NOTES REGARDING THE SULPHITATION-REFINING PROCESS AND PLANT AT UMFOLOZI

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Introduction

During 1959 the Umfolozi Co-operative Sugar Planters Limited started their refinery to produce a refined sugar by means of the melt-sulphitation-process.

The most important advantages of the application of sulphitation of raw-liquor are simplicity of equipment and low consumption of chemicals.

The known difficulty of sufficient ash- and colour-removal from the juice and evaporator-syrup forms a limiting factor for the production of a millwhite sugar of sufficiently high quality to compete successfully with refined sugar. This is especially so in the field of industrial consumption, but also more and more in the retail markets.

Ever-increasing production costs have been met in part for some time, by increased plant capacity; and South Africa has in no way lagged behind in the extension of the individual factory unit. But this has led to the necessity for further activities to provide a finished product on the spot, which saves a good deal of handling and transport as well as fuel expenses. With the introduction of the process under reference further savings have become possible due to the reasons stated above, and the quantity of chemicals, such as lime, sulphur, as also filteraids, etc. can be kept within reasonable limits, when compared with other processes.

Following the usual procedure for the production of raw sugar by defecation (affination being included in the raw-house centrifugal operation), the process is perhaps the shortest way to produce refined sugar.

Process Control

Observations of the behaviour of juices and syrups, as well as those of operations from day to day, show how easy it is to get variations in the quality of the end-product, unless constant conditions can be maintained in respect of concentration, temperature and pH. With the progress made in the application of industrial control equipment and automation, the risk of producing a product not always up to standard, has been eliminated almost completely. Personally I would go as far as to say that it is doubtful if a good quality refined sugar could be made by this process if the factory had to depend upon the more elementary systems of control and the human element, as was the case a few years ago.

The temperature and brix-control need not present great obstacles to the operator, but alertness to sudden changes can never replace the instrument.

This is the case with pH, the value of which cannot be sensed and the only means for sufficiently accurate measurement is the pH meter. Earlier types often presented difficulties by quick fouling of the electrodes, but this has been overcome. Instruments of more recent construction are very accurate and so sensitive that they will show immediately the faintest difference and combined with suitable amplifiers and relays will open or shut a valve for the admission of the required reagent, in our case, milk of lime.

Milk of lime is added through a so-called Splitter-box, which is operated by a small electric motor. This motor is started, stopped and reversed by a relay, activated by the amplified e.m.f. of the cell formed by the electrodes of the pH meter. The pH meter-cum-controller, which is automatic, maintains the pH within very narrow limits to the pre-set value by passing on the impulses, caused by the changing pH to the motor of the Splitterbox. Thereby more or less lime is added to the liquor.

Therefore, we do not only have the instrument to show the prevailing pH continuously, but it also makes the necessary adjustments, all the time, to maintain a constant value at a pre-determined level, called the Setpoint. There are deviations, no doubt, but these are not so much due to the lack of sensitivity of the instrument, and can be traced back to time-lag in the complete response of the chemicals, the dosing apparatus, and the lack of suitable and immediate inter-mixing of the materials. It goes without saying that these conditions can be improved upon. In our case we have not found any insurmountable obstacles in this respect and the necessary adjustments to the plant have been and are being made in due course.

Production of SO_2 Gas

The sulphur burner forms an important part of the installation. It is worked with molten sulphur, contained in pits in which steam coils melt the rock-sulphur and keep it at a temperature of about 135°–140°C. At this temperature the sulphur is least viscous, and may be pumped without any difficulty into the burner. Steam-jacketed piping is essential to prevent re-solidifying of the sulphur in the pipelines. The sulphur enters the burner through a nozzle, by which it is sprayed on to a splashtile and flows down a checker-work of firebricks, the heat of which ignites the sulphur. When starting up, the burner is preheated to a sufficiently high temperature by means of an oilburner. The gas produced is cooled in two stages, first in stainless-steel piping, atmospherically, and then by passing through a water-cooled Karbate cooler. The purpose of the cooling is two-fold, viz. to prevent hot gas from destroying the sugar in the sulphitation, and to remove any SO_3 formed in the combustion. This second point presents a peculiar problem, as the gas, while cooling, tends to be even further converted to SO_3 and thence to H_2SO_4, because of the presence of moisture in the air and in the sulphur. Only a very rapid cooling to below the so-called critical zone can check this continued oxidation.

