Agrointek Volume 20 No 3 2026: 628-637

p-ISSN: 1907-8056
e-ISSN: 2527-5410

AGROINTEK
Jurnal Teknologi Industri Pertanian

journal homepage: journal.trunojoyo.ac.id/agrointek

Thermal properties of crystalline coconut sugar using differential scanning calorimetry for rotary dryer process design

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Article history
Received:
2 October 2025
Revised:
7 July 2026
Accepted:
8 July 2026

Keyword
Crystalline coconut sugar;
Differential scanning calorimetry;
Drying process;
Rotary dryer;
Thermal analysis

ABSTRACT

Crystalline coconut sugar is widely used in the food industry due to its natural sweetness and nutritional content. However, its amorphous nature, high hygroscopicity, and tendency to undergo thermal degradation pose major challenges in drying and storage processes. Understanding the thermal behaviour of crystalline coconut sugar is crucial for effective drying and the design of suitable equipment. This study employed Differential Scanning Calorimetry (DSC) to characterise the thermal transitions of crystalline coconut sugar. Thermal characterisation was performed using DSC, 30 – 300°C at 10 °C/min to determine the glass transition temperature (Tg), melting point (Tm), and enthalpy change (ΔH). The results indicated that the Tg of crystalline coconut sugar represents the lower temperature limit that should be avoided to prevent stickiness and caking during the drying process, ranging from 90 to 100°C, whereas Tm represents the upper temperature limit that must be considered to avoid caramelisation and degradation, ranging from 165 to 200°C. These findings provide a scientific basis for determining the optimal temperature range for rotary dryer operation, which lies between 60 and 80°C. The Gordon–Taylor model demonstrated high agreement between predicted and observed Tg values. The DSC thermal analysis in this study provides a scientific foundation for selecting process parameters such as temperature, residence time, and air flow rate. This study highlights the importance of DSC characterisation as a crucial step in linking material properties to engineering design, thereby ensuring product quality and process efficiency in the drying of crystalline coconut sugar.

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DOI 10.21107/agrointek.v20i3.31967

INTRODUCTION

Crystalline coconut sugar is produced from the sap or nectar of various palm trees, such as coconut, areca, and nipa. In Asia, this sugar has long been used as a traditional sweetener. Due to its natural characteristics, minimal processing, and health benefits, crystalline coconut sugar has been gaining increasing attention in the global market. Its glycemic index is lower compared to cane sugar. In addition, crystalline coconut sugar also contains many essential vitamins and minerals (Sarkar et al. 2023).

The sap collected from the coconut tree is subsequently filtered and then heated or cooked until it thickens. Once it reaches a consistent thickened state (endpoint), it is cooled and ground to form fine granules or crystals. The resulting crystalline coconut sugar is then sieved to obtain a uniform particle size. The moisture content of crystalline coconut sugar is typically around 7% and must be reduced to a maximum of 2% for export standards (Sahat 2017) and 3% according to Badan Standardisasi Nasional. 2021. The sequence of crystalline coconut sugar production processes can be seen in Figure 1.

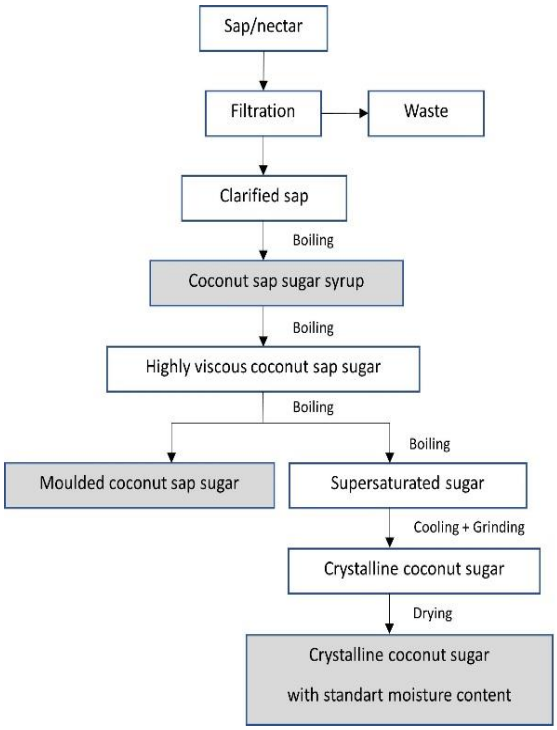


Figure 1 Crystalline coconut sugar production flowchart: modified from Saputro et al. (2019)

