

# SOME NOTES ON THE OPERATION OF VACUUM CLARIFIERS

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The Vacuum Clarifier installation at Umzimkulu was introduced to the Sugar Industry in July 1966, and has been in operation since October 1965. Since then 100,000 tons of sugar have been manufactured from juices clarified by this "cold" phospho-defecation process. As a starch removal process it has been highly efficient, producing sugars which have been well received in refineries both at home and overseas.

In order to maintain a high standard of efficiency however, it is necessary to observe basic principles, which, while being of an elementary nature can lead to poor results if they are overlooked.

The object of this short description of the process therefore, is to give future operators the benefit of some of our experiences, and to remove some of the "mystery" of this entirely new, but recently proven technique.

The Process Stages are as follows:

Mixed Juice is heated to 60°C without prior liming.  
Liming to pH 8.1 or higher as required.  
Mono-calcium phosphate added to reduce pH to 7.4.  
Coagulating agent added.  
Vacuum Flotation of precipitates.  
Separation of mud from clear juice.  
Treatment of mud and filtration.  
Treatment of clarified juice.

## Temperatures

The primary object of the process is the removal of undissolved starch from the juice before it is heated above the starch solution temperature which is in the region of 67°C.

The juice is therefore heated to 60°C in the primary heaters. This temperature is considered safe and, provided the velocity through the heater is high enough, should give no local over-heating.

At this temperature it is also fairly safe to add lime to high pH values without excessive colour formation by the destruction of reducing sugars.

Temperatures of starch-containing material should at no time be raised to more than 65°C, or be mixed with juices or other products of a higher temperature.

Lower temperatures should be avoided, as the growth of *Leuconostoc* is accelerated, and apart from the loss of sucrose, this growth can be a serious problem in the clarifier, pipelines and pumps.

## Liming and Phosphating

After heating to 60°C, the juice is limed to a pH of 8.1 or higher as required, and is then phosphated back to pH 7.4 with a solution of mono-calcium phosphate (3°Be). This final pH with phosphate is regulated to give a syrup pH of 6.8–7.0.

By increasing the primary pH above 8.1 there is a corresponding increase in the amount of phosphate required to lower the pH to 7.4. This results in an increase in the volume of precipitate formed, and gives an improvement in the clarity and brightness of the juice. The brightness of the clarified juice in turn can be related to the starch removal which has been found to be poor when clear juice is even slightly turbid.

Increasing the volume of precipitate excessively will increase chemical costs unnecessarily. Once a satisfactory pH has been established it needs to be increased only in exceptional cases such as when excessively muddy cane is being processed. (To illustrate this a laboratory test was carried out on juice to which 10% of mud had been added. Lime was added to 10.0 pH, phosphate to 7.4 pH, then the juice was coagulated and subjected to vacuum. It was found that only a few of the larger grains of sand were not floated, but the juice was brilliantly clear. This would be rather a costly way of removing heavy field dirt, which should be first removed by a battery of Dorr-Clones or other means before being treated.)

As starch analysis is of necessity time consuming it may be an aid to process control to utilise a photo-electric turbidity meter to give immediate indication of changes in clarity.

## Coagulation

This stage of the process is the key to the successful flotation of precipitates. The reason why coagulation is so important is explained as follows: Juice passing through vibrating screens, pumps, and scales is well saturated with air (i.e. air in solution). If after treatment with lime and phosphate this were subjected to vacuum, the air would be brought out of solution and rise to the surface rapidly, raising only a minute part of the loose floc which adheres to the micro-bubbles.

During coagulation the loose floc is drawn together to form larger globules of mud. These are closely knit so that when air is released from solution in the mud it is trapped and so lifts the coagulated portion of floc to the surface with it.

With incomplete coagulation the floc, being less dense, allows the micro-bubble to escape and rise to the surface showing up as a white froth on the mud layer. When this happens, juice clarity is invariably impaired even though a part of the mud has been floated. This is the result of poor dispersion of the coagulant in the juice.

Uniform coagulation is essential and is achieved by very rapid mixing of flocculant with the juice so that every particle of floc comes in contact with the correct amount of flocculant.

In order to apply a dosage of 5 ppm of coagulant 1 gallon of 0.5% solution is required per 1000 gallons of juice. Dilution of this solution to 0.05% ensures a more accurate dispersion. It is recommended that clarified juice rather than water should be used for dilution, to avoid an additional load on the evaporator.

