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Effects of Grain Size, Reducing Sugar Content, Temperature and Pressure on Caking of Raw Sugar

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ABSTRACT

This work aimed to study the factors contributing to cake formation of raw sugar. They were grain size, reducing sugar content, temperature, and pressure during storage. From the water vapour adsorption isotherms, the grain size was found to have the largest influence on the hygroscopic properties of raw sugar. At 30°C, the largest (>1.000 mm) and smallest (<0.425 mm) raw sugar grain had the highest and lowest critical relative humidity (CRH) or caking point at 79.5% and 73.2%, respectively. Scanning electron micrograph (SEM) revealed that raw sugar, exposed to the 67.89% RH and 30°C, established a contact between crystals and fine particles. The fine particles also play a role as binder. With the consolidation pressure of 1.5 kg/cm^2 on a sugar pile, the temperature within the pile did not significantly increase enough to stimulate the cake formation. These results indicate that key factors in preventing the caking of raw sugar is the control of grain size to be greater than 0.425 mm and RH to be less than 67.89% at 30°C during storage.

Key words: caking, raw sugar, water vapour adsorption isotherms

INTRODUCTION

Due to severe caking of Thai raw sugar during transportation to Japan and Russia in 2003, there has been a loss in exported Thai raw sugar business. Consequently, there is a need to investigate the factors contributing to cake formation. Caking is a spontaneous agglomeration phenomenon due to the moisture adsorption and moisture migration from areas of high moisture to areas of low moisture in a sugar pile (Rastikian and Capart, 1998; Billings *et al*., 2005). Adsorbed water is formed as a thin layer of syrup covering the sugar surface and serving as a liquid bridge between sugar crystals. At this stage, wetted adjoining sugar crystals are called lump and the flowability of sugar is reduced. The wetted sugar desorbs water when the relative humidity drops, and then the recrystallization occurs. The liquid bridges are turned to be solid bridges, which results in cake formation (Cleaver *et al*., 2004).

This work investigated the effects of grain size, reducing sugar content, temperature, and pressure on caking of raw sugar in order to find a way to prevent caking during bulk storage and transportation. Water adsorption isotherms, indicating the relationship between moisture content and equilibrium relative humidity or water activity, and scanning electron microscopy were utilized.

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MATERIALS AND METHODS

Raw sugar

Raw sugar was collected from 8 sugar factories located in 4 different regions of Thailand.

Water vapour adsorption isotherms (WVAI)

Microclimate method was used to determine WVAI at different grain sizes, reducing sugar contents, and temperatures. Various relative humidities (RH) were achieved with different saturated salt solutions (Greenspan, 1977). Dry sugar samples of about 2 g were filled in a small glass container, placed in a big glass container containing salt solutions. After closing, the container was placed in an incubator at fixed temperature. After the equilibrium relative humidity (ERH) was reached, the moisture content of the sugar samples was analyzed by oven drying method (AOAC, 2000). Then WVAI describing the relationship between moisture content and ERH was constructed.

Effect of grain size

All raw sugars were thoroughly mixed to simulate storage conditions at port silo. Then they were sieved to separate the samples according to their sizes: <0.425, 0.425-0.600, 0.600-0.850, 0.850-1.000, and >1.000 mm. Prior to use in the adsorption experiment, the raw sugar was oven-dried at 105°C for 4 hr. The investigation of the effect of grain size was conducted at 30°C.

Effect of reducing sugar contents and temperature

Reducing sugar contents of raw sugar from each factory was analyzed using Dinitrosalicylic (DNS) method (AOAC, 1990). Only the raw sugar with the minimum and maximum reducing sugar contents was used in the experiment carried out at the temperatures of 30° C and 37°C.

Effect of consolidation pressure

The compression machine and temperature recorder used in this experiment were shown in Figure 1. Mixed raw sugar was fully filled in the block with 10×10×10 cm in size, which was compressed with pressure at 1.5 kg/cm^2. The measurement of temperatures of the mixed raw sugar at 2 positions: 1 cm (lower probe) and 5 cm (upper probe) above the inside bottom of the block was performed everyday at 4.30 a.m., 10.30 a.m., 4.30 p.m., and 10.30 p.m. for 3 months. The sample was then removed from the block and examined with zoom microscope (OLYMPUS model SZ-PT).

Change in crystal structures at various RH

The change in crystal structures of mixed raw sugar exposed to different RH was examined using the scanning electron microscopy.