Moisture content has a significant impact on product quality, including texture, taste, and shelf life (Monteiro 2024). Excessive moisture results in crystalline coconut sugar that tends to be sticky, leading to clumping and a less crispy texture. Higher moisture content also makes the sugar taste less sweet, as water reduces the concentration of flavour. High moisture levels during storage can trigger fermentation or chemical changes that produce a sour and stale taste. Moreover, excessive moisture creates favourable conditions for the growth of microorganisms such as moulds and bacteria, which accelerate spoilage and make the product unsafe for consumption (Lee and Robertson 2022). Drying is the process of removing moisture from a material using various techniques, with the main objective of maintaining quality and extending shelf life by reducing moisture content to the desired level (Ritonga et al. 2025). Efforts to address challenges in the drying process of crystalline coconut sugar have been carried out through the development and research of dryers to achieve optimal drying conditions.

Crystalline coconut sugar is highly sensitive to temperature changes, particularly during the drying process. Crystalline coconut sugar is mostly amorphous (Nurhadi et al. 2020), and during drying, it may have non-uniform moisture distribution due to its irregular physical structure, which can result in some parts retaining more water than others. Amorphous crystalline coconut sugar tends to be unstable at high temperatures, which can lead to caramelisation and thermal degradation. Therefore, the drying process must take into account the glass transition temperature (Tg). The glass transition temperature (Tg) is the temperature at which an amorphous material, such as crystalline coconut sugar, changes from a solid state to a soft and sticky condition (Yermia et al. 2025).

The Tg of crystalline coconut sugar is crucial because it is directly related to the physical stability of the material during drying. For product safety and quality, it is essential to estimate the Tg of sugar mixtures accurately. Several models can be used to mathematically describe the Tg of component mixtures. The Gordon–Taylor equation (Equation 1) is commonly used for prediction (Gordon and Taylor 1952) based on the free volume theory and the additive properties of fundamental thermophysical parameters (Schugmann and Foerst 2022). The glass transition temperature Tg_1 of dry sugar is obtained from DSC curve estimation, and Tg_2 from water;

w_1 is the weight fraction of dry sugar, w_2 is the weight fraction of water, while k is the interaction constant, for sugar–water mixtures often using a value of k ≈ 5.2 (Wang et al. 2021, Drake et al. 2018, Chen et al. 2000)

$$Tg_{mix} = \frac{w_1 T_{g1} + k w_2 T_{g2}}{w_1 + k w_2} \quad (1)$$

A drying temperature above the Tg value makes the sugar particles sticky, agglomerate, and adhere to the walls of the dryer. The design of a crystalline coconut sugar dryer must ensure that the drying temperature remains below the Tg to maintain product quality. The machine design should include a temperature control system to prevent Tg from being reached during the drying process.

A rotary dryer is a drum-shaped drying machine that rotates, with a heat source that can come from electricity, gas, or biomass combustion (Wang et al. 2015). Drying in a rotary dryer occurs when the material comes into direct contact with hot air from the combustion chamber, which is driven by a blower at a specific air velocity into the rotating drum equipped with lifters, causing the crystalline coconut sugar to be continuously agitated during drying. This process allows for efficient heat transfer and ensures uniform drying across all particles (Delele et al. 2015).