Process

The process is as follows:

Raw-sugar of 99.1–99.3 is melted continuously in a horizontal cylindrical melter of the Prins-Steuerwald type to a solution of 68° Brix at 78°C.

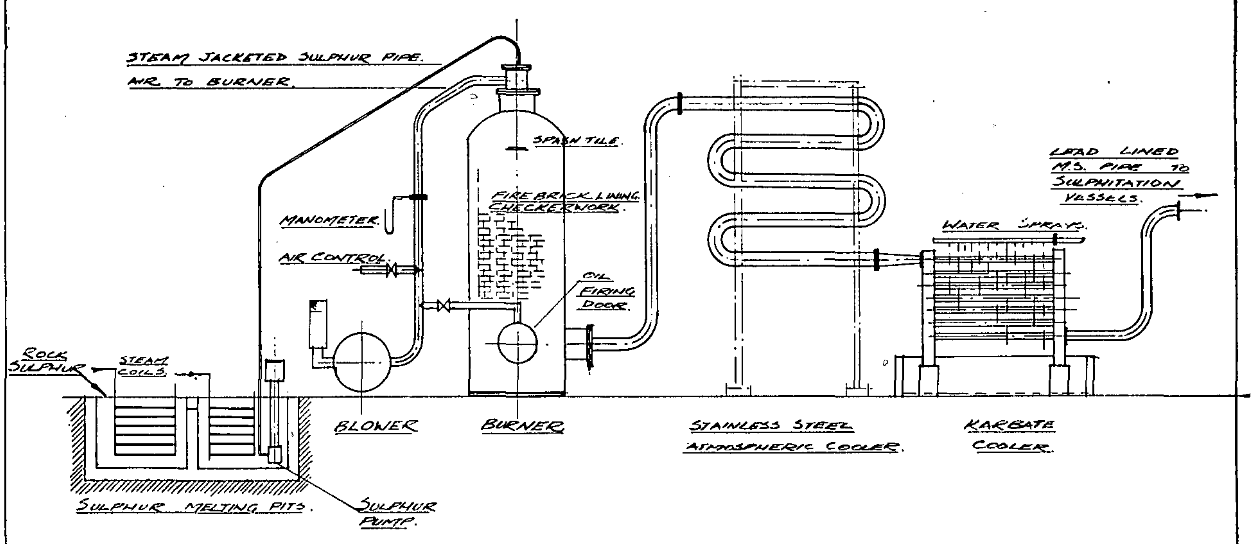
A Thermo-Controller maintains the temperature by opening and closing a pneumatically operated steam valve. Steam is admitted directly into the raw-liquor.

A Density-Controller does the same to a water-valve and admits or shuts off the water required to maintain the specified degree of Brix.

Part of the raw-melt thus made, circulates back to the mingler into which the raw sugar is fed from an overhead bin, via a vibrator feeder. Thereby fluctuations in Brix and temperature are largely eliminated and levelled off, and the task of the automatic controllers is diminished, as they have now to make adjustments between much narrower limits.

The raw-liquor is pumped into the sulphitation system, which consists of two tanks provided with gas distributors near the conical bottom and an external circulation system. The circulation system supplies the means of getting intensified reaction contact, but experience has shown that it does not create the desired degree of inter-mixing. Correct pH is maintained by a controller after each vessel, adding milk of lime through Splitter boxes.

The flow of SO_2 is not altered on purpose. Any fluctuations occurring therein are caused incidentally, and the required adjustments of pH are made exclusively by the changes in the flow of milk of lime. There remains, of course, the possibility of increasing or reducing the flow of gas, but once the best operational conditions have been established, no alterations to the flow of gas are normally required. The distribution of the gas to the individual sulphitation vessels is in the ratio of approximately 70 per cent to the first vessel and 30 per cent to the second vessel. This is achieved by orifice-plates of corresponding apertures in each of the two SO_2 inlet pipes.



