



Differences in the thermal behavior of beet and cane sucrose sources

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ABSTRACT

Sucrose is a major worldwide commodity, produced mainly from sugarbeet and sugarcane. Despite the nearly identical chemical composition of these sugar sources, some differences in aroma and performance in products have been reported in the literature. However, little research exploring thermal behavior differences was found. By employing thermal analysis methods, this research reveals significant thermal behavior differences both between and within beet and cane sugars. Beet samples exhibited only one large endothermic DSC peak ($T_{\text{monset}} = 188.45 \pm 0.43$); whereas twenty-seven of the thirty-one cane samples exhibited two endothermic DSC peaks, one small peak ($T_{\text{monset}} = 153.62 \pm 6.04$) preceded by one large peak ($T_{\text{monset}} = 187.33 \pm 1.72$). However, the four remaining cane samples, containing either high ash content or processing added impurities, exhibited only one large endothermic DSC peak. Understanding the thermal behavior differences between and within sucrose sources is of substantial importance to the food industry, especially in applications involving heat, such as baking, extrusion cooking, pasteurization, and drying.

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1. Introduction

Sucrose, commonly termed sugar or table sugar, is an important commodity worldwide and is produced using mainly sugarbeet (*Beta vulgaris*) and sugarcane (*Saccharum officinarum*) plant sources. Though both white refined beet and cane sugars are commonly reported to contain greater than 99.8% sucrose (Dowling, 1990; Schiweck et al., 1994; Morgan, 1999; Asadi, 2007; Godshall, 2013), differences in their instrumental aroma profile (Acree et al., 1976; Parliament et al., 1977; Monte and Maga, 1982; Marsili et al., 1994; Pihlsgard, 1997; Magne et al., 1998), sensory properties (Urbanus et al., 2014a,b), and product performance (Urbanus et al., 2014a,b) have been reported in the literature.

Some of the differences between beet and cane sugars are due to the plant materials themselves, while others are due to processing differences. For example, due to the difference in their CO₂ fixation pathways, sugarbeet, a C3 plant, and sugarcane, a C4 plant, exhibit a difference in their carbon isotope ratio (¹³C–¹²C), where the ratio is approximately 25% in beet sugar and 11% in cane sugar (Bubník

et al., 1995). The selective rejection of ¹³C is greater in the C3 pathway than in the C4 pathway (Fernelius, 1984). Two other differential plant material markers are raffinose and theanderose. Though raffinose is present in both sugar sources, levels are higher in beet sugar compared to cane sugar, as quantified using chromatography (Vaccari and Mantovani, 1995; Morel du Boil, 1997; Eggleston, 2004). Theanderose is present only in cane sugar and is thought to be a natural constituent of the sugarcane plant (Morel du Boil, 1996). Both raffinose and theanderose affect sugar crystal growth and morphology (Liang et al., 1989; Morel du Boil, 1992).

In regards to processing differences, sugarcane usually undergoes a two-step refining process, with raw sugar produced near plantations and transported to refineries for further purification; whereas, sugarbeet undergoes a one-step refining process, which includes the addition of SO₂ (Asadi, 2007; Godshall, 2013). In the United States, sulfitation is routinely used in beet sugar processing, but not in cane. A number of studies conducted by the Sugar Processing Research Institute, Inc. (SPRI, New Orleans, LA) have focused on the identification of and comparison between beet and cane sugars in regards to a number of physical and chemical properties, such as moisture content, conductivity ash, pH, odor, color, invert sugar, and polysaccharide content (Godshall, 1986, 2013 and Godshall et al., 1994). According to Godshall (2013)

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Nomenclature

ΔC_p	Change in heat capacity (J/g °C)
ΔH	enthalpy of melting (J/g)
DSC	differential scanning calorimetry
HPLC	high performance liquid chromatography
TCD	total color difference
TGA	thermal gravimetric analysis
T_g	glass transition temperature (°C)
$T_{m\text{onset}}$	onset melting temperature (°C)
$T_{m\text{peak}}$	peak melting temperature (°C)

moisture content (0.01–0.02%) and conductivity ash (0.01–0.03%) values are similar for both sugar sources, whereas, the pH range was somewhat wider for beet sugar (6.5–8.0) compared to cane sugar (6.2–6.7).

The crystallization and melting behavior of sucrose has been under investigation for a number of years. However, one aspect of the melting behavior of sucrose that needed further study was the wide variation in melting temperature reported in the literature for sucrose, as well as other sugars, such as glucose and fructose. Though a consistent, uniform melting temperature is expected for a crystalline material, the melting temperature for sucrose has been found to vary widely, as reported in earlier, as well as more recent literature. Examples of early studies include work by [Shah and Chakradeo \(1936\)](#) and [Powers \(1956, 1958\)](#). [Shah and Chakradeo \(1936\)](#) reported a sucrose melting point of 182 °C, but also gave a table of melting point values from the literature ranging from 160 to 189 °C. These authors mentioned a number of possible factors responsible for the reported melting point variation, including “slow heating and consequent partial decomposition into glucose and fructose or even partial hydrolysis into glucose and fructose due to the presence of traces of water,” differences in the melting point determination methods, and sucrose purity. Based on their experiments, [Shah and Chakradeo \(1936\)](#) concluded that the sucrose melting point was dependent upon the purity of the sample alone. [Powers \(1956, 1958\)](#) reported the presence of water, in the form of mother syrup (or liquor) inclusions, inside the sucrose crystal structure and in his 1958 article implicated these inclusions as an explanation for the widely varying melting point of sucrose reported in the literature, given in the article as 160–186 °C.

