SOME NOTES ON THE DETERMINATION OF THE GRIST OF SUGARS WITH REFERENCE TO THE MEAN APERTURE--CO-EFFICIENT OF VARIATION CONCEPT

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It is possible to obtain most substances in the form of discrete particles, but whether these particles are grown from a small neucleus, as in the case of sugar, or reduced to this small size from larger particles, it is only under the most carefully controlled conditions that all the ultimate particles will be of the same size. Even the shapes of these ultimate particles are not readily made to conform exactly to any predetermined configuration, and for a long time, efforts have been made to find a simple and easily comprehended method of expressing the overall picture of the variations in sizes and shapes of the particles in a given mass of material.

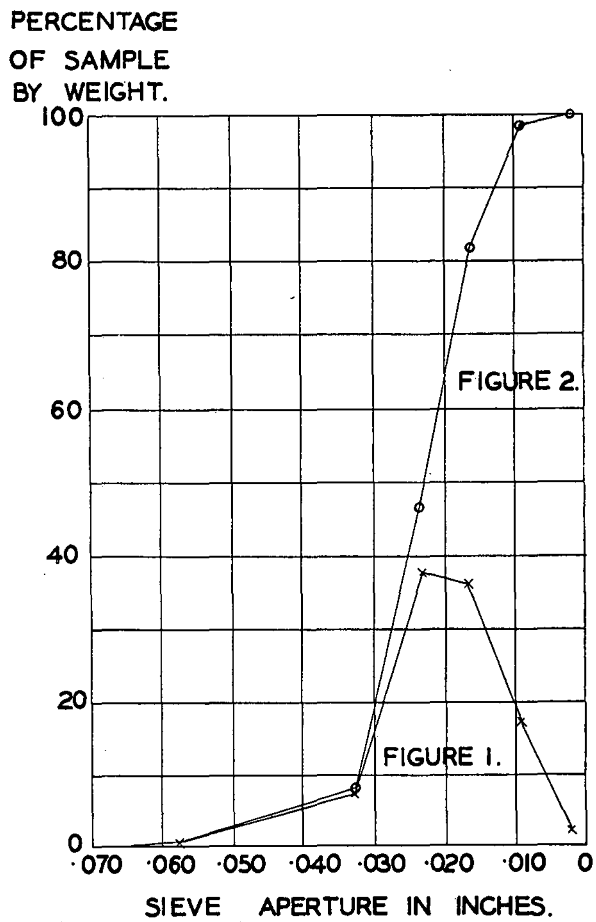
In case these efforts may be thought of as just another manner in which a scientist or technologist can justify his spending (some would even be unkind enough to say wasting) a lot of time, there are many instances of the practical use of a close appreciation of the variation in shapes or sizes from the desired or average value.

To take two examples only from our own industry, in the case of raw sugar manufacture, it is important that the magma used for seeding should be of as uniform a size as possible and without conglomerates, so that the final product will also have crystals of uniform size and shape, since this facilitates the spinning and drying of the massecuite. At the moment, this procedure of magma seeding lies within the province of the sugar miller, but it is quite possible that in the not too far distant future, some form of magma seeding may be used in the refinery. In the case of refined sugar, it is the variations in size and shape of crystals which are responsible for the attractive or otherwise appearance of the finished product.

At a previous S.A.S.T.A. Congress, Dr. Douwes Dekker[1] read a paper connected with the properties of raw sugar, in which he stressed the desirability of obtaining raw sugar in the form of single crystals without conglomerates and of as regular a size as possible. The reason for this, he pointed out, was that the improved purging, washing and drying would reduce the molasses film to as small an amount as possible, the advantage of this being that most deterioration problems find their source in this unwanted substance.

The actual irregularities of grain size also have an effect on deterioration from the physical standpoint, as opposed to the chemical factors usually considered, since the small grain readily fills up the spaces between the large grains, and this process continues with every small vibration until the pile of sugar would eventually consolidate even with a minimum of chemical deterioration.

