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Implication of water state on glass transition temperature in hot air-dried carrot slices

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ABSTRACT

This study assessed and correlated the water state and the glass transition temperature (T_g) of carrots treated by hot air drying at 50, 60, and 70 °C. Results showed that within carrots with high moisture content (X_w, $4.15 \geq X_w \geq 1.01$ g water/g dry matter), free water in vacuoles dominated in tissues and the T_g remained practically constant (−48.4 ± 1.04 °C). And when the X_w of carrots was ≤0.41 g water/g dry matter, the T_g was increased by 6.79 and 22.7 °C with the relaxation time (T_{22}) of the immobilized water in the cytoplasm/extracellular space decreasing by 12.4 and 11.8 m, respectively. Moreover, correlation equations indicated that the T_g had a negatively linear relationship with the T_{22} in dried carrots. These results suggested that the T_g were elevated appreciably by reducing the relaxation behavior of immobilized water in hot air-dried carrots. The more pronounced variation of the microstructure (e.g., vacuole disruption, cell wall dissociation, and turgidity loss) of tissues could be responsible for these results.

1. Introduction

As well known, glass transition temperature (T_g) is a critical reference parameter to predict the quality, stability, and safety of food systems. It is defined as a critical temperature, where an amorphous system shifts from the glassy state to the rubbery one, or the opposite process. At the glassy state ($T < T_g$), the molecular mobility of materials is extremely slow (about 10^{-12} m/s) (Roos, 2010). If the materials become glassy, this would allow them rigid and physicochemical changes are greatly restricted. In contrast, at the rubbery state ($T > T_g$), the molecular mobility exponentially increases and the viscosity dramatically decreases (Roos, 2010). These features collectively cause the various time-dependent and viscosity-related structural transformations, like collapse, "stickiness", shrinkage, etc, during food processing and storage (Ruan et al., 1999; Kurozawa, Hubinger, & Park, 2012). Therefore, foods should be stored at the temperatures below their T_g to avoid loss in quality during the storage.

Water acts as a strong plasticizer. High water content and water activity can reduce the T_g of food materials, such as lactose-whey protein system (Maidannyk & Roos, 2017), β-cyclodextrin/water binary system (Zhou et al., 2015), wheat gluten and maltodextrin (Shimazaki, Tashiro, Kumagai, & Kumagai, 2017), etc. Fresh fruits and vegetables have higher water content. This makes their T_g often lower than the common storage temperature (−20 °C). Thus, fresh tissues are subjected to a rubbery state, easily causing a severe loss of qualities. Hence, vegetables could be dehydrated to shift the T_g to the values close to room temperature (because of the reduction of moisture content) and thus extend their shelf-life during storage. Hot air drying is a conventional and widely applicable drying technology for foods (Moraga, Talens, Moraga, & Martínez-Navarrete, 2011). To our knowledge, the detailed information of the changes of T_g in fruits and vegetables during hot air dehydration is still unclear.

Additionally, nuclear magnetic resonance (NMR) is a non-destructive technique which is helpful to have more insight about microscopic information regarding with water mobility in biological tissues. Based on the analysis of the spin-spin relaxation times (T_2), the water state within raw fruits and vegetables is usually categorized into "free water" in vacuole, "immobilized water" in cytoplasm and extracellular space, and "bound water" bounded to cell walls according to the mobility (Shao & Li, 2011; Xin, Zhang, & Adhikari, 2013). They will suffer dramatic changes during the dehydration process. For instance, vacuolar water (the most mobile water) decreases together with the increases of water in cytoplasm and intercellular space in strawberry due to the osmotic treatment

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(Cheng, Zhang, Adhikari, & Islam, 2014). Tylewicz et al. (2016) have indicated that pulsed electric field pretreatment can cause water redistribution between different compartments in freeze-dried apples. Some water could shift from vacuole into the extracellular/cytoplasm spaces, producing an appreciable increase in its relaxation time.

These changes of the water state in tissues can have a dramatic impact on the T_g. Our previous study has found that the T_g approaches a constant value when free water (the most mobile water) dominates in raw carrot tissues, probably owing to the very low viscosity of dilute solution. And the T_g increases with the decreased immobilized water in far-infrared dried carrots with low water content (Xu, Li, & Yu, 2014). Xin et al. (2013) have also showed that changing the states of water using osmotic dehydration can directly affect the T_g of broccoli. Concretely, the values of the T_g in the osmotically dehydrated broccoli are raised compared with the untreated samples. It can be seen that the water state in tissues is also a crucial contributor to influencing the T_g. However, to our knowledge, more works are still concentrated on the simplified water content/temperature state diagrams or the simplified water activity/temperature state diagrams to predict the glass transition phenomena. The reports about the effect of water state on the T_g in dehydrated products of fruits and vegetables is limited to the two ones above. Hence, a fundamental understanding of the relationship between water state and T_g is still greatly necessary for predicting the storage and processing stability of dehydrated products.

