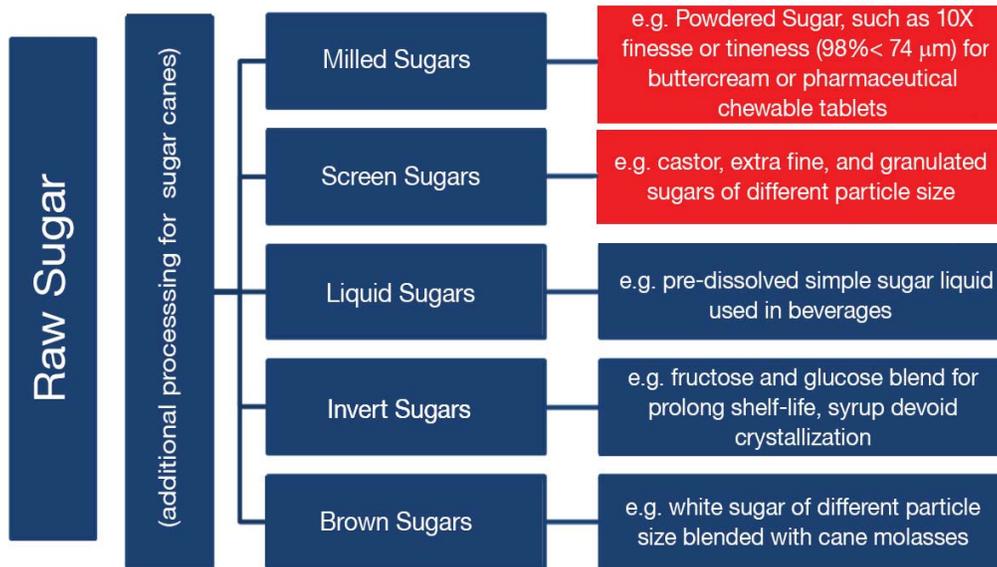


Particle Characterization and Quality Control of Sugar Crystals and Powdered Sugar AN175

Sugar (sucrose) is naturally present in fruits and vegetables in various concentrations. Among all plants, sugar cane and sugar beet contain the highest fraction of sucrose, 12-14% and 16-18% respectively. Extraction of sugar from sugar cane and sugar beet both require similar processing where the juice undergoes carbonation to remove impurities, filtration to attain thin juice sans sediments, thickening at low temperature to achieve brown syrup consistency, and crystallization into raw sugar for exportation to local refineries [1]. Sugar refining is the secondary processing where specialty sugars are made (see Figure 1). In this work, particle size of crystal sugar and commercial powdered sugar will be discussed.



- Long measurement, maintenance, and cleaning time
- Poor performance for fine particles – typically for particles <44 μm (325 mesh)
- Cross sample contamination
- Low automation [2]

For these reasons, dynamic imaging analysis and/or laser diffraction techniques are quickly replacing traditional sieving techniques in the sugar industry.

Materials and Methods

Dynamic Imaging Analysis – Sugar Crystals

One of the important requirements when transitioning from sieving to

dynamic imaging analysis is to maintain continuity with historic data. In order to do so, the users must keep in mind that particles are often not round or symmetrical. For example, a needle shaped particle of 300 μm diameter by 800 μm length will pass through the same sieve mesh as a spherical particle of 300 μm in diameter. First, image analysis allows measurement of particle width, thus providing an often good fit to sieve results (where the mesh passes particles by particle width). A final correction is then applied through shape factor. Users can effortlessly utilize the CAMSIZER's built in algorithm to make correlation possible.

Laser Diffraction – Powdered sugar

Powdered sugar is milled from white granulated sugar crystals to various degrees of fineness. 6X, 10X, and silk sugars are most commonly seen [3]:

- 6X – defined as 93.5% < 200 mesh (74 μm)
- 10X – defined as 98% < 200 mesh (74 μm)
- Silk Sugar – defined as 97% < 20.5 μm

Figure 1: Raw sugar is refined to different sugar ranges for various applications. Milled sugars and screened sugars (in red) are analyzed with the LA-960 and the CAMSIZER, respectively.