In this same paper, Dr. Douwes Dekker introduced, I believe, to Natal the concept of Specific Grain Size, which is one of the methods mentioned earlier of reducing to a description in simple terms the variations in size of the particles of any material.



Without wishing to become involved in arguments concerning the merits and demerits of this idea, I should now like to place before you another concept, that of Mean Aperture and Co-efficient of Variation which was evolved by Philip Lyle, and is in common use in the Tate & Lyle refineries, both in the laboratory, and in the pan house.

This concept is described in the *International Sugar Journal* in an article by Powers,[2] and it is extremely simple in use since only two sieves are necessary once certain prerequisites have been determined.

The first requisite concerns these sieves, and it is the fairly obvious one that they should have a high standard of accuracy and their aperture sizes should so be chosen that the one of larger aperture should retain about 10 per cent. of the sugar, and the one of smaller aperture should allow about 10 per cent. of the sugar to pass, when the sieving operation has been allowed to proceed for such a time that any sieve has passed all the sugar that it is capable of passing.

The second requirement concerns the sugar itself and is more easily understood if one considers mathematically and graphically a complete gristing with a full range of sieves. Fig. 1 represents a typical grist analysis of Hulett's Refined Sugar, plotted with the sieve apertures as abscissae and the percentages remaining on each sieve as ordinates. This shows a rather wide peak with a spread each side into coarse and fine fractions, and the aim of a sugar refiinery is to produce refined sugar which has as high a peak as possible with a narrow but symmetrical spread on each side.

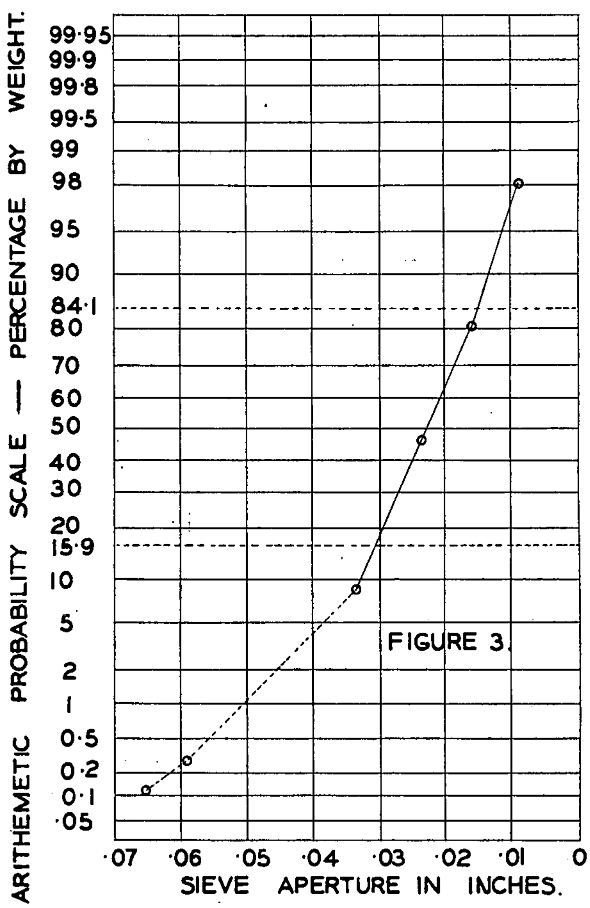
If, instead of plotting the actual percentages retained by each sieve as ordinates, one plots the cumulative percentages, that is, the percentage retained by each sieve, plus the percentages retained by all sieves having a bigger aperture than the particular one in question, the graph assumes the from of Fig. 2, which is seen to resemble a straight line, with a certain amount of fading-away at each end, i.e. in the 0 to 10 per cent. and the 90 to 100 per cent. regions.

If the sieving results used for Fig. 2 are further plotted on arithmetic probability graph paper, the line becomes much more closely a straight line, and within the region 10 to 90 per cent., it can be assumed to be straight within the limits of the experimental errors involved in the actual sieving determinations. This gives the graph in Fig. 3.

