RAW SUGAR QUALITY

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ABSTRACT

Thirteen quality criteria considered to be important in the evaluation of raw sugar as a raw material for a sugar refinery are listed. A brief review is given in elaboration of each criterion for the purpose of initiating discussion rather than attempting comprehensive coverage.

Reference is made to advantages of up-dating certain analytical practices, particularly with respect to pol/sucrose relationships.

Attention is drawn to the need for keeping economic considerations in their correct perspective. The relevance of three situations is indicated — the actual value of raw sugar as an independent item, the operating costs of a refinery as a separate financial organisation and the marginal cost of the refining process when carried out as an on-site extension of raw sugar production.

INTRODUCTION

The subject of this paper is of substantial concern both to milling and refining interests in the relation of seller and customer. The relationships were at one time very frequently as between 2 sections of the one organisation when ownership of sugar estates and associated mills was in the hands of European or American concerns operating associated refineries in centres of high population density. The relationship became more tenuous with the coming of independence of countries growing cane and today there are also countries growing cane for raw sugar production and which never were under overseas control. As a result there is an active market into which it has been sought to inject a measure of control through the medium of the International Sugar Agreement.

It is undoubtedly a fact that the criterion of greatest concern to both buyer and seller is the price. This has varied substantially over a range for which a factor of 6 or even more has been known between the lows and the highs. By comparison quality variations are largely marginal.

Nevertheless quality variations are important and reliability from a particular source engenders confidence in the customer and substantially improves relationships.

Whilst raw sugar of concern to sugarcane technologists is immediately associated with the product from sugarcane, as a term of international concern raw sugar may also refer to a corresponding product from sugar beet. Although beet factories are usually associated with the direct production of sugar of retail market grade, they are also important to the raw sugar market in certain areas. In many countries cane sugar also appears directly from a mill as a marketable white sugar for domestic consumption. Occasionally this may even be of the best refined sugar quality.

This paper is restricted to a consideration of the quality requirements of raw sugar from a sugarcane primary source.
It is well known that as well as there being variations in quality from different sources, purchasers also are by no means uniform in their requirements. Refineries which focus attention on a high rate of throughput tend to prefer a high polarising raw sugar. On the other hand refineries which may not even operate for the full 12 months of a year tend to prefer a lower polarising raw sugar which will give them more material to work on. There are different reasons for such decisions and they are not subjects for general discussion.

Much of the information relevant to the refining of raw sugar, especially in relation to costing and value considerations, has been of a confidential nature which has largely restricted specific discussion. On the other hand many small windows have been opened from time to time into refinery operations by way of published information — both technical and otherwise. The observant and enquiring person can find all he needs to know.

This paper is not a review of such publications and omission of references is no reflection on the quality or importance of any piece of published information. The views expressed are entirely the opinions of the author for which he accepts full responsibility and concedes alternative opinions, but these are submitted as a basis for discussion. It would be invidious to attempt a comprehensive list of the many processing and technical aspects covered in textbooks and original publications.

QUALITY SPECIFICATIONS

There is no single criterion which can adequately represent the desired quality of raw sugar. In fact at least 13 criteria have been identified and these are listed as a starting point:

1. Polarisation
2. Moisture content
3. Total impurities content
4. Concentration of specific impurity groups
5. Concentration of specific impurity components
6. Colour value of whole crystal
7. Colour distribution within crystal
8. Concentrated coloured inclusions
9. Average crystal size
10. Uniformity of crystal size
11. Crystal shape
12. Mono-crystalline character — conglomerate effect
13. Filterability

It is not possible to discuss exhaustively each criterion and comment is limited, intending at times to be provocative.

