PAPER # 771

COLOUR MEASUREMENTS IN RAW AND AFFINED SUGARS

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ABSTRACT

Application of ICUMSA measurement at 420 nm is commonly used in laboratory to the washed sugar coming out of the affination centrifugals.

Works carried-out at BEGHIN/SAY Nantes refinery, aimed to establish an accurate measurement and rapid communication of colour characteristics.

A basic laboratory colorimeter was firstly used for stationary samples of different raw sugars and brown dry affined sugars. Laboratory works were done with a wide range of colour from very low coloured washed sugar to raw sugar entering into the refinery.

Absolute colour measurements were performed with values expressed by the L*a*b* and L*C*H* colour-coordinate systems, and statistical correlations with ICUMSA method have been determined.

Secondly, an on-line colour instrument, based on real-time reflectance measurement, was tested on a dry washed sugar, on which the colour control in solution form doesn't match perfectly with visual specifications. Currently, the continuous measurement of colour is used to monitor affination centrifugals with a good accuracy.

Details of operational procedures and results obtained are reported.

COLOUR MEASUREMENT IN RAW AND AFFINED SUGARS

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INTRODUCTION

Colour measurements currently in use

The ICUMSA methods for measuring colour in order to determine the quality level of sugar in crystal form use two distinct analytical procedures.

The measurement of colour of sugar in solution can be applied to all types of sugar from brown to white. [1]

The measuring of aspect, by comparison with a standard scale, is used to visually grade the degree of whiteness of refined sugars. [2]

These laboratory methods require a considerable amount of time from the sampling to the analysis and this can create difficulties in decision making when operating a non-stop production process.

The aim of this study is to examine methods and equipment for measuring colour, used both in laboratory and at the production level, which can accelerate the appreciation of the quality of brown or affined sugars and provide data and values which can be used by the production team.

Production Control

The continuous control of the whiteness of white sugar crystals, in correlation with the colour in solution, is an application which is currently mastered, on either wet or dry sugar. The control of batch centrifugals can also be optimised with the possibility of identifying, on the wet sugar conveyor, the whiteness of the sugar coming out from each machine.

As for the affination of raw sugar, routine tests are generally carried out in the simplest possible manner by comparing the aspect of a random sample with a standard sugar of an ideal colour.

Obviously the personnel in charge of these practices must have a combination of particularly keen sight and an excellent appreciation of different shades of colour. However, in order to be efficient, they must also benefit of adequate conditions:

Uniform sample surface
Controlled lighting
Constant environment
Absence of lumps

All of these ideal conditions are unfortunately not always met in an industrial environment where the activities of the production team are more and more oriented on supervising processes and co-ordinating different tasks.

COLOUR MEASUREMENT

Method

A preliminary study of different colourimeters with regard to repeatability, reproducibility and reliability enabled us to select the equipment the best suited to the visual analysis of brown sugar.

The very simple tristimulus colourimeter, proved to be the most appropriate for measuring the colour of sugar crystals both in the laboratory and in an industrial situation.

The use of a wide diameter measuring head permits the observation of a larger area, and obtains an average reading which takes into account the surface irregularities due to the crystalline nature of brown and affined sugars. The equipment can be used on raw samples requiring no particular preparation beforehand.

Observations were carried out using a standard CIE type D 65 illuminant which reproduces daylight conditions.

All of the colour measurements given are the average result of three automatic, consecutive colour readings on the observed sample.

The standard colour systems L*a*b* and L*C*H* were selected in order to carry out comparative statistical studies between the ICUMSA colour in solution form and the colourimeter values,

Sample preparation

For marketing reasons, only dried affined sugar for specialities, on which colour of final product is included in customer specifications, was concerned in these studies.

At Nantes refinery, the affined sugar coming out of continuous centrifugals is characterised by its relatively small crystal size with a mean aperture of about 0.55 mm.

The ideal sample for colour measurement has a regular surface, is opaque and of uniform colour. Unfortunately all of these conditions are not reached in brown sugar, where the measurements can be greatly influenced by the size of the crystals, the natural variation of internal and external colour and the level of humidity.

The processes of preparation and presentation of the samples require special attention on the part of the operators in order to ensure accurate results. The regularity of the surface to be measured, and the compactness of the sugar are two points which it is especially important to master.