Over the years, researchers have offered a number of possible explanations for the wide melting temperature range of sucrose, as well as other sugars, reported in the literature, including impurities ([Hirschmüller, 1953](#); [Kamoda, 1960](#); [Beckett et al., 2006](#)), polymorphism ([Kishihara et al., 2001](#); [Lee and Lin, 2007](#); [Lee and Chang, 2009](#)), superheating ([Tammann, 1910](#); [Hellmuth and Wunderlich, 1965](#); [Wunderlich, 1990b](#); [Magoñ et al., 2014](#)), liquefaction (used to explain the large variation observed in the melting parameters for isomerizable materials, such as fructose, glucose, and galactose) ([Tombari et al., 2007](#)), and thermal decomposition and/or mutarotation ([Hurttta et al., 2004b](#); [Lappalainen et al., 2006](#)) in addition to melting. However, according to [Lee et al. \(2011a\)](#), these explanations do not completely account for the observed variation. Rather, [Lee et al. \(2011a,b\)](#) demonstrated that analytical grade Sigma cane sucrose melting parameters tended to increase substantially with increasing heating rate (more than expected from thermal lag alone), leading to their conclusion that the initial loss of crystalline structure in sucrose is associated with the kinetic process of thermal decomposition, which, in the case of sugars, is often termed caramelization. The research by [Lee et al. \(2011a,b\)](#), connecting heating rate dependency and thermal decomposition in

sucrose, generated significant discussion in the literature ([Roos et al., 2012, 2013](#); [Schmidt et al., 2012](#)).

Another unique observation, related to the wide range of sucrose melting temperatures, is the appearance of a small endothermic event just prior to the large endothermic peak observed in some sucrose DSC curves. The presence of this small endothermic event in sucrose can be observed in a number of recent articles, which have included one or more of their DSC curves (e.g., [Lee et al., 2011a,b](#); [Saavedra-Leos et al., 2012](#); [Mathlouthi and Roge, 2012](#); [Magoñ et al., 2014](#); [Kinugawa et al., 2015](#)).

A variety of factors have been reported to affect the presence and magnitude of the small peak. [Beckett et al. \(2006\)](#) reported that the appearance of the small endothermic peak is highly dependent on the purity of the sucrose. For example, the magnitude of the small peak decreased when the sucrose was recrystallized in the presence of potassium chloride (KCl). They also observed that the appearance of the small peak was affected by the recrystallization conditions employed. For example, by introducing a stirring step or increasing the recrystallization temperature, the magnitude of the small peak decreased. [Kawakami et al. \(2006\)](#) reported that the DSC curves of recrystallized amorphous sucrose also exhibited two peaks, with the peaks varying in magnitude depending on the %RH and temperature of the recrystallization conditions. However, upon annealing of the recrystallized amorphous samples, the magnitude of the first (small) peak decreased and ultimately disappeared.

A number of hypotheses have been proposed in the literature to explain the presence of the small endothermic DSC peak in sucrose. These hypotheses were grouped into six categories and summarized in [Table 1](#). Though there may be merit to some of these hypotheses, preliminary research from our laboratory ([Lu et al., 2013](#)) focused on a new factor that appears to substantially influence the presence and magnitude of the small endothermic DSC peak in sucrose – the plant source from which the sucrose was extracted. Little previous research was found tying the existence of the small peak to the plant source of the sucrose. Also, little previous research was found that mentioned studying the melting behavior of beet sucrose ([Kamoda, 1960](#); [Beckett et al., 2006](#)). Unfortunately, most studies do not report the plant source of the sucrose used. Therefore, the objective of this research was to investigate the thermal behavior differences between beet and cane sucrose sources using three thermal analysis methods, Differential Scanning Calorimetry (DSC), Thermal Gravimetric Analysis (TGA), and Ampule Heating.

2. Materials and methods

2.1. Materials

Two analytical grade crystalline sucrose samples were purchased from Sigma-Aldrich Co. (St. Louis, MO; #S0389, ≥99.5%) and Fisher Scientific Inc. (Pittsburgh, PA; S5-500; Certified ACS, purity not reported). Both analytical grade sucrose samples are isolated from sugarcane. Ten beet and 10 cane sucrose samples were obtained from SPRI (New Orleans, LA). Origin information for these sugars is provided as supplementary material. One beet and one cane sample were obtained directly from United Sugar (US) Corporation (Clewiston, FL). The remaining beet and cane samples were obtained from markets in the United States and abroad. One cane sample (called Lump Candy, single crystal sugar) was purchased in the United States, but, according to the label, was produced in Guangdong, China. The samples obtained abroad included: two cane sugar samples from the same market in Beijing, China, two cane samples from markets in Mexico and Brazil, one beet and one cane sample from a market in Canada, and one beet sample from a market in Sweden. Sample information for all

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