These conditions are difficult to achieve with static drying methods, such as oven drying. The design and operation of the drying process are fundamentally aimed at increasing efficiency, reducing energy consumption and costs, enhancing processing capacity, and simplifying processing procedures (Nelwan et al. 2025). In convective drying of agricultural products like crystalline coconut sugar, various strategies have been developed to achieve these goals, including the regulation of temperature, humidity, and airflow rate. However, the implementation of these strategies is constrained by the need to maintain product quality and minimize potential quality degradation. The objective of this study is to analyse the thermal characteristics of crystalline coconut sugar for the development of an effective drying process and appropriate equipment design.

METHOD

Materials and equipment

The main material used in this study was 100 g of commercial crystalline coconut sugar obtained from a local producer. The crystalline coconut sugar was sieved through 14 mesh screens to obtain uniform particle sizes. The moisture content of the samples varied 0.77%, 2.62% and 4.03% w.b. were randomly obtained from a local producer. The samples were stored in airtight containers at room temperature, 28 ± 2 °C and RH 60 ± 5 until testing. The equipment consisted of a DSC – 60 Plus (Shimadzu), a computer equipped with LabSolutions TA software, sample pans, a sealer, and an analytical balance.

Procedure of thermal analysis by DSC

Thermal characterisation was carried out using differential scanning calorimetry (DSC-60 Plus, Shimadzu Corporation, Kyoto, Japan), equipped with LabSolutions TA software. Crystalline coconut sugar was sealed in closed aluminium pans (aluminium seal) to prevent mass loss during heating. The analysis was performed at atmospheric pressure (≈ 1 atm or 101.3 kPa) under an air atmosphere with a gas flow rate of 10 mL/min. The temperature program started at 30 °C and was increased at a rate of 10 °C/min up to a maximum temperature of 300 °C without a hold time (hold time = 0 min). During heating, enthalpy changes of the sample were recorded to identify thermal transition phenomena, including glass transition (Tg), melting (Tm), and enthalpy changes (ΔH).

Data analysis

The DSC measurement data were analysed to obtain the main thermal parameters of crystalline coconut sugar. The glass transition temperature (Tg) was determined from the change in specific heat capacity (Cp), which appeared as a baseline deviation in the DSC curve, with the reported values including Tg onset and Tg midpoint. The melting temperature (Tm) was identified from the endothermic peak in the curve, while the melting enthalpy (ΔHm) was calculated from the area under the corresponding endothermic peak.

RESULT AND DISCUSSION

Thermal properties of crystalline coconut sugar

Crystalline coconut sugar with a moisture content of 0.77% (w.b.), as shown in Figure 1 and Table 1, exhibited several thermal transition points that describe the physical characteristics of the material. The first transition, occurring at 136 – 182 °C, was observed as a baseline shift with a peak at 167 °C, which can be interpreted as the glass transition (Tg). The enthalpy required for this phase change (ΔH) was relatively high at 74.03 J/g. This event indicates the transformation of crystalline coconut sugar from an amorphous phase into a more molecularly mobile state. The second transition, detected at 201–205 °C, appeared as an endothermic peak with a maximum at 203 °C.

This peak corresponds to the onset of partial melting, which is likely attributed to the sugar fraction with a semi-crystalline structure and lower thermal stability (ΔH = 44.57 J/g). The third transition, occurring at 207–209 °C, was observed as a second endothermic peak with a maximum at 208 °C, which indicates the main melting point (Tm) of crystalline coconut sugar, with an enthalpy value of 14.92 J/g. The relatively small ΔH suggests that most of the amorphous structure had already undergone prior changes, resulting in lower energy requirements for the main melting process. These results indicate that crystalline coconut sugar with a moisture content of 0.77% (w.b.) exhibits good thermal stability up to 200 °C, before undergoing complete melting above 208 °C.