RESULTS AND DISCUSSION

Effect of grain size

WVAI of mixed raw sugar with different grain size distributions is shown in Figure 2. Between 32 and 75% ERH, all sugar samples adsorbed negligible amount of water. The greatest amount of adsorbed water vapour was only 1.44 g per 100 g dry basis for the raw sugar with the smallest size. Above 75% ERH, the moisture contents increased significantly to reach the point where the dissolution of raw sugar occurred. From WVAI, the critical relative humidity (CRH, caking point) of raw sugar with the size distribution, <0.425, 0.425-0.600, 0.600-0.850, 0.850-1.000, and >1.000 mm, was determined to be 73.2, 77.5, 77.7, 77.8 and 78.5%, respectively. Thus, the small raw sugar grain is more likely to cake than the large raw sugar grain due to more surface areas. This result corresponds to the research on the effect of grain size on caking of white sugar by Mathlouthi and Roge (2003).

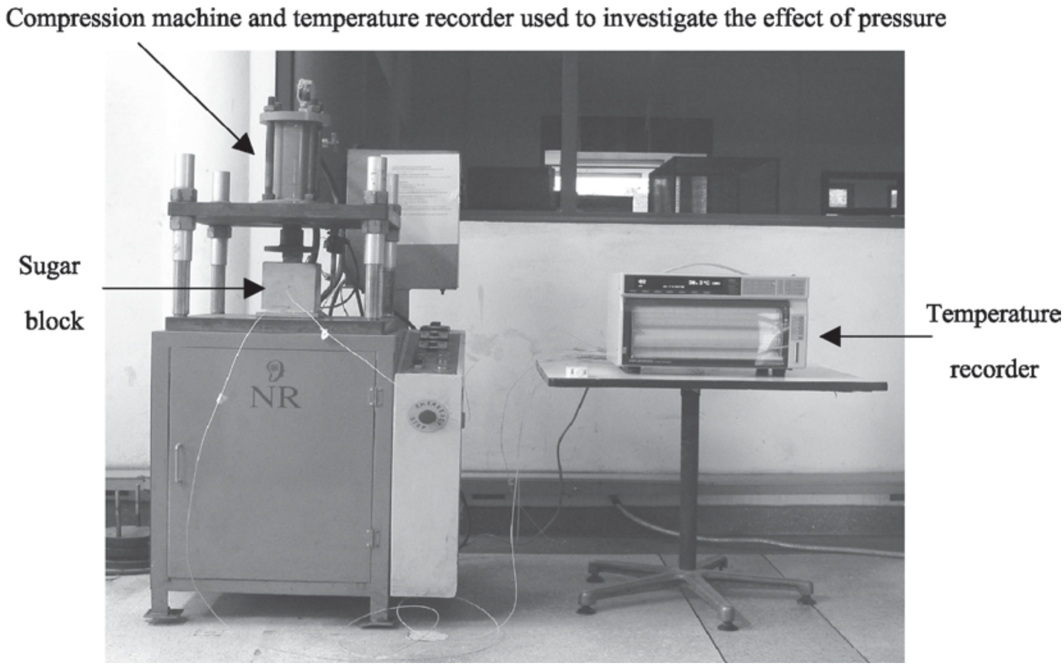

Figure 1 Compression machine and temperature recorder used to investigate the effect of pressure on caking of raw sugar.

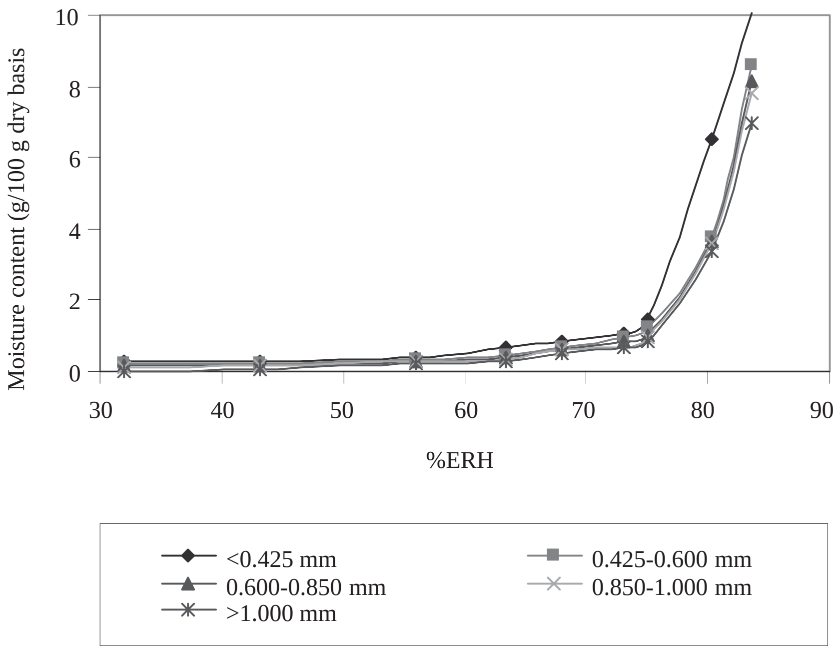

Figure 2 Water vapour adsorption isotherms of mixed raw sugar at 30°C as a function of grain size distributions.

