

SUCROSE PARTICLE SIZE REDUCTION - DETERMINATION OF CRITICAL PARTICLE DIAMETERS CAUSING FLOWABILITY DIFFICULTIES

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Abstract

Sugar is an essential food powder used in great quantities in households and food industry. Besides providing sweet taste to food products, it is also responsible for prevention of microbial growth and texture enhancement. In industry surrounding, problems arise during conveying of sugars, especially sucrose, due to its particle size, shape, moisture and glass transition properties. From a food safety aspect, it is important to monitor these properties through out industrial handling of sucrose in order to stop possible microbial contamination, dusting and production process stoppage.

In this research, commercially available sucrose crystals were ground and different particle size fractions were collected using conventional sieving analysis. Obtained fractions were analysed as follows: median particle diameter was determined using laser diffraction particle sizing, bulk density was determined by jolting volumeter method and flow properties (cohesiveness, caking properties and changes in compaction behaviour with different conveying speeds) was determined by powder rheometer method using Powder Flow Analyser (Stable Micro Systems, UK). Fractions obtained by sieving were in particle size range from 800 μm to 50 μm .

Fractions of particle sizes below 200 μm started to exhibit flow difficulties - increased cohesion index and stronger susceptibility to caking. Mathematical correlation between particle sizes, bulk densities, cohesion and caking properties was also defined using Eureka Formulize software package (Nutonian, USA).

Crystalline form of sucrose showed different behaviour at different flow speeds in comparison with pulverized fractions, and proved to be more suitable for industry purposes.

Key words: Sucrose, Particle size reduction, Flow properties.

1. Introduction

European regulation defines "sugar" or white sugar as purified and crystallised sucrose of sound and fair marketable quality [1]. Sugar is a mainstay in baking, candy and confectionery production and is used as a sweetener for the food and beverage industries. Sucrose is table sugar, granular in appearance, and is the most popular of the various sugars for flavouring. Powdered, or confectionery sugar, has a powdery structure.

Sugar, as an important ingredient of most food products, affects the flowability and the rheology of the final product as well. Whether it is used in crystal or in powdered form, it is known to be sensitive to environmental conditions, especially temperature and humidity [2]. Granular sugar is free flowing, hygroscopic, agglomerates, and is mildly abrasive and friable. On the other hand, powdered sugar is also hygroscopic, but non-free flowing, heat sensitive and aeratable. The major powder issue with regard to food ingredient powders is maintaining the stability of ingredient functionality from production right through to the final powder application. Other powder issues are also important in delivering the powder, and they include the ability to handle and transport ingredient powders, dust problems, dust fire and explosion hazards, allergy problems, creation of desirable powder particle properties, and the ability to dissolve the powder when required [3]. Particle size characterization is of great importance if we take into consideration that powders with smaller particles exhibit poor flow properties and usually cause handling problems, with powders being more cohesive as the particle size decreases [4]. It has been found that particle size distribution significantly contributes to flowability and cohesive properties [5].

The aim of this paper was to determine the sugar particle diameter which is critical for sugar flow properties and their impact on bulk characteristics of different particle size fractions. Hausner ratio, cohesion index, caking profiles, mean cake strength and reconstitution properties were determined after milling to the appropriate

particle size in order to determine which form of sugar is more appropriate for industry handling and processing.

2. Materials and Methods

2.1 Materials

Crystalline sucrose samples (Sugar Factory Osijek, Croatia) were purchased at a local supermarket and stored at an ambient temperature of 20 °C and 50 - 55% of relative humidity prior to milling and testing.

2.2 Methods

2.2.1 Sample preparation

In order to obtain different particle size fractions of sucrose powder, samples were milled using Gorenje SMK 202 grinder (Gorenje, Slovenia). Milling time was adjusted according to the desired particle size at 0, 1, 5 and 15 sec. Immediately after grinding, samples were sieved using standardized DIN sieves (Fritsch, Germany) with pore openings from 830 to 50 µm, in order to separate different particle size fractions. After sieving fractions were analyzed by laser diffraction particle sizing, jolting volumeter and a powder rheometer.

