

SOME NOTES ON THE TECHNOLOGICAL ASPECTS OF DEFECATION—CLARIFICATION

By A. VAN HENGEL
Sugar Milling Research Institute.

Introduction

There was a time when nearly all the raw sugar in the world was made by the same process: cold defecation. During the past few decades, however, numerous workers have experimented with many changes of the basic "cold liming and heating, to near or over boiling point—process." Many investigators have proposed new processes, which very seldom involved the use of any new chemical reaction and were nearly always merely changes in the sequence of liming and heating or the number of times that a certain sequence was repeated. Davies¹ published an article on fractional liming and double heating in which he described the advantages obtainable with his method over "cold liming". This F.H. & D.L. process, however, is one of the better known modifications to which I feel it is hardly necessary for me to add to at the moment.

Practical Defecation

The practical process superintendent in the sugar mill requires an answer to the question: which treatment should be given to a mixed juice in order to achieve the following results:

- (a) the clearest possible clarified juice,
- (b) the lowest volume of muds,
- (c) muds of easy filterability,
- (d) the achievement of the above in the cheapest way and with the aid of the simplest possible machinery.

It is, however, most confusing to study international literature in an attempt to find a way of working which guarantees good uniform results with respect to the first 3 points mentioned above. Most, if not all, processes proposed are based on data collected at one factory during a short period of the season and still the question remains whether the good results were caused by the new process or by the fact that supervision during the trial of the process was very much intensified. Moreover, many results of investigations are contradictory. Another weak point is that not all processes are judged by the same yardstick. We all know, for example, that neither clarity nor purity rise² are trustworthy criteria. So, the man seeking a suitable process to apply may choose one of many and he makes his selection in the hope that he has chosen the process which will serve him best under the prevailing conditions at his factory. To my mind it is obvious that this almost random method of choice will very seldom give optimum results and for this reason he will

soon want to experiment in order to find a method which gives him better results. In the following pages, I intend to discuss some of the basic requirements of a defecation installation which ensures sufficient flexibility with respect to changing conditions to permit the use of every known defecation process.

Basic Requirements

Irrespective of the sequence, there are three major treatments of the juice which together form the defecation process:

- (1) *Liming*: Milk of lime of certain density is added to the juice, in this way raising the pH. Chemical reactions take place resulting in the precipitation of a flocculant material.
- (2) *Heating*: The mixed juice, with or without liming is heated and physical and chemical reactions occur.
- (3) *Settling*: The limed and heated juice is given time to separate in three layers of a pronounced difference in appearance, viz. a supernatant layer of scums, a layer of more or less clear juice in the centre and a layer of muds on the bottom.

Two aspects of "Liming" should always be kept in mind, viz. the thorough mixing of juice and lime and the addition of sufficient lime in order to obtain a predetermined pH. Therefore "Liming" will be seen as "Mixing" and "pH measurement".

Mixing, heating and settling are unit-processes and the general technology is applicable. To my mind, it is hardly worthwhile discussing the merits of different ways of applying the defecation process as long as the three simple unit processes are not carried out in the best technological way, which is usually also the simplest.

As heating nowadays is done in modern heat exchangers only, and open defecation pans have fallen into disuse, there is not very much to say about this subject. Different authors^{2,4} advocate juice velocities of approx. 6 ft./sec. to achieve a high rate of heat transfer and minimum incrustation. However, it will be clear that at this velocity the juice will be very turbulent during its passage through the tubes. According to Davies⁵, this might have a detrimental effect upon the breaking up of the flocs.

Mixing is a subject which is of greater importance to us now. It is not necessary to go too deeply into the theory of mixing but a few basic principles will not be out of place here:

- (1) Bring the chemicals to be mixed into as intimate contact as possible before mixing. In the case of a large tank with a stirring device, then the two chemicals should not be added at opposite sides of the tank but every effort should be made to mix them as soon as possible with each other and the bulk of the tank.
- (2) Avoid "dead spots" and "dead corners" where no proper mixing takes place. This principle rules out every tank that is not cylindrical, half-cylindrical, conical or round in shape. Think for a moment of a crystallizer: nobody has ever seen a square one.

