PRODUCTION OF SULPHUR-FREE SUGAR BY PHOSPHATATION

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Abstract

The present investigation deals with a major chemical modification to the existing double sulphitation process of sugar manufacture. Laboratory and pilot plant trials were conducted for clarification of sugarcane juice with phosphoric acid, and the results were compared with those obtained with the existing double sulphitation process. The sugar produced by the phosphatation technique is free from sulphur, low in ash and has a better storage quality. The cost of producing sugar by this method is *prima facie* on the higher side. Yet, considering the fact that sulphur-free sugar fetches a premium price and has intangible gains, the method deserves further investigation aimed at bringing down the cost of production and proving the intangible gains.

Keywords: sulphur-free sugar, phosphatation, sulphitation, quality

Introduction

At present, the demand for natural food products that contain minimum chemicals (as per FDA approval) is increasing day-by-day in the domestic as well as the international market. In the field of medicine, it is evident that an increasing number of people are shifting from ‘allopathic’ to ‘ayurveda’, and the same trend can be observed in the food industry. Our existing method of sugar manufacture involves double sulphitation. Sugar produced by this process contains about 20 ppm or more sulphur. Also, our sugar does not produce a clear solution when dissolved in water, due to the presence of calcium sulphate ($CaSO_4$) and polysaccharides. It therefore becomes difficult to sell our food, plasma and beverages industries on the domestic and international markets.

In the double sulphitation process, clarification of juice is done with the help of lime and sulphur dioxide gas (SO_2). The SO_2 does not dissolve completely, and creates environmental hazards. In this process, the observed rise in CaO content from mixed juice to clear juice is in the range of 400-500 ppm; this leads to the formation of scale in the evaporators and also retards the process of pan boiling by reducing the RS:ash ratio. At the stage of syrup sulphitation the SO_2 is used as a bleaching agent for lowering the colour of syrup, but at the same time the formation of a fine precipitate of $CaSO_4$ can be observed, caused by the difficulties in eliminating the SO_2. It is therefore desirable to explore some modification to the method of sugar manufacture.

The use of phosphoric acid and phosphate salts to form calcium phosphate precipitate for the purification of sucrose solution has been known for a long time. The purifying effect seems to be due to the adsorptive power of calcium phosphate, and it has often been observed that calcium phosphate clarification is very effective in eliminating colloids and finely dispersed matter from the clarified juice.

In the nineteenth century, phosphatation was introduced in the refining industry to clarify the raw sugar solution prior to bone char treatment to produce white sugar directly from sugarcane.

Phosphoric acid is used in Java to neutralise the filtrate of the second carbonation in the liquor carbonation process. Neutralisation of the alkaline filtrate (pH 8.5-9.0) with phosphoric acid is preferred to neutralisation with sulphur dioxide, because of the undesirable presence of sulphates in the refining liquids, and when golden syrup or a certain type of soft sugar is made.

In Taiwan, Sang *et al*. (1971) studied the carbonisation-phospatation process on a pilot plant scale vis-à-vis the existing carbonation-sulphitation process and obtained satisfactory results in regard to quality of sugar, i.e. sugar that was free from sulphur, low in ash and had comparatively less colour development during storage.

In Uttar Pradesh in India, Mishra (1977, 1978) studied the possibility of phosphatation of unfiltered second carbonated sugarcane juice in the laboratory as well as in the factory. He found that the application of phosphates in the factory process was helpful in reducing the scale problem, it led to less colour development in post-clarification operations and it improved the exhaustibility of molasses. His analytical data showed less destruction of reducing sugars, higher juice purities, a higher P_2O_5 content, lower colour and lower ash in a sugar that was free from sulphur and had better storage quality.