1. POLARISATION

Whilst this is the main index of quality it is of course only a physical measurement known usually to be slightly less than the actually desired sucrose content and hence a small bonus to the refiner. The question might be raised — is the industry now sufficiently sophisticated to require a sucrose analysis rather than polar? The answer may well be “yes” in the case of well-developed refinery interests, but must still be “no” in certain developing countries especially those more recently entering the international market.
To require specification of analytical requirements by a qualified umpire would probably represent more inconvenience than would be justified. Qualified umpires have their use when a dispute arises between buyer and seller but their use is not a particularly convenient way of dealing with a continuing problem. It has been difficult enough to define an acceptable analytical technique simply for polarisation, but perhaps thought could be given at this stage to the requirements for a "quantum jump" to sucrose analysis itself.

2. MOISTURE CONTENT

This is mainly used in terms of Dilution Indicator or Safety Factor as a criterion of keeping quality. In this context it is not the moisture content alone which is the criterion of keeping quality but rather a specified relationship between moisture content and total impurities (actually non-pol).

The moisture content itself does have some relevance in the context of caking either in bulk or in bags but is not a sole criterion of this very important undesirable behaviour characteristic. Environmental conditions are complementary and environmental variability is probably more important than many conditions.

Unfortunately we usually know only approximately the real moisture content of raw sugar due to the inadequacies of the conventional oven-drying techniques. From the point of view of Dilution Indicator and its importance the effects of this error may be considered marginal. However when it comes to estimating total impurities or "other organic matter" (OOM) then the fractional error in these estimates becomes substantial.

3. TOTAL IMPURITIES CONTENT

The impurities are those substances which are non-pol but preferably non-sucrose and which do not include moisture. Because, total impurities are determined by difference-analysis the use of pol instead of sucrose introduces a significant percentage error in the impurities concentration — usually an inflationary error. On the other hand the error introduced by using an oven-dried moisture content compensates probably very closely. Any net error will be further magnified when studying the group which is wholly dependent on difference analysis, viz. the "other organic matter" (OOM).

If a change is made from pol to sucrose then the moisture should ideally be corrected at the same time or vice versa.

It is common practice in all fields of analysis, be it foodstuffs or metals or other materials, to focus attention on impurity concentrations when dealing with nearly pure conditions. Traces of inorganic impurities are much easier to analyse with precision in the case of a nearly pure metal than the traces of mixed materials of organic origin which are lumped together as OOM in a sugar analysis.

The main use of the group figure for total impurities content is to give us a first indication of the probable molasses loss in a refinery from a particular raw sugar.

Much emphasis has been placed on recovery in a refinery and Oliver Lyle drew the curtain back to reveal something of the way refiners used to think on this subject.
Empirical formulae of earlier years have progressively been discarded and simple application of the SJM formula will give the recovery of sucrose to be expected from any raw sugar, in which the "J" represents the purity of the raw sugar.

This formula, when so applied, does not take into account the undetermined and other losses which are almost entirely related to refinery procedures. They should not be a factor in estimating potential yield from a particular raw sugar, but should receive some assessment when evaluating the actual yield to be expected.

The use of "true" rather than "apparent" purities substantially improves the precision of applicability, particularly in relation to the final molasses purity.

Otherwise the formula is fundamentally correct being merely a mathematical expression of a projected materials balance.

There is however, a very important question arising from this statement — what molasses purity should be taken? As already indicated it should be true purity and not apparent purity. But to go to further stages of sophistication poses serious problems some of which will be considered under sections dealing with specific impurities.

This subject was extensively reviewed in 1956 by Honig3 whose preferences at that time were pragmatically very strongly in favour of using apparent purities. The author equally as strongly but respectfully disagrees and especially so with refinery situations where the relatively high purity of the raw material makes a 40 to 50% difference in the estimated molasses loss depending on whether true or apparent values are used. By this standard the corresponding effect in the case of the S or J components of the formula or the other refinery losses are only marginal.

However there are now many refinery situations in which the emphasis on yield is of much less importance than was at one time the case. This may result from diversification of products or simply from a very favourable price situation for molasses. Occasionally it may even result from a political reason for wishing to produce more molasses from a particular sugar without purchasing a lower grade sugar in the first place. Also, political decisions can jump ahead of cost optimisations.

