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ORIGINAL ARTICLE

Physicochemical properties and stability of sucrose/glucose agglomerates obtained by cocrystallization

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Abstract

Glucose is used as ingredient in a wide variety of food and pharmaceutical products such as sport drinks, syrups, and tablets, among others. However, this monosaccharide can react easily with several substances (e.g., amino acids, peptides, and proteins) causing brown coloration and decomposition. In the current work, the cocrystallization of glucose with sucrose is presented as a useful strategy to enhance the physicochemical properties and stability of glucose. The sucrose/glucose agglomerates showed low water content (0.1–1.5 wt %) and water activity (0.4–0.7). Besides, all products showed values of dynamic angle of repose between 40° and 45°, which are characteristics of free-flowing powders. The sucrose/glucose agglomerates were blended with sodium bicarbonate (5:1 wt/wt) and the resulting mixtures were analyzed by differential scanning calorimetry before and after 12 days of storage at 75% RH and 20 °C. The thermal parameters were maintained almost unaltered for the formulations containing cocrystallized products.

Practical applications

Glucose constitutes one of the most used ingredients in food and pharmaceutical industries as a sweetener, cryoprotectant, and excipient, among others. However, this sugar tends to uptake water from the surrounding environment affecting its handling properties. In the current work, the cocrystallization process (an emerging encapsulation method) is presented as a useful way of enhancing the physical properties of glucose. Through the encapsulation of glucose by cocrystallization it was possible to improve the handling and technological properties of this sugar (e.g., flowability and stability). In addition, the interaction of glucose with sodium bicarbonate (a model of food ingredient) was prevented. These results could be potentially applied in both food and pharmaceutical industries.

1 | INTRODUCTION

Cocrystallization is an emerging encapsulation process which has gained growing interest in the last years; thanks to its operational simplicity. In this process, the perfect sucrose crystalline structure is modified and a porous matrix formed by irregular agglomerated crystals is obtained which hold and protect active ingredients. Through the cocrystallization process, the handling properties of several food ingredients such as natural extracts, minerals, flavors, honey, fruit juices, and others have been improved (López-Córdoba, Deladino, Agudelo-Mesa, & Martino, 2014; López-Córdoba, Gallo, Bucalá, Martino, & Navarro, 2016; Sardar & Singhal, 2013).

The agglomerates obtained by cocrystallization can be used as sugar-based ingredients in food formulations (e.g., chocolate, mints, soft drinks, chewing gum, toffee, and fudges) and also as pharmaceutical excipient for direct compression (Awad & Chen, 1993; Bowe, 1998; López-Córdoba et al., 2016, 2015).

Carbohydrates constitute one of the most used food ingredients produced worldwide. These materials are commonly employed in food and pharmaceutical industries as sweetener, cryoprotectants, pharmaceutical excipients, and among others (Bowe, 1998). Carbohydrates may also be used as encapsulating agents of active compounds (Bhandari & Hartel, 2002; Sardar & Singhal, 2013). In this field, sucrose has important advantages because of its low cost and availability.

Glucose is commonly used in the food industry as a key ingredient in sport drinks, jams, desserts, candies, syrups, and other products. It is naturally occurring and is found in fruits and other parts of plants in its free state.

Glucose may be crystallized as either α-glucose monohydrate, anhydrous α-glucose, or anhydrous β-glucose. Frequently, anhydrous glucose absorbs significant amounts of moisture to form the monohydrate during storage (Scholl & Schmidt, 2014). This fact can lead to products with poor flowability and caking, and consequently, to lost-production time and decreased customer satisfaction (Scholl & Schmidt, 2014). As a primary source of energy for living organisms, glucose is commonly used therapeutically in fluid and nutrient replacement. Oral rehydration salts are dry mixtures of powders based on glucose, sodium bicarbonate, sodium chloride, and potassium chloride (WHO, 2006). Several authors have reported that sodium bicarbonate, used to produce or maintain an alkaline pH in these salts, is the main cause of the brownish discoloration of the glucose resulting from spontaneous polymerization, or caramelization, of certain intermediate furfural compounds (Izgu & Baykara, 1981; Santoro, Hackmann, & Moudatsos, 1993). Thus, encapsulation by cocrystallization could be an economic and flexible alternative to solve glucose instability in powder mixtures (e.g., oral rehydration salts).