A clearly defined procedure was established for each type of sugar so as to obtain the best possible reproducibility of measurements.

Correlation between ICUMSA and colourimetric values.

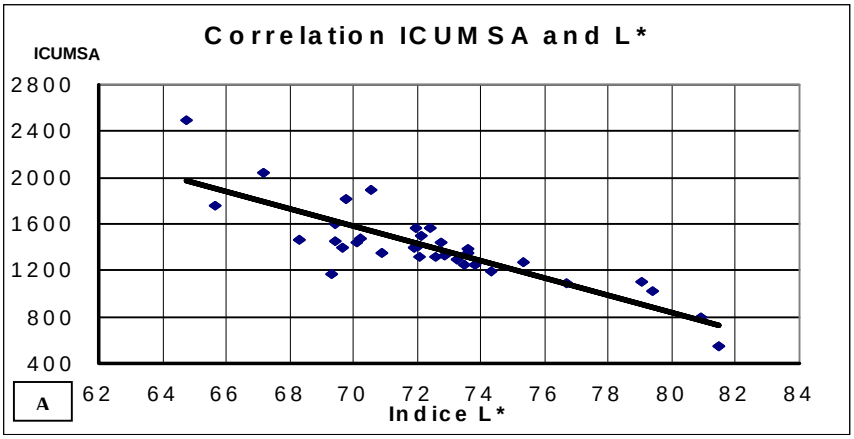
The study was carried out on different types of sugar whose colour index range on the ICUMSA scale from 600 to 2800.

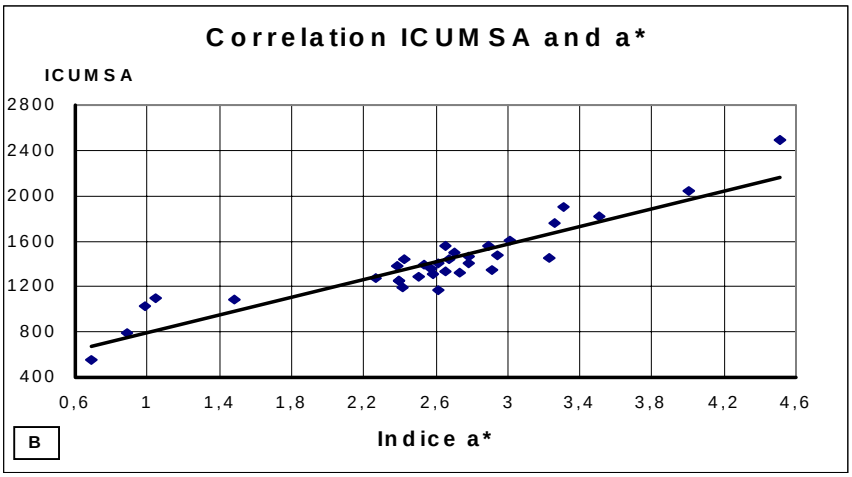
Firstly the statistical validation between the ICUMSA and the 3 elements of the L*a*B* colour system model was explored. The measurements carried out on a vast number of samples of affined sugar, which had been dried beforehand, enabled the drawing up of the statistical graphs which are presented hereafter on Figure 1.

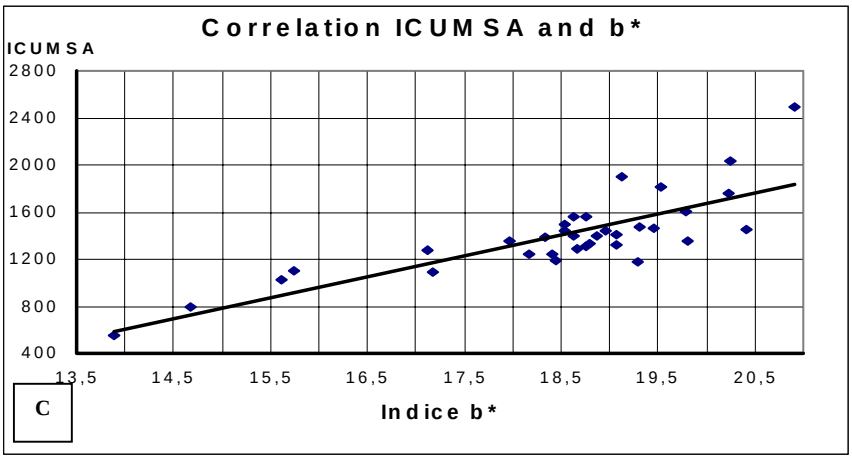
Figure 1A shows the statistical linear regression between ICUMSA and the L* parameter which characterises the lightness of the colour. It can be noticed that the dispersion of values around the regression curve becomes considerable for the higher colour values.
The correlation coefficient of 0.843 is considered to be low, and indicates that the lightness has no direct relation with the ICUMSA.

