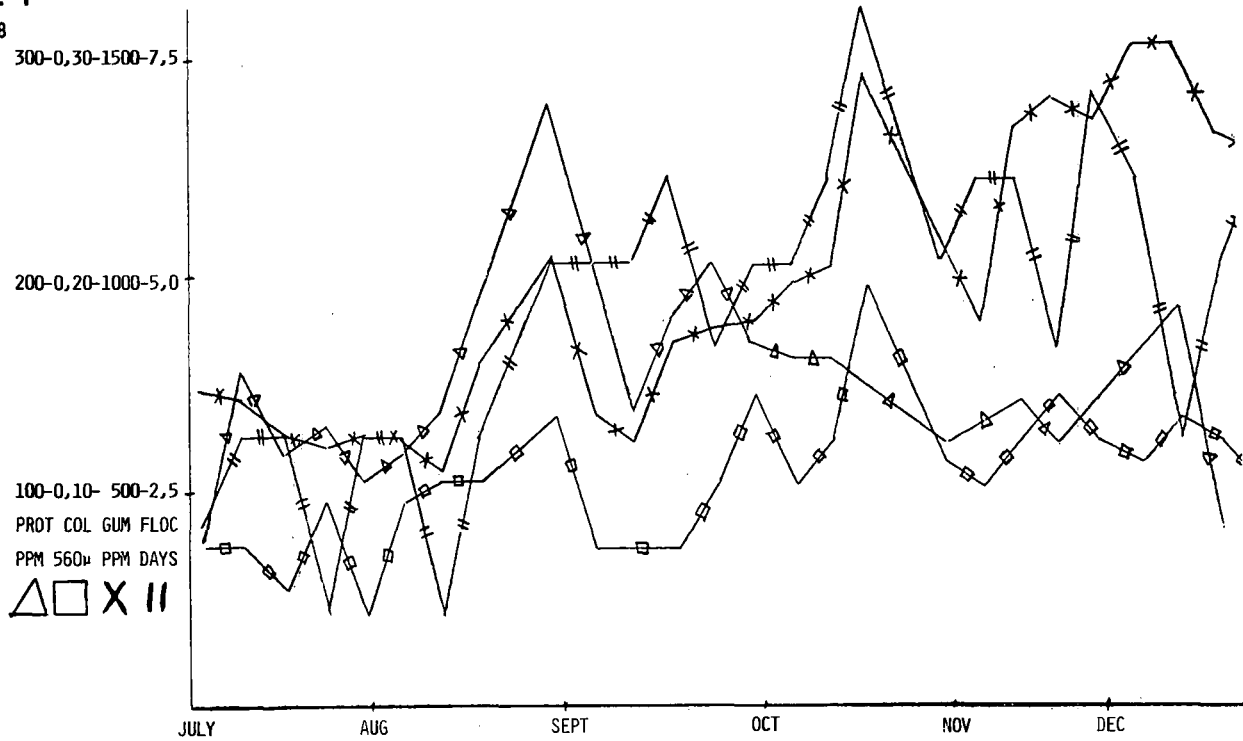


FIGURE 2
UC 1978

