Journal of Food Engineering 125 (2014) 1–6

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Water phase transition under pressure and its application in high pressure thawing of agar gel and fish ☆

S.M. Zhu [a], G.M. Su [a], J.S. He [a], H.S. Ramaswamy [b], A. Le Bail [c], Y. Yu [a,*]

[a] College of Biosystems Engineering and Food Science, Zhejiang University, Key Laboratory of Equipment and Informatization in Environment Controlled Agriculture, Ministry of Agriculture, 866 Yuhangtang Road, Hangzhou 310058, China
[b] Department of Food Science, McGill University, 21111 Lakeshore Road, St-Anne-de-Bellevue, QC H9X 3V9, Canada
[c] GEPA-ENITIAA (UMR CNRS 6144-SPI), Rue de la Géraudière BP 82225, F-44322 Nantes Cedex 03, France

ARTICLE INFO

Article history:
Received 10 May 2013
Received in revised form 25 September 2013
Accepted 13 October 2013
Available online 20 October 2013

Keywords:
High pressure
Phase transition
Thawing
Calorimeter
Fish

ABSTRACT

Experiments were carried out with a high-pressure (HP) differential scanning calorimeter (DSC) and a HP unit using frozen agar gel (3%, w/w) and Atlantic salmon (*Salmo salar*). Small samples (0.54–0.7 g) were prepared for HP DSC tests. Frozen samples of agar gel and salmon muscle in cylinders (47.5 mm diameter, 135 mm length) were subjected to water immersion thawing (WIT) (20 °C) and HP thawing at 100, 150 and 200 MPa with a water temperature of 20 °C. Phase transition temperature of agar gel was close to the phase diagram of pure water. Melting temperature of salmon was significantly lower than phase diagram of pure water probably due to the presence of solutes and cellular structures in fish. HP DSC tests demonstrated a good correlation between temperature (T) and average pressure (P): $T = -1.22 - 0.0946P - 0.000115P^2$ ($R^2 = 0.99$, $n = 10$). High pressure caused a depression of the ice-melting temperature resulting in an accelerated thawing process. The reduction of melting plateau time can be predicated by using Plank's model. For frozen agar gel, the total thawing time was 50.3 ± 2.7, 36.4 ± 2.2 and 30.8 ± 1.8 min, or 73, 53 and 45% of WIT time (68.7 ± 4.3 min) at 100, 150 and 200 MPa, respectively. For frozen fish, the total thawing time was 58.9 ± 2.8, 41.8 ± 4.7, 37.2 ± 2.6 and 33.8 ± 1.9 min for WIT, HPT at above pressures, respectively.

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1. Introduction

Freezing and thawing processes are important technologies for the preservation and quality of frozen foods. Retention of fresh-like quality is the primary focus of freezing preservation and consumer expectations. Traditional freezing process is generally slow, resulting in large extra-cellular ice crystal formation, which causes texture damage, accelerates enzyme activity and increases oxidation rates (Bello et al., 1982; Ngapo et al., 1999). Thus, improvement in the freezing process is often related to increasing the freezing rate based on high efficiency refrigeration systems. Thawing is generally slower than freezing, causing further damages to frozen food texture. Less attention has been paid to using novel thawing technologies to preserve the tissue structure and product quality. Indeed, due to microbial and enzyme activities, a minimal ambient temperature should be ensured for the thawing process. Rapid thawing at low temperatures can help to prevent the loss of food quality during the thawing process (Okamoto and Suzuki, 2002). This is obviously a challenge for traditional thawing processes, because a lower ambient temperature results in less temperature difference between the frozen sample and the surrounding which is the main driving force for the thawing process.

Elevated pressure depresses the freezing point of water from 0 °C to −21 °C at about 210 MPa (Bridgman, 1912). This phenomenon allows a frozen sample to be thawed at temperatures as low as −21 °C (at 210 MPa). Freezing and thawing processes at high pressure are innovative processes with a great potential for the food industry. The decrease in freezing point under pressure significantly enlarges the temperature difference between the frozen sample and the ambience, and thus effectively increases the driving force and the rate of thawing. According to Plank's model, the freezing/thawing time is inversely proportional to the temperature difference between test sample and its ambient condition (International Institute of Refrigeration, 1986). For a thawing process carried out at 20 °C ambient temperature, the conventional thawing gives a temperature difference of about 20 °C, while for high pressure thawing (HPT) at 200 MPa the difference will be approximately twice as much. Thus, theoretically HPT time at 200 MPa can be half that of conventional immersion thawing time.

☆ *Industrial relevance*: Rapid thawing at low temperature can prevent the loss of food quality. This is a big challenge for traditional thawing processes. High pressure can depress melting temperature and significantly accelerate the thawing process. The result from this study is helpful for a better understanding of water phase transition in foods and its potential application in the HP thawing of food products.

* Corresponding author. Tel./fax: +86 571 88982181.
E-mail address: yyuzju@zju.edu.cn (Y. Yu).

0260-8774/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.jfoodeng.2013.10.016

If the thawing is carried out at 4 °C, the HPT time can be as low as one fifth of conventional thawing time because of the enlarged relative difference in temperature between the frozen sample and the ambient temperature. Thus, HPT process offers considerable potential for rapid thawing applications.

