STUDIES OF LONG-TERM STORAGE OF HIGH QUALITY RAW SUGAR

By

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Abstract

ALTHOUGH existing contracts do not provide sufficient economic justification to increase raw sugar quality, the trend of manufacturing better quality raw sugar in the mills will likely continue. Relatively few studies have been conducted on storage of VHP (very high pol) and VLC (very low colour) sugars, especially when the storage period approaches 9–10 months. Monitoring of commercial sugar shipments indicated that, after an initial relatively safe period of storage, sugar colour might double or even triple in a short period of time. Large experimental piles of VHP and VLC sugar were monitored in two sugar mills with different crystallisation sequences (conventional and double magma) for two consecutive seasons. Temperature and relative humidity probes were placed up to 15 m inside the piles. Samples were taken periodically to evaluate the effects of storage conditions on colour, purity, invert content and other parameters. It was concluded that sugar of high quality stores better compared to conventional sugar. However, even VHP sugar can double its colour during long-term storage. Sampling near the surface of the sugar pile (up to 1.5 m inside the pile) is not representative of the bulk of sugar. It has been found that sugar temperature follows the ambient trend as deep as 3 m inside the pile. Sugar within 1.5 m of the surface that was not subjected to temperature increase stored well compared to sugar in the core of the pile. It is unclear what conditions trigger colour increase in storage. Changes in temperature profiles of raw sugar during storage in commercial warehouses indicate that some exothermic reactions take place in the core of the piles that result in colour increase and pol reduction. The reactions take place even when the initial sugar moisture and temperature meet the requirements accepted for safe storage (safety factor below 0.25 and temperature below 30°C). Lower sugar pH may be one of the reasons that makes sugar less stable in storage. Options of cooling sugar before or during the storage will be considered in future research.

Introduction

The increasing price of fossil fuels creates an impetus to manufacture better quality raw sugar in the mills that utilise renewable fuel (bagasse). In an analysis of impacts of high quality raw sugar on refinery parameters, Al Ghurair and Hikmat (2005) demonstrated that the transition from conventional raw sugar (98.4 pol and 3800 ICUMSA colour) to VHP sugar (99.4 pol and 800 ICUMSA colour) resulted in the elimination of the affination process, a reduction in low grade massecuite handling and consequent savings in energy and utilities. Various specifications of high quality raw sugar are used throughout the world resulting in different terminology. A detailed summary of the specifications and contracts can be found in Rein (2007).

Most Louisiana sugar mills use a conventional three boiling scheme and produce raw sugar within the range of specifications: pol 98.5–99.0 and colour 2000–3000 IU (pH 8.5). Several mills that use a ‘double magma’ boiling scheme have the capability of producing VHP and VLC sugar, but existing contracts do not provide sufficient motivation to do it. It is noteworthy that the values for colour measured at pH 8.5 are sometimes twice as high compared to values obtained at pH 7.
(following ICUMSA procedure). Louisiana millers produce sugar that is close to VLC specifications if the standard ICUMSA procedure is applied. The VHP and VLC sugar specifications for the purpose of the current paper are: colour 2000 IU (pH 8.5) and polarisation exceeding 99.2.

Because of the relatively short grinding season in Louisiana, raw sugar may be stored for 9–10 months. Historic data on conventional quality sugar indicate that colour increases significantly during storage with a relatively low decrease in pol (Kochergin and Saska, 2008). A typical curve illustrating colour increase in storage of conventional sugar is shown in Figure 1. It is generally accepted that a correlation exists between the colour and pol of the raw sugars. The expectation is, therefore, that production of VHP sugar automatically results in VLC sugar. This statement may be valid for freshly produced sugar, but does not necessarily apply for stored sugars.

![Fig. 1—Colour increase in storage typical for Louisiana sugar mills; shipment dates range from November through September of the following year.](image)

Figure 2 presents the data collected from four Louisiana mills during production and storage seasons. Although the trend line follows the expectations, the low value of the correlation coefficient indicates that colour-pol relationships have to be treated with caution.

Consequently, production of low colour sugar does not guarantee colour preservation during storage.

The majority of the information on changes in sugar quality during long-term storage is related to refined sugar where sugar is stored in silos and bins without contact with ambient air (Mikuš and Budiček, 1986).

Limited data are available on storage of raw sugar, especially of high quality. Early investigations of quality change of raw sugars in long-term storage (Kopfler, 1933) demonstrated four to five-fold colour increase after 6–7 months of storage of sugar with 97 pol and 0.6–0.7% moisture.