2.2.2 Particle size

Fractions obtained by milling and sieving were analysed by laser diffraction method in order to determine median diameter, Sauter diameter and span of the obtained fractions. Mastersizer 2000 instrument coupled with the Scirocco 2000 dry dispersion unit (Malvern Instruments, UK) was used at feed pressure of 1 bar and 50% feed rate, which was increased if observed that the sample flowed poorly through the feed unit. Laser obscuration was kept at 2 - 6% [6, 7]. Three parallel measurements were performed.

2.2.3 Bulk density and Hausner ratio

Bulk density was determined by a laboratory made jolting volumeter. Bulk density after 10, and 1250 taps was determined according to a method described by [8] and Hausner ratio was calculated as a ratio between bulk density at 1250 taps and bulk density at 10 taps [9]. All measurements were performed in triplicate.

2.2.4 Cohesion properties

Sucrose fractions cohesion properties were measured by a powder rheometer - Powder Flow Analyser coupled with TA.HDPlus Texture Analyser (Stable Micro Systems, UK). This method is known to be one of the newest in powder analysis [10] and it is based on measuring the force exerted on the base of the instrument caused by blade movement through the powder cylinder. Cohesion properties were assessed by quick test which determines the cohesion index of the powder,

compression and decompression force. After 2 conditioning cycles, cohesion property is determined by integrating the negative part of the force = $f(\text{distance})$ curve. This area represents a cohesion coefficient which, divided by the sample weight, gives the cohesion index value by which the powders are categorized as free flowing (CI = 11), easy flowing (CI values between 11 and 14) cohesive (CI values in the range 14 - 16), hardened (CI 16 - 19) or extremely cohesive (CI value larger than 19) [10, 11]. Measurements were done in triplicate.

2.2.5 Caking properties

Caking properties were also determined by a powder rheometer - Powder Flow Analyser coupled with TA.HDPlus Texture Analyser (Stable Micro Systems, UK). After two initial conditioning cycles, the powder is compacted five times and the height of the powder cake in the cylinder is recorded. After the fifth cycle, the blade cuts through the cake and the force measured on the base of the cylinder divided by initial column height is recorded as mean cake strength. All the measurements were repeated three times.

2.2.6 Reconstitution properties

Dispersibility

The dispersibility of the powder is the time in seconds taken to disperse a given amount of powder into a given amount of water of a given temperature [8]. Approximately 3 g of the sample were dispersed in 100 mL of distilled water heated to 40 °C. At the moment the powder touched the water surface, stop watch was started and the water containing the sample was stirred manually, making approximately 25 circular movements within 15 sec. The sample was stirred until all the lumps were dispersed and the clock watch was then stopped. Dispersibility was expressed as the number of seconds from when the stirring began until all the lumps were dispersed. Measurements were done in triplicate.

Wettability

Wettability is defined as time, in seconds, necessary for a given amount of powder to penetrate the quiet surface of the water. For the purpose of the measurement, a funnel with a height of 100 mm, lower diameter of 40 mm and an upper diameter of 90 mm, was made. The purpose of the funnel was to distribute the powder as evenly as possible on the water surface. 100 mL of distilled water at 40 °C was used for wetting of approximately 3 g of instant coffee powder. Stop watch was started immediately as the powder hit the water surface and stopped when all the powder was wetted. Wettability was expressed as time in seconds required to achieve complete wetting [12].

2.2.7 Data analysis

Statistica v.10 (StatSoft, USA) and Spearman correlation coefficients with a probability level of 0.05, as a method of nonparametric statistics were used to analyse the influence of particle size on physical properties of sucrose fractions. If a significant influence of particle size on a physical property was determined by Spearman correlation coefficients, data was analysed further using Eureka Formulize software v.0.99.7 (Nutonian, USA) to create a mathematical correlation between interdependent properties.

3. Results and Discussion

3.1 Influence of particle size on Hausner ratio, cohesion and caking properties

Interdependence of Hausner ratio and $d(0.5)$, $D[3,2]$ and span of the sieved fractions is shown in Figure 1.