This is the second requirement of this present concept and it leads to the simple mathematical fact that only two points on the graph are required to fix completely the straight line, and this is the explanation for the sieving determination only being carried out with two sieves as detailed in the first requisite.

Once the two sieving results have been obtained, and the graph has been drawn, the Mean Aperture can be read directly from the graph, since it is defined as the size of sieve aperture which would retain 50 per cent. by weight of the sample and would permit 50 per cent. by weight to pass.

The mathematical theory of statistics is now brought into use, and this states that in a normal distribution, there are two sizes, one larger than, and one smaller than, the Mean, each of which differs from the mean by a certain amount, and this certain amount is called the Standard Deviation. Furthermore, 15.9 per cent. of the sample will be bigger than this standard deviation above the mean size, and 15.9 per cent. will be smaller than the standard deviation below the mean size. It is an easy matter



therefore, to see that the Standard Deviation can be found from Fig. 3 by reading off the sieve apertures corresponding to 15.9 per cent. retained, and 84.1 per cent. retained, and the difference between these two sieve sizes will be twice the Standard Deviation.

In actual use in Tate & Lyle's refineries, and in Hulett's South African refineries, the necessity of drawing the graph and reading off the Means and Standard Deviations, has been obviated by the working out of a set of tables which relate the required results directly to the percentages remaining on the two sieves.

Tate & Lyle consider that the characteristics of the grain size distribution of a sugar are better described by the ratio of the Standard Deviation to the Mean Aperture rather than the Standard Deviation itself, and it is this ratio expressed as a percentage which is termed the Co-efficient of Variation.

Thus the present concept can be summarised as follows: The Mean Aperture is the calculated aperture which would retain 50 per cent. by weight of the sample and allow the other 50 per cent. to pass, and the Co-efficient of Variation is the difference between the two calculated aperture sizes, which would retain 15.9 per cent. and 84.1 per cent. respectively divided by twice the Mean Aperture and multiplied by 100.

In the actual example taken for Figs. 1, 2 and 3, the Mean Aperture is read from the graph to be 0.022 inches. The aperture retaining 15.9 per cent. is read off as 0.029 inches and the aperture retaining 84.1 per cent. is read off as 0.014 inches. Thus the Co-efficient of Variation is calculated as:

$$\frac{0.029''-0.014''}{0.022''\times 2}\times 100=34$$

Hence the sugar taken for this example would be described as having the following grist analysis: M.A. 0.022", C.V. 34.

Tate & Lyle have fixed specifications for their several sorts of refined and castor sugars, and without going into details, it is sufficient to say that the pansmen are fully aware of this simple method of expressing the appearance of the sugar they have boiled, and they also know whether it is necessary for them to make any adjustments to their boiling technique to improve their next pan.

Acknowledgements

I should like to express my thanks to the management of Hulett's South African Refineries for permission to publish this short paper and to my colleagues for their encouragement.

I should also like to place on record my delight at being the means of bringing to an end a long series of sustained silences by the staff of Hulett's South African Refineries in so far as the reading of papers is concerned, and I would like to express the earnest hope that more regular contributions will be made at future Congresses of the S.A. Sugar Technologists' Association.

Summary

A discussion is entered into concerning sizes and shapes of particles, with particular reference to sugar, and mention is made of the concept of Specific Grain Size.

It is then concerned with the Mean Aperture-Co-efficient of Variation concept, as evolved and used by Tate & Lyle, with definitions of the terms, and a description of the method of obtaining these values by a single sieving using two carefully chosen accurate sieves.

[1] Douwes Dekker, K. (1952): Some Notes on the Properties of Raw Sugar in connection with Deterioration during Storage. Proc. S.A. Sugar Tech. Ass. 26, p. 40.

[2] Powers, H. E. C. (1948): Determination of the Grist of Sugars. Int. Sugar Journal. Vol. L, No. 594 (June, 1948), p. 149

The Chairman, Mr. Rault, said that the subject of this paper was of some importance in standardising our final product.