Introduction

Quality control is an essential part of the sugar production. Sugar particle size specifications affect product dissolution rate, flowability, dust generation, and directly affects applications such as the spread of cookie dough or compaction strength of a chewable tablet. Traditionally, sugar manufacturers have chosen to use sieving analysis due to its low initial cost and its seemingly simple operating procedure. Sieve shakers vibrate and cause sample to pass through a series of sieve meshes stacked on top of each other until the narrowest dimension of a particle falls through (Figure 2). The sieving technique, however, has inherent disadvantages:



Figure 2: A Rectangular shaped sugar crystal compared to the square aperture of the sieve opening.

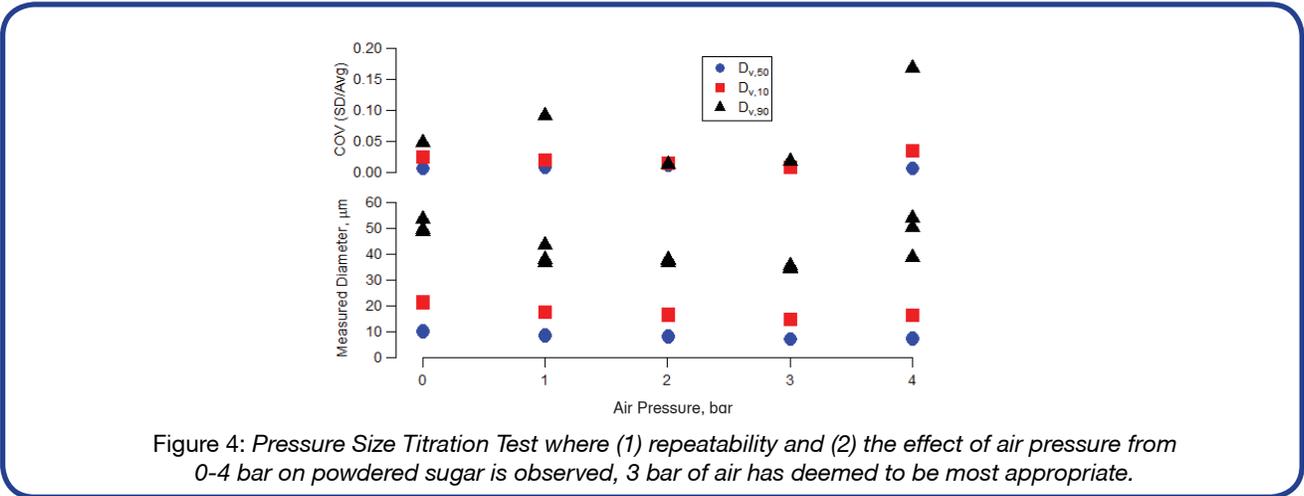
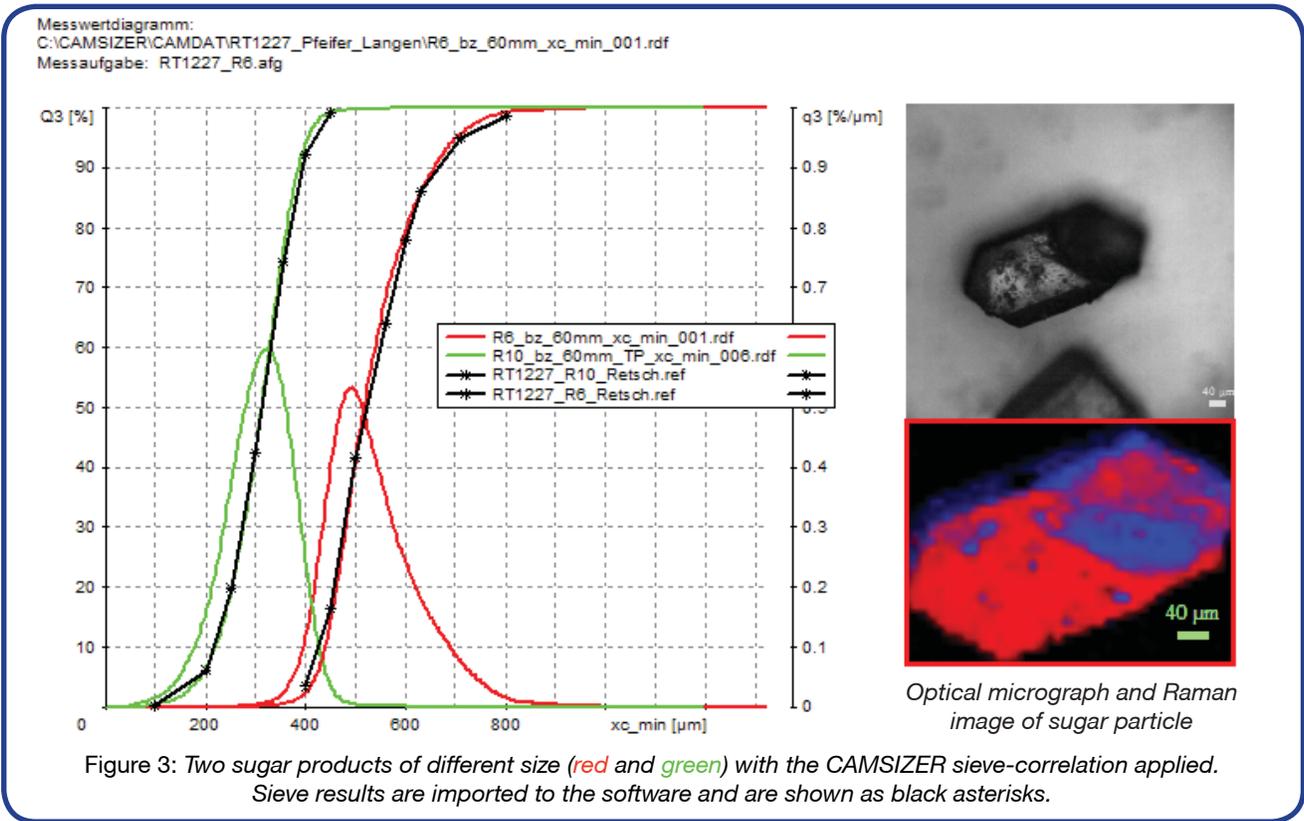
- Low repeatability and reproducibility – up to 30% difference from sieve to sieve [2]