Practically, in a sugar pile with various grain sizes stored in a silo exposed to the high relative humidity, small grain sugar adsorbs the greater amount of water and then water migrates to the large grain size sugar, causing the sugar pile to lump finally (Roge, 2003). Consequently, in order to prevent the caking of this mixed raw sugar, it is essentially to keep the RH of the air in a warehouse under 73.2% RH. This is practically impossible to do in Thailand since normally the raw sugar is piled in a warehouse without the control of RH and the RH in the rainy season is quite high (>80%). The other solution is to get rid of the sugar with small grain size from the pile since it aggravates the caking process. The separated small sugar can be remelted and brought back to the process with no loss.

Effect of reducing sugar contents and temperature

WVAI of raw sugar with different reducing sugar contents at 30°C and 37°C is shown in Figure 3. Similar to the WVAI shown previously, between 32% and 75% ERH, all raw sugar samples adsorbed very small amount of water. At higher ERH, the moisture content of the raw sugar suddenly increased. The CRH of each sugar sample was determined to be 79.5, 78.9, 78.3, and 78.1% for the raw sugar with 0.15% reducing sugar content (RS) at 30°C, 0.60%RS at 30°C, 0.15%RS at 37°C, and 0.60%RS at 37°C, respectively. Consequently, the raw sugar sample with 0.60% reducing sugar content and being kept at 37°C has the greatest tendency to cake. Reducing sugar mostly located on the surface of raw sugar in the form of syrup (molasses) is more hygroscopic than sucrose. Therefore, the CRH decreases with increasing reducing sugar content. Temperature is also found to be another factor contributing to caking. As temperature increased, the CRH decreased. This is because water adsorption takes place quickly as a result of water molecule possessing high kinetic energy. However, when carefully consider these CRH values, they are not

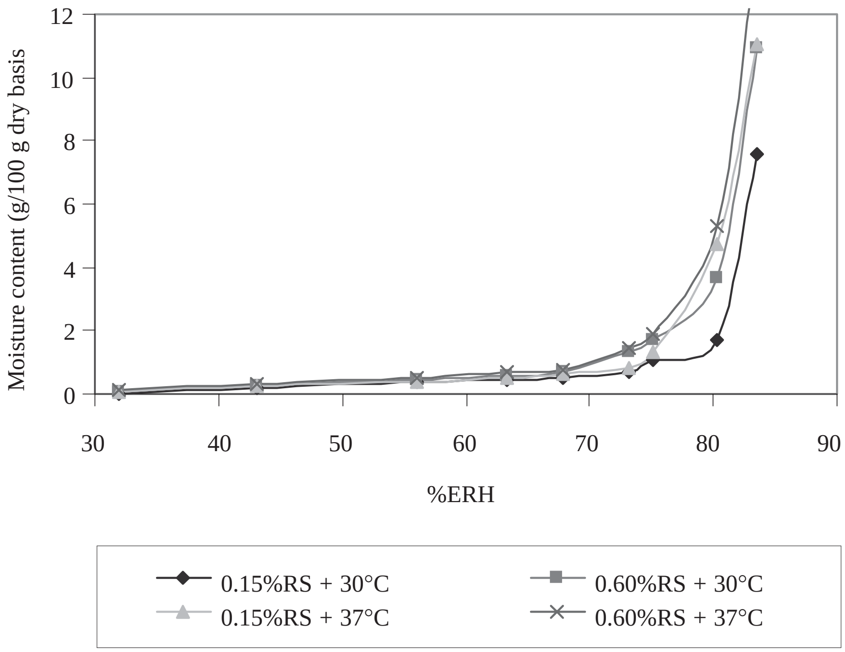

Figure 3 Water vapour adsorption isotherms of raw sugar as a function of reducing sugar contents and storage temperature.

practically different. Therefore, the maximum amount of reducing sugar allowed by the Office of the Cane and Sugar Board at 0.80% is a reasonable value for storing sugar at 30-37°C. When compare CRH from this experiment to those from previous experiment, it can be seen that the grain size has greater effects on caking than reducing sugar contents and storage temperature.

