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Time-dependent changes in suspensions of sucrose powder in saturated sucrose solution

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Abstract

Viscous and pseudoplastic fluids were prepared at 1:2.1, 1:2.3 and 1:2.5 proportions of saturated aqueous sucrose solution to sucrose powder. These specimens gradually became more solid-like upon storage for two weeks in closed containers. Scanning electron microscopy revealed time-dependent changes in microstructure accompanying the redistribution of sucrose molecules in these specimens. These changes included: (1) an increased particle size from approximately 5–25 μm, (2) the appearance of highly faceted, single crystalline particles of sucrose and (3) coarsening and densification of sucrose particle networks. Pronounced changes in textural parameters were observed within 24 h of storage and are likely associated with changes in microstructure accompanying the redistribution of sucrose in the specimens.

Keywords: Sucrose; Crystallization; Texture profile analysis

1. Introduction

Several publications focus on the chemistry and applications of sucrose (Krutosikova & Uher, 1992; Belitz & Grosch, 1999, ch. 5). Sucrose is commonly used as a sweetener, a food preservative, a food texturiser and a decorating aid. Significant amounts of sucrose are also used in the manufacture of inverted sugar. Control of the rheology of aqueous suspensions of sucrose powder and other foodstuffs is essential in a variety of industrial applications involving fluid pumping, pipeline transport, mold filling and more (Steffe, 1996). For example 18% sucrose dissolved in sugar syrup is used for manufacturing caramel candies in which the sucrose does not crystallize from solution upon cooling. Fondants are formed at higher concentrations of sucrose in sugar syrup (Lees & Jackson, 1973).

In our recent papers, we measured rheological properties of aqueous suspensions of saturated saccharide solutions and granular starches (Sikora, 2001; Sikora, Mazurkiewicz, Tomasik, & Pielichowski, 1999). In these systems, we observed time-dependent transitions from pseudoplastic fluids to intractable, solid-like behaviour. The starch variety, the starch concentration and the type of saccharide in solution strongly influenced the kinetics of these transitions. We also evaluated time-dependent transitions from pseudoplastic fluids to intractable, solid-like behaviour in suspensions of granular starches and saturated aqueous solutions of mineral salts (Lii, Tomasik, Hung, & Lai, 2002). In contrast, permanent pseudoplasticity in slurries of micrometric alumina powder in saturated aqueous solutions of mono- and disaccharides was observed by Kim, Schilling, Tomasik, and Auh (2000). The rheology of such suspensions did not change with time, because alumina powder was unable to dehydrate saccharides in their saturated solutions.

In our subsequent paper (Sikora, Tomasik, Schilling, & Sady, 2002) we showed further examples of

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time-dependent pseudoplasticity in blends of edible food powders (potato starch, corn flour, wheat flour, milk powder and cocoa powder) suspended in saturated aqueous sucrose solutions. We further evaluated time-dependent textural parameters in these specimens. The role of the size and shape of suspended powder particles and their swelling ability and/or dissolution in water was evaluated.

In the aforementioned studies, we composed pseudoplastic blends from saturated saccharide solutions with micrometric powders that belong to four categories: (i) water-insoluble and incapable of dehydrating sucrose in saturated solutions (alumina); (ii) water-insoluble but capable of dehydrating sucrose in saturated solutions (starch); (iii) partly water-soluble, forming molecular solutions (e.g. cocoa powder, flours) and (iv) partly water-soluble, forming colloidal solutions and/or emulsions (milk powder). In this study, we present a system composed of saturated sucrose solution to which fully water-soluble sucrose powder was admixed. In this manner, we complete a comprehensive set of studies in which all possible systems are evaluated. Sucrose was selected as the fully water-soluble powder in order to make this study useful for the food industry.

2. Materials and methods

2.1. Materials

Crystalline sucrose was purchased from Aldrich (St. Louis, MO, USA). Sucrose powder was manufactured by Sugar Enterprise Sroda (Sroda Wielkopolska, Poland) and followed Polish Standards PN-86/A-74860 for sugar powder.

