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Application of compression test in analysis of mechanical and color changes in grapefruit juice powder as related to glass transition and water activity

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ABSTRACT

Physicochemical and structural properties of grapefruit juice powder were studied as affected by water activity. Powdered juice was obtained by freeze-drying and equilibrated at different water vapor pressure atmospheres in order to give samples with water activity in the range of 0–0.84. The mechanical properties of the powder were measured by confined compression tests and the compressed samples, which presented uniform surface and thickness, were subjected to color analysis. The maximum force attained during the compression tests and the color coordinates could be quantified with good reproducibility. The results were related to water activity and to glass transition temperature. The occurrence of mechanical changes in the powder was shown to precede significant color changes with increasing water activity. Considering the susceptibility to stickiness, the stability limit was observed at $T-T_g \approx 2$ °C, with a high degree of mechanical changes being detected at $T-T_g \approx 16$ °C, whereas for significant color changes this critical temperature difference was around 32 °C.

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1. Introduction

Natural fruit juices, and particularly citrus juices, are considered nutrient dense beverages, as they are concentrated sources of nutrients (Rampersaud, 2007). Powdered juices might constitute good alternatives to convenient and healthy food products or ingredients to formulated foods. Nevertheless, drying and storage of powdered fruit juices presents technical difficulties due to their hygroscopic and thermoplastic behavior at high temperature and/or humidity, a characteristic that is associated to their composition (Adhikari, Howes, Bhandari, & Troung, 2004). Most of the soluble solids in fruit juices are low molecular weight sugars such as sucrose, glucose and fructose, as well as organic acids such as citric, malic and tartaric acid. Rapid water removal during freeze-drying or spray-drying, which are the methods usually employed in producing powdered fruit juices, results in an amorphous matrix that is susceptible to glass transition related changes, including stickiness, caking, and collapse (Aguilera, Del Valle, & Karel, 1995; Chen, 2007; Foster, Bronlund, & Paterson, 2006; Phanindrakumar, Radhakrishna, Mahesh, Jagannath, & Bawa, 2005; Venir, Munari, Tonizzo, & Maltini, 2007), as well as color changes (Acevedo, Schebor, & Buera, 2006; Lievonen, Laaksonen, & Roos, 2002; Ling, Birch, & Lim, 2005; Miao & Roos, 2006).

The glass transition is characterized by the change from the glassy to the rubbery state, with a certain characteristic of a thermodynamic second-order phase transition over a temperature range. This temperature range is characteristic for each material and may extend over 10–20 °C for amorphous sugars, or up to over 50 °C for some food polymers. The glass transition temperature (T_g) is taken as the onset or midpoint temperature of such a range and it is usually plasticized by water. When amorphous food materials reach their glass transition temperature, by increasing temperature and/or increasing water content, various time dependent structural transformations may occur as a consequence of the drastic decrease in the viscosity and increase in molecular mobility above T_g (Levine & Slade, 1989; Roos, 1995a, 2003; Roos & Karel, 1991; Slade & Levine, 1991). The highly porous materials prepared by freeze-drying are susceptible to post-drying collapse, characterized by a loss of structure and, in particular, a drastic decrease in porosity, affecting aroma retention, caking and stickiness, rehydration capacity and final moisture distribution (Levi & Karel, 1995). There is a strong dependence of rates of collapse on the quantity ($T-T_g$), and the effect of water is mainly associated with the depression of T_g that leads to the increase in ($T-T_g$) at constant temperature (Aguilera et al., 1995; Fitzpatrick et al., 2007; Foster et al., 2006; Paterson, Brooks, Bronlund, & Foster, 2005). According to Roos (1995b), stickiness, caking and collapse, appear to be related

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Nomenclature

A	parameter in Eq. (4)
a^*	CIELab color coordinate
a_w	water activity
a_{whalf}	parameter in Eq. (7)
b^*	CIELab color coordinate
$C_{ab}{}^*$	chroma
F_l	parameter in Eq. (7), N/kg
F_{max}	maximum force attained during the compression test, N
F_u	parameter in Eq. (7), N/kg
$h_{ab}{}^*$	hue angle, degrees
k	parameter in Eq. (5)
L^*	CIELab color coordinate
m	sample mass, kg
T	temperature, °C
T_{gs}	glass transition of the anhydrous solids fraction, °C
T_{gw}	glass transition of pure amorphous water (−135 °C)
X	water content, dry basis
X_m	monolayer water content, dry basis
X_s	solids fraction
X_w	water fraction
ΔE^*	total color difference
λ	parameter in Eq. (7)

phenomena. When a freeze-dried matrix reaches a critical temperature, which is related to T_g, a sequence of deleterious events is observed. Initially, an incipient liquid state of a lower viscosity at the particle surface occurs, which results in stickiness. Caking of sticky powders results because of interparticle bridging, causing a loss of structure and decrease in sample volume. Collapse may be considered as an extended caking phenomenon that results in liquefaction, reducing the macroscopic volume towards that typical of the liquid state and concomitant loss of porosity.