SO_2 PRODUCTION PLANT

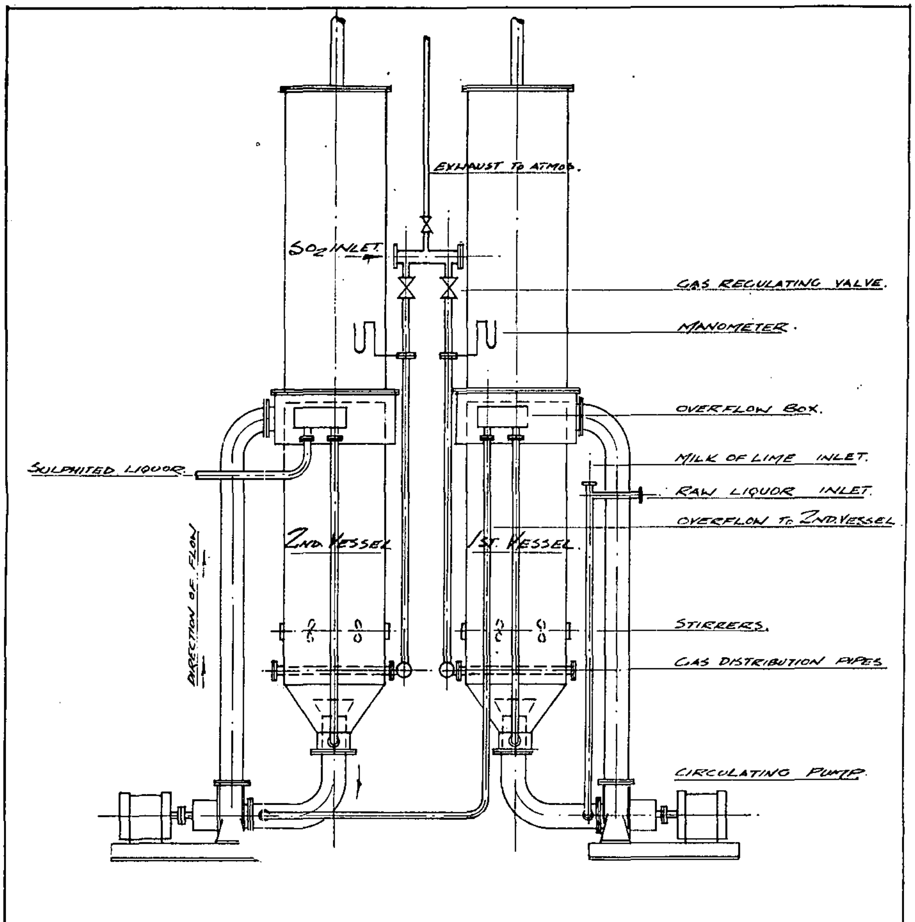
It is obvious that the quantity of calcium sulphite formed in the processing is important for the extent to which the clarification, i.e. the removal of undesirable matter from the liquor, is required. Therefore, a certain quantity of gas must be admitted per ton of Brix, and for a higher or a lower throughput the gas required will vary. This is adjusted by setting the valves for the admission of air to the sulphur burner with the aid of a differential-pressure gauge, and changing the speed of the pump for the supply of sulphur to the burner.

The sulphited liquor, leaving the sulphitation vessels, is still subject to a certain completion of reaction, and/or a completion of the required structure or formation of calcium sulphite crystals, and therefore the liquor is sent to a retention tank, where an agitator keeps the material in motion, so as to sponsor the right crystal-formation and the adsorption of more of the impurities.

At the same time the temperature, which was 78°C during the melting and can be taken to have dropped by a few degrees only while the liquor was under treatment in the sulphitation tanks, is being raised to 90°C, the danger of inversion now having been eliminated, as the pH is in the region of 7.1–7.2. This rise is necessary in order to precipitate as much $CaSO_3$ as possible, which substance is less soluble at elevated temperatures.

In the original process, allowance was made for the incorporation of vegetable carbon in the clarification and a tank has been installed between the second sulphitation vessel and the retention tank.