Hausner ratio represents a quick way of getting an insight into flowability of a certain powder. In this paper, Hausner ratio was determined by a jolting volumeter method and results shown in Figure 1A clearly exhibit that sugar fractions containing smaller particles show higher Hausner ratio values. The highest Hausner ratio value was recorded for particle fractions below $100\ \mu\text{m}$ and ranged from 1.32 to 1.33, which categorizes them in the range of poorly flowing powders [13, 14, and 9]. Since Hausner ratio was calculated by dividing tapped bulk density with loose bulk density, it was logical to expect its dependence on the bulk density determination method. The bulk density was also influenced

by the manner in which particles interacted with each other in the measuring cylinder and the amount of air contained between particles. Smaller particles have the ability to cling more closely together, forming a denser layer occupying a smaller volume, therefore exhibiting higher bulk density values [15]. In this case, fractions with low particle sizes formed large lumps with a great amount of air captured between lumps while being poured into the measuring cylinder. The air entrapped between lumps was pushed out of the measuring cylinder by the vibrations which enabled small particles to pack very close together, giving high values of tapped bulk density. Based on such behaviour of the powder during testing, tapped density and loose density ratio for small particle fractions was high, as shown in Figure 1A. Hausner ratio for size fractions larger than $100\ \mu\text{m}$ ranged mostly from 1.0 to 1.1, which makes these fractions freely flowable [9]. Based on these results, particle sizes lower than $100\ \mu\text{m}$ should be avoided in processing and handling of sugars. Crystalline form proved to be more reliable in this case.

Interdependence of Hausner ratio and particle size distribution span visible in Figure 1B showed that a wider span also means higher Hausner ratio values. For span values significantly higher than 1, Hausner ratio was found to be significantly ($p < 0.05$) higher than those for span values equal to or lower than 1, which confirmed previously stated benefits of a narrow particle size distribution [9].

Cohesion index values as related to particle median diameter, Sauter diameter and span are shown in Figure 2.

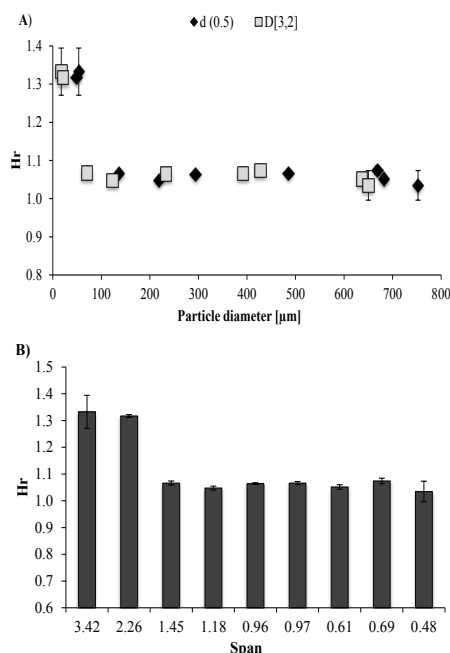


Figure 1. Influence of: A) $d(0.5)$ and $D[3,2]$ on the Hausner ratio of analysed sugar fractions and B) span on the Hausner ratio of analysed sugar fractions

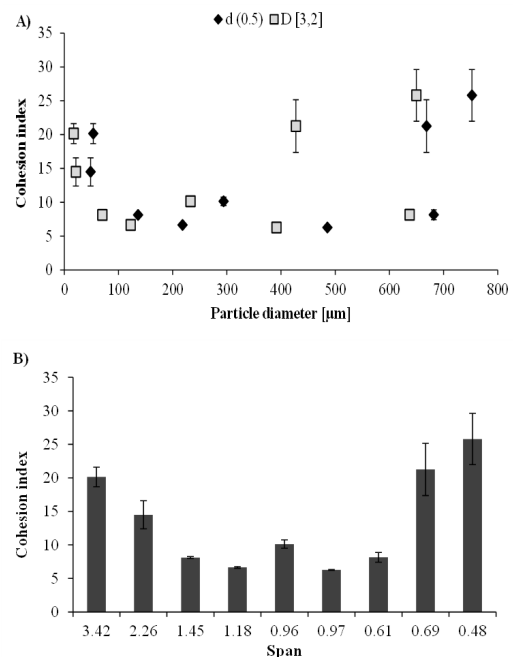


Figure 2. Changes of cohesion index values as related to median ($d(0.5)$) and Sauter ($D[3,2]$) diameter (A) and span (B)