Taking all the above points into consideration, an illustration of a mixer which would serve the purpose ideally is shown in Fig. 1.

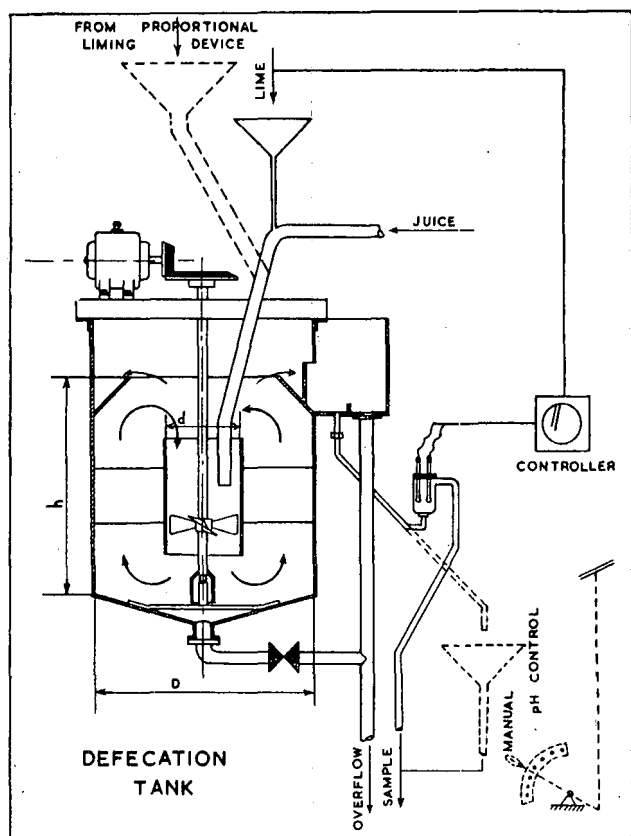


Fig. 1.

According to Perry,⁶ this type of mixer will give an excellent performance provided the tank is well-shaped. Basically, a propeller is surrounded by a tube which has small clearance from the propeller tips. The same author states that this kind of mixer compares very favourably with the more common "straight arm or blade paddle mixer," irrespective of whether the blades are flat or tilted. It was found that a propeller mixer took $\frac{1}{3}$ of the time and 25% less power than the blade paddle mixer to achieve the same degree of mixing. It will be clear that this

comparison would be even more unfavourable in the event of the tank being square. It will be noted that the lime and juice are fed in the direction of the flow and will be mixed immediately by the action of the propeller. Moreover, thorough mixing will occur with the juice circulating in the vessel. The overflow ensures that at any given time the amount of juice discharged will be equal to the volume added. Thus a continuous system is created.

We still have to decide on the size of the vessel as this determines the average retention time of the juice in the vessel. The reports of different workers⁵ on the time necessary to obtain maximum degree of purification are contradictory but seem generally to favour short contact time with a low density milk of lime. This suits our design very well and we take 2 minutes retention time as an average. Taking the example of a factory crushing 100 T.C.H. the following data can be calculated: 100 T.C.H. equals 100 tons of Juice per hour if weight of cane equals weight of juice. The expected amount of juice per second will be:

$$\frac{100 \times 2000}{1.06 \times 62.4 \times 3600} = 0.85 \text{ cu.ft./sec.}$$

(assuming 1.06 is the average specific gravity of mixed juice).

With an average retention time of 2 minutes, the capacity of the tank should be:

$$2 \times 60 \times 0.85 = 100 \text{ cu.ft. nett.}$$

It is good practice to take $D=h$ and with sufficient accuracy:

$$D = \sqrt[3]{\frac{4 \times 100}{\pi}} = 5 \text{ ft.}$$

d is generally taken as $\frac{1}{3}D$ and hence $d=20"$.