Vegetable carbon is to be added to the liquor in order to improve its colour, and consequently the colour of the sugar made from this liquor. It also reduces the ash-content to some extent. No data are available from Umfolozi regarding this subject, as carbon has not yet been applied to the clarification. It is thought, however, that it may be advisable to add the carbon at a later stage, i.e. after filtration. Adding it to the mixture of a sugar solution and calcium sulphite precipitate might cause a fair percentage of the carbon to be occluded by the precipitate, before it can adsorb colour from the



SULPHITATION-VESSELS.

liquor proper. This will have to be seen in practical operation, and should it prove to be the case, certain alterations in the lay-out will have to be made.

From the retention tank the liquor is pumped to the filter-feed tank in which it is kept at the right temperature to suit speedy filtration, i.e. about 85°C. Filtration is done in three Sparkler Leaf Filters over cotton duck or twill which is precoated with Hyflo-supercel and the filtrate is then passed over Stellar Candle Filters for a "polishing" treatment of the liquor.

The filtercake from the leaf filters, following sweetening off with hot water, is sluiced down by hot water and dropped into so-called "pulping" tanks from where it is pumped into the sump of a string filter. Water is sprayed on to the cake formed on the string filter and the total filtrate is returned to the sweetwater tank. From this tank it is piped to the melter for raw-melt. The cake from the string filter is dropped into a tank with stirring gear and pulped to a slurry, after which it is pumped out of the factory as waste. Originally filtration was only done over the leaf filters, but this was considered insufficient and two Stellar filters were installed, which have given satisfaction and have proved themselves already, although they have been in operation for only a few weeks. The Stellar filter cake is returned to the leaf filter feed tank.

The filtrate, leaving the Stellar filters, is pumped to the panfloor and worked off to sugar. Boiling is done under reduced vacuum (about 22-23 inches of vacuum) and a two or two-and-a-half massecuite system is applied. Final run-off is returned to the raw-house and boiled away into A-massecuites. The massecuites are spun in 1450 r.p.m. centrifugals of diameter 40 inches.

Sugar is dried in a rotary sugar-drier and coarse grain and lumps sent to a reject-melter. A fine crystal fraction is separated and elevated into a seed-bin on the panfloor. When available, this seed is drawn into the pans into a footing of concentrated liquor, forming the footing for the massecuite. Thereby the necessity for graining all the time is avoided, and a much more regular crystal is produced in the massecuite. This facilitates spinning, improved drying, yields more crystal in the bag and saves a good deal of time and steam. It also makes the factory less dependent on the skill of the individual panboiler and there is less difference in the appearance of the sugar boiled by different panboilers. Graining is reverted to only when insufficient seed is available, but usually this brings forth a good supply of seed for the following cycles, especially if the grain has been kept sufficiently small, so that most of the sugar produced from a grained strike returns as seed.

This will not affect the throughput to any great extent, as liquor drawing is proceeding as usual or even faster than when working on seed, because more grain can be formed and except for the period of actual graining, boiling can be kept very fierce.

When working with seed, the panboiler need not worry so much about secondary grain, should this appear, especially in the first stages of the boiling, as it will develop into fine crystals which will be sieved out in the screening of the sugar and return as seed.