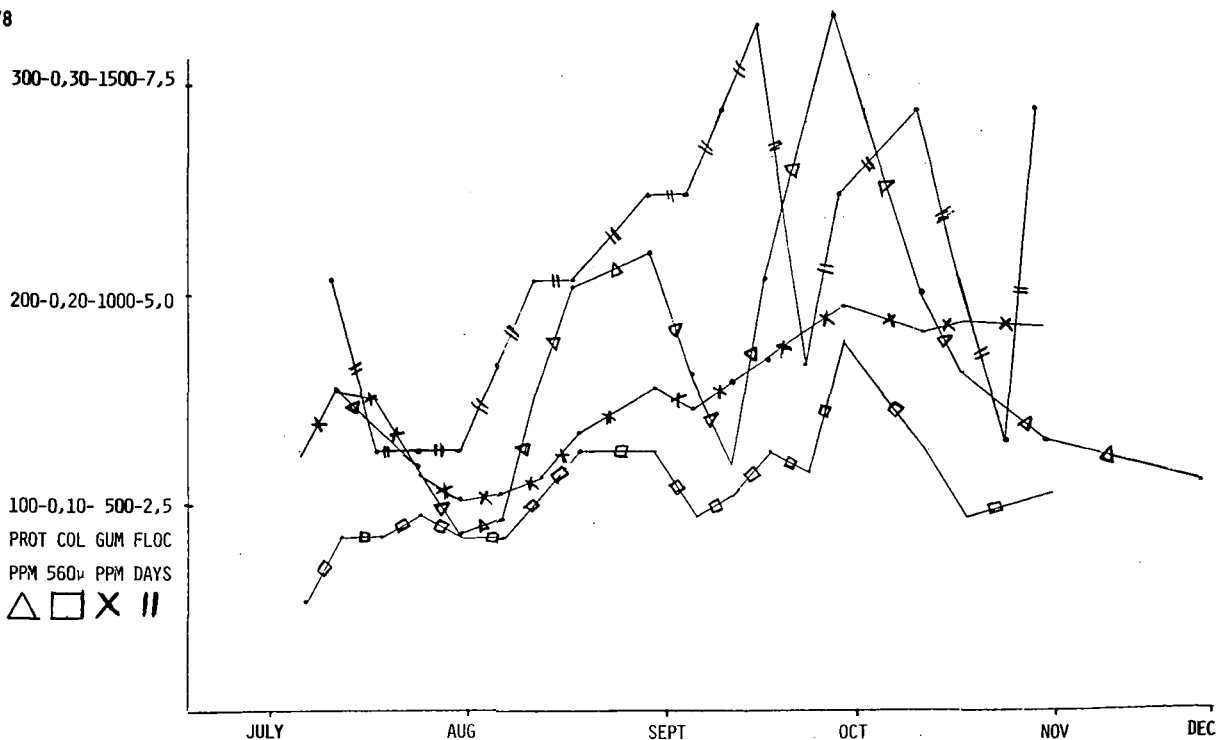


FIGURE 3
UF 1978