A good circulation and mixing will be obtained when the dilution is 20-fold, in other words, 20 gallons of total juice are recirculated for each gallon fresh juice entering the system. So, in the case of our example the juice displacement must be:

$$20 \times 0.85 = 17 \text{ cu.ft./sec.}$$

The speed of flow in the centre pipe will accordingly be:

$$\frac{17 \times 4}{\pi \times 1.67^2} = 7.8 \text{ ft./sec.}$$

If the propeller is revolving at 300 R.P.M., the distance to be covered by the fluid per 1 R.P.M. will be:

$$\frac{7.8 \times 12 \times 60}{300} = 19"$$

This 19" distance is equal to the pitch of the propeller and Perry⁶ gives nomographs to determine the necessary horsepower (see page 1561-1562). This in our case, will be approximately 2 H.P. and taking

into account the mechanical efficiency of propellor and drive, 5 H.P. will be ample.

Apparently a mixing plant can be made by very simple means but we have still to resolve the question of the determination and setting of the required pH.

It will be clear that our mixing vessel is also a reaction tank and hence the pH of the overflowing juice can be regarded as a yardstick for the pH in the vessel. Moreover, due to the thorough mixing, there will be virtually no time lag between the addition of the lime and the response by the pH-meter or pH-paper, provided the sample is taken immediately at the overflow. The next step is to have the pH determined by an automatic pH-controller and to accomplish the addition of the lime automatically. Figure 1 shows how the sampling should be done both with and without a pH-controller. Most factories are not equipped with automatic pH-controllers and although regrettable, other means of controlling the lime dose must be found. Another way of liming is to add the lime flow proportionately and reset the proportionality ratio by hand if necessary. The Java Experimental Station constructed a very simple and very reliable apparatus for lime proportioning. The apparatus has already been described by Perk² and the principle is shown in Fig. 2.

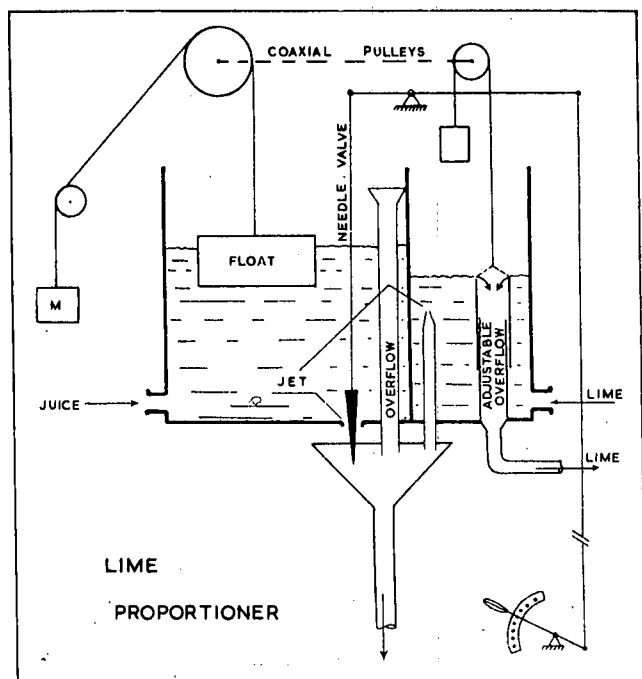


Fig. 2.