In the present work, sucrose/glucose agglomerates were successfully obtained by cocrystallization. The agglomerates were characterized in terms of their water content, water activity, flow properties, water sorption properties, and thermal stability. Moreover, the effect of the cocrystallization of glucose with sucrose on its solid state stability in presence of sodium bicarbonate (a model of food ingredient) was evaluated.

2 | MATERIALS AND METHODS

2.1 | Materials

Anhydrous glucose (Anedra, Buenos Aires, Argentina), commercial sucrose (Ledesma, Buenos Aires, Argentina), and sodium carbonate (Parafarm, Buenos Aires, Argentina) were used.

2.2 | Preparation of the sucrose/glucose agglomerates by cocrystallization

The agglomerates were fabricated as reported by López-Córdoba et al. (2014; Figure 1). The different formulations used are shown in Table 1. Briefly, supersaturated sucrose syrups with and without the addition of glucose were heated to 132 °C under constant stirring at 500 rpm until the beginning of cocrystallization was achieved (Vertical agitator IKA Labortechnik, Staufen, Germany). Then, the mixtures were removed from the hot plate and cooled to room temperature under stirring at 700 rpm. Finally, the samples were dried at 40 °C for 15 hr and then ground and sieved through a 500 μm mesh. The products without glucose addition (i.e., recrystallized sucrose) and the cocrystallized products with 10 g and 15 g of glucose added per 100 g of sucrose will be referred to as "F1," "F2," and "F3," respectively (Table 1).

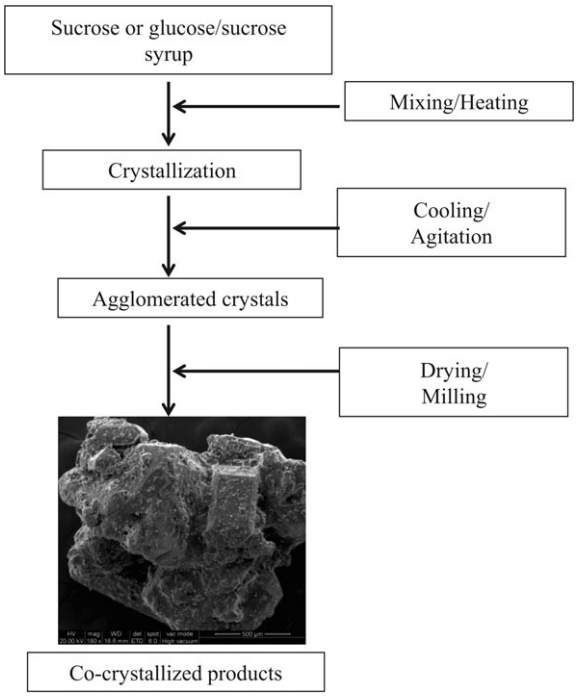

FIGURE 1 Schematic diagram of the cocrystallization process

2.3 | Moisture content and water activity

Water content (%) was determined gravimetrically according to AOAC methods in a vacuum oven at 70 °C (AOAC, 1998). Water activity (A_w) was measured employing AquaLab Series 3 TE (United States) equipment.

2.4 | Water gain (%)

The agglomerates were placed in desiccators conditioned with a supersaturated solution of sodium chloride (75% RH) at 20 °C. Samples were taken at different times and the changes in the mass of the samples were recorded until constant value. The water gain (%) of the powders was calculated as follows:

$$\Delta W(\%) = \left(\frac{W_t - W_0}{W_0}\right) \times 100, \quad (1)$$

where W_t and W_0 are the weights at each time t and before exposing the agglomerates to 75% RH, respectively.

2.5 | Dynamic dewpoint isotherms generation

The moisture adsorption profiles of the agglomerates (1–2 g of sample) under dynamic conditions were measured using an Aqualab Vapor Sorption Analyzer (VSA 1020, United States) at 20 °C (Scholl & Schmidt, 2014).

TABLE 1 Formulations of the agglomerate products

Ingredient	F1	F2	F3
Sucrose (g)	100	100	100
Glucose (g)	–	10	15
Distilled water (ml)	20	20	20

2.6 | Flowability

The dynamic angle of repose was measured to characterize the flowability of the samples. This parameter was evaluated using a cylindrical chamber, which was rotated slightly until the slipping of the powders took place (Solids handling study bench, CEN, Armfield, United Kingdom). The dynamic angle of repose is the angle with respect to the horizontal formed by the agglomerates (USP 30-NF 25, 2007).