Carrot, as a root vegetable, contains appreciable amounts of nutritional components (e.g. carotenes, vitamins, fiber content) and is widely consumed worldwide at a relatively low cost. Given these advantages, we took carrot as a model system to assess the changes of the water state (using nuclear magnetic resonance) and the T_g (from differential scanning calorimetry analysis) in hot air-dehydrated carrots and establish their relationship using correlation analysis and path analysis. These findings could provide the theoretical basis for enhancing the storage and processing stability of dehydrated products of root vegetables.

2. Materials and methods

2.1. Sample preparation

Carrots (*Daucus carota* L.) were harvested in November in Shanghai (China). Fresh carrots without any physical damage were bought from a local market. Carrots were cut into slices (6 ± 1 mm thickness and 32 ± 2 mm diameter) and randomly placed in a thin layer in an electrical thermostatic drying oven (Shanghai Yaoshi Instrument Factory, Shanghai, China) with 10–15 kg mass loading. Then, samples were dried at 50, 60, and 70 °C for 14, 10, and 10 h, respectively, until reaching a final moisture content lower than 0.20 g water/g dry matter. A recirculating air maintains the constant temperature (±1 °C) and the uniformity of the temperature at every corner (±2.5%) in the oven. Some samples were taken out every 2 h for analysis.

2.2. Moisture content

Carrot samples (initial mass, $\boldsymbol{W_1}$, g) were dried in an oven at 105 °C to attain a constant mass ($\boldsymbol{W_2}$, g). Moisture content ($\boldsymbol{X_w}$, g water/g dry matter) was calculated using Eq. (1). Data were reported as the mean value of three replicates for each treatment.

$$X_w = \frac{W_1 - W_2}{W_2} \quad (1)$$

2.3. Nuclear magnetic resonance (NMR)

Based on our previous study (Xu, Li, & Yu, 2014), a NMI 20-Analyst system (22.6 MHz; Shanghai Niumag Corporation, Shanghai, China) was used to monitor the water state in carrot samples. Briefly, approximately 2 g of samples was applied to this study. Three duplicate NMR measurements (total of six runs) were carried out and a mean value taken.

2.4. Differential scanning calorimetry (DSC)

The thermal transitions of carrot samples were assayed in a differential scanning calorimeter (DSC 204 F1 Phoenix®, Netzsch, Germany) according to our previous report (Xu et al., 2014) with some modifications. The heat flow and temperature of instrument were calibrated using distilled water (melting point (mp) = 0 °C, melting enthalpy (ΔH_m) = 333.88 J/g) and indium (mp = 156.60 °C, ΔH_m = 28.45 J/g). An empty sealed aluminum pan was employed as a reference. Nitrogen gas (50 mL/min) was used as the purge gas to avoid the condensation of water around samples during running. Samples (about 10 mg) were cooled from the load temperature (room temperature) to −60 °C at 10 °C/min and equilibrated at −60 °C for 6 min. Finally, samples were scanned from −60 to 40 °C at 10 °C/min to determine the glass transition temperature (T_g). The onset (T_{go}), mid-point (T_{gm}), and end (T_{ge}) of the glass transition were assessed by using the TA universal analysis software provided with the DSC instrument. The midpoint of the transition was taken to be the T_g (Fig. 1). Data were reported as the mean value of three replicates for each treatment.

2.5. Microstructure

According to the method reported by our previous study (Xu et al., 2015) with slight modification, specimens of carrot samples were prepared. Briefly, ultrathin sections (70 nm thickness) were obtained using an ultra-microtome (Leica Ultracut UC6, Germany). Specimens were double-stained with uranyl acetate and lead citrate, and examined in a Tecnai G2 Spirit Biotwin transmission electronic microscopy (TEM, FEI, Hillsboro, OR) at an accelerating voltage of 18 kV.

2.6. Statistical analyses

Pearson's correlation analysis was performed by combining the data from all the carrot samples to address the relationship

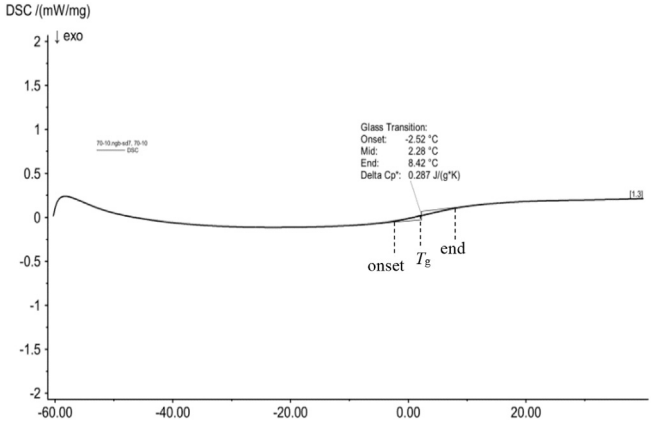

Fig. 1. Typical DSC thermogram showing the glass transition temperature (**Tg**) of hot air-dried carrots at 70 °C for 10 h.