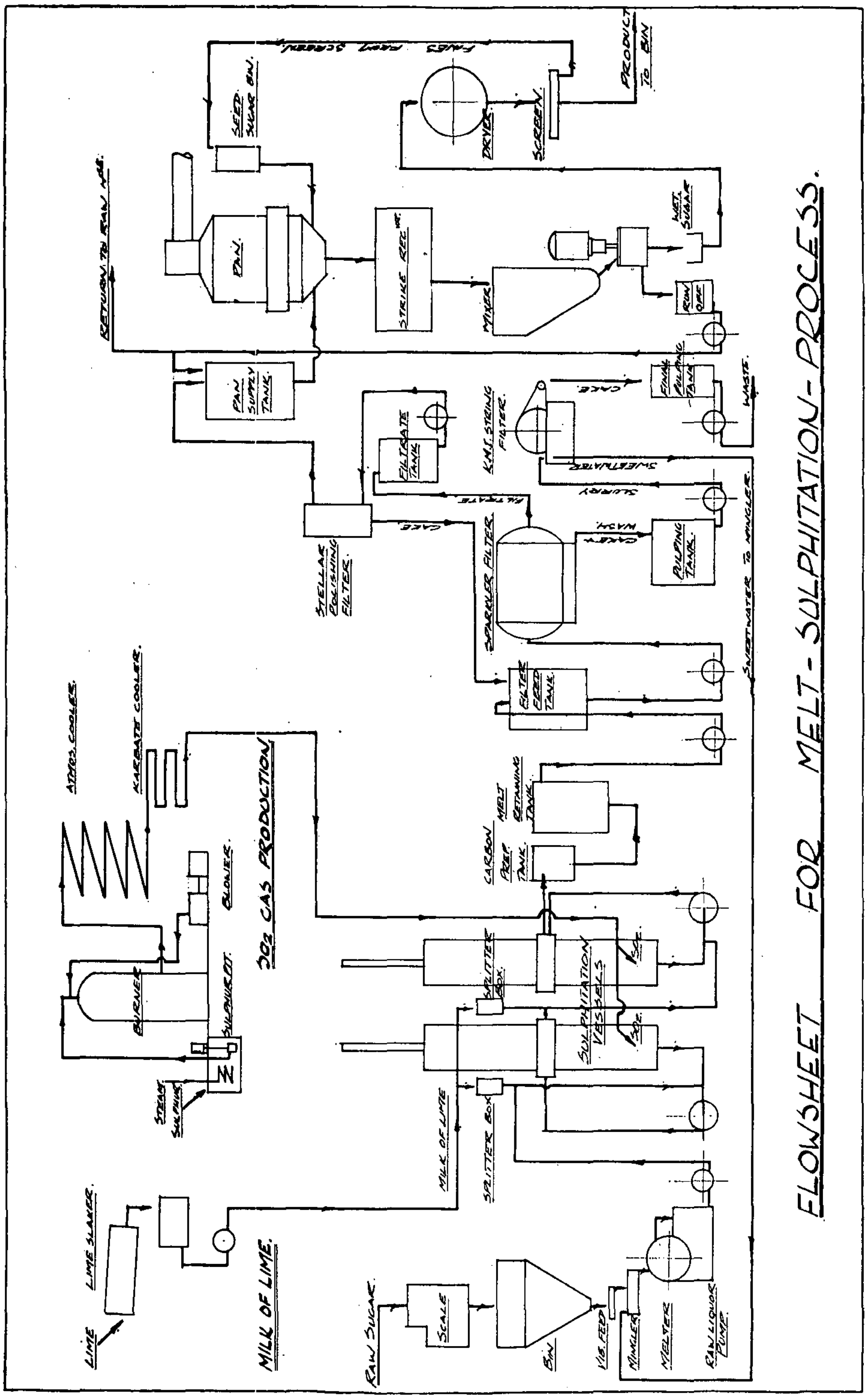
There should not be too many secondary crystals though, as this would affect the centrifugal work and hence the quality of sugar for bagging.

Operating Experience

Operating experience was gained during the past season and several problems had to be dealt with and have gradually been overcome.

The first problem arose from the SO_2-production plant. The nozzle through which the molten sulphur is pumped into the combustion chamber kept choking up with a substance so far undertermined, but suggested by experts to be mainly Carsulph, mixed with impurities from dust and dirt contained in and deposited on the rocksulphur in storage. Insufficient and unsteady supply of sulphur to the burner caused the temperature to drop below the minimum of 550°–600°C resulting in sublimation in the cooling system, choking of the gas pipes and lack of gas to the sulphitation vessels. Very irregular operation was caused by this, and since one of the most important conditions for a successful operation is a steady flow with a very constant pH, the processing was not at all satisfactory. Changing the type of nozzle to a very simple one with a much larger aperture has remedied the trouble.