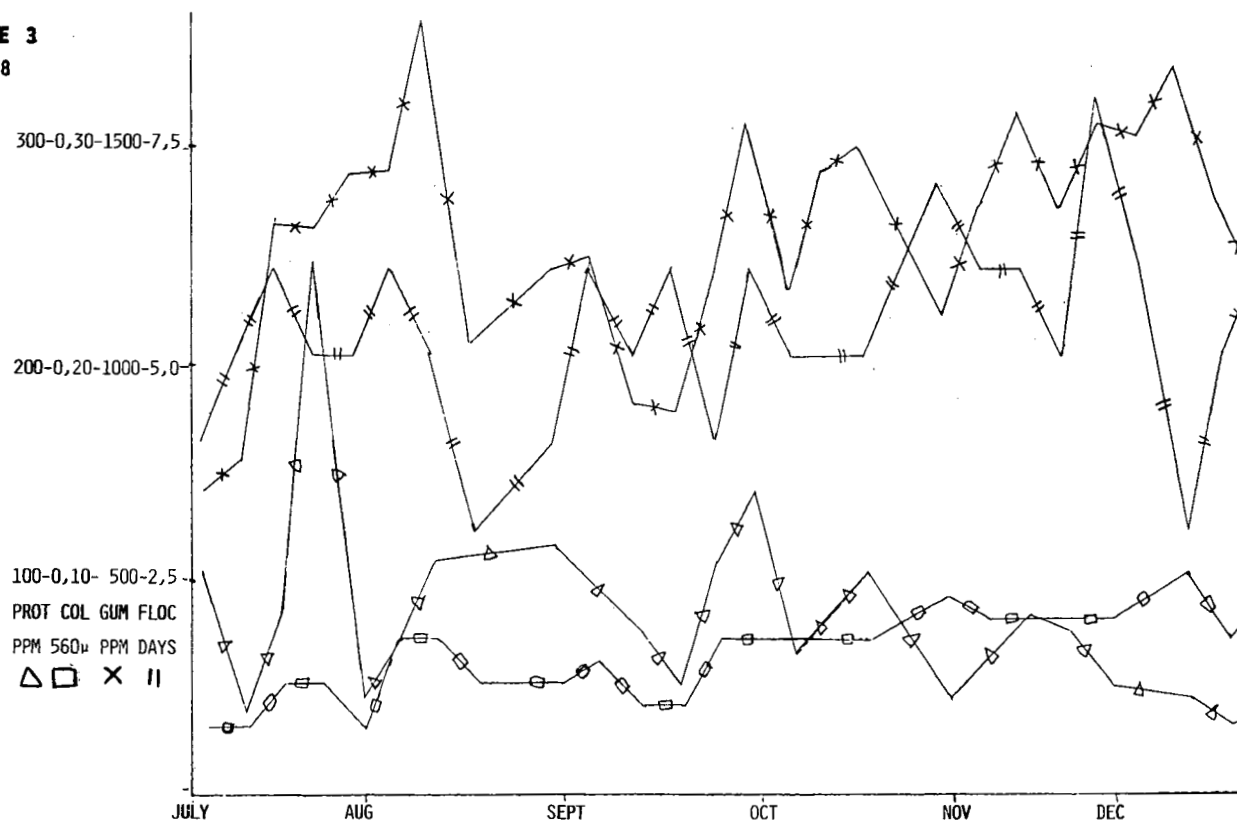
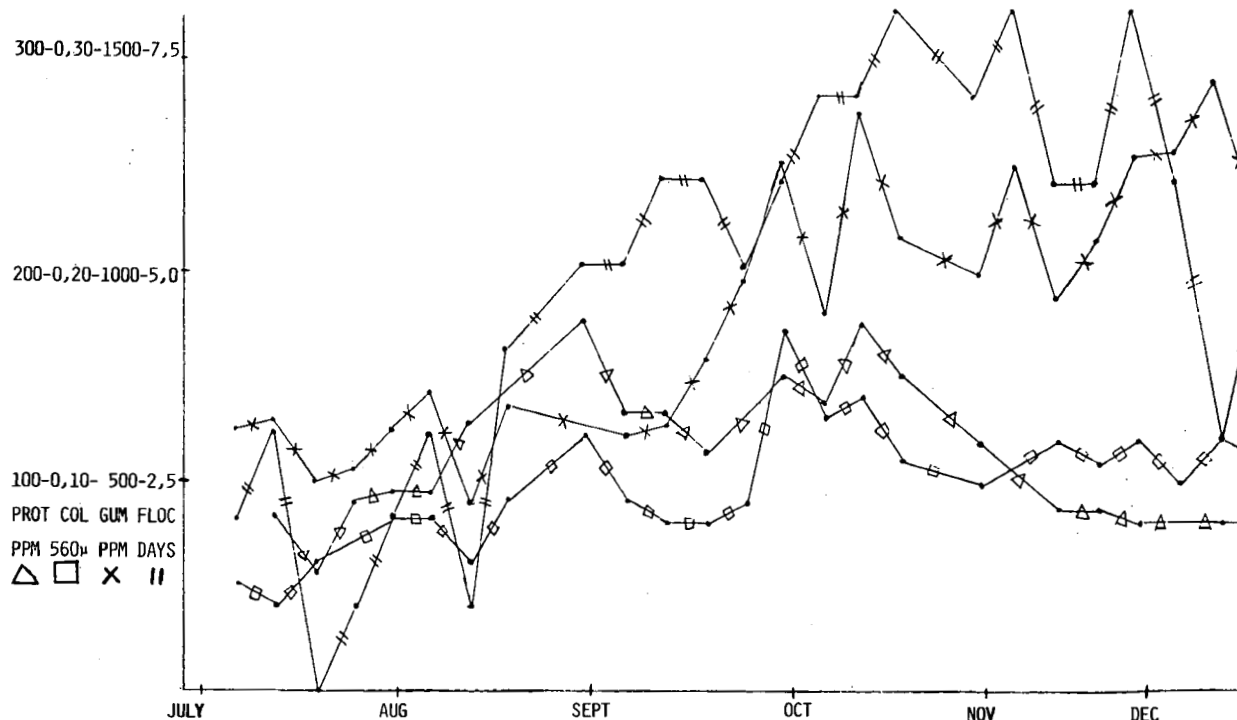