Based on cohesion index values, different sucrose particle size fractions can be categorized as follows: fractions smaller than 50 μm , 710 μm and 800 μm as extremely cohesive, fraction 50-100 μm as cohesive, and all the other analysed fractions as free flowing.

Generally speaking, sugar can be defined as a free flowing powder in its crystalline form. Based on previous research, particle sizes below 200 μm start to show sticky and cohesive properties [3]. In this case, particle sizes below 50 μm proved to be extremely cohesive, while particle sizes between 50 and 100 μm exhibited cohesive properties. All particle ranges from 100 to 630 μm were characterized as free flowing. A further phenomenon was also observed: crystalline form of sucrose above particle sizes 710 and 800 μm also proved to be extremely cohesive (Figure 2A). This phenomenon could be explained by particle to particle and blade to particle friction and crystal breakage. According to previously published research, crystalline materials under the influence of friction undergo crystal breakage if fraction energy and the fraction force to which the particle has been exposed exceeds critical levels [16]. In this case, force exerted from the blade of the instrument on the tested powder in the measuring cylinder was sufficient to cause crystal breakage and changes in particle shape and size, which resulted in rise of cohesion index values.

A similar trend was observed for the impact of span on cohesion index values. Fractions with higher particle size distribution span had higher cohesion index values. In the span range from 1.45 to 0.61, cohesion indexes were the lowest, with an exception of very small span values, which again showed higher cohesion index. Samples with the highest span values were also the samples with the lowest particle size, which meant that the combination of a low particle size and a high span lead to a high cohesion index and poor flowability. On the other hand, the latter 2 samples with span values 0.69 and 0.48 were the crystalline samples with the highest particle diameters, where the crystalline form of the sample had an important influence on the flowability behaviour of these powders, due to friction and crystal breakage.

Caking is defined as a phenomenon in which particles of amorphous powders are progressively deformed until they stick to each other, eventually forming large agglomerates [17]. Influence of median particle size, Sauter diameter and particle size distribution span is shown in Figure 3.

Mean cake strength is determined during the caking test using the powder rheometer and is defined as the average force required to cut the cake formed in the testing cylinder. Generally speaking, powders with smaller particle sizes have higher tendencies to cake. In this case, fractions with the smallest particle sizes

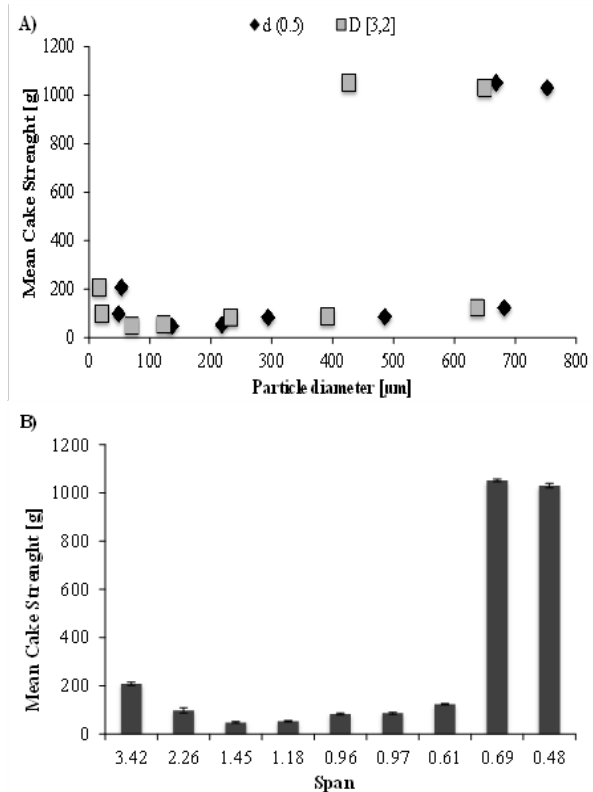


Figure 3. Influence of median and Sauter diameter (A) and particle size distribution span (B) on mean cake strength of sucrose particle size fractions