2.2. Sample preparation

A saturated aqueous stock solution of sucrose was prepared from redistilled water (100 g) and crystalline sucrose (201 g) at 20 °C. Individual specimens were prepared by the addition of sucrose powder to stock solution. In units of grammes of stock solution per gramme of sucrose powder added, specimen concentrations were 1:2.1, 1:2.3 and 1:2.5. Specimen concentrations were empirically determined in order to cover a maximum range of compositions at which flow behaviour could be observed. The lowest concentration of sucrose powder in a given specimen corresponded to a transition from highly fluid state to pseudoplastic, viscous behaviour. The highest concentration of sucrose powder in a given specimen corresponded to a transition from pseudoplastic fluid to semisolid paste. Specimens were stored for 2 weeks at ambient temperature in tightly closed containers.

2.3. Texture profile analysis

Texture profile analysis (TPA) was performed on the above specimen with a computer-controlled TA-XT 2 penetrometer manufactured by Stable Micro Systems (Haslemere, England). This instrument pushed a standard P1S spherical ball (25.4 mm diameter) into a test specimen to a depth 25 mm at a rate of 1 mm/s at 20 °C and subsequently moved the ball to the starting position. After a time interval of 5 s, the ball was subsequently pushed into the same specimen at the same speed and depth as above and finally removed. The measurements were duplicated on specimens immediately, 24 h later and seven days later. The computer-controlled instrument recorded penetration data from which food texture parameters, such as hardness, fracturability, adhesivness, springiness, cohesiveness, chewiness, resilience and stringiness, were determined. The physical meanings of these parameters are explained in the literature (Steffe, 1996).

2.4. Scanning electron microscopy

As-received sucrose powder was sputter-coated with gold and subsequently evaluated by scanning electron microscopy (JSM-5400, Jeol USA Inc., Peabody, Massachusetts). Specimens of sucrose powder in stock solution underwent 3 weeks of storage in tightly sealed containers and were subsequently gold sputter-coated and evaluated by scanning electron microscopy.

2.5. Rheological measurements

Rheological measurements were performed on the above samples with a Searle-type rheometer (RheoStress RS 75 Gebrueder Haake GmbH, Karlsruhe, Germany) equipped with a parallel plate system. In order to avoid drying of the examined blends during rheological measurements, mineral oil was applied to cover the edges of the samples having contact with the open air.

3. Results and discussion

During the addition of sucrose powder to saturated sucrose stock solution, three regimes of behaviour were noted: (1) highly fluid, (2) viscous and pseudoplastic and (3) semisolid (Fig. 1). Upon first addition of powder, a given specimen formed more viscous, pseudoplastic fluid. Upon further addition of powder, the specimens became more pseudoplastic. The transition to pseudoplastic fluid occurred when the proportion of solution to sucrose powder reached 1:2.1. This pseudoplastic behaviour was retained upon further addition of stock solution until a critical proportion of stock solution to sucrose powder (1:2.5) was reached. Higher concentrations beyond this

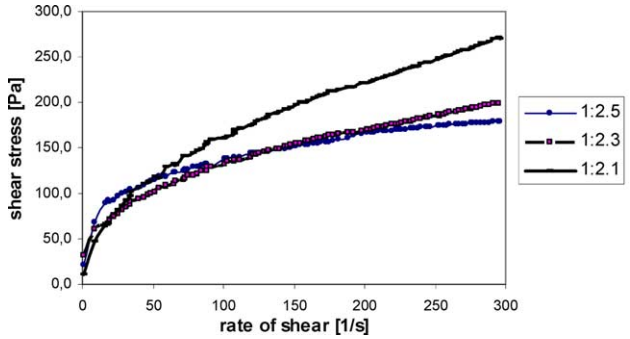

Fig. 1. Flow curves of the blends of saturated sucrose solution with sucrose powder.

critical limit resulted in the formation of hard, untractable solid (Fig. 1). Fairly viscous and pseudoplastic specimens gradually became more viscous upon storage for two weeks in closed containers. After two weeks of storage, these samples exhibited solid-like behaviour. Simultaneously, the surfaces of these specimens exhibited a layer of transparent fluid (sucrose solution). We shall hereafter refer to these specimens as aged.

SEM indicated that the sucrose powder in viscous and pseudoplastic specimens underwent a significant change in morphology during 2 weeks of aging (Fig. 2). As-received sucrose powder has an average particle size of approximately 5 μm. However, upon aging, the average particle size is increased to approximately 25 μm. Furthermore, the aged particles appear highly faceted, suggesting that each particle is a single crystal.