Crystalline coconut sugar with a moisture content of 2.62% (w.b.), based on DSC analysis, also exhibited three major thermal transitions during heating, as shown in Figure 2 and Table 2. The first transition occurred within the temperature range of 151.88–172.04 °C, with a peak at 161.70 °C and an enthalpy of 20.30 J/g, indicating a mild exothermic event. This change is generally associated with the release of bound water or partial recrystallisation of some sample components. In the DSC curve (Figure 2), three exothermic events (sharp downward peaks) were observed, but no glass transition was detected, which is usually represented by a subtle baseline shift (small endothermic event without sharp peaks). The DSC trace from 30 °C to 150 °C remained flat without the characteristic baseline deviation for Tg. The presence of sucrose, glucose, fructose, and other compounds can form an amorphous mixture with a low Tg (approximately 60–90 °C) (Saavedra-Leos et al. 2012).

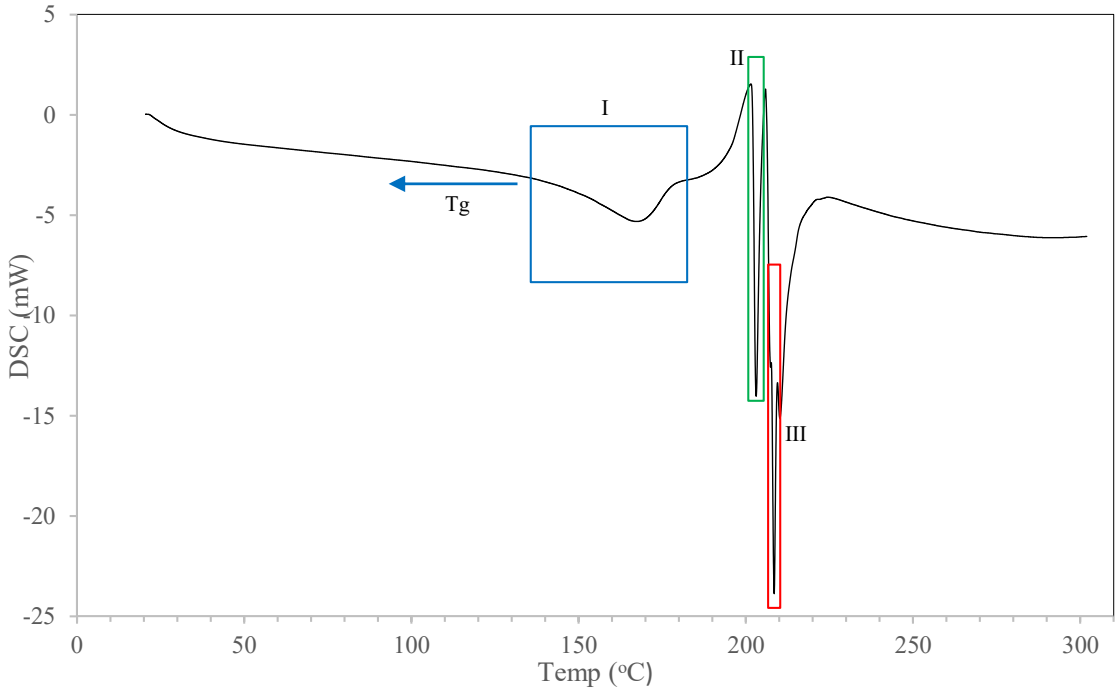


Figure 2 DSC thermogram of crystalline coconut sugar 0.77% w.b. moisture content

Table 1 DSC analysis results of crystalline coconut sugar at 0.77% (w.b.) moisture content

DSC condition	Transition I	Transition II	Transition III
Start	136.52 °C	201.47 °C	207.40 °C
End	182.68 °C	205.76 °C	209.38 °C
Peak	167.33 °C	203.10 °C	208.49 °C
Onset	149.72 °C	202.06 °C	207.82 °C
Endset	177.58 °C	204.46 °C	209.05 °C
Heat	- 280.57 mJ	-168.92 mJ	- 56.55 mJ
	- 74.03 J/g	- 44.57 J/g	- 14.92 J/g
Height	- 2.10 mW	- 15.40 mW	- 10.78 mW
	- 0.55 mW/mg	- 4.06 mW/mg	- 2.84 mW/mg