4. CONCENTRATION OF SPECIFIC IMPURITY GROUPS

The sugar industry has become accustomed to grouping impurities in raw sugar into 3 main categories — reducing sugars, ash and an omnifarious term such as Other Organic Matter to include everything that is left over after the other group analyses have been performed. OOM also includes the net result of all of the errors in the other measurements.

The reducing sugar and ash concentrations have frequently been used in various relationships as a guide to anticipated yield from a particular raw sugar.

Douwes-Dekker4 went one stage further in the case of raw sugar processing to produce the statistically based formula which carries his name and relates to 1938 - 39 results in Java. His formula uses the concentrations of the reducing sugar — perhaps we should use the term hexose — and ash in the total non-sucrose component.
With the development of present day computer techniques the author has run statistical studies of numerous raw factory and refinery molasses with the object of discovering the magnitude of distribution of influence between all 3 groups of impurities. The regression equation is simply a development of the Douwes-Dekker relationship from which Statistical Melassigenic Factors for each of the 3 groups of impurities have been determined by standard regression analysis techniques. The author has found the procedure generally useful for raw cane sugar products but not satisfactory when raw beet sugar is mixed in certain refinery circumstances.

The calculations are generally beyond the capacity of many refineries especially on a routine basis unless quite sophisticated computer back-up facilities are suitably programmed and organised for use.

The work involved ceases to be of value as soon as “yield” depreciates in importance. Such data do have value to provide primary indicators for research studies related to melassigenesis in showing up the relative importance of the 3 areas and on which area to focus attention.

More simple statistical formulae include those of the Queensland Sugar Research Institute:

Expected True Purity = $40,67 - 17,80 \log (RS/Ash)$.

The author prefers to use his own statistically derived relationship which gives very similar answers:

Expected True Purity = $40 \times (RS/Ash) - 0.2$

Hugot prefers still further simplification and also quotes a large number of other formulae:

Clerget Purity = $40 - 4 \times (RS/Ash)$

The usefulness of statistical formulae depends on the principle of “what has been before is likely to happen again next time.” Statistical techniques enable us to introduce precision analysis and hence define the probabilities of future behaviour and the limits of risk that are involved,

Whilst the actual constants in a statistical formula derived only from raw sugar mill data may not be quite the same in a refinery situation they do afford a starting point for discussion and RS/Ash ratio is equally as important in either context.

Sulphur dioxide is a special constituent which is not common in raw sugar but might be used if a premium is being sought for low colour value. It is unlikely that traces in a raw sugar would persist into the refined crystal after the chemical treatment given for purification.

5. CONCENTRATION OF SPECIFIC IMPURITY COMPONENTS

Attention is drawn from time to time to the influence of specific components of raw sugar on one or more stages of refinery operations e.g.

1. Starch affecting filterability.
2. Dextran affecting filterability and perhaps product crystal shape.
3. Amino acids affecting final molasses purity and hence yield.

These are simply taken as 3 examples to which others may be added and to which no doubt many more will be added as our detailed knowledge of processing develops.

The effects of several specific impurity components will be discussed under other headings. As references have already been made to “yield” predictions
Amino acid specification by chromatographic techniques is now a well developed if sophisticated analytical procedure, of uncertain routine value in usual refinery processing and of no economic value if “yield” is a processing criterion of lesser importance. It would have research value, and could have some routine value in a raw sugar mill where minimising final molasses purity is of economic importance.

6. COLOUR VALUE OF WHOLE CRYSTAL

The colour value of a shipment of raw sugar is very important to any sugar refinery. A major cost factor is that which is involved in the removal of colour. Much can be written about the measurement of colour, the sources of colour components, the effects of colour components and various techniques of removal.

In the experience of the author the colour values of raw sugars on the international market vary over a very wide range — from 2 000 to 10 000 ICUMSA units, there may be substantial variation from a particular country and colour can increase with time when stored at normal ambient conditions experienced in equatorial and tropical temperature zones.