FIGURE 4
IL 1978



The concentration and composition of mineral fraction of potable waters may vary seasonally as shown above, and no doubt occurs in other parts of the world. It may thus be asked whether the floc behaviour of a "soft drink" should be solely ascribed to its sugar component or whether its water fraction plays a significant part in it.

Summary and Conclusions

Although VHP sugars received from various mills have been found to differ in respect of floc-forming tendency, there is no obvious relationship with the gum, colour and nitrogenous content. It is possible that the individual process/

equipment differences and perhaps geographical location of sugar cane used, may be responsible for the variable patterns noted in respect of impurity concentrations and also flocculation phenomena in VHP sugars received in bulk at the S A Sugar Terminals in Durban.

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FIGURE 5
COLOUR 1978

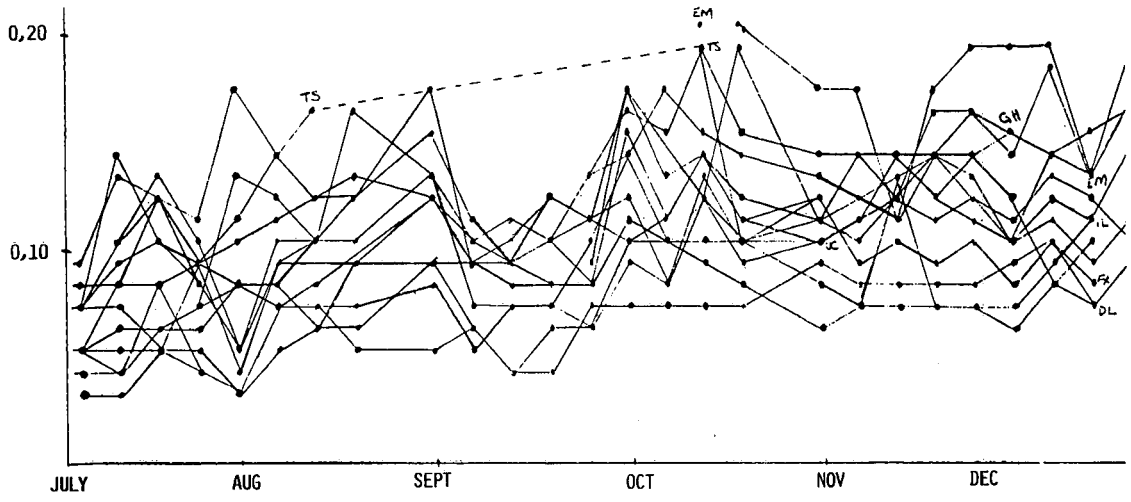


FIGURE 6
GUMS 1978

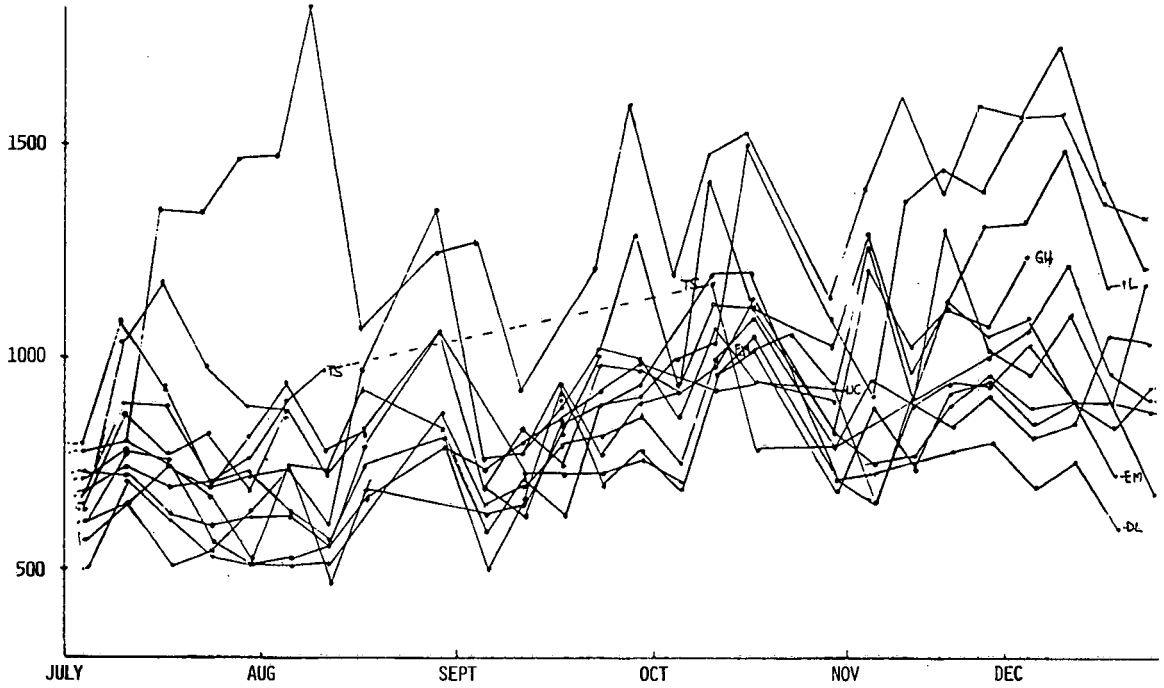


FIGURE 7
FLOC 1978

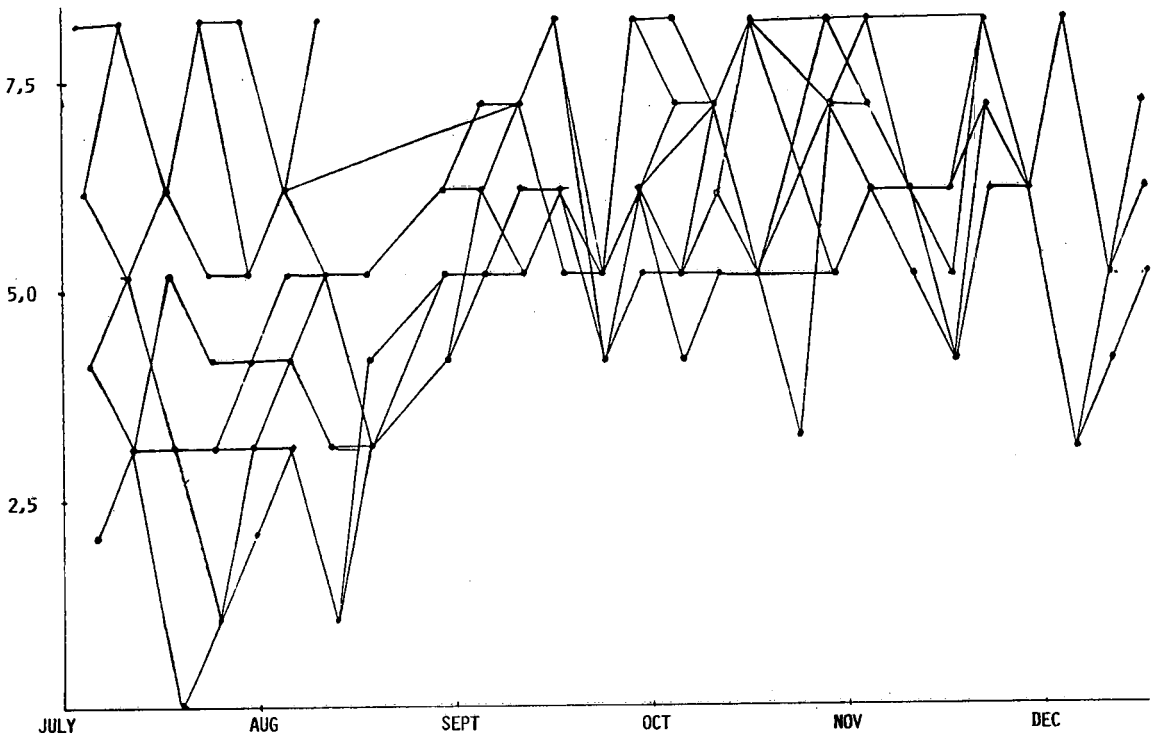
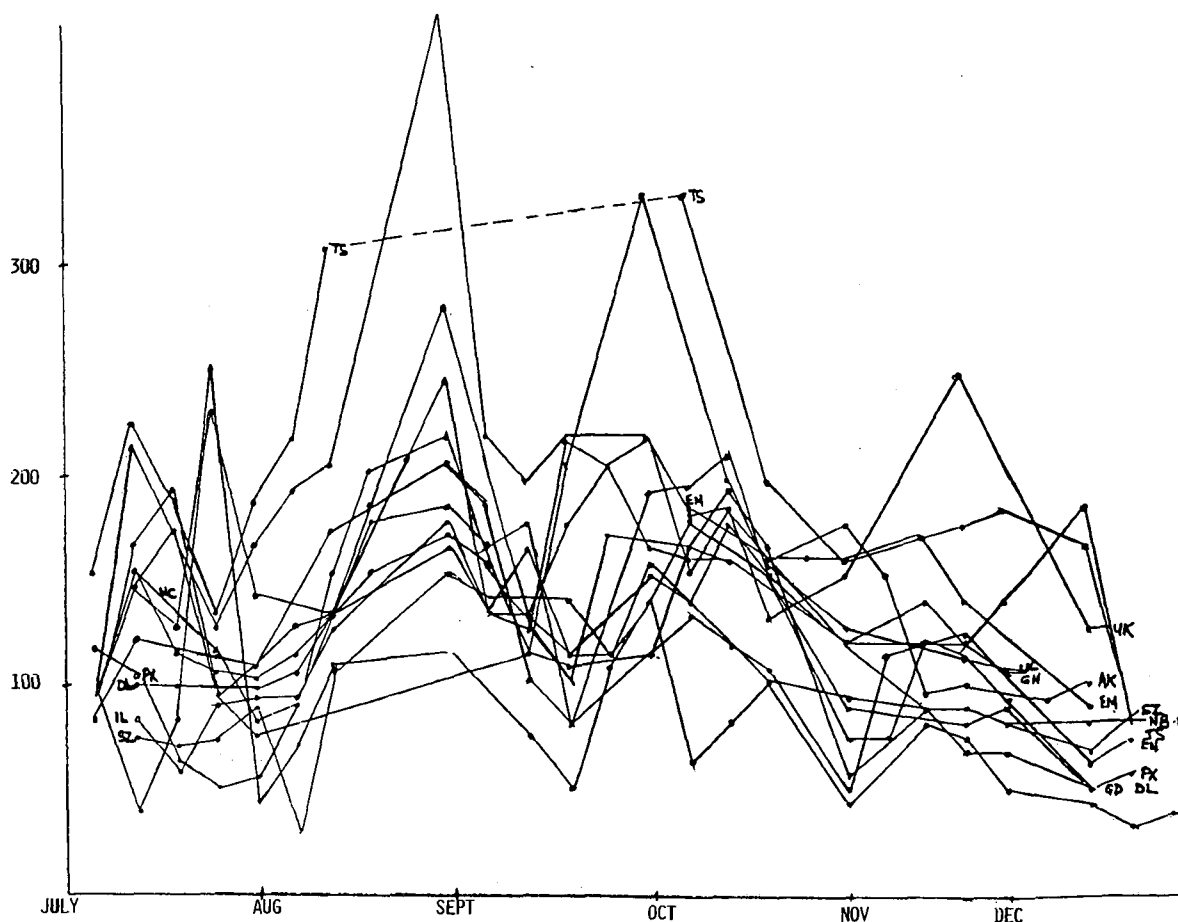


FIGURE 8
PROTEIN 1978



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