2.7 | X-ray diffraction and differential scanning calorimetry

X-ray diffractograms were recorded at 2θ between 10° and 30° in a X'Pert PRO (The Netherlands) diffractometer operated at 40 kV and 40 mÅ. The studies of differential scanning calorimetry (DSC) were performed using a Q100 unit (TA Instruments, United States). The samples (3–5 mg) were placed in aluminum pans and heated at 10 °C/min from 50 to 220 °C in a nitrogen atmosphere. An empty aluminum pan was used as reference.

2.8 | Preparation of physical mixtures of cocrystallized products with sodium bicarbonate

Physical mixtures (1:5 wt/wt ratio) containing sodium bicarbonate and cocrystallized products with glucose (F2 and F3) were prepared (M1 and M2, respectively). In addition, blends containing sodium salt and raw glucose (M0) were also made at the same ratio (1:5), for comparison. The weight ratio 1:5 wt/wt was chosen based on the sodium bicarbonate: Sugar balance established for oral rehydration salt by the World Health Organization (WHO) and the United Nations Children's Emergency Fund (WHO, 2006).

The physical blends were placed in Petri dishes and stored at 75% RH and 20 °C during 12 days. Samples were taken out before and after storage and analyzed by DSC as mentioned above (Section 2.8).

2.9 | Statistical analysis

All results were reported as mean ± *SD* of at least three replicates. Analysis of variance was carried out to compare the means using SYSTAT Inc. software (Evanston, United States). The Tukey test was also performed using a level of 95% confidence (α = 0.05).

3 | RESULTS AND DISCUSSION

3.1 | Moisture content and water activity

Glucose addition led to higher water amounts in the agglomerates, values of 0.17 ± 0.05, 0.75 ± 0.10, and 1.43 ± 0.27% were obtained for F1, F2, and F3, respectively. A similar behavior was reported by Bhandari and Hartel (2002) and it was attributed to the presence of low molecular weight sugars (e.g., glucose or fructose) which delayed the crystallization of sucrose. Besides, F1 and F2 showed A_w values between 0.40 and 0.55 (p > .05); while, F3 showed a statistically significant increase in the water activity, with respect to the product without glucose (F1), exhibiting A_w values of around 0.64.

TABLE 2 Values of flowability and deliquescense point (RH_0) of recrystallized sucrose (F1) and cocrystallized products with 10 g (F2) and 15 g (F3) of glucose added per 100 g of sucrose

Sample	Angle of repose (°)	RH_0 (%)
F1	41.8 ± 4.7	88.3 ± 0.1
F2	43.1 ± 3.9	81.9 ± 0.1
F3	42.2 ± 4.1	80.2 ± 0.1

3.2 | Flowability and water uptake ability

All samples showed values of dynamic angle of repose ranging between 40° and 50° (Table 2). Similar results were reported when zinc sulfate salt was cocrystallized with sucrose (López-Córdoba et al., 2016). It is well-known that repose angles ranging between 40° and 50° are indicative of powders with optimal handling properties, whereas powders with repose angles above 50° are consider very cohesive materials (Geldart, Abdullah, Hassanpour, Nwoke, & Wouters, 2006; Santomaso, Lazzaro, & Canu, 2003). Values of dynamic angle of repose for raw sucrose and glucose have been found around 39° and 57°, respectively (Amefia, Abu-Ali, & Barringer, 2006). Therefore, it can be highlighted that the cocrystallization process constituted a useful strategy to improve the flowability of glucose.

Figure 2 shows the kinetic of water gain of the agglomerates (F1, F2, and F3) stored at 75% RH and 20 °C. All products showed negligible water uptake ability under these storage conditions (i.e., <0.5 wt %). According to Newman et al. (2008), these agglomerates could be classified as slightly hygroscopic. The ability of water vapor uptake of the cocrystallized products increased for the higher added glucose amount (F3; Figure 2). As it is well-known, the water adsorption of these products is attributed to the links between the hydrogen present in water and the hydroxyl groups available in sugar molecules. In this sense, the cocrystallized products with glucose (F2 and F3) have a higher amount of OH groups available to interact with water, compared with recrystallized sucrose (F1).