The big advantage of this apparatus is that its driving force is gravity, that it contains few moving parts and is easily accessible during actual working. It was shown by Perk that the proportionality could be altered by inserting a needle in the lime nozzle, thus partially restructuring the flow of lime,

while still maintaining a revised proportionality. Later it was found that it was much more practicable to insert a needle in the main juice nozzle, thus preventing the clogging of the much smaller lime nozzle. Restriction of the flow of juice will increase the level in the juice compartment and subsequently in the lime compartment. Thus restriction of the flow of juice increases the flow proportionate lime dose. Although this method ensures rather smooth regulation of the pH, the question of the determination of the pH still remains. There can be no doubt that the use of a lime proportioner in conjunction with pH-paper is by far inferior to automatic pH control. If the lime proportioner is used in conjunction with a pH-meter instead of pH-paper, much better results can be expected. At the moment there are several makes of rugged non-electronic pH-meters on the market which sell at very reasonable prices. The pH-meters consist of an Antimony electrode and an Ag-AgCl reference electrode. They are very rigid and can be used continuously if some precautions are taken.

They are guaranteed to 60°C but I have used them satisfactorily during long periods at 75°C without any apparent damage. Fig. 3 shows a photograph of a pH-meter of the type described. One of these meters is in the possession of the S.M.R.I. and having been lent on different occasions to factories, has been very favourably commented upon.

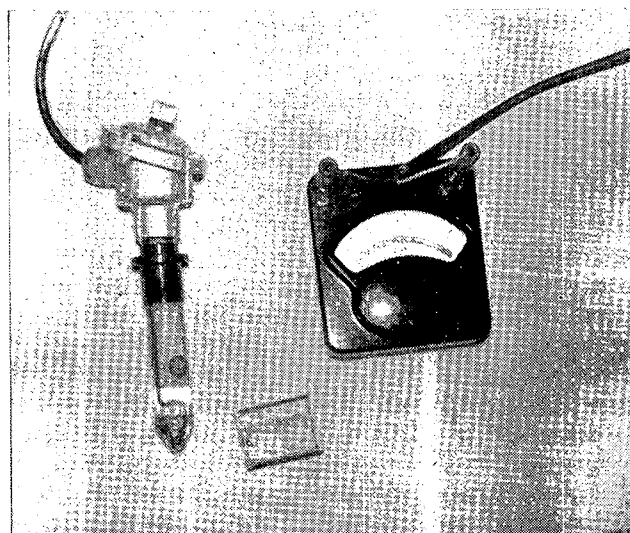


Fig. 3.

Two check curves are shown in Fig. IV. It will be noted that if a check point is chosen near enough to the desired average pH, it is possible to make readings which differ less than 0.06 pH per one unit of pH with the true pH.

However, the best possible way of controlling the pH is to have an automatic pH controller moving

the needle valve in the lime proportioner. The advantages are:

- (a) If the flow of juice changes, the pH-meter need not react because the flow of lime remains proportional to the flow of juice.
- (b) the pH-meter only reacts when the nature of the juice necessitates the resetting of the amount of lime in order to reach a predetermined pH.

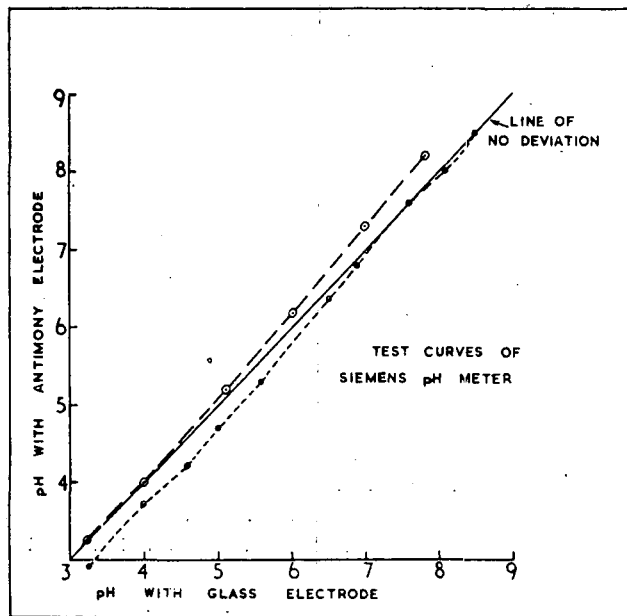


Fig. 4.