Finely milled powdered sugars have a higher surface area, which leads to a higher tendency for moisture absorption and clumping. About 3-5% of anti-caking agent is often added to help maintain flowability. During processing, it is important to ensure the efficiency of milling is transferrable from pilot scale to full production. For this reason, the laser diffraction technique is preferred over dynamic imaging analysis for powdered sugar because of its powerful dispersion ability and its exceptionally short measurement time. The need to understand particle morphology for finely milled powders is also of lesser value. The laser diffraction technique offers a quick feedback mechanism to monitor the milling process that cannot be replaced by imaging. Ultimately, a desired powdered sugar product has a narrow particle size distribution to minimize particle separation and to maximize powdered sugar dissolution when dissolution is a challenge such as during the creation of buttercream icing due to the high fat content.

Results and Discussion

Two sugar crystal samples from the same manufacturer were submitted for analysis. The data was collected with the CAMSIZER dynamic imaging system; each measurement took three minutes. Sample R10 measured distinctively larger than R6 as expected and when the particle size distribution obtained from imaging analysis (with the sieve matching algorithm applied) were compared to sieve results (black asterisks), there is a near perfect agreement between the two techniques (Figure 3). Note that the percentage of aggregated particle is another quality measure in sugar crystals. Aggregation is related to the presence of molasses. This is another shape specification that is not readily obtainable using sieving technique.