3.3 | Dynamic dewpoint sorption profiles

The effect of the glucose addition on the dynamic dewpoint sorption profiles of the products is shown in Figure 3. All samples showed

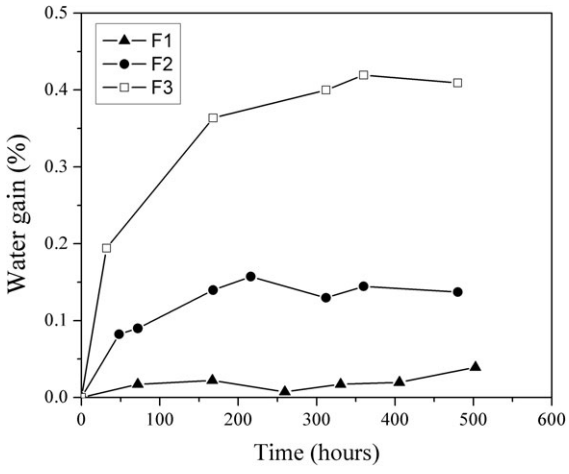

FIGURE 2 Kinetic of water gain of recrystallized sucrose (F1) and cocrystallized products with 10 g (F2) and 15 g (F3) of glucose added per 100 g of sucrose under storage conditions of 75% RH and 20 °C

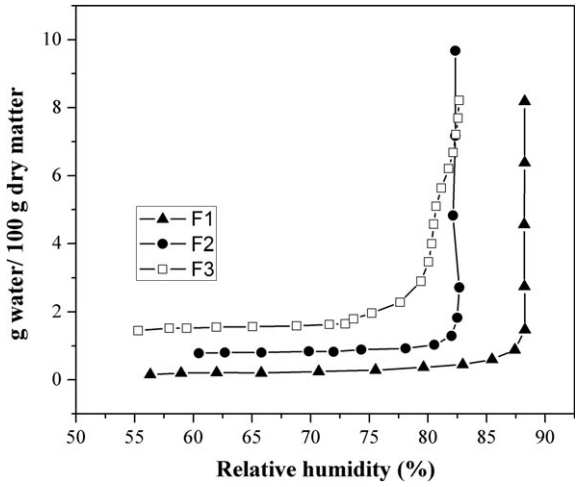

FIGURE 3 Dynamic dewpoint isotherms of recrystallized sucrose (F1) and cocrystallized products with 10 g (F2) and 15 g (F3) of glucose added per 100 g of sucrose

isotherms with similar form, considered as type III according to Brunauer-Emmett-Teller (BET) classification. This isotherm-type is characteristic of systems containing high-soluble solids content (Brunauer, Deming, Deming, & Teller, 1940; Mathlouthi & Rogé, 2003). The isotherm curves showed two well-separated zones: At RH between 50% and 80% (Zone I) the products showed negligible changes in their moisture amount, whereas at RH values higher than 80% (Zone II), the samples began to adsorb larger amounts of water from the atmosphere until dissolution (Burnett, Thielmann, & Booth, 2004). This solid–liquid phase change that occurs at a characteristic relative humidity (i.e., the critical relative humidity, RH_0) is known as deliquescence. The RH_0 is unique to each crystalline solid system and has been used as a very important indicator of storage stability (Lipasek, Ortiz, Taylor, & Mauer, 2012). RH_0 values of the products were measured from the dynamic dewpoint isotherms by the second derivative method (Scholl & Schmidt, 2014). The products without glucose (F1, recrystallized sucrose) showed higher RH_0 values than the cocrystallized products with glucose (F2 and F3; Table 2). This behavior was attributed to the glucose presence which increased the amount of hydroxyl groups available to interact with the hydrogen present in the water molecules, leading to decrease the deliquescence point of the cocrystallized products. Salameh, Mauer, and Taylor (2006) also observed deliquescence lowering when working with mixtures of several model food ingredients (e.g., sucrose–glucose, sucrose–citric acid, sucrose–glucose-citric acid, and among others). These authors found that physical mixtures of glucose and sucrose (1:1) showed deliquescence at significantly lower RH_0 values (79%) compared with the individual ingredients, glucose (91%) and sucrose (89%), regardless of the concentration of each ingredient in the blend.

3.4 | X-ray diffraction studies

X-ray diffraction patterns of the different formulations evaluated (F1, F2, and F3) are shown in Figure 4. The diffractograms of all samples were matched with the X-ray pattern of crystalline sucrose, showing signals at 2θ (°) = 11.7, 12.7, 18.8, 19.6, 24.8, 25.2, and 38.3 (JCPDS, 1999). In addition, the agglomerates containing glucose showed new peaks in the 2θ region between 11° and 14°, which were attributed to the glucose presence (Figure 4).