The last unit process to be considered is "settling". Modern industry has passed far beyond the stage when individuals concerned with processing experimented with different sizes and constructions of settling tanks. Nowadays units are bought, built by big firms which have had an enormous amount of practical experience. This, however, results in the building of subsiders eminently suited to normal average circumstances in all sugar growing countries. These average circumstances do not always prevail in Natal, where even in small areas distinct differences can exist. Therefore some caution must be exercised when accepting data concerning the performances of subsiders. Care should be taken that so-called simplifications of the basic design do not interfere with the proper working of the apparatus.

Which Process ?

Having previously considered what was, technologically, the best way of handling the juice during the defecation process, consideration must now be given to the sequence of liming and heating. As already stated, it is very difficult, in fact almost impossible to give detailed recommendations with regard to the "best process". And that even more so, because many advantages can only be achieved at the cost of some disadvantages.

I would like to draw attention to the precaution taken at some factories with regard to the breaking up of the flocs. Liming is done after heating to the boiling point and even mechanical mixing is avoided. Davies⁵ mentions that flocs are of insufficient mechanical strength to withstand moderate industrial handling. He also thinks that a modern high velocity heater has a detrimental effect upon the flocs due to the turbulence in the tube and regards an open steam coil defecation pan as more beneficial. He advocates flow by gravity after initial liming. However, it has been demonstrated by Behne⁵ that the rates of reactions are increased by boiling and in order to complete the reactions between lime and phosphates as rapidly as possible the temperature of the juice should be raised to boiling point or just above. A final heating with some moderate counter pressure enables us to raise the temperature above boiling point which increases the speed of the chemical reactions. Also, the high temperature creates heavy flashing in the flash tank ensuring that the temperature of the juice entering the subsiders will be constant, viz. boiling point. This will greatly reduce any turbulence due to convection inside the clarifier. Again Davies⁵ thinks that this is an advantage, because he considers that "the gentle movement caused by convection would tend to roll the flocs together", thus providing collision and adhesion. However, it is just as conceivable that any such movement of this sort is deleterious, because the clarifier is not built to condition flocs but to settle them, which will be in the simplest and quickest way when there is no turbulence.

It is generally agreed that cold liming gives a clearer juice but higher mud volumes. Clear juice is not a very useful measurement of the purification but the mud volume is of the utmost practical importance. Moreover, any growth of bacteria is rapidly terminated when the juice is heated to above 160°F. This must be seen as a great advantage, because bacteria can add highly undesirable colloids to the juice. Also, it can be stated that several authors place importance upon the fact that the juice is heated at relatively low pH's, so as to allow the precipitation of proteins at or near their isoelectric point.² However, considerable inversion may take place at low pH's and raised temperatures and therefore the process of heating should be a short one and the liming process should follow suit.

It is worthwhile also to consider the disadvantage of liming at boiling point if a final heating is applied. In order to raise the temperature a few degrees, a heater should be available, which in this case is an extra one, because the juice was already at boiling point. Therefore liming is often done at temperatures between 75° and 85°C because such a temperature can easily be obtained with vapour while the additional rise in temperature required can be achieved by one heater only.

Another controversial factor is time. Some technologists advocate a retention time of over 20 min. in order to ensure that all reactions are complete. Other authors write that all major inorganic reactions take place fairly rapidly. However, taking into account the very long time that the juice is kept in a clarifier where there is ample time available to complete any reactions, it seems unnecessary to keep the juice in process for any length of time at a temperature under boiling point, even more so because the modern clarifiers are fitted with so-called flocculation chambers.

From the above we conclude that in most cases and with normal average juices, the best results would be obtainable when:

- (1) The juice is heated to approx. 160° to 190°F in order to stop bacteriological contamination immediately and to increase the speed of the chemical and physical reactions taking place.
- (2) Liming is done to a certain predetermined pH in a mixing tank of adequate construction, so as to avoid extreme high or low pH's for any length of time.
- (3) The final heating will be to a temperature well over boiling point (about 215°F) with good flashing afterwards. The juice will enter the clarifier exactly at boiling point, which ensures highest possible chemical reaction velocity and lowest possible turbulence.