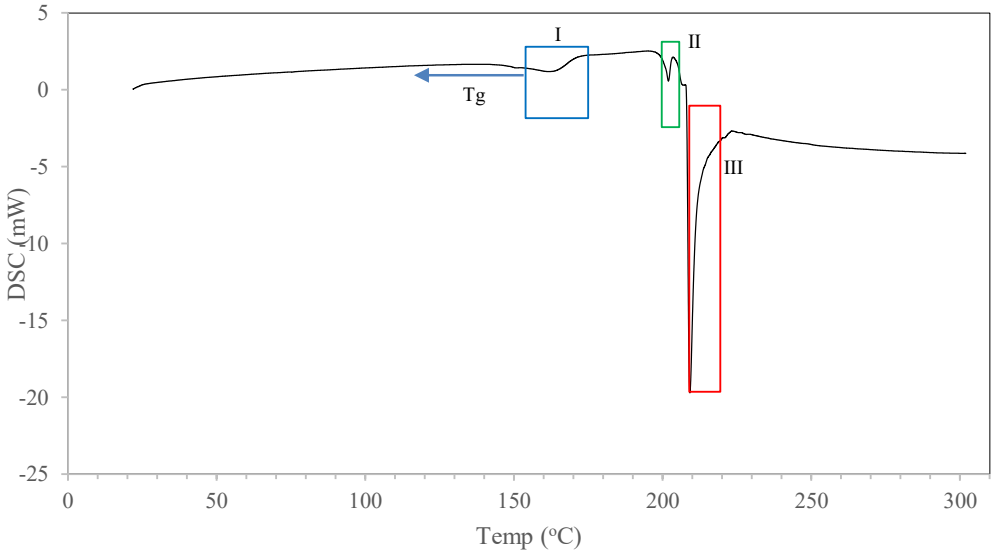


Figure 3 DSC thermogram of crystalline coconut sugar 2.62 % w.b. moisture content

Tg is often masked by water evaporation or early recrystallisation, making it difficult to detect. The Tg of crystalline coconut sugar at this moisture content is likely within the 60–90 °C range, but does not appear clearly due to overlap with other phenomena. The second transition appeared in the range of 197.56–203.54 °C, with a peak at 201.97 °C and a relatively small enthalpy (9.49 J/g), which may be interpreted as a polymorphic change or the onset of slight degradation of organic components such as sucrose or other sugars. The third and most significant transition occurred between 207.77 and 218.95 °C, with a peak at 209.18 °C and a very high enthalpy of 188.67 J/g, representing a major exothermic event attributed to thermal decomposition, caramelisation, or Maillard reactions that release substantial energy. The high enthalpy value of this transition indicates that temperatures above 200 °C are critical for the thermal stability of the material, as they may lead to permanent changes in the chemical structure and functional properties of the sample. The thermal transitions of crystalline coconut sugar as determined by DSC analysis are strongly influenced by moisture content (Jothi et al. 2018) and the composition of reducing sugars (Maidannyk et al. 2022). The first thermal transition of crystalline coconut sugar with a moisture content of 4.03% (w.b.) occurred within the temperature range of 132.24–165.39 °C, with an endothermic peak at 151.56 °C and a heat absorption of 22.15 J/g. This phenomenon indicates the initial melting of simple sugar fractions or amorphous components present in crystalline coconut sugar, reflecting the presence of sugar mixtures with different thermal stabilities.