Materials and methods

A lime dosage optimisation experiment was conducted by applying different doses of lime in the pH range of 7.5 to 9.8 neutralised with phosphoric acid. For the mixed juice treatment, 10-litres of the process mixed juice plus filtrate (10%) was brought to the laboratory, and each litre was separated into six parts. One part was used for the analysis of untreated juice, and remaining five parts were heated separately to 70ºC and treated with milk of lime of 10 Bx at 1.0, 1.5, 2.0, 2.5 and 3.0 V/V%. The pH of the resultant mixture was 7.5, 8.2, 9.2, 9.6 and 9.8 respectively. The juice was neutralised in each case with 10% phosphoric acid, and again heated to boiling and then transferred separately into one-litre glass measuring cylinders for settling. After settling for about half an hour, the clear juice in each case was analysed for Bx, Pol, purity, P_2O_5 content, ICUMSA colour, CaO content, turbidity, reducing sugar, starch, dextran and mud volume.

For the plant scale study, a temporary arrangement was made for phosphoric acid addition near the sulphiter. A 10 % solution of phosphoric acid was prepared and kept handy. The juice in the sulphiter was limed to a pH between 9.5 to 9.8 with the help of milk of lime as practised in the factory. The prepared phosphoric acid solution was added continuously so as to neutralise the juice. The process continued for 17-18 hours. During this period, samples of mix juice, clear juice, syrup and stickwise sugar were analysed for Bx, Pol, purity, P_2O_5 content, ICUMSA colour, CaO content, turbidity, reducing sugar and conductivity ash.

Results and discussion

The laboratory experiment had revealed that colour of the clear juice (9100 IU) at pH 9.2 was comparable with that of the processed clear juice. Also, its CaO content was similar to that of the mixed juice. By contrast, the same parameter in the clear juice was found to be increasing with the existing process. Other parameters such as purity, RS, P_2O_5, mud volume and polysaccharides seemed comparable with the process clear juice. Detailed observations from the laboratory experiment are given in Table 1. The trends observed in the laboratory study prompted a similar experiment to be conducted at plant scale. Suitable arrangements were made for the phosphoric acid to be added near the sulphiter. The addition of phosphoric acid continued for about 17-18 hours (2 shifts). During this period, regular analyses of mixed juice, clear juice, syrup and sugar were made for brix, pol, purity, colour, turbidity, CaO, P_2O_5, RS and conductivity ash. The results from the factory experiment are given in Tables 2 and 3.

Table 1. Improvement in quality of sugar produced.

Parameter	Phosphoric acid process	Double sulphitation process
pH	6.7 to 6.8	5.7 to 6.2
Turbidity of 50% solution	1.0 to 1.5%	1.0 to 2.5%
Colour by method GS2/3-10	85-120 IU	60 to 95 IU
Sulphur content	Nil	Above 20 ppm
Haziness of sugar solution	Slightly lower	Slightly higher
Overall appearance	Lack-lustre crystals	Lustrous crystals

Table 2. Results of laboratory experiments on clarification with lime and phosphoric acid.

Sr. No.	Parameter	Mixed juice pH 5.24	Shock liming pH					Process clear juice
			7.50	8.18	9.22	9.62	9.80	
01	Milk of lime 10º Bx(v/v) (ml)	-	10	15	20	25	30	-
02	Phosphoric acid 10% (w/V) (ml)	-	1.55	4.45	8.55	11.15	12.35	-
03	Brix (%)	17.60	17.55	17.39	17.38	17.73	17.42	16.18
04	Pol (%)	14.76	14.71	14.53	14.66	14.75	14.55	13.60
05	Purity (%)	83.90	83.53	80.98	84.19	83.24	83.54	83.10
06	Colour (IU)	11516	11119	10455	9105	11005	12310	13150
07	Turbidity (%)	-	29.89	12.68	3.42	2.38	7.32	12.1
08	CaO (ppm)	1155	925	895	825	805	775	1310
09	P_2O_5 (ppm)	340	42	28	30	23	22	15
10	RS (%)	0.18	0.19	0.22	0.21	0.22	0.21	0.26
11	Dextran (ppm)	412	224	217	99	73	65	133
12	Mud volume (ml)	-	225	265	315	410	420	-

Table 3. Analysis of process samples during phosphoric acid addition.