The correlation between the ICUMSA and the colourimetric parameter a* illustrated in figure 1B is much better than that obtained with L*, with an even dispersal of values around the regression curve.
This a* parameter, which is one of the characteristic coordinates of chromatic colour value, reaches a correlation of 0.918 with the ICUMSA.

The correlation between parameter b*, which represents the second axis of chromatic colour values, and the ICUMSA, figure 1C, is rather unsatisfactory. We can see a dispersal of measurements around the linear regression which increases as colours increase, in the same way as the results obtained with the brightness indicator L*. The correlation coefficient 0.805 is too low to exploit this criteria for statistical use.







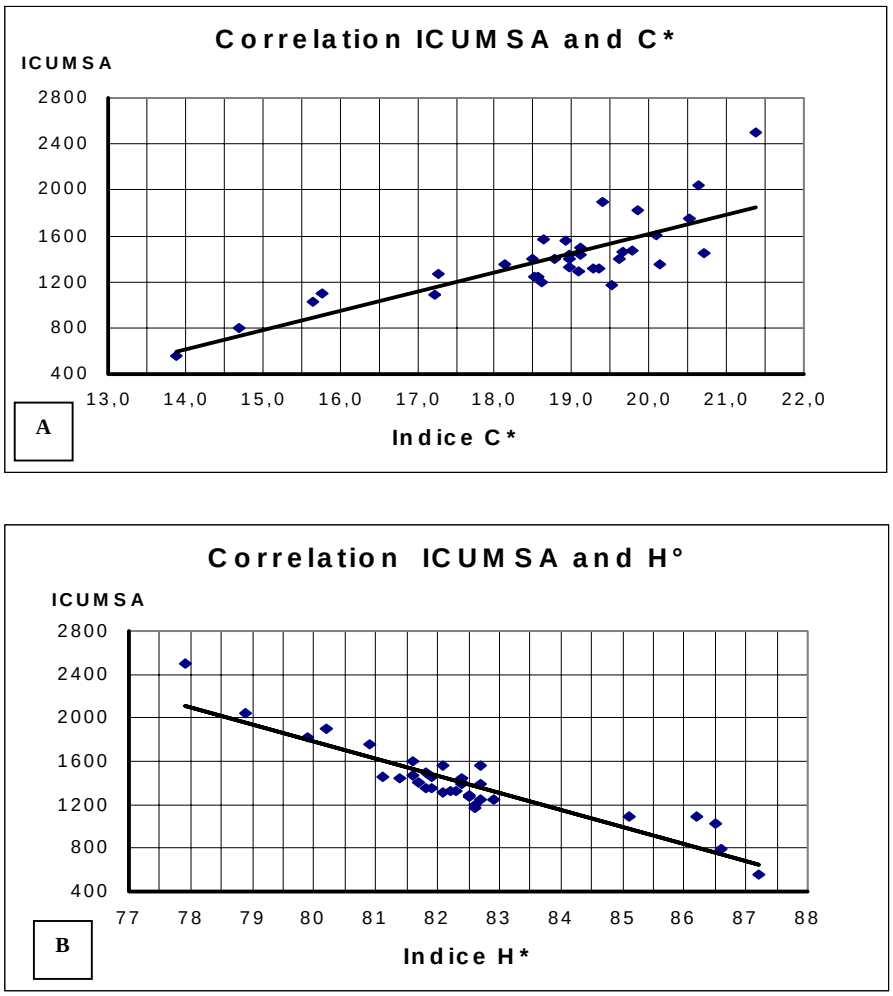
❖ **Figure 1: Correlation curves between ICUMSA and L*a*b* colour system**

The colourimetric model L*C*H*, which is defined with the same diagram as the model L*a*b* but using cylindrical coordinates, was then statistically compared with the ICUMSA measurements.