To ensure that the maximum amount of air is in solution we have made it a rule that cold water only is used for dissolving the flocculant to give the 0.5% solution.

### Separation of Mud from Clear Juice

As the prepared juice passes into the clarifier, it comes into the reduced pressure zone and, as described above, the mud floats to the surface leaving the clear juice below. The mud is continually skimmed into several mud troughs, from where it flows to the extraction pump to be pumped to the filter. The clarified juice is also continuously withdrawn by means of an extraction pump.

Control of levels is essential to ensure that mud is continuously removed and that no accumulation takes place. If this is not done the small vessel soon fills with mud, which results in a heavy carry-over within a short time.

While the Umzimkulu system of extraction of mud and clear juice is by means of pumps, we realise that it is not the ideal. We have no recourse but to use them because of the present location of the clarifier.

Given adequate height the recommended method is to allow for juice and mud to be extracted by means of barometric legs from which they can be gravitated to the next point of treatment. This applies especially to the mud. If this is pumped the floc is so badly damaged that further coagulation at the filter station becomes necessary.

### Treatment of Mud

Filtration of mud is normal and the procedure need not be altered except with regard to temperature. Where filters are operated with a continuous overflow which is re-cycled through a reheating tank it will be appreciated that heating here would result in re-solution of starch. Filter cake wash water must be kept at the safe temperature of not more than 65°C.

This in itself will tend to keep the filter sweet, as the temperature drop through the cake layer in the drying cycle should be kept at a minimum. Danger

of inversion increases when muds are kept long enough for temperatures to drop appreciably.

It may be necessary to add lime to muds to re-adjust pH which tends to drop rapidly in juice which has cooled off. Periodic cleaning and sterilisation with formalin is recommended.

### Further Treatment of Clarified Juice

*Single Stage Clarification:* Clarified juice at Umzimkulu has been passed to the evaporator after final heating since the process was originally brought into operation. Some comments here are necessary. It has always been felt that with the "cold" clarification, the elimination of proteins would not be effected as temperatures have to be kept low because of starch.

The proteins have been accepted as the lesser of two evils. They have caused heaters to scale up to a point where temperatures for protein removal could not be attained. This caused scaling in the first vessel of the evaporator. By periodic cleaning of heaters during the week this unpleasant feature was overcome, and should be completely eliminated with secondary clarification.

During recent weeks tests have been carried out with a view to further clarification. The heated Mixed Juice (60°C) was limed to pH 8.5, then phosphated to 8.0, coagulated in the normal way, and clarified in the vacuum clarifier. The clear juice was now phosphated to pH 7.4, then heated and pumped to the settlers (two 14' Bach clarifiers). Settling rates were exceptionally high, with no carry-over of mud. This is remarkable as the two clarifiers were handling juice at the rate of 140 tons per hour with ease, whereas with the defecation (hot liming) process 75 tons per hour could be handled with difficulty.

These tests have shown the way for future procedure, and indicate that the original concept of utilising the process as an intermediate step lends itself to greater flexibility without increasing production costs unduly, retaining the advantage of low retention times. A variation which suggests itself in the secondary stage is sulphitation and this should be utilised by factories already equipped to put it into operation.

In the secondary stage the hot mud from the settlers should either be filtered separately or cooled to safe temperatures before mixing with "cold" mud from the vacuum cell.

It is hoped that this short paper will be of assistance to those who are about to use this process.

### Discussion

**Mr. van Duyker:** Mr. Rabe suggests that the Separan be diluted with clear juice instead of water down to 0.5%. We were using clear juice but, having double clarification, we were phosphating to 8 pH, clarifying in the Rabe Clarifier and then re-phosphating to 7.4. This juice was then used again for dilution and affected the Separan dosage to the Rabe Clarifier, destroying its coagulating effect. We therefore either added phosphate after extracting the dilution juice or did not add phosphate at all.

**Mr. Ashe:** I would like to know the cost per ton of sugar for liming and phosphating in the secondary clarification.

Also, is the mud from these clarifiers sent back to the filters and mixed with the mud from the Rabe Clarifier or is it treated separately, otherwise it appears that the hot mud will redissolve starch?