Effect of consolidation pressure

The measured temperature profiles are shown in Figures 4 and 5. At each time of the days, the temperature of raw sugar at the upper probe position was quite close to that at the lower probe position. Also the temperature inside the block was close to the outside temperature at 4.30 a.m. and 10.30 p.m. (data not shown), but lower than the

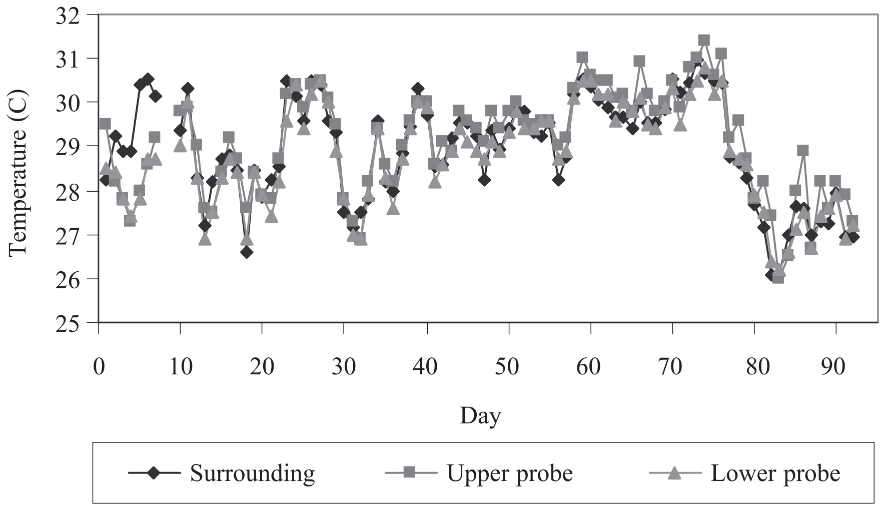

Figure 4 Temperature profiles of raw sugar at different positions and surrounding under pressure of 1.5 kg/cm^2 at 4.30 a.m.

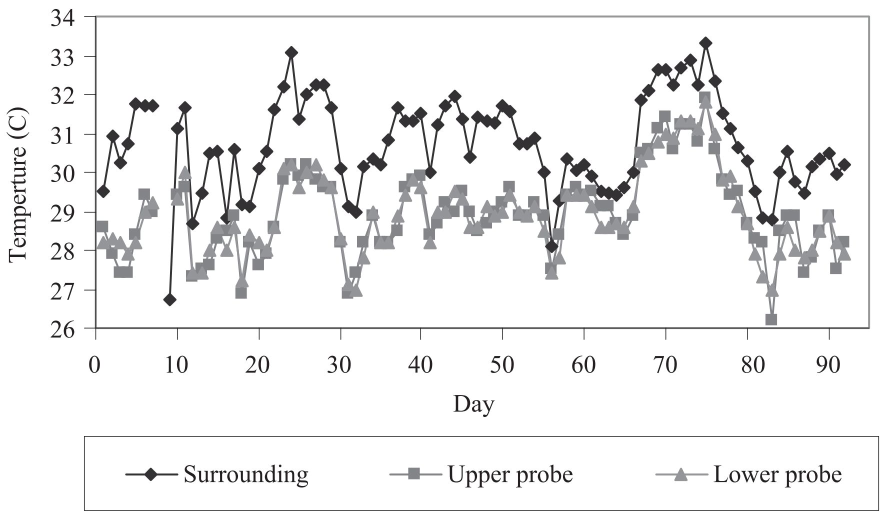

Figure 5 Temperature profiles of raw sugar at different positions and surrounding under pressure of 1.5 kg/cm^2 at 10.30 a.m.

outside temperature at 10.30 a.m. and 4.40 p.m. (data not shown). After 3 month storage, pictures from zoom microscopy (Figure 6) revealed that sugar crystals were partially broken to small particles and their surfaces became rougher. The results indicate that surrounding temperature, not pressure, has an effect on sugar temperature and caking.