It is likely that redistribution of sucrose molecules between the solid powder and the saturated solution took place during this two-week period of aging. The redistribution is likely driven by coarsening (reduction of total free energy by minimization of surface area). In this manner, as a single sucrose molecule diffuses from a solid powder surface into saturated solution, one molecule of sucrose in solution undergoes dehydration and crystallizes (Fig. 2).

Crystal growth reduces the specific surface area of sucrose (i.e. the surface area per gramme of sucrose powder) and densifies the solid sucrose microstructure. This could explain the formation of a liquid film on the specimen surfaces during two weeks of aging.

Textural studies are presented in Table 1. Increases in hardness, fracturability, gumminess and chewiness paralleled increases in the content of sucrose powder in the fluid. Increases in these textural parameters were particularly marked on passing from 1:2.3 to 1:2.5 concentrations. In each sample, the hardness and fracturability increased by approximately 200% after 24 h of storage. Although raising the powder concentration significantly increased hardness and fracturability, scanning electron microscopy did not reveal significant changes in microstructure as a result of raising the powder concentration.

Simultaneously, changes in the concentration of sucrose powder sharply reduced the adhesiveness while mildly decreasing cohesiveness. Time-dependent increases in adhesiveness were more concentration-dependent. The largest increases in adhesiveness (111%) occurred in the most diluted (1:2.1) specimens, whereas the adhesiveness increased by hardly 10% in the most concentrated specimens. Cohesiveness decreased by approximately 100% in all three fluids upon 24 h of storage. Resilience, springiness and stringiness were only slightly influenced by time of storage and concentration of sucrose powder.

Changes in hardness, fracturability, adhesiveness and cohesiveness were reduced by approximately an order of magnitude within the subsequent 7 days. The pronounced changes in textural parameters within the first 24 h are likely associated with changes in microstructure accompanying the redistribution of sucrose in the specimens. Undoubtedly, this microstructural evolution also included redistribution of water molecules, thereby influencing the properties of lubricating films between powder surfaces. Similar time-dependent effects of a sudden increase in the viscosity, hardness and fracturability were formerly observed in pseudoplastic fluids composed of saturated sucrose solution and one each of the following edible powders: potato starch, wheat flour and corn flour, cocoa powder and milk powder (Sikora et al., 2002).

Increases in hardness and fracturability, as well as decreases in adhesiveness and cohesiveness, are generally

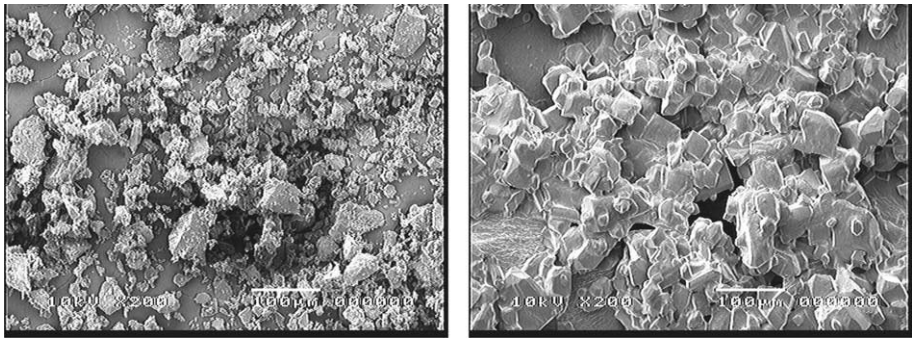

Fig. 2. Scanning electron micrographs of freshly prepared 1:2.1 saturated sucrose solution–sucrose powder. The micrograph on the right was obtained after a two-week storage of that solution at ambient temperature. The magnification of each micrograph is 200×.