3.5 | Melting endotherms by DSC

Figure 5 shows the DSC thermograms of the products formulated as detailed in Table 1.

The values of melting enthalpies (ΔH) and onset (T_{on}), peak (T_p) and endset temperatures (T_{end}) are shown in Table 3. The sample F1, that is, only recrystallized sucrose, showed an endothermic peak around 190 °C characteristic of sucrose melting (Bhandari & Hartel, 2002; Sardar & Singhal, 2013). The cocrystallized products with glucose (F2 and F3) showed a shift to lower values for the T_{on} and T_p temperatures of the melting endotherm, but only a small change in T_{end} temperatures (Table 3). Therefore, broader melting peaks assigned to sucrose were observed (Figure 5). Moreover, the ΔH values associated with the sucrose melting peaks strongly decreased

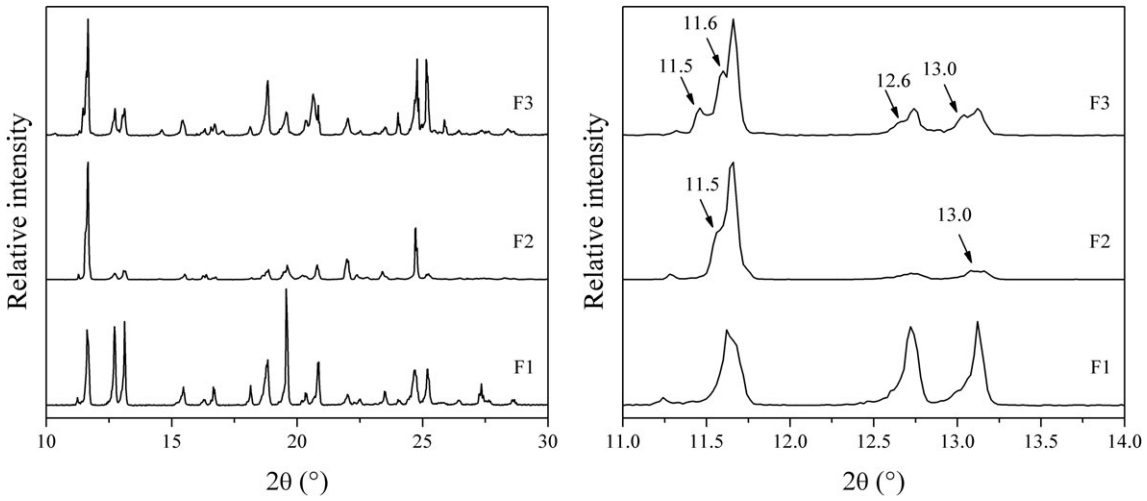

FIGURE 4 X-ray diffraction patterns of the recrystallized sucrose (F1) and cocrystallized products with 10 g (F2) and 15 g (F3) of glucose added per 100 g of sucrose. Insert shows the 2θ region between 11° and 14°

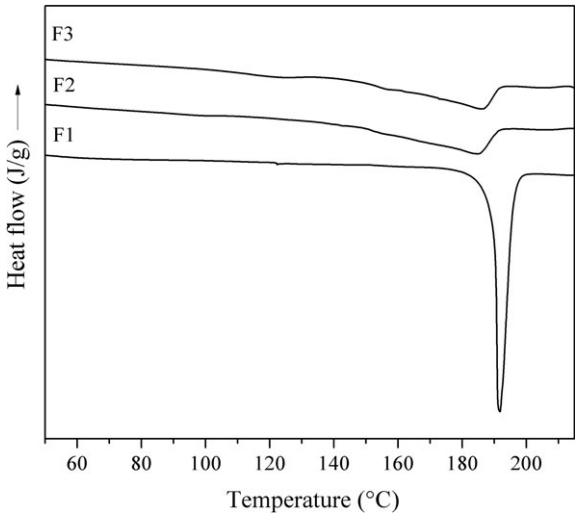

FIGURE 5 DSC thermograms of recrystallized sucrose (F1) and cocrystallized products with 10 g (F2) and 15 g (F3) of glucose added per 100 g of sucrose

as the glucose concentration was increased (Table 3). Bhandari and Hartel (2002) previously reported a similar effect of glucose or fructose addition on the broadening and reducing melting enthalpy of the sucrose melting peak which was attributed to the inhibition of sucrose crystallization by effect of the presence of these monosaccharides.