The DSC curve of crystalline coconut sugar at 4.03% (w.b.) moisture content (Figure 3) and its analysis (Table 3) showed no baseline shift that

typically indicates Tg, suggesting that Tg may lie below the range of the first thermal transition. The second transition appeared between 185.43 and 196.63 °C, with a peak at 194.76 °C and a heat absorption of 9.47 J/g, representing the main melting of sucrose. The onset temperature of 192.55 °C and the endset of 195.93 °C are consistent with the literature on the melting point of pure sucrose, reported at 185–190 °C (Toda et al. 2018), although slightly shifted due to the presence of water and minor sugars in the matrix. The third transition occurred between 197.75 and 223.13 °C, with a peak at 199.92 °C and a very high heat absorption of 213.63 J/g, indicating the onset of decomposition or caramelisation. This event is accompanied by significant energy release, reflecting the formation of compounds from non-enzymatic browning reactions that contribute to the characteristic colour and flavour of crystalline coconut sugar.

Correlation between moisture content and glass transition temperature (Tg)

Tg of crystalline coconut sugar is strongly influenced by its moisture content (Linnenkugel et al. 2022). Water acts as a plasticiser, a small molecule that enhances the mobility of sugar molecular chains. As moisture content increases, water molecules penetrate the sugar matrix and disrupt part of the hydrogen bonding between sugar molecules and increasing molecular mobility.

Table 2 DSC analysis results of crystalline coconut sugar at 2.62% (w.b.) moisture content

DSC condition	Transition I	Transition II	Transition III
Start	151.88 °C	197.56°C	207.77 °C
End	172.04 °C	203.54 °C	218.95 °C
Peak	161.70 °C	201.97 °C	209.18 °C
Onset	153.56 °C	200.56 °C	208.14 °C
Endset	169.99 °C	202.85 °C	211.35 °C
Heat	- 42.43 mJ	-19.84 mJ	- 394.31 mJ
	- 20.30 J/g	- 9.49 J/g	- 188.67 J/g
Height	- 0.60 mW	- 1.63 mW	- 19.56 mW
	- 0.29 mW/mg	- 0.78 mW/mg	- 9.36 mW/mg

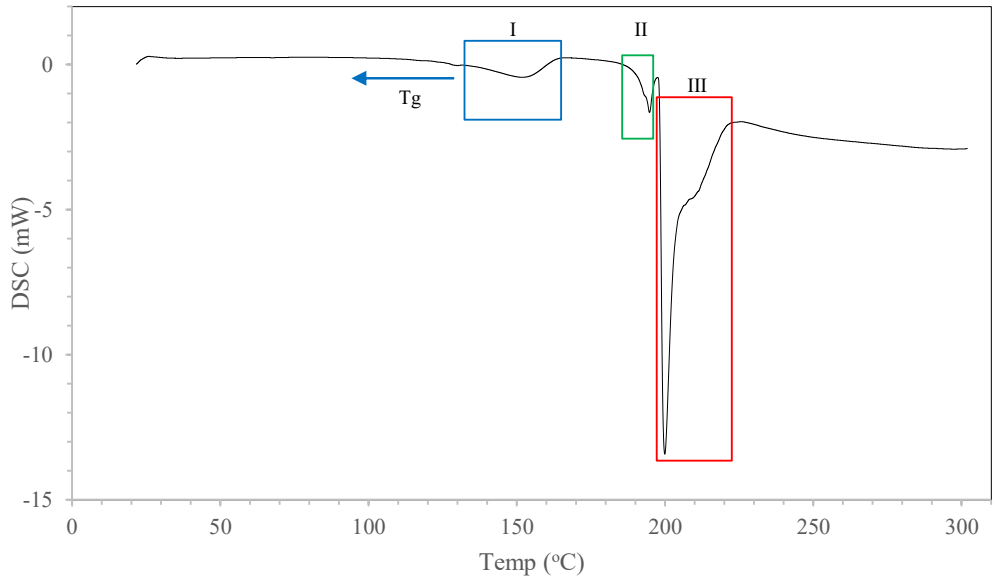


Figure 4 DSC thermogram of crystalline coconut sugar 4.03 % w.b. moisture content

Table 3 DSC analysis results of crystalline coconut sugar at 4.03% (w.b.) moisture content