Preferential destruction of glucose observed in the tests implied microbiological origin of sugar deterioration. The present analysis of degraded sugar syrup collected from the floor of a raw sugar warehouse showed a fructose/glucose ratio of 2, confirming rapid destruction of glucose. In a discussion on raw sugar storage, Honig (1963) attributed sucrose deterioration to bacterial activity, emphasising the importance of producing low moisture sugar for good storability.
Information on long-term storage effects on high quality raw sugar is scarce. Interpolating between two extreme cases of storage of white and conventional raw sugar, it is logical to assume that high quality sugar would store without significant colour increase. Investigating storage behaviour of VHP sugar, Kimmerling (1975) reported that ‘with the exception of moisture pick-up in the crust and a very slight colour deterioration therein, no deleterious effects were observed on VHP sugar stored in the silo for six months’. However, no quantitative data, besides sugar moisture and ambient relative humidity, were presented. South African technologists reference storage of 99.3 pol sugar for 6 months with a colour increase of 5–20% (Rein, 2007). Chen and Chou (1993) referenced a Hawaiian report that VLC sugar could be stored safely at low moisture. Data on accelerated storage of raw sugar in small closed containers indicate that colour of VHP sugar can be increased by simply holding it at elevated temperature, e.g. 70°C (Kochergin and Saska, 2008). Any colour changes at this temperature would most likely have chemical rather than bacterial origins. Most researchers agree that the colour changes happen within the thin film of molasses surrounding the sugar crystals. Payne (1987) insists that colour changes are happening within sugar crystals as well. The rate of such changes would be conceivably slower than that in the molasses film.

The main goal of the current study was to monitor storage of VHP and VLC sugar in factory warehouses. It was important to assess if sugar specifications could be kept constant throughout the grinding and storage seasons.

**Experimental procedures**

**Grinding season 2007–08**

Enhanced washing procedures were used at two Louisiana sugar mills to produce VHP and VLC sugar. Both mills started production of higher quality sugar in the beginning of November, 2007. Sugar was allowed to stay in the warehouse for the maximum practical storage time (about 300 days) before shipping. Five thousand tonnes of VHP/ VLC sugar were produced at Mill A with a conventional three-boiling configuration. Mill B with a ‘double magma’ boiling configuration produced two smaller piles next to each other in the same warehouse. Each pile contained about 300 tonnes of VHP/VLC and conventional quality sugar, respectively. All produced sugar satisfied the safety factor requirements for storage (safety factor below 0.25). Sugar samples were taken every 3–4 weeks at three different depths from each pile, namely, top crust (15–20 mm), immediately under the top crust (>20 mm) and at a depth of 0.3–0.4 m. Each sugar sample was prepared by blending 5–6 samples taken from positions around the pile about 3 m apart. A special sectioned
auger was used to take samples from 0.3–0.4 m deep in each pile. It was difficult to obtain samples from larger depths due to friction. After several months of storage, another sampling procedure was added at Mill A. Samples were collected about 1.5–2 m inside the pile at the bottom using a front end loader. The following parameters were measured for each sample: pol, moisture, ash, fructose, glucose and colour at pH 7 and pH 8.5.

HOBO data loggers (model U12-011, Onset Computers, Bourne, MA, USA) were installed right above the surface of the pile and at 1.5 metres depth, respectively, to record temperature and relative humidity (RH). The sensor submerged in sugar was placed inside a perforated pipe covered with a porous plastic screen to protect the sensor from immediate contact with sugar. Thus, air parameters at equilibrium with raw sugar were measured. Battery operated sensors recorded the RH and temperature every two hours throughout the whole storage period.

 Grinding season 2008–09

The test program was continued during the 2008–09 grinding season. Mill B (double magma boiling configuration) produced 5000 tonnes of VHP and VLC sugar in the beginning of November 2008 and stored it until June 2009. From the results of the previous season, it was difficult to conclude whether the samples were truly representative of the entire bulk of sugar in the pile. Sampling procedures were modified to obtain samples deeper into the pile. A gas-powered auger model EA-400 (Echo Inc., Lake Zurich, IL, USA) was used to obtain composite samples from a 1.5 m depth. The auger was driven into the sugar pile through an opening in the bottom of a stainless steel bucket. When the auger was slowly removed without changing rotation, the bucket gradually filled with sugar representing a 1.5 metre cross-section of the pile. Two samples were collected from different spots to produce a composite sample for analysis. The samples were analysed for pol, purity, ash, glucose, fructose, colour and moisture.

To evaluate temperature and RH changes in the core of the pile, five RH-temperature sensors were installed inside a sectioned 10 cm diameter pipe positioned according to the diagram in Figure 3. Four sections of the pipe at various depths had cut-outs covered with plastic screens. The sensors were located at the depths of 0, 3, 7, 5, 10 and 15 m, respectively. The probe at 0 m was monitoring ambient conditions. Recorded data were periodically downloaded to a computer through connected USB cables.