3.6 | Physical mixtures of cocrystallized products with sodium bicarbonate

Physical blends of sodium bicarbonate with glucose (M0) or with cocrystallized products (M1 and M2) were prepared as detailed in Section 2.9 and analyzed by DSC. The values of melting enthalpies (ΔH) and onset (T_{on}), peak (T_p) and endset (T_{end}) temperatures are shown in Table 4. The systems containing sodium bicarbonate and raw glucose (M0) showed a thermal event with a melting range from 125 to 183 °C. After 12 days of storage, this event was found shifted to lower temperatures and a reduction of around 37% in the ΔH value was observed (Table 4). It is well-known that several physical and chemical interactions can take place in powdered mixtures, prompting the degradation of active compounds (Byrn, Xu, & Newman, 2001). The main factors influencing these reactions include the temperature, the moisture content, the ingredients and the physical state of the mixtures (amorphous vs. crystalline).

The stored systems containing sodium bicarbonate and cocrystallized glucose with sucrose (M1 and M2) did not show significant changes in their values of ΔH, T_{on}, T_p, and T_{end}, with respect to their initial values (Table 4). These results suggested that the cocrystallization process constituted a useful means to decrease the solid-state reactivity between glucose and sodium bicarbonate. Thus, the total amount of energy (ΔH) needed to melt the cocrystallized products was maintained almost unaltered after storage.

4 | CONCLUSION

Sucrose/glucose agglomerates were successfully obtained by cocrystallization. The products showed desirable handling characteristics such as low water content and water activity, low hygroscopicity, and good flowability. Glucose addition led to higher water contents in the cocrystallized products. Moreover, DSC results showed that ΔH values associated with the sucrose melting peaks decreased as the glucose concentration was increased. These results could suggest that glucose retarded the crystallization of sucrose. X-ray diffraction analysis of the agglomerates with and without glucose showed that important changes in the crystal structure of the sugar did not take place. The sucrose/glucose agglomerates were incorporated to blends containing sodium bicarbonate, like oral rehydration salts, and evaluated before and after storage at 75% RH and 20 °C. Thermal analysis proved that cocrystallization helped decrease reactivity between glucose and bicarbonate, because no changes in enthalpy values of agglomerates were found even at storage conditions. The results

TABLE 3 Values of melting enthalpies (ΔH) and onset (T_{on}), peak (T_p) and endset temperatures (T_{end}) extracted from DSC thermograms of the different formulations

Formulation	T_{on} (°C)	T_p (°C)	T_{end} (°C)	ΔH (J/g)
F1	190.15 ± 0.15	191.78 ± 0.02	203.34 ± 0.47	127.75 ± 1.62
F2	154.09 ± 2.11	185.70 ± 3.07	198.57 ± 3.80	106.50 ± 2.26
F3	153.24 ± 2.88	183.58 ± 2.37	197.60 ± 1.77	73.48 ± 0.87

F1: Recrystallized sucrose; F2 and F3: cocrystallized products with 10 g (F2) and 15 g (F3) of glucose added per 100 g of sucrose.

TABLE 4 Values of melting enthalpies (ΔH) and onset (T_{on}), peak (T_p) and endset (T_{end}) temperatures extracted from DSC thermograms for physical blends of sodium bicarbonate with raw glucose (M0) or with cocrystallized products (M1 and M2) before and after 12 days stored at 75% RH and 20 °C

System	Storage	T_{on} (°C)	T_p (°C)	T_{end} (°C)	ΔH (J/g)
M0	Before	124.79 ± 0.84	137.05 ± 0.31	183.13 ± 0.78	251.50 ± 1.55
	After	109.24 ± 1.19	132.37 ± 3.39	156.04 ± 1.40	158.45 ± 4.17
M1	Before	161.32 ± 6.12	179.60 ± 7.97	194.23 ± 2.04	75.91 ± 4.98
	After	154.72 ± 0.28	176.21 ± 2.25	193.34 ± 0.78	69.84 ± 3.27
M2	Before	161.61 ± 0.01	180.35 ± 1.87	194.79 ± 5.65	74.24 ± 5.76
	After	155.29 ± 3.27	176.23 ± 2.28	192.79 ± 0.94	65.11 ± 5.14

showed that the cocrystallization process is an effective way to produce sucrose/glucose agglomerates with improved physicochemical stability.

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