The parameters for the lightness L* are identical in the two colourimetric models and are illustrated by the same linear regression on Figure 1A.

The regression curve between ICUMSA and parameter C* ,figure 2A, is of the same sort as that obtained with parameter b*. The correlation coefficient is equal to 0.803, and does not permit statistical use of this criteria.

The third parameter H* ,Figure 2B, has a slight resemblance to parameter a* but with a dispersal of values which increases in parallel to the colour. The correlation coefficient obtained is 0.9088.



❖ **Figure 2: Correlation curves between ICUMSA and L*C*H* colour system**

Application

The results obtained with the two colour models are quite similar.

The differences in lightness between sugar crystals constitute an obstacle to the statistical use of the colour measurement index L*. We can suppose that the irregular aspect of the sample and the complexity of the structure of sugar crystals necessitate a measuring method which would be more difficult to set up in an industrial situation.

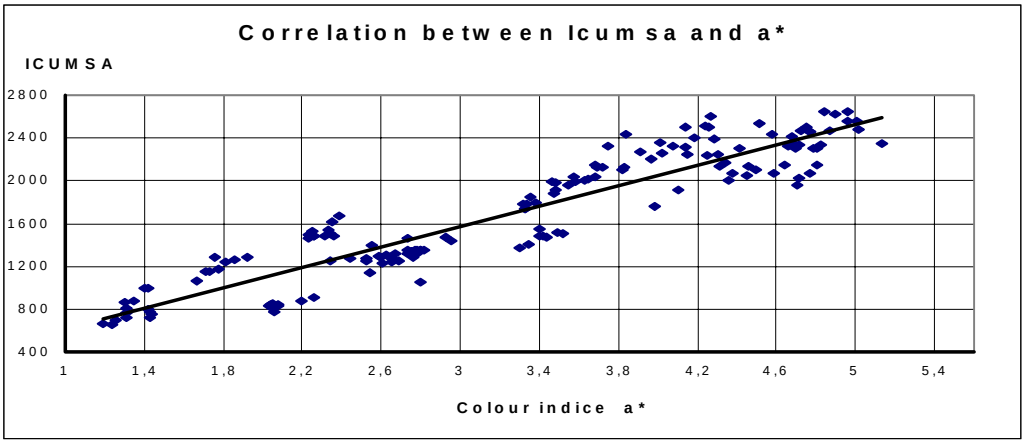
Only the values a* and H* correlate correctly with the ICUMSA colour.If we accept that the precise definition of colour requires a close relation between the three parameters of the two colour systems, the use of a tristimilus colourimeter is unsatisfactory as a replacement for the ICUMSA method.

On the other hand, the very short time required to obtain the parameter a*, which has the highest correlation with the ICUMSA method, could constitute an efficient assistance for supervising the affination centrifugals.

Using only this a* parameter, we established the correlation with the ICUMSA method, on a wider variety of colours of sugar ranging from affined sugar with a light colour of 600 IU to a raw sugar at 3000 IU.

We can see below the linear correlation obtained on figure 3. The correlation coefficient determined with this extended variety of brown sugars is of 0.932 and high enough for statistical use.In the range of 1100-1800 ICUMSA, we obtain a precision of colourimetric a* values of +1/-0.1 which correspond to an interval of 50 ICUMSA.

We have considered that this relationship was satisfactory for an industrial application on production processes for brown sugar.



❖ **Figure 3: Correlation of ICUMSA with colourimetric index a* for raw and affined sugars**

IN LINE COLOUR MEASUREMENT

Equipment

The in line colour measurement currently used to control the whiteness of refined wet or dry sugar, was tested on brown sugar obtained after affination of raw sugar.

The trials, as for tristimulus colorimeter, were carried out on dry, affined sugar destined for specialities requiring accurate colour control.

