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Freezing by immersion in liquid CO_2 at variable pressure Response surface analysis of the application to carrot slices freezing

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ABSTRACT

In order to demonstrate the potentials of high pressure carbonic immersion (HPCI) freezing in food fast freezing, effects of HPCI freezing on moisture, drip loss, hardness, nutritional components and microstructure of carrot slices were investigated. Response surface methodology analysis indicated that, the decompression time was the most significant factor affecting the central temperature, followed by pressure and retention time ($p < 0.05$). HPCI freezing at pressure of 6 MP, initial temperature of 10 °C, retention time of 3 min and decompression time of 5 min produced less drip loss and better nutrition retention, but more moisture loss in samples compared with liquid nitrogen (LN) immersion freezing or −80 °C Ultra Low Temperature Freezer (ULTF) freezing. The scanning electron microscopy (SEM) images and visual observation indicated that samples in HPCI freezing showed less tissue damage as compared to samples frozen in −80 °C ULTF freezing or LN immersion freezing. HPCI freezing is a promising way for fast freezing treatment of food.

Industrial Relevance: In the food industry, freezing is one of the common and excellent methods for long term preservation of foods. And it is generally accepted that fast freezing better preserves local structure. High Pressure Carbonic Immersion Freezing (HPCI), named by contrast to the spray-freezing of liquid carbon dioxide, can accelerate the freezing rate and make some quality attributes of food better than those in liquid nitrogen (LN) or liquid carbon dioxide spray freezing. Available data provided in this study will benefit the fast freezing food industry.

1. Introduction

Freezing is one of the common and excellent methods for long term preservation of foods as it maintains original color, flavor and nutritive values in foods (Chassagne-Berces, Fonseca, Citeau, & Marin, 2010). In the food industry, it is generally accepted that fast freezing better preserves local structure. Air blast freezing, plate contact freezing, fluidized-bed freezing and liquid nitrogen (LN) are the common methods to obtain optimum freezing rate for food products. However, the high freezing rate achievable by these methods is controlled by the thermal conductivity of foods with a low value. Therefore, new methods are being proposed and developed, such as dehydrofreezing (Ando, Kajiwara, Oshita, & Suzuki, 2012), high-pressure assisted freezing (Kurth, Wiedmer, & Entzeroth, 2012; Tironi, de Lamballerie, & Le-Bail, 2010), high-pressure shift freezing (Otero & Sanz, 2006), applications of antifreeze protein and ice nucleation protein (Kiani & Sun, 2011). High-pressure assisted freezing means phase transition under constant pressure while high pressure-shift freezing means phase transition due to a pressure release (Fernández, Otero, Guigon, & Sanz, 2006). None is ideal in every respect; especially, these techniques are associated with high investment costs and ongoing costs.

Freezing with liquid carbon dioxide or liquid nitrogen had been investigated. Peters, Smith, and Brisson (2010) developed an apparatus for CO_2 flash freezing that formed CO_2 hydrate directly as a dessert mixture was frozen. In the spraying process the CO_2 evaporates, absorbing its heat of vaporization from the dessert mixture, which in turn frozen the mixture. Expansion of carbon dioxide causes quickly frozen due to the Joule–Thomson expansion cooling (Henczka, Bałdyga, & Shekunov, 2006). That is, as a gas expands, the average distance between molecules grows. Because of intermolecular attractive forces, expansion causes an increase in the potential energy of the gas. If no external work is extracted in the process and no heat is transferred, the total energy of the gas remains the same because of the conservation of energy. The increase in potential energy thus implies a decrease in kinetic energy and therefore in temperature (Reif, 1965). The more the pressure decreased, the more the temperature decreased. Harnkarnsujarit and Charoenrein (2011) reported that the structural collapse coincided with surface cracking of freeze-dried mangoes in liquid nitrogen freezing, and found the effect of structural collapse on stability of β-carotene in different freeze-dried mangoes. However, there is no report in the literature about the

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freezing behavior of carbon dioxide when foods are firstly soaked or immersed in a high pressure carbon dioxide chamber, and then the high pressure carbon dioxide is released from the chamber into the ambient or a lower pressure chamber. Contrast to the spray-freezing of liquid carbon dioxide, the High Pressure Carbonic Immersion Freezing (HPCIF, or HPCI freezing) is probably a suitable name for this novel freezing technique. Compared to LN or liquid carbon dioxide spray freezing, HPCI freezing can accelerate the freezing rate and make some quality attributes of food better because the carbon dioxide has thoroughly penetrated into the food in high pressure chamber, once upon the pressure is released the carbon dioxide inside the food makes it rapidly and evenly frozen, reducing or eliminating the product rupture due to freezing stress usually induced by temperature difference between the surface and the inside of food in LN or liquid carbon dioxide spray freezing.

In this paper, we are particularly interested in the performance of HPCI freezing. The detailed contents are (1) to demonstrate a new freezing method, namely, High Pressure Carbonic Immersion Freezing (HPCI Freezing); (2) to detect the central temperature of carrot slices changing with treatment pressure, retention time and decompression time; (3) to obtain the optimal freezing condition and mathematical model of HPCI freezing for carrot slices; (4) to present the quality indices of products as compared with the other two conventional freezing methods—LN immersion freezing and freezing in a refrigerator at −80 °C; (5) to compare the tissue structure through scanning electron microscopy of freeze-dried carrot slices and visual observation.