When sugar of “refined” quality is produced in association with raw sugar in a conventional sugar mill a different slant on costing of colour removal is needed as compared with a conventional separate refinery. The reason for this is the substantial difference in the cost of fuel. In the separate refinery chemical colour removal is of major importance, but where steam is available from a bagasse-fired generator, chemical colour removal need be of less significance and could in fact be dispensed with except for the simple juice defecation procedures. This would involve special boiling formulae taking advantage of the fact that a crystallisation step removes about 95% of the colour in a sugar crystal.7

The author has also found it quite advantageous to use colour as a simple impurity indicator in many situations by evaluating a colour/impurity ratio according to the following formula:—

\[
\frac{\text{Colour Value of Sugar}}{100 - \text{Pol}^* - H_2O}\]

Strictly the denominator should be multiplied by 10 where colour values relate to 1 000 rather than 100 parts of dry substance, otherwise the colour/impurity index may be determined from Stammer, ICUMSA or other colour values according to personal preference.

It has been observed that for many raw sugars a value of about 50 has been obtained for the above relationship using Stammer Units and Pol, and with a standard deviation of 8%. Where samples have been observed to have only about half of this concentration sulphitation or some other special decolourising technique in the raw sugar factory is suspected.

Colour may be largely inherited from the juices in the cane and partly derived from thermal decomposition of the sugars during processing. By

* The author prefers to use sucrose.
following a colour balance through the refinery process, not only can local development of new colour be identified, but it is possible to relate to any proneness of raw sugar from particular sources to colour development in the refining stages.

For a constant colour/impurity ratio and a fixed value for dilution indicator it may be mathematically shown that the colour of a raw sugar is linearly related to the polarisation but with a negative coefficient.

7. COLOUR DISTRIBUTION WITHIN THE CRYSTAL

The refiner prefers to have as much as possible of the colour of raw sugar concentrated in the surface film of the crystal enabling it to be removed in the affination process. Certain chromophores however seem able to build into the crystal lattice and no amount of affination or surface washing will remove these.

The best analytical specification for this is simply to perform a colour measurement of the affined crystals.

On the other hand the refiner does not necessarily always remove all of the surface film during affination or he may at times remove more than just the surface film for reasons other than colour penetrating the crystal structure.

8. CONCENTRATED COLOURED INCLUSIONS

Whilst these impurities are equally as impossible to remove by affination as the previous colour materials they are incorporated in the crystal in a different way and enter the crystal differently. They are usually concentrated in the heart of the crystal and originate during nucleation, their presence being ascribed largely to nucleation being effected at a too low value of purity.

The concentration of these colour materials will be included in the analytical figure for affined sugar and will not be differentiated from the coloured materials distributed more uniformly in the lattice structure. Concentrated inclusions will also be associated with impurities other than coloured substances and the whole affined crystal will be of a lower purity value.

The most satisfactory identification is, however, mainly qualitative in nature viz. to view the crystals under the microscope and a semi-quantitative measure can be obtained by observing the size of the inclusion relative to the size of the whole crystal. It is not necessary to use affined crystals for this observation.

9. AVERAGE CRYSTAL SIZE

This is best determined from a sieve analysis from which the Mean Average (MA) size or size of the 50% cumulative plot may readily be estimated using probability ruled graph paper.

The result is of interest to the refiner mainly at the affination stage. The surface film, being a function of surface area is less for larger crystal sizes. Centrifuging of affination magma is also easier with larger crystal sizes. A figure of 0,75 mm represents a compromise between the desire of the refiner for a large crystal and the extra cost to the raw sugar producer in boiling a larger size.

It should also be recognised that a larger crystal size has a smaller specific surface area and is more easily obtained at higher purity in raw sugar processing which has been confirmed by colour observations made by the author.
10. UNIFORMITY OF CRYSTAL SIZE

It is equally as important to have a uniform crystal size as to have a large average size. Small crystals fit into the spaces between large ones and reduce the affination qualities both by reducing the flow rate of syrup during separation and also providing more surface area to be affined for the same weight of crystal. Specifying maximum amounts of small sized crystals is one way of providing a measure for this criterion, but there would seem to be merit in following the more widely used measure of Coefficient of Variation (CV) for this purpose. This is being used for refined sugar size specifications and raw sugars can be equally as well specified.