An in line colourimeter of the type NELTEC Colour Q 800 was placed directly above the brown sugar conveyor, the power supply unit and the computer needed to perform the measurements were installed in a control room so that it could be supervised directly by the operators.

A statistical model of correlation between the NELTEC colour measurement values and ICUMSA colour was then determine and validated by a significant number of samples.

Method

The main objective of this test was to demonstrate the efficiency of in line measurement on the visual aspect of brown sugar.

With the ICUMSA methods, the colour is measured in liquid form after filtering through a membrane of standard porosity of 0.45 microns. This filtration eliminates a part of the colouring substances and does not permit the reconstitution of the total colour values of brown sugar.

For this reason, two statistical models were made up, the first referring to the ICUMSA colour with filtration, the second referring to the ICUMSA colour without filtration.

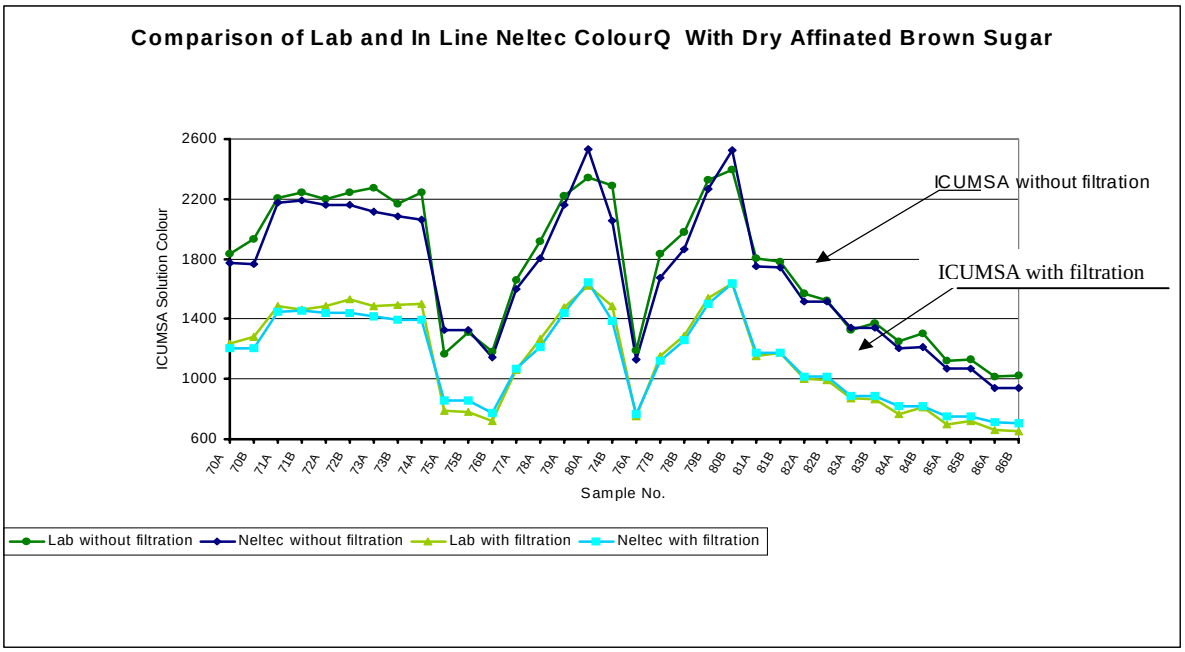
The statistical correlation curves were performed with dry, affined sugar with the following average characteristics:

- Residual moisture 0.20 %
- Minimum colour 600 IU
- Maximum colour 1800 IU
- Average crystal size 0.50 mm

Figure 4 illustrates the important difference in the colour values between the ICUMSA measures on the same sample with and without filtration. This difference increases considerably with the colour of brown sugar, varying from Δ=600 ICUMSA for affined sugar, to Δ > 1000 ICU.MSA for raw sugars.

The origin of the raw sugar and the type of colour can also modify the importance of this difference.

But all along the test it has been shown the relevant relation between the ICUMSA values and the NELTEC colourimeter values on the overall colour variation of the samples.