Change in crystal structures at various RH

SEM showing crystal structures of the mixed raw sugar during storage at various %RH are shown in Fig. 7. At 32.00% RH (see Figure 7 (a)), the raw sugar was in a pendular stage. The crystals were in almost a typical shape which was being rectangular. Crystal surfaces were partially covered with dust or micro-particles formed during

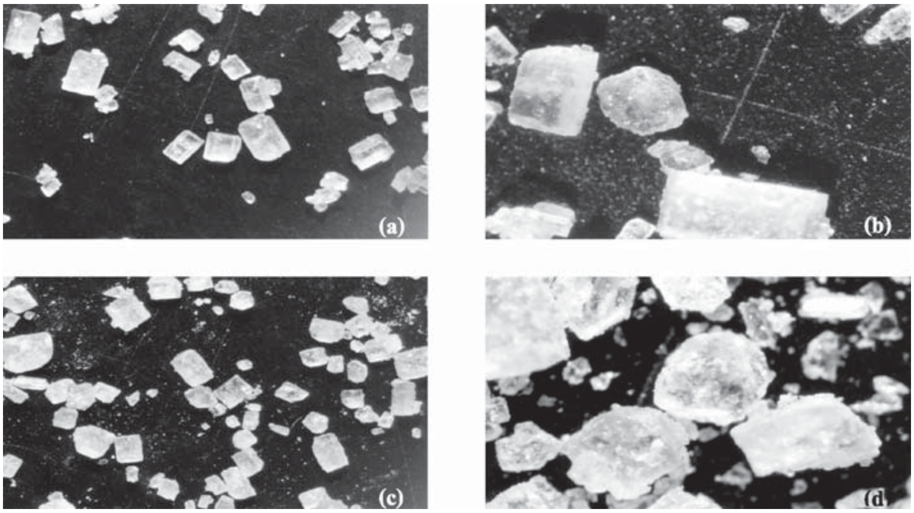

Figure 6 Light micrographs of raw sugar crystal structures before consolidation test (a and b) and after consolidation test for 3 months (c and d).

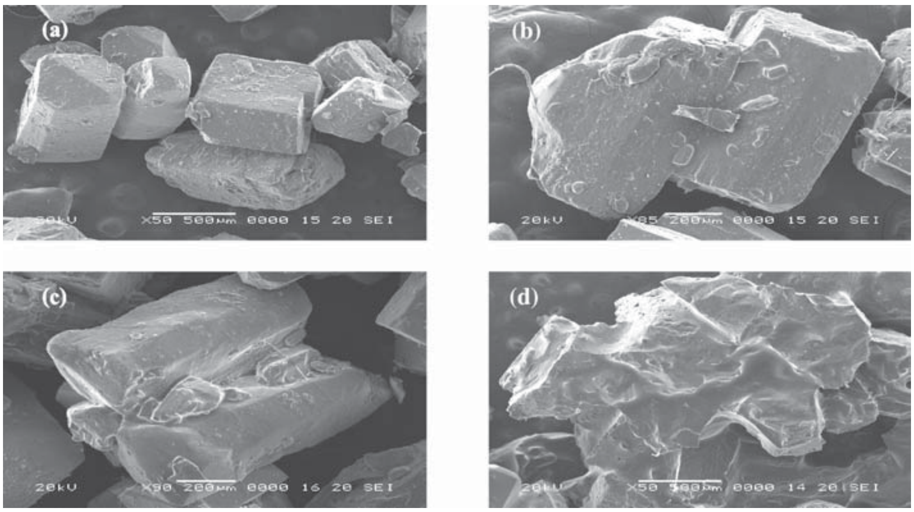

Figure 7 Scanning electron micrographs of raw sugar crystals during storage at 32.00%RH (a), 67.89%RH (b), 75.09%RH (c), and 83.62%RH (d).

the breaking of conglomerates. Voids of air between sugar crystals were present. At 67.89%RH (see Figure 7 (b)), the raw sugar was in a funicular stage. There was a permanent contact between crystals and voids of air between sugar crystals were reduced. At 75.09%RH (see Figure 7 (c)), the raw sugar was in a capillary stage. Due to larger difference in water vapour pressure, raw sugar in this condition adsorbed greater amount of water. Thereby, the surface dissolution occurred and the liquid bridges between sugar crystals were established. Moreover, dust or fine particles played a role as binder between raw sugar crystals. At 83.62%RH (see Figure 7 (d)), the raw sugar was in a drop stage. There was an increase in the amount of adsorbed water with an increase in RH. It is evident that the dissolution was predominant.

CONCLUSIONS

The study of effects of grain size reducing sugar content of raw sugar, temperature and pressure of storage conditions on caking of raw sugar shows that the removal of small size sugar (<0.425 mm) is particularly important for control and prevention of compaction of sugar. In addition, SEM pictures show that at 67.89%RH, the mixed raw sugar started to dissolve to form a lump and fine particles played a role of binder. Therefore, it is recommended to keep the air in a warehouse of this mixed raw sugar under 67.89% RH to prevent caking.

ACKNOWLEDGMENT

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