This value can readily be obtained from the cumulative size analysis and conveniently derived from the graphical plot on paper with probability rulings. Thus:

\[
CV = \frac{a_{16} - a_{84}}{2 \text{ MA}}
\]

where
- \(a_{16}\) = size of crystal at 16% of cumulative weight
- \(a_{84}\) = size of crystal at 84% of cumulative weight
- \(\text{MA}\) = size of crystal at 50% of cumulative weight

Whilst recognising certain merits for a low CV value it is perhaps worth recording that multi-sized crystals pack into a smaller volume which can have some economic value from the point of view of storage and transport facilities.

11. CRYSTAL SHAPE

A well-formed sugar crystal is slightly more spherical than a cube and is frequently referred to as a pseudo-cube. If we define sphericity as the surface area of a sphere of the same volume as the crystal relative to the actual surface of the crystal then a well-formed sugar crystal has a sphericity of 0.87 whereas a true cube is 0.81. The value of 0.87 is for the crystals of pure sucrose grown by Kucharenko in his classical kinetic studies. Drinnen studied shapes of various Australian raw sugars from the 1936 season and these more closely approximated the true cube with an average sphericity of 0.82.

Vavrinecz classified crystal shapes on the basis of a simple length to breadth ratio. This provides perhaps the quickest first approximation of shape and Vavrinecz values for this ratio from 0.8 for broad twins to as high as 10.0 for some unusually elongated crystals have been observed.

The author has indicated the importance of a high shape factor from the point of view of good bed porosity in the affination stage. However the shape of a particular crystal is also a very good indicator of the environment in which it has grown and consequently of the likely effect of its own surface film when this becomes the environment during refinery treatment.

A detailed study of the actual faces is much more revealing from this point of view but is not a simple exercise. Vavrinecz has recorded a number of important effects of environment on shape, likewise Sutherland, and Smythe have pointed out the special effects of certain polysaccharides in relation to refinery studies. Dextrin as an enzymatic product of starch hydrolysis in the sugarcane itself or in early stages of processing is known later to influence crystal shape during the boiling process. It is believed that other polysaccharides in
the soluble fraction of starch or resulting from process hydrolysis of starch or of its soluble fraction can likewise affect the shape of the subsequent crystal.

The author anticipates publishing17 the results of a range of studies recently conducted on relative growth rates of the 15 faces of the sucrose crystal, with related molecular bond distribution and relative energy contributions. From these studies predictions of environmental influences from crystal morphology is also in an advanced stage of development.

To complete the record the reminder is worth recording that inadequate circulation during crystallisation can also be an important factor in shape distortion.

12. MONO-CRYSTALLINE CHARACTER

In more common terms of the nomenclature of sugar technology this really means that conglomerate crystals in raw sugar are undesirable to the refiner. These may result from poly-nuclear development due to incomplete dispersion of powdered seed material or as a secondary development due to inadequate circulation mainly in the final stages of high grade boiling or in the crystallisers. Secondary development of conglomerate can also take place before, during and even after drying.

The refiner legitimately objects to conglomerates of any type, chiefly because of the difficulties they introduce in the affination processes. Not only does the extra surface area involve more surface film, but an important proportion of this film is in crevices from which it is not as readily removed as from the surface of a well-shaped single crystal.

It is not easy to provide a simple measurement of the amount of conglomerate other than by microscopic observation — counting crystals, and classifying them in simple terms. Many of the conglomerate crystals appear in the largest fraction of a sieve analysis but this is not a simple guide on its own but requires associated microscope inspection.