❖ **Figure 4: Calibration curves for the in line colourimetre with dry affined sugar.**

The precision of the in line measurement for the two different curves was determined by the following formula (SEP = Standard Error Prediction)

$$SEP = \sqrt{\Sigma\,(\text{Lab Colour} - \text{Neltec value})^2 / N} \quad [3]$$

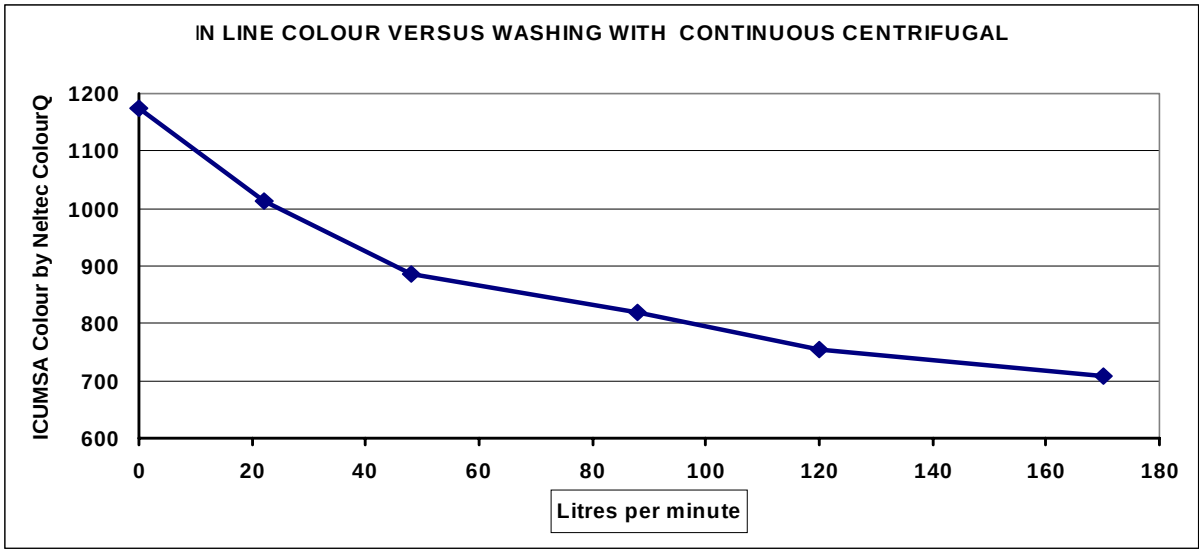
Precision without filtration	93 ICUMSA
Precision with filtration	39 ICUMSA

Application

After the calibration phase of the in line colour control system, the stability of the model was tested over a two month period during which no significant variations were detected.

In the affination process, the control of washing in the centrifugals is probably the critical stage which needs to be mastered in order to obtain a consistent quality of affined sugar.

Figure 5 below represents the evolution of colour, measured by the in line colourimeter, according to the amount of the washing water introduced into the continuous affination centrifugals. We can notice the excellent capacity of the measuring system to give an quasi instantaneous and perfect visualisation of the influence of the adjustments to the process.



❖ **Figure 5: Colour Measurements with Variation in Washing in centrifugals.**

Finally the advantages achieved with continuous control of colours include:

A more efficient monitoring of the centrifugals.
A greater regularity in the colour of affined sugar.
A reduction in the number of laboratory analyses.

CONCLUSION

The use of colourimetric measuring to control brown sugar, instead of ICUMSA methods, is a simple technique requiring minimum handling of samples , and providing accurate and reproducible results within a very short time limit.

Colour measurement, on stationary samples, with a standard tristimulus colourimeter using the colour system model L*a*b* provides a classification of sugars which is as close as possible to a visual analysis. Thanks to a simplified operating process these tests can be carried out either in a laboratory or directly in the factory by the production team.

In line measurement also provides accurate and reliable results for a range of colours from brown to affined sugar.

This real time colour measurement, opens vast possibilities for improving affination operations with a strict control on the quality of the products.

This initial study, carried out on dry sugar destined for specialities will be followed up on wet sugar exiting from the centrifugals and prior melting.

References

[1] ICUMSA methods GS 1-7 and 2/3-9 (1994)

[2] ICUMSA method GS 2-11 (1994)

[3] Nielsen B.C. (1998) In line colour measurement by NELTEC colour Q instrument.