(lower than 50 μm and 50 - 100 μm) had slightly higher mean cake strength in comparison with particle size fractions in the range from 100 to 630 μm . A significant rise in mean cake strength was detected with large, crystalline particles (Figure 3A). Namely, part of the force required to cut the cake formed in the measuring cylinder is used not solely on the cake disruption, but on crystal breakage, as well. Since the force required to break crystals is usually higher than cake breakage force, these samples exhibit a high mean cake strength value as a result of both forces used during the measurement process. Corresponding results were also recorded for span values (Figure 3A). As previously mentioned, low span values correspond to crystalline particle fractions of 720 and 800 μm and exhibit high mean cake strength values due to crystal breakage during the measurement. These results are in accordance with cohesion index results, based on which it can be concluded that optimum median and Sauter diameters have to be combined with span values and are larger than 100 μm and lower than pure crystalline size of approximately 630 μm .

Besides mean cake strength values, caking test provides an insight into caking susceptibility by definition of cake height ratio values during five testing cycles which result in caking profiles shown in Figure 4.

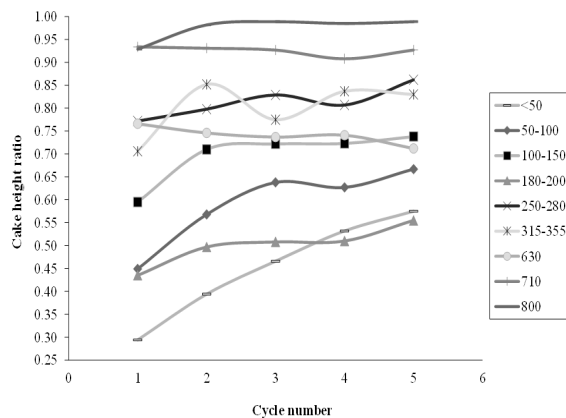


Figure 4. Caking profiles of analysed sucrose particle size fractions

Caking profile interpretation differs in three basic trends: an unchanging cake height ratio with rising cycle numbers means that the tested powder is not susceptible to caking; rising cake height ratios are specific for powders strongly susceptible to caking and decreasing cake height ratios are also characteristic for powders which do not form a cake during testing [11, 18].

Sucrose particle size fractions smaller than 355 μm all showed an increasing cake height ratio, which made them susceptible to caking (Figure 4). Sucrose powder comprised of particles smaller than 50 μm showed the steepest climb of the cake height ratio with increasing cycles, since such small particles had a higher interparticle surface and were, therefore, subjected to more possibilities of particle to particle bondage. Particle size fractions above 630 μm behaved differently during measurement. In comparison to smaller

fractions, the profiles in some cases showed only a slight climb in cake height ratios (800 μm), relatively unchanging values in the case of 710 μm fraction and a slight drop for 630 μm fraction (Figure 4). Such differences in the behaviour of large particle size fractions can, once again, be explained by its crystalline form and changes in shape due to friction caused when the rotating rheometer blade traveled through the measuring cylinder. Cake height ratio profiles have to be examined in combination with mean cake strength data shown in Figure 3, in order to know whether the cake formed during testing breaks down easily. In this case, the fraction below 50 μm showed the highest mean cake strength in comparison to other powdered fractions, which is in accordance with steeply rising cake height ratio profile shown in Figure 4. As for crystalline fractions, high mean cake strength was not in accordance with cake height ratio profiles, which confirmed the thesis about using a part of the compaction force for crystal breakage.