13. FILTERABILITY

This measurement can be a controversial subject. It involves simply measuring the rate of filtration of a syrup prepared by dissolving the raw sugar in water. The conditions of concentration, temperature, pressure, type and concentration of filter aid must be carefully controlled as well as standardisation of filter medium. Various designs of equipment have been proposed and used from time to time. The author is currently well satisfied with results from the CSR type unit.18

The author is not as satisfied with the prediction value of results obtained only from raw sugar.

The test is intended to provide the refiner with a prediction of the manner in which a particular consignment of raw sugar will behave during filtration after carbonatation.

It has long been suspected that the filterability test is not as reliable as the industry would like it to be from the point of view of behaviour prediction. Attempts to come closer to obtaining a reliable test for the purpose have not been wholly satisfactory — such as performing the test on affined rather than raw sugar.
The author has set up various laboratory experiments with the object of studying the actual carbonatation process itself and observing the filtration characteristics of the product. These have indicated that the filtration characteristics of the calcium carbonate precipitate itself can be substantially affected by the procedure of carbonatation as well as by the nature of impurities in the raw sugar.

The fact that a refiner experiences filterability problems after carbonatation need not always be the fault of the raw sugar. By always keeping conditions of carbonatation the same in the refinery it may however be legitimate to throw at least some blame on to variations of raw sugar quality. Unfortunately the raw sugar supplier is not in a position to know whether the refiner might have had problems related to his own techniques or not.

Whilst the author recognises the importance of refinery filterability reflected in the effect of changes in behaviour of the calcium carbonate precipitate, he is of the opinion that the industry has come far enough now to recognise the limitations of the raw sugar test. Lyle has given us enough information to be able to estimate the relative cost of the filtration step in the full refinery costing exercise. There are no reasons to believe that the proportion involved has changed significantly since the time these cost data were recorded.

On the other hand it is recognised that the smooth operation of a refinery can be upset by a sudden change for the worse in the filtration process. To focus attention on known raw sugar causes of poor precipitation of calcium carbonate — such as protective colloids — is considered by the author to be a better approach. The refiner likewise can benefit himself from studies he could make in relation to the effects of variations in his own procedure on the filtration characteristics of the calcium carbonate precipitate and the mechanism involved in the procedure.

CONCLUSIONS

In assessing the quality of raw sugar in relation to the cost of refining any particular shipment it is necessary to keep the various costs in their right perspective.

The actual refining cost is approximately 15% of the final retail price of refined sugar, on the other hand it is less than 10% if the operation is conducted in the same sugar mill as that in which the raw sugar is produced.

If a separate refinery encounters sugars which increase its own costs by 10% this would have very little influence on the final retail price. But an increase of this amount might well be enough to make the difference between profit and loss and unless rectified might even put it out of business.

On the other hand a raw sugar miller operating on his own raw sugar and encountering the same type of difficulty will be affected to a much lesser degree if he considers his financial costs in relation to his over-all process.

Big fluctuations in international raw sugar prices were often a very valuable bonus to independent refiners when prices were low and little regard needed to be paid to the quality of the raw sugar. However with tightening of the raw sugar price structure and an increasing tendency for governments to build in control features to cover speculative activities attention should revert more specifically to the significance of quality criteria in the raw product.
CALIDAD DEL AZÚCAR CRUDA

F. H. C. Kelly

RESUMEN

Se da la lista de trece criterios de calidad consideradas de importancia para la evaluación de azúcar crudo como materia prima para una refinería de azúcar. Se hace una breve revisión de la elaboración de cada criterio con el objeto de iniciar un debate más que con el fin de pretender cubrir totalmente el tema.

Se hace referencia a las ventajas de actualizar algunas prácticas analíticas especialmente con respecto a relaciones de polí sacarosa.

Se llama la atención sobre la necesidad de tener una perspectiva adecuada con respecto a las consideraciones económicas. Se indica la relevancia de tres situaciones: el valor del azúcar crudo como un artículo independiente, los costos de operación de una refinería como una empresa separada y el costo marginal del proceso de refinación cuando se lleva a cabo en el mismo sitio como un agregado a la producción de azúcar cruda.