Analytical procedures

Standard ICUMSA procedures were followed for measuring sucrose polarisation, brix and conductivity ash. Sugar colour was analysed according to the ICUMSA Method GS1/3-7(2002) at pH 7.0, and at pH 8.5; the latter measurement is required by contracts accepted in the USA. Glucose and fructose were analysed using Dionex DX-500 HPLC with Carbopac PA-100 columns and a
pulsed amperometric detector following ICUMSA Method GS1/2/3-4(1998) with lactose as the internal standard. All HPLC measurements were performed in duplicate.

Sugar moisture was determined by drying at 105°C under vacuum, for four hours. Initially, sugar pH was measured at about 16 brix, which was a convenient dilution for colour measurement. As a result, sugar pH values were not recorded at constant brix.

Separate dilutions were made for samples obtained later in the season, and the measurements were performed at 50 brix.

**Results and discussion**

**Grinding season 2007–08**

Colour changes in the samples collected 0.3–0.4 m below the top crust are shown in Figure 4. As expected, sugar colour in Mill A (using conventional three-boiling scheme) was almost twice as high as in Mill B with a ‘double magma’ configuration, and was very close in quality to conventional sugar produced in Mill B.

Rates of colour rise were similar in both cases. Since sugar colour was expected to be kept at or below 2000 ICUMSA units (pH 8.5), the results at Mill A were slightly higher than desired. Little change was noticeable in colour of sugar stored at Mill B throughout the season.

Data demonstrated that additional washing in a ‘double magma’ configuration allows production and maintenance of the sugar quality within the desired limits. However, wash water requirements and the associated sugar losses need to be evaluated.

![Fig. 4—Colour changes during 2007–08 grinding season at 0.3–0.4 m depth and at 1.5 m depth)](image)

Measurements at different depths in the pile are illustrated by curves in Figure 5. In all cases, the colour of the top crust changed significantly, with the most pronounced effect after 3–4 months of storage. The first several months corresponded to the time of storage when the ambient temperature was lower than that of the sugar.
Fig. 5—Colour changes at various depths in VHP sugar (Mill B).

Top crust (about 15–20 mm) has higher colour and moisture than the rest of the sugar. The crust is formed of fine dust particles that settled after sugar slinging.

Measurements showed that product sugar crystals (800 µm) have four times less colour than the fine fraction (less than 250 µm).

The results in Figure 4 show changes in colour at various sampling depths at Mill B. Colour of sugar directly under the crust is no different from sugar at a 0.3–0.4 m depth.

The same trend was observed in all sampled piles. The crust appears to work as a protective layer; this phenomenon was also mentioned by Kimmerling (1975).

Depending on ambient conditions, crust may be very hard (observed in January and February) or soft, but always different from the rest of sugar.

The changes in ambient conditions (temperature and RH during the trials) are reflected in Figures 12 and 13.

A reduction in both sugar polarisation and apparent purity was observed in all stored samples (Figures 6 and 7). The pol of conventional sugar in Mill B was slightly higher than that in Mill A, although the rate of deterioration was similar. Differences in degradation rates are difficult to discern due to the data dispersion. However, the pol and purity drop of 0.3–0.4 points should be considered significant.

Changes in concentration of reducing sugars (Figure 8) do not reveal if sucrose deterioration was caused by inversion or degradation reactions that have gone further.

Trends in Figure 9 represent the ratio of fructose to glucose (F/G) in stored samples at various depths.

While the F/G ratio fluctuated around unity (which was expected for sugar inversion), the crust samples showed drastically different F/G ratios, consistently below this level, reaching a value of 0.3.

This indicates that fructose was preferentially consumed in browning reactions.
Fig. 6—Changes in sugar polarisation (depth of 0.3–0.4 m and 1.5 m).

Fig. 7—Changes in sugar purity (depths of 0.3–0.4 m and 1.5 m).
Fig. 8—Change in the concentration of reducing sugars (depths 0.3–0.4 m and 1.5 m).