The methods used to detect collapse and shrinkage ranged from visual observation (To & Flink, 1978) and measurements of specific volume (Levi & Karel, 1995; Prado, Buera, & Elizalde, 2006; Venir et al., 2007) to measurements of internal porosity (White & Bell, 1999). Techniques and instrumentations developed for quantifying the degree of stickiness, caking and agglomeration in food powders have been reviewed by Boonyai, Bhandari, and Howes (2004), who concluded that efforts on developing an accurate, simpler, and cheaper technique to characterize the stickiness behavior of food powders are still needed.

Boonyai, Howes, and Bhandari (2007) developed a thermal compression test that involves the application of compression force in a thermally controlled sample cell attached to a texture analyzer as a method to investigate the glass–rubber transition of food powders. The technique was validated against standard DSC and TMA methods. Özkan, Walisinghe, and Chen (2002) applied a penetration test based on the measurement of force required to penetrate powder compacts to characterize stickiness and cake formation in whole and skim milk powders. Compression tests were also used by Al Mahdi, Nasirpour, Banon, Scher, and Desobry (2006) to quantify caking intensity of dried skimmed milk and wheat flour, expressed as the maximum force calculated from force/compression curves.

Krokida, Maroulis, and Saravacos (2001) pointed out that when compared to other drying methods, freeze-drying seemed to prevent color changes in foods, resulting in dried products with improved color characteristics. Nevertheless, storage of freeze-dried foods above their glass transition temperature induces reactions of color deterioration such as enzymatic and non-enzymatic browning (Karmas, Buera, & Karel, 1992; Miao & Roos, 2006). According to Acevedo et al. (2006), the dependence of browning rate with relative humidity of freeze-dried systems of different compositions and structures was governed by solid–water interactions and by structural characteristics of the systems. Rates of non-enzymatic browning of solid food models were shown to increase at temperatures 10–20 °C above the T_g (Lievonen et al., 2002).

Color measurement in bulk powder presents some drawbacks. Instrument manufacturers point out that when measuring powder color with a spectrophotometer, the measurement value varies depending on the density of the powder and the surface conditions. To avoid errors, special methods are required such as placing a fixed amount of powder into a container of a fixed shape and size and maintaining a fixed surface quality (Konica Minolta Sensing, Inc., 2003). Stickiness and collapse undergone by amorphous food powders during exposure to high water vapor pressure atmospheres may affect color measurement, confounding the effects of browning reactions themselves and of the structural changes suffered by the powder.

The objective of the present work was to evaluate the viability of using mechanical compression as a simple and convenient empirical method for characterizing freeze-dried pink grapefruit juice in terms of stickiness development and color changes as affected by glass transition and water content.

2. Materials and methods

2.1. Material and sample preparation

Grapefruit (*Citrus paradisi*) of the pigmented variety Star Ruby was obtained in the local market (Valencia, Spain). Fruits were washed and peeled with careful removal of the albedo. The pulp was cut and triturated in a bench top electrical food processor (Thermomix TM 21, Vorwerk, Spain) and passed through a coarse sieve (which is an accessory of the Thermomix) in order to remove most of the fruit rag. This procedure resulted in about 800 mL of juice with the following characteristics: 9.20 ± 0.01 °Brix, measured in an Abbe refractometer (model 3 *T*, Atago, Japan); 9.74 ± 0.02 g total solids/100 g total mass determined by the gravimetric method in vacuum oven at 60 °C to constant weight; water activity (a_w) of 0.985 ± 0.003 measured in a water activity meter (model FA-st lab 1, GBX, France); pH of 3.06 ± 0.03 measured using a pH meter (model SevenEasy Conductivity, Mettler-Toledo, Switzerland); titratable acidity of 1.74 ± 0.01 g citric acid/100 g total mass, determined by titration to pH 8.2 with NaOH 0.1 mol equi/L.

Assuming that the main soluble solids of grapefruit var. Star Ruby are sugars and citric acid (Peiró-Mena, 2007) and taking into account the analyzed amount of total solids and citric acid, the sugars content of the sample used for the study will be around 7.4/100 g total mass. Moreover, as pointed out by Peiró-Mena (2007), the main sugars of grapefruit var. Star Ruby are sucrose:-fructose:glucose in mass ratio of 54:25:21.

The juice was rapidly frozen at −25 °C in thin layers for 48 h before freeze-drying in a Telstar Lioalfa-6 Lyophyliser at 10^{-2} Pa for 24 h. The dry product was ground in a mortar and resulted in a powder with water content of 3.24 ± 0.01 g water/100 g total mass, determined by the gravimetric method, in a vacuum oven at 60 °C, to constant weight. Samples of about 0.6 g of the grapefruit juice powder spread over watch glasses of 50 mm in diameter,

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