2. Materials and methods

2.1. Preparation of carrot slices

Fresh carrots were purchased from a local market and stored at 4 °C until use within one week. Carrots were washed and sliced into a uniform circle with a diameter of 3 cm and thickness of 5 mm. Samples were subjected to vapor blanching at 95 ± 5 °C for 1 min, and then cooled in ice water for 5 min prior to being frozen. After frozen, the samples were packed in polyethylene bags and thawed in a cold chamber at 4 °C overnight before various indices were examined.

2.2. Temperature determination in freezing of LN and −80 °C ULTF

The central temperatures of the samples were determined with a T-type thermocouple (diameter: 0.3 mm, scope: −200–150 °C, accuracy: ± 0.75 °C, TC6-T, Shanghai Feilong Instrument & Electronic Co. Ltd., Shanghai, China) linked to an automatic recorder (ST4000, Jiangsu Shun Tong Automation Instrumentation Co. Ltd., Jiangsu, China). The T-type probe was inserted into the center of the samples before they were put into LN bath or −80 °C Ultra Low Temperature Freezer (ULTF) (DW-86 L388, Haier Co. Ltd., Qingdao, China). The samples in LN bath and in −80 °C ULTF lasted for 35 s and 13 min when the center temperature reached −18 °C.

2.3. HPCI freezing system

The HPCI freezing system used in the experiment (Fig. 1) was described by Bi, Wu, Zhang, Xu, and Liao (2011). Commercially-available CO_2 of 99.5% purity was purchased from Beijing Jingcheng Co. (Beijing, China), and was passed through an active carbon filter before entering the pressure vessel. The carrot samples were put into the vessel, and then the vessel lid was sealed with screws. The vessel was pressurized by the plunger pump to the required pressure level, and the required pressure was held for the required treatment time. Then the depressurization was performed by opening the pressure relief valve at CO_2 outlet on the pressure vessel. The temperature reduction of the product immediately after depressurization was below 0 °C due to Joule–Thomson cooling effect depending on applied pressures.

The critical pressure of supercritical fluid CO_2 is 4.5 MPa when temperature is 10 °C. In order to ensure that the CO_2 in the vessel was in the supercritical state, the initial temperature of vessel was decreased to 10 °C by decompression of supercritical CO_2 before each experiment started. And then, five pieces of carrots (about 25 g) were used for each experiment, and each experiment was repeated three times. The mean values from each of the three independent experiments were expressed as an overall mean ± standard deviation of the mean calculated.

2.4. Response surface design of carrot slices in HPCI freezing process

The central temperature of carrot slices changing with independent variables X_1 (treatment pressure, MPa), X_2 (retention time, min), and X_3 (decompression time, min) was respectively detected. After the end of the freezing, two frozen carrot slices were fetched out immediately from the vessel and cut into two halves with a knife along the central point, respectively, and then four pieces of halves were arrayed to form a center face, when the laser spot (with diameter of less than 1 mm) of a non contact portable Fluke 527 infrared thermometer (mini handheld infrared gun) (±1.5% accuracy, Victor-VC-306B, Shanghai, China) spotted on the center face the central temperature was displayed on the digital screen. Based on the single factor experiments, a response surface methodology (RSM) was applied to determine the optimal working condition of HPCI freezing system for carrot slices.

The complete design at three variation levels in the freezing process (Table 1) consisted of 17 experimental points including five replications of the center points, and the triplicates were performed at all design points in randomized order.

Experimental data were fitted to a second-order polynomial model and regression coefficients obtained. The generalized second-order polynomial model used in the response surface analysis was as follows:

$$Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} b_{ii} X_i^2 + \sum \sum_{i<j=1}^{3} b_{ij} X_i X_j \quad (1)$$

where Y is the predicted response, b_0, b_i, b_{ii}, and b_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively, and X_i, and X_j are the independent variables. SAS 8.0 software (version 8.0, SAS Institute Inc., USA) was used for the Box–Behnken design (BBD) and for generating response surfaces and contour plots, as well as for calculating determination coefficient (R^2) of the Eq. (1).

2.5. Determination of quality attributes

2.5.1. Moisture

For moisture determination, 3–4 g of homogenized samples was weighed onto an aluminum weighing dish and dried in a convectional heating oven (DZF-6050, Shanghai CIMO Medical Instrumental Manufacturing Co. Ltd., Shanghai, China) at 105 °C for at least 16 h until reaching constant weight. The above experimental procedure was repeated three times for each moisture determination.

2.5.2. Drip loss

Frozen samples were laid over an absorbent paper and thawed at 4 °C (Gonçalves, Abreu, Brandão, & Silva, 2011). Drip loss (DL) was then evaluated by periodically weighting the absorbent paper until a constant value was reached:

$$DL = (W_t - W_o)/W_s \times 100\% \quad (2)$$

where W_0 and W_t are the weight of the dry and wet absorbent paper (g), respectively, and W_s is the weight of the frozen sample (g).