**Mr. Rabe:** The tests with secondary clarification were carried out at the end of the season merely to test the equipment so costs are not yet available. However, the process appears to be costly and a possibly better method would be sulphitation of the clarified juice but I am not sure how effective or practical this would be and would appreciate advice from the S.M.R.I. on this point.

As far as mud is concerned, it is mentioned in the paper that the mud has to be cooled before mixing the cold mud from the vacuum cell, or it could be filtered separately.

We intend at Umzimkulu to run the secondary mud through the Eimco belt so that the filtrate can be returned straight to the evaporator.

**Mr. Robinson:** Darnall was treating the juice in the vacuum clarifiers, phosphating it and settling it in the Bach clarifiers and such a small amount of mud was produced that it was returned straight to the cold mud. Juice was limed to 8.6, phosphated back to 8.2, passed through the vacuum clarifier then pumped to the Bach clarifiers without further treatment. A small amount of mud collected in the Bach clarifiers and it was flushed out for five minutes every hour.

I notice Mr. Rabe limes first, then phosphates. Darnall used to do the opposite but could not get more than 80% starch removal and it was more expensive. Better results were achieved when we changed to liming first and phosphating afterwards.

**Mr. Rabe:** We tried phosphating first because it is so much easier but we reverted to liming followed by phosphate.

Regarding mud in the Bach clarifier, there is so little that almost certainly no starch will be dissolved if it is mixed with the cold mud. At times, at Umzimkulu, in order to keep sucrose in filter cake down we have used a lot of dilution water, which we added as cold water to the secondary mud, thus reducing its temperature.

**Dr. Graham:** During the coming year the S.M.R.I. hope to carry out systematic tests at Tongaat which will include an investigation of phosphating and

liming procedure. This should clarify the whole problem.

**Mr. Rault:** You mention sulphitation of a clear juice at a pH of 8. Our experience with double carbonation was that our final pH was about 8.4 to 8.5, due to a potash alkalinity and we were able to sulphite the second carbonation juice to about pH 7 without any precipitation. We got very little encrustation in our evaporators, which had a capacity of 9 pounds per square foot per hour.

**Mr. Rabe:** One factory, using sulphitation at the moment, is also going to apply the vacuum clarification process at a much higher pH, then sulphite down with the addition of lime to give a secondary precipitation which will be settled out in the normal way.

**Mr. Ashe:** Since you changed to the new process at Umzimkulu the ash content of your sugar has increased considerably. If a secondary process such as sulphitation or carbonation is used might there not be an even bigger increase?

**Mr. Rabe:** With this cold clarification process, because of the solubility of lime salts the ash would tend to be higher without a secondary clarification process. When we phosphated the clarified juice to lower the pH and to get secondary clarification the phosphate in sugar increased, but sugar ash is no higher than it was before the introduction of this process.

**Dr. Matic:** In our work at the S.M.R.I. we found that scale obtained from the heaters and evaporator contained protein and calcium phosphate. If sulphitation is used the calcium salts, being soluble, will not precipitate. Therefore if a factory using the Rabe process introduces sulphitation after liming there will be an even bigger increase in ash because only the calcium phosphate already in the juice will precipitate.

As far as carbonation is concerned, sulphiting will bring down the pH but is generally applied only after eliminating calcium carbonate formed. If this is done before elimination of calcium carbonate residual ash will be very high.

**Mr. Buchanan:** The author says that for Leuconostoc he applied quaternary ammonium compounds without success. What quantities were used and was it to prevent or remove growth?

**Mr. Rabe:** I cannot remember the dosage, but we first applied by drip for prevention and then used it by the gallon but once the Leuconostoc was established we could not remove it.

**Dr. Graham:** In a paper to be read by Mr. Jennings later to-day he gives .02% as the crystal ash figure for Umzimkulu whereas the average for Hulett's refinery is .10%. You therefore appear to be producing a low ash, not a high ash, sugar.

**Mr. Rabe:** Our sugar is no higher in ash than any other Natal sugar. When we introduced secondary clarification and added additional phosphate the phosphate figures increased to 60 ppm instead of the normal 30 ppm.

**Dr. Graham:** Although your lime salt figure may have increased other salts are possibly being eliminated because you end up with a low crystal ash figure.

**Mr. Rabe:** It should be explained that the sugar

ash determined by the S.M.R.I. is made on un-affinated sugar, which will vary with polarisation. The crystal ash determined by Hulett's gives a true reflection and should be used when making comparisons.