Table 1
Parameters from texture profile analysis*

Period of storage/Increase (%)	Hds[a]	Fra[b]	Adh[c]	Spr[d]	Coh[e]	Chew[f]	Res[g]	Str[h]	Gum[i]
Sample 1:2.1									
Instantly	201.7 ± 7.4	201.7 ± 7.4	−1720 ± 52	1.049 ± 0.004	0.723 ± 0.027	152.8 ± 0.6	0.008 ± 0.001	29.1 ± 0.1	145.8 ± 0.1
After 24 h	586.2 ± 39.7	559.5 ± 34.6	−3633 ± 166	1.027 ± 0.03	0.355 ± 0.029	212.6 ± 3.5	0.005 ± 0.001	29.6 ± 0.0	207.0 ± 2.8
Increase	*190.6 ± 32.7*	*177.4 ± 28.4*	*−111.2 ± 16.6*	*−2.1 ± 0.7*	*−103.7 ± 26.4*	*39.1 ± 2.9*			*42.0 ± 2.0*
After 7 days	896.9 ± 11.1	657.8 ± 250.2	−4753 ± 74	1.018 ± 0.005	0.278 ± 0.01	253.4 ± 1.4	0.006 ± 0.001	29.7 ± 0.1	249.0 ± 2.5
Increase	*53.0 ± 13.2*	*17.6 ± 55.4*	*−30.8 ± 8.4*	*−0.9 ± 0.8*	*−27.7 ± 10.9*	*19.2 ± 2.7*			*20.3 ± 3.1*
Sample 1:2.3									
Instantly	457.1 ± 14.4	452.8 ± 18.7	−3954 ± 113	1.023 ± 0.001	0.653 ± 0.011	305.1 ± 4.4	0.006 ± 0.001	29.5 ± 0.0	298.2 ± 4.6
After 24 h	1280.8 ± 0.2	1282.8 ± 0.2	−8059 ± 287	1.017 ± 0.001	0.338 ± 0.007	439.7 ± 8.1	0.006 ± 0.001	29.6 ± 0.0	432.4 ± 8.3
Increase	*180.2 ± 9.1*	*183.3 ± 12.3*	*−103.8 ± 13.5*	*−0.6 ± 0.2*	*−93.2 ± 7.4*	*44.1 ± 4.8*			*45.0 ± 5.1*
After 7 days	2124.9 ± 142.1	1704.2 ± 278.6	−10395 ± 144	1.014 ± 0.002	0.239 ± 0.015	512.6 ± 3.8	0.008 ± 0.001	29.6 ± 0.0	505.6 ± 2.9
Increase	*65.9 ± 10.9*	*32.9 ± 21.7*	*−29.0 ± 6.6*	*0.3 ± 0.3*	*−41.4 ± 12.6*	*16.6 ± 3.0*			*16.9 ± 3.0*
Sample 1:2.5									
Instantly	1760.9 ± 48.4	1760.9 ± 48.4	−13545 1 ± 1032	015 ± 0.001	0.569 ± 0.045	1018.4 ± 107.3	0.009 ± 0.000	29.6 ± 0.0	1003.3 ± 106.5
After 24 h	5613.0 ± 80.6	5613.0 ± 80.6	−16232 ± 2079	1.002 ± 0.006	0.252 ± 0.026	1414.8 ± 120.0	0.016 ± 0.001	29.5 ± 0.2	1412.1 ± 127.6
Increase	*218.8 ± 13.7*	*218.8 ± 13.7*	*−19.8 ± 6.7*	*1.3 ± 0.7*	*−123.0 ± 48.7*	*38.9 ± 29.6*			*40.7 ± 31.0*
After 7 days	8439.5 ± 7.7	8439.5 ± 7.7	−20077 ± 683	0.946 ± 0.011	0.215 ± 0.003	1717.0 ± 4.7	0.025 ± 0.001	29.9 ± 0.2	1814.5 ± 24.9
Increase	*50.4 ± 2.5*	*50.4 ± 2.5*	*23.7 ± 23.0*	*5.9 ± 1.9*	*−17.2 ± 13.9*	*21.4 ± 11.6*			*28.5 ± 14.7*

[a] Hardness.
[b] Fracturability.
[c] Adhesiveness.
[d] Springiness.
[e] Cohesivensess.
[f] Chewiness.
[g] Resilience.
[h] Stringiness.
[i] Gumminess.
* All experiments were duplicated.

not beneficial to food quality. However, the stability in springiness and stringiness are beneficial properties of these fluids.

4. Conclusions

Viscous and pseudoplastic specimens can be formed at certain concentrations of saturated aqueous sucrose solution and sucrose powder. A gradual transition toward solid-like behaviour is caused by a slow redistribution of sucrose molecules to form dense, single crystals. This redistribution is accompanied by changes in hardness, fracturability, adhesiveness and cohesiveness.

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