A commercially available 10X Powdered Sugar was purchased and tested. To measure powdered sugar at its



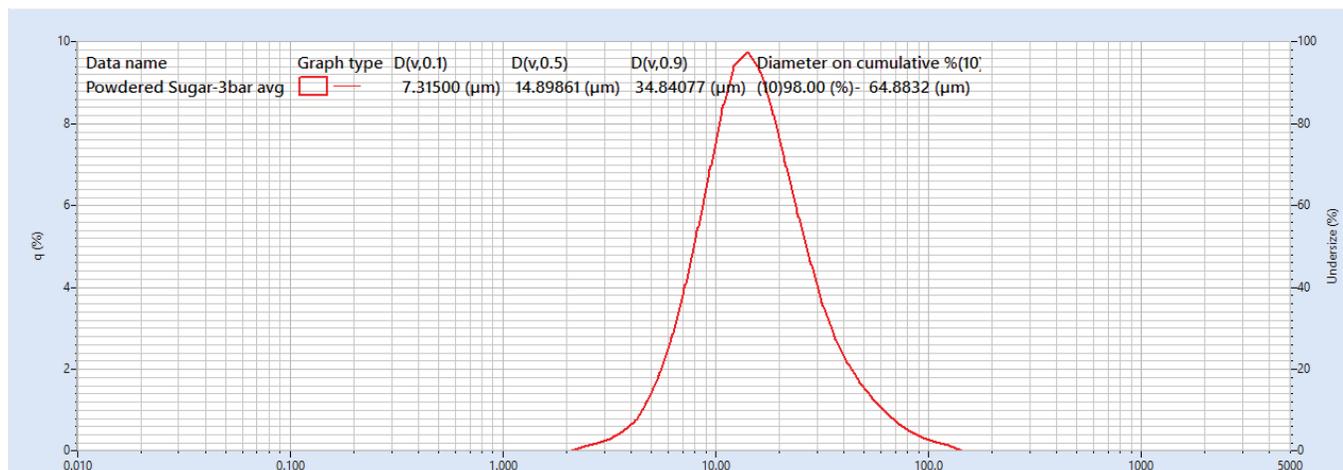


Figure 5: The particle size distribution of 10X powdered sugar dispersed at 0.3MPa of air pressure

un-agglomerated state, some sort of energy assistance for particle dispersion is required. Powdered sugar is set to be measured three repeats each at 0.01 MPa (≈ 0 bar), 0.1 MPa (1 bar), 0.2 MPa (2 bar), 0.3 MPa (3 bar) and 0.4 MPa (4 bar). In doing so, two important parameters can be observed: (1) sample repeatability indicating particle separation and (2) the effect of air pressure on size.

At 0.3 MPa (3 bar) of air pressure, excellent repeatability and a relatively narrow Gaussian distribution is observed. The three measurements at 0.3 MPa were then averaged and displayed below (Figure 5, next page.)

The particle size distribution of 10X powdered sugar measured just as expected as 98% of the particles are smaller than 64.8 μm with a $D(v,10)$ of 7.32 μm , $D(v,50)$ of 14.9 μm , and $D(v,90)$ of 34.8 μm . The result matches typical milling criteria set by manufacturers: 98% < zoomesh [74 μm].

Summary

The particle size of sugar products is important. This study shows the ease of transitioning from sieving technique to dynamic imaging analysis and the effect and importance of air pressure dispersion on the analysis of milled powdered sugar. Laser diffraction and dynamic imaging systems have proven to be an essential and effective replacement of sieves for quality control of sugar and sugar substitutes.

*Both dynamic image analysis (sizes from 1 μm to 30 mm) and laser diffraction techniques (10 nm to 5 mm) are best suited for different applications and measurement needs despite of the overlap in size ranges.

References

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2. HORIBA Scientific Applications Note: Sugar on the CAMSIZER AN141
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