Sr. No.	Sample	CaO (ppm)	Cond. (µs)	RS (%)	P_2O_5 (ppm)	Turbidity (%)	Colour (IU)
01	Mixed juice	1175	835	0.24	365	-	14908
02	Clear juice	1125	825	0.21	20	11.3	8055
03	Syrup	3554	1285	0.38	-	25.3	8465

The salient outcomes from this process are as follows:

CaO content:	There was no rise in CaO content from mixed to clear juice.
Conductivity:	A reduction in conductivity from mixed to clear juice was observed.
Colour:	About 50% reduction in colour from mixed to clear juice was obtained. There was no colour rise from clear juice to syrup.
Turbidity:	Comparable turbidity of clear juice was observed vis-à-vis the conventional process.

Future plan of work

Further to the laboratory work and plant scale results obtained at BCSSK Ltd. Nanded, it seems worthwhile to conduct a large scale trial to corroborate the benefits observed during the pilot study. It will be necessary to conduct the trial for a period of at least two months, using the existing double sulphitation process for one month and the modified process for one month. This will enable an evaluation of the technical and economical benefits.

Benefits of the phosphatation process

- The lower CaO content in clear juice is beneficial in reducing scale formation. There is scope to reduce the number of cleanings and also the quantity of chemicals used for cleaning.
- The scale formed by this process is comparatively soft, which helps to reduce the time required for cleaning.
- A reduction in conductivity helps improve the RS:ash ratio, which results in improved exhaustion of massecuits during pan boiling. There is scope for a reduction in sugar loss in molasses of about 2.0-2.25 units, which will result in a 1% rise in recovery.
- Sugar produced by this process, being free from sulphur, is acceptable by the food, pharmaceutical and beverage industries.
- Health conscious consumers are prepared to pay a premium price for sulphur-free sugar.
- As far as storage is concerned, conventionally produced sugar deteriorates due to the release of SO_2 and the colour increases. In the new process, no such problems exist.
- As a result of lower salt concentration, the quality of molasses is better in the proposed phosphatation process compared with the double sulphitation process.
- The nutrient content of press-mud obtained with the proposed method will be better because of its increased phosphate content. This will improve soil fertility. In contrast, the press-mud from the double sulphitation process contains sulphur, which will tend to acidify the soil.
- The shelf life of sugar produced by the phosphatation method is substantially better. The increase in colour during storage over a period of six month was only 5.8%, and 11.85% over 12 months (Table 4) compared with a 25-30% increase observed in conventional double sulphitation sugar.

Table 4. Analysis of sugar produced.

Sr. No.	Sample	Strike No.	pH	Colour (ICUMSA units at 420 nm)		
				fresh	6 month	12 month
01	S-30	1129	6.42	80	87	92
02	M-30	1129	6.37	83	86	88
03	S-30	1128	6.67	117	124	133
04	M-30	1128	6.46	123	138	140
05	S-30	1131	6.91	115	117	125
06	M-30	1131	6.42	123	128	128
07	S-30	1132	6.46	121	133	142
08	M-30	1132	6.54	122	123	133
09	M-30	1133	6.68	108	116	128
10	S-30	1134	6.63	119	132	146
11	M-30	1134	6.58	131	142	148

Limitations of the new process

- The cost of phosphoric acid is higher than that of sulphur. Therefore, cost of producing sugar with the proposed technique will be Rs16-17 per bag higher than the existing double sulphitation process.
- Mud produced with the phosphatation method is slightly spongy, and may require more space in the clarifier. Therefore, clarifier capacity will have to be increased.

Financial aspects of the project

Expenditure for 1 lakh MT crushing with existing double sulphitation process:

Sulphur:	50 MT @ Rs4,000/MT	Rs200,000
Antiscalant:	1 MT @ Rs100/kg	Rs100,000
Phosphoric acid:	1 MT @ Rs32/kg	Rs 32,000
Hydrogen peroxide:	400 Kg @ Rs20/kg	Rs 8,000
Total expenditure		Rs340,000

Expenditure for 1 lakh MT crushing with phosphoric acid method:

Phosporic acid:	60 MT @ Rs32/kg	Rs1,920,000
Hydrogen peroxide:	2 MT @ Rs20/kg	Rs 40,000
Total expenditure		Rs1,960,000

Additional expenditure with the proposed phosphoric acid method:
Rs1,960,000 minus
Rs 340,000
Rs1,620,000, which is equivalent to Rs16-17/bag.

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