DSC condition	Transition I	Transition II	Transition III
Start	132.24 °C	185.43 °C	197.75 °C
End	165.39 °C	196.93 °C	223.13 °C
Peak	151.56 °C	194.76 °C	199.92 °C
Onset	136.23 °C	195.93 °C	198.18 °C
Endset	163.00 °C	202.85 °C	203.87 °C
Heat	- 60.04 mJ	-25.67 mJ	- 578.94 mJ
	- 22.15 J/g	- 9.47 J/g	- 213.63 J/g
Height	- 0.56 mW	- 1.25 mW	- 12.83 mW
	- 0.21 mW/mg	- 0.46 mW/mg	- 4.73 mW/mg

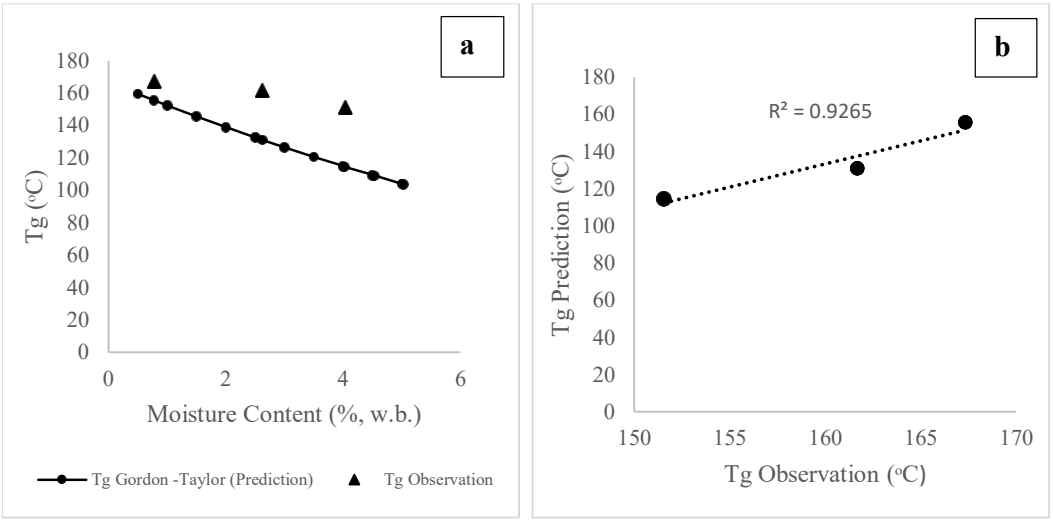


Figure 5 Tg value based on moisture content (a); Observed vs Predicted of Tg of crystalline coconut sugar (b)

Consequently, the glass transition temperature (Tg) shifts to lower values. Conversely, at low moisture content, stronger intermolecular interactions within the sugar matrix result in a more rigid structure, leading to higher Tg values (Takeda et al. 2023). Experimental analysis using DSC to obtain Tg data at various moisture contents can be evaluated with predictive models such as the Gordon–Taylor equation to estimate the Tg of mixtures, as illustrated in Figure 4. Figure 4a shows that the solid circles represent Tg values predicted by the Gordon–Taylor equation over the moisture content range, whereas triangles indicate experimentally measured Tg values obtained by DSC in the peak temperature at transition I. Although the Gordon–Taylor model slightly underestimated the experimental Tg values, both datasets exhibited the same decreasing trend with increasing moisture content.

The Gordon–Taylor equation is a widely applied semi-empirical model used to predict the glass transition temperature (Tg) of amorphous mixtures, such as sugar–water systems. The observed decrease in Tg of crystalline coconut sugar with increasing moisture content underscores the role of water as a plasticiser, thereby reducing the physical stability of amorphous materials (Schugmann and Foerst 2022). Figure 4b shows strong agreement between the experimental and Gordon–Taylor-predicted Tg values (R^2 = 0.9265). The predicted values were consistently lower than the experimental measurements, indicating a slight underestimation by the Gordon–Taylor model.