Fig. V shows a flowsheet in which the foregoing is shown schematically. The different possibilities are:

(a) With the aid of a pH-controller.

If the valves B and C are closed and the valves A and D open, the juice would follow a straight course from the scale through the heaters and from there to the defecator. The defecator would overflow into a constant-level tank and a pump working at constant maximum flow in order to prevent the breaking of the flocs as far as possible, would bring the juice to the flashtank and subsider via the heaters.

(b) With the aid of a lime proportioner

When the valves A and D are closed, B and C open, E closed and F open, then the pH is controlled by the lime proportioner.

(c) With the aid of pH controller and lime proportioner

The splitterbox can be by-passed completely and the controller linked up with the needle in the juice discharge-jet of the lime proportioner.

(d) The fractional liming and double heating process

If the valves A, B, C and D are kept in the same position as under (b) but E is opened and F closed and a lime proportioner as well as a pH-controller are used, it will be clear that the lime proportioner would add milk of lime to the cold juice and the pH controller would add the final dose of milk of lime.

It will be appreciated that several items of the machinery as shown in the flowsheet can be omitted according to the process required. In case (a) the installation of a lime proportioner and in case (b) a splitterbox is not necessary. However, it will be clear that the flowsheet gives a universal set-up except in cases where the gravity flow is regarded as being necessary.

BIBLIOGRAPHY

- ¹ Davies, J. G., and Yearwood, R. D. E: International Sugar Journal, 38 (1936), 298.
- ² Payne, J. H.: Principles of Sugar Technology edited by P. Honig, Vol. I (1953).
- ³ Hugot, E.: La Sucrierie de Cannes (1950).
- ⁴ Tromp, L. A.: Machinery and Equipment of the Cane Sugar Factory (1946).
- ⁵ Valentine, K. S. & MacLean, G.: Chemical Engineers Handbook edited by J. H. Perry (1941) (1527-1583).
- ⁶ Davies, J. G.: Principles of Sugar Technology edited by P. Honig (1953), 563.
- ⁷ Perk, C. G. M.: Automatic Pre-Liming. South Afr. Sugar Jnl., 38 (1954), 371.
- ⁸ Behne, E. R.: Dept. Agr. Brisbane, Queensland, Bur. Sugar Exp. St., Techn. Comm., No. 4 (1941), 85.