Another difficulty was that part of the sulphur did not burn up on its way down from the splash-title to the bottom of the burner. This also caused sublimation and it was found that some channelling of the liquid sulphur took place. It was overcome by placing additional checker-brickwork inside the burner, thereby increasing the total burning area and better combustion was achieved. This is not necessarily a fault of the plant as such, but partly caused by the fact that the plant had to work below guaranteed minimum SO_2 production for a successful operation. About 150–200 lbs. of sulphur per hour average was burnt to produce the SO_2 for the actual throughput, whereas the burner, which is of the Monsanto-cascade type has been designed for about 300 lb. per hour minimum, this being the smallest possible capacity for a guaranteed good operation. The difficulty seemed to be the contact space and burning area to ensure complete combustion of



FLOWSHEET FOR MELT-SULPHITATION-PROCESS.

sulphur by the air. To improve this the above mentioned extra bricks were placed in the burner, while for the next season cooler-piping will be jacketed and the compressed air for the burner will be passing through the jacket, before entering the burner, so that a more spontaneous and a more complete combustion of the sulphur can be expected, with the higher air-inlet temperature, and the consequently higher combustion-temperature in the burner.

The cooling caused by the air flow through the jackets of the atmospheric cooler section will assist to overcome the increase in gas-temperature by the preheated air and possibly also give a more rapid cooling of the gas to below the point of SO_3 formation, so that this can be kept at a minimum.

Early in the season it was found that the formation of filter-cake was such that enormous volumes were experienced. This caused the filters to be filled up with cake the thickness of which was far in excess of the expected size. This could be explained to some extent as being due to very erratic operations in the first few weeks. Sometimes liquor was being circulated for a long while through the sulphitation vessels, and the gassing was not always interrupted, causing an excess of calcium sulphite formation. But after the initial difficulties had been overcome and more or less normal operation could be maintained, it was found that the cake was still generally more voluminous than expected. The filters were supposed to run on stream for up to 12 hours or thereabouts for the formation of filtercakes of up to 1 or 1.25 inch thickness, but it was found that after a six hour run they had to be taken off, the thickness of the cake being as desired and not too much to cause difficulty in sluicing down, as experienced when a thicker cake had been formed.

This would indicate that the specific volume of the produced calcium sulphite filtercake is fairly large and therefore of a porous structure which should facilitate the filtration and also sweetening off. For the coming season an extra filter will be installed to have capacity under all circumstances, e.g. in case it is desired to change the filtersheaths for a clean set.

The formation of sufficiently-sized crystals of calcium sulphite appears to be influenced by the pH of the liquor and fluctuations seem to have some effect, so that filtrates were found to be less sparkling from time to time. With the installation of polishing filters, following the primary filtration, fine liquor of a high quality could be had, resulting in an improved quality of the sugar.

Some of the cloudiness observed from time to time in the primary filtrate can be attributed to a sudden drop in pressure in a filter, when a clean filter was put on stream along with it. The newly started filter would offer less resistance to the liquor so that the pressure of the feedpump would suddenly be reduced and the cake might crack, resulting in some liquor passing through the cracked surface and causing cloudy filtrate for a while. To overcome this each filter will be fed by its own pump in the future.

Originally the cake as produced from the filter was conveyed on a belt, but the material proved to be too sticky for this method and was abandoned. The next move was the pulping of the cake with muddy juice from the clarifiers of the raw-house, the mixture being returned to the raw-house filters so that there was no need for the separate disposal of the refinery-filtercake. Unfortunately this caused the formation of a very fine and hard scale in the 3rd and 4th vessels of the evaporator plant, which impaired the operation of this section of the factory, so that it had to be abandoned too.

Further consideration to this problem may be given in the future, together with certain contemplated alterations in the raw-house filtration-plant.

It was found that corrosion of the mild-steel surfaces in the various tanks of the plant, which were not protected, caused a slight colouring of the liquor. To stop this effect all tanks will now be treated with a corrosion-resistant coating.

Conclusion

In spite of various problems that had to be tackled during the first season of operations with the new refinery the quality of the sugar was as good as any produced elsewhere and with forthcoming alterations and improvements the Company is confident of an even better quality.

The writer wishes to express his thanks to the Management of the Umfolozi Co-operative Sugar Planters, Limited, for the permission to present this paper.

For discussion on this paper see page 125.