This phenomenon is crucial to understand in the context of drying and storage, as the decrease in Tg caused by increasing moisture content can elevate the risks of stickiness, agglomeration, and reduced product stability. Consistent with Kawai et al. (2014), who reported that moisture content plays a critical role in maintaining the stability of sugar-based products, as sugar–water systems exhibit low Tg due to plasticization effects.

Table 4 Drying conditions based on DSC analysis

Parameters	Moisture content (%, w.b)			
	0.77	2.62	4.07	7.0
Tg crystalline coconut sugar, °C	167.3	161.7	151.6	110
Tg (Tg mix), °C	155.7	131.2	114.6	84
Inlet temperature rotary dryer, °C	< 130	< 120	< 100	< 80
Material temperature, °C	< 100	< 100	< 90	< 73

Therefore, moisture control is a critical factor in preserving the quality of amorphous products such as crystalline coconut sugar. Crystalline coconut sugar is a mixture of various sugars and contains minor sugars beyond pure sucrose (sucrose 80–93%; reducing sugars ~3%) Badan Standardisasi Nasional. 2021. Water and small-molecule sugars (glucose and fructose) contribute to lowering Tg, while sucrose maintains a higher Tg. Thus, the Gordon–Taylor model can be applied to predict Tg; however, it should be noted that systematic deviations may occur. Experimental DSC data are therefore necessary for validation, whereas the Couchman–Karasz model tends to systematically underestimate the Tg of sugar mixtures (Schugmann and Foerst 2022).

Rotary dryer drying conditions

The drying of crystalline coconut sugar in a rotary dryer should be designed based on thermal data (DSC) to prevent melting, non-enzymatic browning, and undesirable crystallographic changes that impair product quality. The chemical and thermal characteristics of crystalline coconut sugar indicate the presence of sugar components with glass transition and melting behaviours distinct from pure sucrose. The DSC results indicated that lowering the moisture content increased Tg, suggesting greater physical stability. However, excessively high drying temperatures may accelerate thermal degradation and undesirable browning of sugar-rich materials. Therefore, the operating temperature of 60–80°C represents a balance between achieving sufficient moisture removal to increase Tg while minimizing quality deterioration. This temperature is also supported by previous studies reporting efficient moisture evaporation within the same temperature range (Tiarasari et al. 2023). The use of DSC to identify Tg and melting peaks enables process design with a safety margin, such as operating temperatures ≤ Tg + 10–20 °C when the product is partially amorphous, to minimizing phase transitions during drying (Saraiva et al. 2023).

Parameters for drying crystalline coconut sugar using a rotary dryer include the drum rotation speed (rpm) and the lifter configuration, which should ensure effective particle–vapour exchange without particle adherence to the drum wall. The airflow rate is controlled to maintain a low inlet air relative humidity, maximising the evaporation driving force. Temperature and humidity profile modelling for industrial-scale rotary dryer design should be based on DSC data to ensure that particles never experience temperatures exceeding the thermal limits determined by DSC. Table 4 presents the drying conditions for crystalline coconut sugar based on DSC analysis, assuming an initial moisture content of approximately 7% and a target final moisture content ≤ 2%.

CONCLUSION

Thermal profile of crystalline coconut sugar was successfully mapped using DSC, including the glass transition temperature (Tg), melting point (Tm), and enthalpy changes (ΔH), providing a basis for understanding the thermal stability of crystalline coconut sugar. DSC data serve as a reference for designing rotary dryers for crystalline coconut sugar, including inlet air temperature, airflow rate, and particle residence time, ensuring effective moisture removal without inducing browning or crystallographic damage. A temperature range of 60 – 80°C is recommended as the optimal compromise between drying efficiency and product stability.

ACKNOWLEDGMENTS

We would like to express our sincere gratitude to Universitas Gadjah Mada for their support and valuable contributions to this research. The authors gratefully acknowledge the Indonesian Education Scholarship, Center for Higher Education Funding and Assessment, and

the Indonesian Endowment Fund for Education for providing facilities and funding for this study.

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