3.2 Reconstitution properties

Besides the behaviour of a certain powder in industry environment, if the product is also used in households in a powdered form, it is of great importance to determine its reconstitution properties. Powdered material with poor reconstitutive properties is considered inappropriate for household usage, not only due to its slow dissolving, but also due to dust problems [9].

In the case of sucrose, it is known as a raw material for industry purposes, but also as a final product used in powdered form. Reconstitution properties (dispersibility and wettability) of different particle size fractions of sucrose analysed in this research are shown in Table 1.

Table 1. Reconstitution properties of different particle size fractions of sucrose

Particle size fraction [μm]	d (0,5) [μm]	D [3,2] [μm]	Span [I]	Wettability [s]	Dispersibility [s]
<50	53.35 \pm 2.89	17.24 \pm 0.54	3.42 \pm 0.19	10.67 \pm 2.05	6.33 \pm 1.53
50-100	48.51 \pm 1.35	21.37 \pm 0.45	2.26 \pm 0.03	20.00 \pm 2.94	10.00 \pm 1.00
100-150	136.06 \pm 0.94	70.33 \pm 0.35	1.45 \pm 0.001	22.67 \pm 2.05	11.00 \pm 1.00
180-200	218.26 \pm 1.57	122.66 \pm 2.25	1.18 \pm 0.01	30.33 \pm 0.94	12.33 \pm 2.08
250-280	293.92 \pm 2.75	233.00 \pm 3.48	0.96 \pm 0.04	15.00 \pm 0.00	11.67 \pm 1.53
315-355	485.63 \pm 11.04	391.65 \pm 13.76	0.97 \pm 0.05	2.33 \pm 0.47	1.67 \pm 0.58
630	681.91 \pm 14.12	637.37 \pm 13.72	0.61 \pm 0.03	< 1	< 1
710	668.40 \pm 3.66	427.36 \pm 17.33	0.69 \pm 0.03	< 1	< 1
800	752.30 \pm 11.96	649.89 \pm 9.26	0.48 \pm 0.05	< 1	< 1

In direct comparison of wettability and dispersibility times, it was visible that wettability times range from less than 1 sec to 30.33 sec, while the dispersibility times were significantly ($p < 0.05$) shorter, with a range from less than 1 to 12.33 sec. Dispersibility testing method required a use of a glass rod for breaking down of lumps formed during exposure of the powder to the water surface, which enabled the powder to break the water surface more rapidly. Minimal wettability times were achieved with crystalline fractions larger than $630 \mu\text{m}$ which easily overcome the surface tension of the water in the testing beaker. The same was observed for dispersibility. In both cases, wettability and dispersibility, increased with particle size to maximum values of 30.33 (wettability) and 12.33 (dispersibility) for particle size fractions $250 - 280 \mu\text{m}$. Above that particle size value, wettability and dispersibility times began to decrease, mostly due to higher percentage of crystalline particles in the tested fractions. In the case of particles smaller than $50 \mu\text{m}$, small particle size resulted in higher active surface of the particles and thus more interparticle bondage. Therefore, small particles formed larger lumps during testing which made it easier to break the surface tension.

Formation of lumps decreased with an increasing particle size and wettability and dispersibility times were higher up to the $250 - 280 \mu\text{m}$ particle size fraction. It is important to emphasize that, in comparison to cohesion indexes and mean cake strength measured rheometrically, reconstitution properties exhibited opposite behaviour - particle sizes which had the smallest cohesion index and mean cake strength values showed the highest dispersibility and wettability times.

3.3 Data analysis

Statistica v.10 (StatSoft, USA) and Spearman correlation coefficients with a probability level of 0.05, as a method of nonparametric statistics were used to analyse the influence of particle size on physical properties of sucrose fractions. Spearman correlation coefficient (r_s) values are listed in Table 2.