Sugar sales are, and would continue to be, on appearance, and laboratory tests should in some measure agree with the superficial judgment of the buyer. Colour determinations are now standardised by the I.C.U.M.S.A., and it is hoped that figures referring to crystal size and its regularity would also be accepted by the consuming public.

He hoped Mr. Hastilow would be successful in educating buyers to appreciate figures expressing the grain-size of sugar, rather than the visual appearance.

Mr. Hastilow said he was not quite sure that the public really knew what they wanted as far as grain size was concerned. He found that some people who asked for a smaller grain-size, when confronted with a more regular grain which was even bigger than before, were completely nonplussed.

Mr. Boyes asked Mr. Hastilow what application this method would have to raw sugars. At Tongaat they prefer to measure crystal sizes under a microscope. He also thought that the actual sieving operations might break up the grain.

Mr. Hastilow replied that the sieves did tend to break up the grain to a slight extent but were more likely to break up congolmerates.

He stated that even by washing raw sugar with 70 per cent alcohol saturated with sucrose, it was difficult to keep the crystal size at its original level.

If, however, the sugar could be sieved, this would be much easier to do than to try and measure large quantities of sugar under a microscope.

Dr. van der Pol enquired of Mr. Hastilow what the average mean variation would be in a raw sugar which would be of good affination properties.

Mr. Hastilow replied that even though the Refinery was interested in the crystal properties of raw sugar from the point of view of good affination it was much more concerned at the moment with the factors causing deterioration and consolidation.

Mr. Beesley said that the two methods of determining grain size (sieve test and photomicrographic) both had their own advantages and disadvantages. For instance, the sieve test was very much quicker than the photomicrographic method and hence was more applicable to good dry sugars. However it was practically impossible to apply it to a C sugar and hence the micrographic method was the best to use in this case.

A vast number of photomicrographic analyses had been carried out at the S.M.R.I. and had clearly shown that this method, where the basis was number of crystals (in the sieve method the basis is weight of fractions), also gave a normal distribution of sizes and that the Mean Crystal Length and Co-efficient of Variation best described the properties of the crystals.

It therefore appeared that the two methods could possibly be correlated by some formula, such that it would be possible to express crystal properties by the M.A.—C.V. concept no matter how the analyses had been carried out.

This would be of immense value and would cover the complete range from dry refined to sticky C sugars.

The Chairman said that possibly through slower growth, it was common experience that graining from a comparatively low purity liquor, produced a very uniform size crystal, whilst the product crystallised from the purer refinery liquors, contained more mixed grains.

A quick method of determining the size and proportion of crystals, specially in a viscous massecuite, would be very valuable, as regularity of grain was an advantage in the purging of massecuites.

He had observed some very regular, although small crystals, in beet factories, but this regularity was obtained by sieving installations at the bagging department, and was not due to more efficient methods of pan boiling.

Mr. Hastilow said he considered the explanation for poor crystal sizes of refined sugar was that the sucrose liquor was so highly super-saturated that sucrose had to be deposited on any face of the crystals, so they did not grow regularly, whereas, with low purity material, where the deposition of sucrose was slower, it was easier to obtain regularly shaped crystals. He considered that the specific grain size could not give a reasonable description of the sugar, whereas the co-efficient of variation could do so.

Mr. Perk replying to Mr. Hastilow drew attention to the fact that the specific grain size was never used alone, where description of a sugar was concerned, but always together with two other characteristics.

In the paper read by Dr. Douwes Dekker at the 26th Annual Congress when discussing "Size and Regularity of Sugar Crystals," Dr. Douwes Dekker mentioned as ultimate characteristics:

(i) the specific grain size of the sugar (s.g.s.), which was calculated from the weights of the fractions by a method described in Appendix II;

(ii) the weight of the main fraction;

(iii) the weight of the finest fraction(s).

(see Proc. 26th Congress; p. 40.)

These three characteristics give a complete picture of the size and the regularity of the sugar concerned.

The Chairman said he was glad to have such a paper because it was high time we departed in the sugar industry from mere subjective opinion, and were able to express conditions and standards by figures.