Fig. 9—Fructose /glucose ratio at Mill A.
Changes were observed in sugar pH (Figure 10). Sugar pH was not routinely measured at the beginning of the storage period. The pH trend showed a gradual decrease toward the end of storage, coinciding with observed pol decreases. The lowest pH was observed in the regular sugar at Mill B, followed by samples at Mill A. Mill B VHP sugar was the closest to neutral pH. These were also the samples that showed minimal deterioration. It is difficult to discern if low initial pH might...
cause enhanced sugar degradation. It is conceivable that low pH in the molasses film could gradually invert sugar causing a further pH decrease. Sucrose inversion consumes water but further degradation reactions actually release water, which should result in increased sucrose moisture. Moisture changes observed during the study (Figure 11) do not provide reliable support to this hypothesis. Moisture content of the samples is also influenced by water migration in and out of the pile. Analysis of highly deteriorated sugar samples always shows increased moisture content. Moisture levels in the crust increased as high as 1–2% at the end of storage.
Analysis of daily records of RH and temperature indicates that the top several metres of sugar act as an ‘insulation’ layer, protecting the bulk from sudden temperature or humidity swings.

The monthly temperature averages represented in Figure 12 show temperatures 1.5 m inside the pile follow the temperature trend outside the pile quite closely (sugar gradually cooled down from an initial 30 to 20°C). When the ambient temperature increased, sugar temperature started to rise as well. However, it never reached the initial sugar temperature. The graph in Figure 13 illustrates a continuous increase of RH both inside and outside the sugar pile (Mill A).

Moisture transfer is a function of vapour pressure, which is closely approximated by absolute humidity. Thus, the difference between absolute humidity of ambient air and the air inside the pile can give an indication of the direction of moisture transfer. The values for absolute humidity in Table 1 were determined from a psychrometric chart based on RH and temperature readings for several points during storage.

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<td>Abs. humidity at 1.5 m depth, kg H₂O / kg dry air</td>
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<td>Abs. humidity of ambient air, kg H₂O / kg dry air</td>
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<td>0.009</td>
<td>0.016</td>
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Initially, absolute humidity of air in equilibrium with sugar was higher than that of ambient air, which meant that sugar tended to release some moisture to the atmosphere which will be migrating out of the pile. Later in the season, sugar was unlikely to lose moisture, because ambient humidity is higher. Sugar moisture levels (Figure 11) did not vary much toward the end of the storage season.

**Grinding season 2008–09**

Analytical results listed in Table 2 show that sugar colour in the composite samples remained virtually unchanged during storage. However, the samples collected from the bottom of the pile before shipping showed that colour almost doubled.

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<th>Date</th>
<th>Moisture %</th>
<th>Pol</th>
<th>Purity %</th>
<th>Fructose %</th>
<th>Glucose %</th>
<th>Total %</th>
<th>F/G ratio</th>
<th>pH ~16 Bx</th>
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This may be explained by reviewing the temperature profiles presented in Figure 14. After an initial stable storage period, sugar temperatures started rising well above the initial values. The reading from 3 m inside the pile followed the ambient temperature trend (Figure 15). The greatest temperature rise was observed at the depths of 5 and 10 m. The probe located near the bottom of the pile floor measured a lower gradient due to heat transfer to a cooler floor surface.

Temperature rise in the pile and corresponding colour change imply that exothermic reactions take place even at low moisture and high pol. Relatively low pH was observed in all samples and no changes in pH were observed during the storage period. Fructose to glucose ratios were also different compared to results from the 2007–08 season. The fact that sugar as deep as 2 m inside the pile can store without deterioration implies that such conditions can be reproduced if mechanisms causing sugar degradation are better understood. The study will be continued with the emphasis on changes that need to be made to production to assure that high quality sugar can be stored reliably without deterioration.
Conclusions

- Sugar of high quality stores better compared to conventional sugar. However, even VHP sugar can double its colour during long-term storage.
- Sampling near the surface of the sugar pile (as deep as 1.5 m inside) is not representative of the bulk of sugar.
- It is unclear what conditions trigger colour increase in storage. Lower sugar pH may be one of the reasons that makes sugar less stable in storage.
- Sugar temperature follows the ambient trend as deep as 3 m inside the pile.
- Exothermic reactions take place inside the pile even when initial sugar moisture and temperature meet the requirements accepted for safe storage (safety factor below 0.25 and temperature below 30°C).
- Sugar within 1.5-m of the surface that was not subjected to temperature increase stored well compared to sugar in core of the pile. Options of cooling sugar before or during the storage will be considered in future research.