As shown in Table 2, all particle size parameters had a significant influence on Hausner ratio values. Hausner ratio decreased with an increase in median and Sauter diameter, while these values decreased with a decrease in span. Statistically speaking, there was no significant influence of particle size parameters on the cohesion

Table 2. Influence of particle size on flowability parameters - Spearman rank order correlation coefficients (r_s)

Param.	Hausner ratio	Cohesion index	Mean cake strength	Wettab.	Dispersib.	Cake height ratio 1	Cake height ratio 2	Cake height ratio 3	Cake height ratio 4	Cake height ratio 5
d (0,5)	-0.68*	0.20	0.45	-0.78*	-0.68*	0.80*	0.82*	0.80*	0.78*	0.68*
D [3,2]	-0.70*	0.18	0.42	-0.75*	-0.66	0.83*	0.85*	0.83*	0.80*	0.70*
Span	0.72*	-0.25	-0.40	0.71*	0.59	-0.87*	-0.83*	-0.85*	-0.78*	-0.72*

* Marked coefficients are significant at $p < 0.05$

Table 3. Models for interdependent variables correlation calculated using Eurequa Formulize software

Model	Equation	Max. error	Mean squared error	Mean absolute error
Hr = f(d(0.5))	$\left(d(0.5)^2 - (d(0.5)) \right)^{\frac{d(0.5)}{(0.5)+(d(0.5))^2}}$	0.05	0.001	0.03
Hr = f(D[3,2])	$Hr = \frac{D[3,2]}{D[3,2] - \sqrt{D[3,2] + \log D[3,2]}}$	0.07	0.001	0.02
Hr = f(span)	$Hr = \sqrt{\sqrt{\text{span} + \frac{\text{span} + \sqrt{\text{span}}}{e^{\text{span} + \text{span}^5}}}}$	0.09	0.001	0.02
Wettability = f(d(0.5))				
Wettability = f(D[3,2])	Wettability and dispersibility modelling resulted in complex sin and cos functions, which proved to be inadequate for very large and very small particle sizes			
Wettability = f(span)				
Dispersibility = f(d(0.5))				

index and mean cake strength. However, it is important to emphasize that both of these parameters acted similarly with a change in particle size. Values between 100 and 630 μm appeared to be the optimal sucrose particle sizes for powder handling. The same can be stated for mean cake strength.

Wettability was influenced by all tested particle size distribution parameters. The influence of particle size distribution parameters diminished when it came to dispersibility, mostly due to the testing method which included mechanical dissipation of the lumps formed on the water surface. All cake height ratios were significantly influenced by median diameter, Sauter diameter and span. Higher median diameter and higher particle size of sucrose led to higher cake height ratios, which could be explained by the prevailing influence of crystal breakage in comparison to actual particle size.

A mathematical correlation between interdependent parameters was calculated using Eurequa Formulize software. Models are presented in Table 3.

Models shown in Table 3 enable calculation of different Hausner ratio parameters in correlation with different particle size distribution parameters. Although statistical analysis showed a significant correlation between particle size diameters and reconstitution properties, wettability and dispersibility modelling resulted in sin and cos functions, which were inadequate for calculation of reconstitution times for very small and very large particle diameters. Furthermore, cake height ratios also proved to be particle size dependant, but the expressions used for modelling of all cake height ratio cycles in one equation are also very complex and required expensive software support.

4. Conclusions

- Median diameter, Sauter diameter and span had a significant influence on Hausner ratio values. Hausner ratio decreased with an increase in median and Sauter diameter, while these values decreased with a decrease in span.

- Values between 100 and 630 μm appeared to be the optimal sucrose particle sizes for powder handling based on the cohesion index and mean cake strength results.

- All cake height ratios were significantly influenced by median diameter, Sauter diameter and span. Wettability was also influenced by all tested particle size distribution parameters. This influence diminished when it came to dispersibility, mostly due to the testing method which included mechanical dissipation of the lumps formed on the water surface.

- It is important to emphasize that, in comparison to cohesion indexes and mean cake strength measured

rheometrically, reconstitution properties exhibited opposite trends - particle sizes which had the smallest cohesion index and mean cake strength values showed the highest dispersibility and wettability times. This led to a conclusion that sucrose used in industrial environment and as a final product in households differed in optimal particle sizes, flowability and reconstititional properties.

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