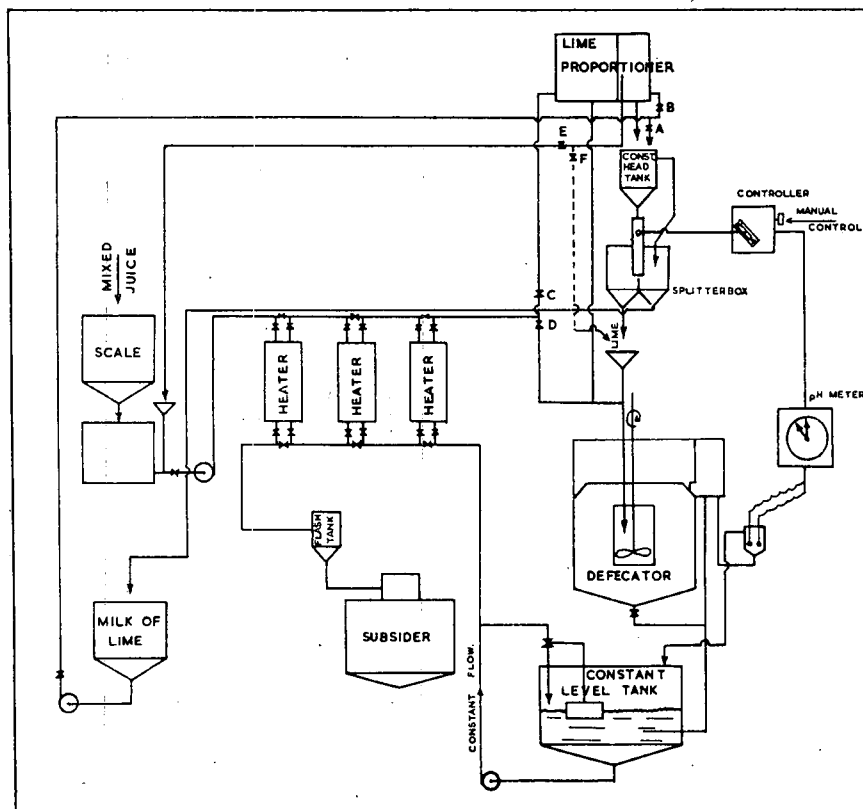


Fig. 5.

Mr. van Hengel paid tribute to all the Factory Managers who had allowed him to obtain the information he required for his paper.

Mr. Rault asked if the portable antimony pH meter was now working satisfactorily.

Mr. van Hengel said he was glad the S.M.R.I. had been able to import one of these meters, because he had used it in Java very successfully. He said it could be regarded as being equivalent to a good glass electrode pH meter. He had seen this latter instrument very much abused however. On occasions, he had seen an operator using pH paper and powdered lime with the same hand, after which the piece of pH paper promptly turned blue. If it is felt that a glass electrode meter is unnecessary, then an antimony electrode can render far better service than pH paper. Only if the pH meter was properly used could good results be obtained. The only maintenance it required was at the week-end stop, when perhaps a quarter-hour would get it cleaned and further, it could not easily be damaged by unskilled labour.

Mr. Alexander said that as far as the antimony electrode was concerned he would like to add a word of warning to would-be purchasers that they first ascertain whether the instrument was suitable for their particular purpose. He suggested that the instrument would be unsuitable over a wide range of pH and that in any case it should be standardised at a point near to the pH at which it was to be used.

Cases of poisoning and pH drift had been reported in sulphited juices.

Mr. van Hengel referring to figure 4, said that it could be seen that quite a considerable mistake had been made when the standardisation of the apparatus was not carried at about the pH it was going to be used. However, it had the advantage over the use of pH paper which was subject to many disturbing factors.

Mr. Alexander asked Mr. van Hengel if in his opinion it was satisfactory to use the antimony electrode on sulphitation juices.

Mr. van Hengel said that it had been used for sulphitation, but it had to be cleaned every two hours or so with hydrochloric acid. No trouble was encountered if this was done.

Mr. du Toit asked what substances would poison the electrode.

Mr. Alexander replied that some makers admitted that sulphites could do so.

Mr. Rault said that the pH figures determined on juices cooled down to laboratory temperatures were not the same as registered on the factory instruments, controlling juice reactions at 60° to 90°C and these two different readings on the same product often caused confusion and mistrust of pH control in the worker's mind.

Dr. Douwes-Dekker said that the antimony electrode was actually used in many countries and, at times, with the sulphitation process. A certain amount of difficulty had been experienced. A simple solution was to wash it every two hours or so with hydrochloric acid. He said that Javan experience had found that antimony electrodes might on occasion give incorrect readings, but this was not due to poisoning. He said that at about 7.5-8.0 pH, the electrode started to give a different set of readings, but if it was checked at the point at which it was normally being used, the antimony electrode gave very good service. He said that the paper had been written as an attempt to assist factories in producing the best possible quality defecation raw sugar. It was sometimes said that the difficulties experienced with Natal raw sugar originated from the introduction of the defecation process. This was not true, complaints had been received when all raw sugar was still made by sulphitation. Sulphitation raw sugar was mainly superior to defecation raw sugar in respect of colour and wax, but this could be expected since the sulphitation method was originally a white sugar classification technique. It was wrong to say, that because some factories produced defecation raw sugar of not as high a quality as they expected, that the defecation process was unsuitable. He thought that the technological application of the process was the important factor. He said that the S.M.R.I. had made a survey of the different ways in which the defecation process has been applied in different factories. After this survey it was thought that considerable improvement could be made in the application of the defecation process in various factories. It would be an advantage to many factories to install the process outlined in the paper, and that at the moment considerable improvement could be carried out in various mills. For example, the mixing of the milk of lime with the juice in square tanks was poor and if this was given all the attention it deserved by Factory Managers the results of defecation would be considerably improved.