Acknowledgments

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ÉTUDES DE L’EMMAGASINAGE À LONG TERME DU SUCRE ROUX DE BONNE QUALITÉ

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MOTS CLEFS: Sucre VHP, Emmagasinage, Sucre VLC

Résumé

BIEN QUE les contrats existants ne fournissent pas une justification économique suffisante pour améliorer la qualité du sucre brut, les usines continueront très probable à produire un sucre de bonne qualité. Relativement peu d’études ont été menées sur l’emmagasinage à long terme du VHP (pol très élevé) et des sucres VLC (couleur très faible), en particulier lorsque la période de stockage approche 9–10 mois. On a suivi les transports outre-mer de sucres commerciaux ; cela a indiqué que, après une période relativement stable, la couleur du sucre peut doubler ou même tripler en peu de temps. On a aussi suivi des sucres VHP et VLC stockés en vrac dans deux usines avec des systèmes de cristallisation différents (magma conventionnel et double) pendant deux saisons consécutives. Des sondes de température et d’humidité relative ont été placées jusqu'à 15 m à l'intérieur des tas. Des échantillons ont été collectés régulièrement pour évaluer les effets des conditions de stockage sur la couleur, la pureté, la concentration de sucres invertis et d'autres paramètres. Il a été conclu que le sucre de haute qualité stocké mieux par rapport au sucre conventionnel. Toutefois, même le sucre VHP peut doubler sa couleur pendant le stockage à long terme. L’échantillonnage près de la surface du tas de sucre (jusqu'à 1,5 m à l'intérieur) n’est pas représentatif de la majeure partie de sucre. Il a été constaté que la température du sucre suit la tendance ambiante jusqu’à 3 m à l'intérieur du tas. Le sucre à 1,5 m de la surface qui n’était pas soumis à une hausse de température se conserve mieux que le sucre dans le coeur du tas. On ne sait pas quelles conditions déclenchent une augmentation de la couleur au stockage. Les changements de température du sucre brut au cours du stockage dans les entrepôts commerciaux indiquent que des réactions exothermiques se déroulent dans le coeur du tas ; cela donne une augmentation de la couleur et une réduction du pol. Les réactions ont lieu même lorsque la teneur initiale en eau dans le sucre et la température sont conformes aux exigences acceptées pour garantir la sécurité du stockage (facteur de sécurité < 0,25 et température inférieure à 30 °C). Un pH bas pour le sucre peut être une des raisons qui rend le sucre moins stable pendant le stockage. Le refroidissement du sucre avant ou pendant le stockage sera considéré dans le futur.
ESTUDIOS DE ALMACENAMIENTO POR LARGO TIEMPO DE AZÚCAR CRUDO DE ALTA CALIDAD.

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Resumen

Los contratos actuales de compra de azúcar, no tienen suficiente justificación económica para incrementar la calidad del azúcar crudo, sin embargo la tendencia actual es la producción de azúcar crudo de mejor calidad. Pocos estudios se han llevado a cabo sobre el almacenamiento a granel de azúcar de alto pol (VHP) y azúcar de bajo color (VLC), especialmente cuando el período del almacenamiento está entre 9 y 10 meses. Monitoreos realizados a cargamentos comerciales de azúcar, indicaron que después de un relativo seguro periodo inicial de almacenamiento, el color del azúcar se puede duplicar o incluso triplicar en un corto tiempo. Grandes arrumes experimentales de azúcar grados VHP y VLC obtenidos a partir de dos esquemas de cristalización (convencional y doble magma) fueron monitoreados en dos ingenios azucareros durante dos zafra consecutivas. Medidores de humedad relativa y temperatura fueron colocados hasta 15 m al interior de las pilas. Muestras de azúcar fueron tomadas periódicamente para evaluar los efectos de las condiciones de almacenamiento sobre el color, la pureza, contenido de azúcares invertidos, y otros parámetros. Se concluyó que el azúcar de mejor calidad se almacenó mejor que el azúcar convencional. Muestras de azúcar tomadas cerca de la superficie (hasta 1.5 m al interior de la pila) no son representativas del comportamiento del azúcar a granel. Se encontró que la temperatura del azúcar sigue la tendencia de la temperatura ambiente hasta una profundidad de 3 m al interior de la pila. Azúcar a 1.5 m de la superficie que no estuvo sujeta a incrementos de temperatura durante el almacenamiento se almacenó mejor que azúcar que estaba en el centro de la pila. No se identificaron las condiciones de almacenamiento que incrementan el color del azúcar. Los cambios en el perfil de temperatura del azúcar crudo durante el almacenamiento en bodegas comerciales, indican que algunas reacciones exotérmicas están ocurriendo en lo más profundo de la pila que ocasionan incrementos de color y reducción de pol. Estas reacciones toman lugar incluso cuando la humedad inicial y temperatura del azúcar cumplen con los requisitos aceptados para un almacenamiento seguro (valor de seguridad menor a 0.25 y temperatura menor de 30°C). Uno de los factores que hace menos estable el azúcar durante su almacenamiento es el pH bajo. Alternativas para enfriar el azúcar antes o durante el almacenamiento.