Mr. Rault said that it was not always possible to install a new plant similar to the one indicated in this paper, but the Sugar Milling Research Institute could help by informing them of the mistakes they might be making with existing arrangements with a view to modifying certain details without having to discard completely a costly installation.

Dr. Douwes-Denker said that instead of pointing out to all the factories where improvements were possible, a more direct approach had been to give them an outline of the process which had given the best results.

Mr. Boyes thought that in applying defecation one should take into consideration the whole process, including the operation of the filters. For instance,

cold liming might give poor results because of a poor filtering station. As far as flocculation was concerned, he wondered if it was advisable to have a flocculating tank before the juice entered the subsider. Some authorities advised pre-flocculation, others said that because the juice had to be subsided for two hours, this was not necessary. It was fascinating to watch flocs rolling together to form larger particles and so he had advised the installation of a flocculating tank, but it was very difficult to judge whether it was carrying out what was desired or not. A point to be studied was the temperature at which the lime should be added and the heating thereafter. He thought that a higher temperature than 215° would be to greater advantage. A more detailed study of the various techniques of carrying out defecation was required. This was essentially the chemical aspect of the problem.

Mr. van Hengel said that he was not confident that in this case the same chemical reactions with lime could be repeated in one factory which did not have the same installation as another. What he had tried to advocate was not the application of a certain process, but the application of certain plant which would allow of the proper application of the defecation process irrespective of the sequence of liming, etc. If the plant was not efficiently designed to duplicate the laboratory tests then the laboratory tests were of no value. He did not think that increasing the temperature of the juice above 215° would improve matters. 215° was a practical temperature to use as higher temperatures would mean the installation of tremendous flash tanks. He thought in the installation of flocculation tanks over the Rapid-Dorr, the time was so short and five minutes or so would have no effect on improving flocculation. He said that in Java they had found that the juice leaving the flash tank and entering the flocculation tank, entered tangentially and caused too much turbulence.

Mr. Perk said that the Deming Clarification Process heated the juice to very high temperatures; this process being used in Hawaii and Mexico before the turn of the century. The juice was first passed through a heat exchanger, the cold juice passing across the tubes of this apparatus, picking up heat from the super-heated juice flowing through the tubes and subsequently through a set of heaters which heated the juice to temperatures between 230° and 250°F. Immediately afterwards the juice was cooled down to 210°F by passing it through the heat exchanger, this time flowing inside the tubes.

Mr. Walsh said that as far as super-heating of the juice to 220°F was concerned, this meant that the juice had to be fed into the flash tank through a valve which dispersed the flocculation. The only way they had found to reduce the pressure was to use a barometric column.

Mr. van Hengel said that he chose 215° because it was only 3° above the boiling point; this excess temperature could be lost through the vertical piping leading from the juice heater to the flash-tank. He agreed that flash-tanks caused a tremendous amount of turbulence but he thought that some flash tanks in this country were effective and caused no difficulty with respect to breaking up of the floc.

Mr. Boyes said that it was quite easy to get the temperature to 225° and through a flash tank reduce the temperature to 212°. He did not think that this would cause any undue turbulence. He also said that while people overseas had the idea that they could break up the floc with flash tanks, they now seemed to have changed their opinion.