Early research attempts into HPT processes were carried out mostly in the medical field. Taylor (1960) reported that a slow freezing rate followed by a HPT process at 225 MPa significantly improved the cell survival ratio of human conjunctiva. Some recent studies demonstrated the advantages of HPT process, including reduction in thawing time and preservation of food quality (Zhao et al., 1998; Le Bail et al., 2002a,b; Zhu et al., 2004a; Park et al., 2006; Alizadeh et al., 2007). Makita (1992) observed that the HPT process of frozen beef at 120 MPa took one-third the time to thaw than thawing process at 0.1 MPa and produced sensory qualities comparable to those of conventional thawed products. Okamoto and Suzuki (2002) observed that there was a slight decrease in thawing loss for pork meat processed with HPT, while discoloration was not recognized with the naked eye up to 200 MPa. Park et al. (2006) demonstrated that HPT treatment effectively improved the quality of frozen pork below 100 MPa.

A HPT process involves compression heating, pressure-dependent temperature change, phase transition and heat transfer. It is more complicated than a conventional thawing process (Knorr et al., 1998). Understanding thermal behaviors during HPT process are important for the application of HPT technology and improvement of product quality, but available scientific information is limited (Le Bail et al., 2002a; Urrutia et al., 2007). Early high-pressure equipment frequently did not contain temperature sensors within the pressure chamber. As a result, temperature changes under pressure were not reported (Ting et al., 2002). Nowadays temperature probes are available for use within pressure chambers, but it's still a challenge to monitor phase transition and pressure–temperature changes in different locations of a frozen food during a HPT treatment due to the limitation of probe flexibility. There are several studies focusing on modeling of HPT processes (Chourot et al., 1997; Denys et al., 2000; Otero and Sanz, 2003; Ousegui et al., 2008). Chourot et al. (1997) modeled the thawing of an aqueous solution (4% NaCl) at pressures of 100–150 MPa. Denys et al. (2000) established a heat transfer model for temperature prediction during HPT processes by taking into account the pressure dependence of the latent heat of a food model called Tylose (methylcellulose gel, about 78% water content). Otero and Sanz (2003) demonstrated the difference in temperature change between high pressure-assisted thawing and high pressure-induced thawing by HPT experiments using frozen agar gel. Ousegui et al. (2008) developed a numerical model for HPT processes that was validated using Tylose slabs ($100 \times 100 \times 25$ mm^3). However, little data is available on phase transition during HPT of real food materials probably due to the difficulty of high-pressure measurements. Guignon et al. (2008) presented several approaches to describe the initial freezing temperature of foods at high pressure. In fact, ice percentage and freezing temperature of partial water in a real food system vary with temperature and pressure (Zhu et al., 2006).

The objective of this work was (1) to monitor temperature and phase transformational changes in model gel (agar) and fish samples during different HPT processes, and (2) to compare the difference of thawing thermal behaviors between water and fish in various HPT conditions.

2. Materials and methods

2.1. High pressure DSC test

A high-pressure (HP) differential scanning calorimeter (DSC) was used in this study. DSC is a useful tool to explore physical properties and phase transition phenomena of water, aqueous solution and organic liquids, which usually works at constant pressure while temperature is changed as a working parameter. In the case of high pressure, however, DSC does not work well due to large heat capacity of the vessels that is necessary to restrain pressure. HP DSC is an alternative measure to overcome this difficulty which works at constant temperature and pressure change. The details of the HP DSC system as well as experimental methods can be found in previous papers (Zhu et al., 2004b, 2006). Low concentration agar gel (3%, w/w) and fresh Atlantic salmon were used in the HP DSC experiment. Agar gel was prepared as described in the following section. Salmon were obtained from a local market (Carrefour, Nantes, France). Small samples (0.54–0.7 g) of these materials were prepared for HP calorimetric experiments. Each specimen was vacuum-packaged in a polyethylene bag (80 μm thick multilayer film) (La Bovida, Nice, France). Packaged samples were stored in a cooler (4 °C) before experiments. Test sample was installed in sample cell as previously described in Zhu et al. (2004b, 2006). Air bubbles were carefully removed from the cell during sample installation by filling them with the pressure fluid. After calorimetric experiments, moisture content of each salmon sample (71.2 ± 1.4%) was determined by drying in a ventilated oven at 103 °C for 24 h.

2.2. High-pressure thawing test

Experiments were carried out using a HP unit (ACIP-3500/1/8 VB, ACB Pressure Systems, Nantes, France) as shown in Fig. 1. The pressure chamber was 80 mm in diameter and 200 mm in height. Distilled water was used as the pressure transmission medium. A copper tube jacket in thermal contact with the outer wall of the vessel was connected to the circulator to enable temperature control.

To obtain temperature signals from the test samples inside the pressure chamber, stainless-steel shielded thermocouple wires (K-type, 0.5 mm in diameter, Omega, Stamford, CT) were fixed through a silver welded threaded nipple. The pressure was measured through a pressure transducer connected on the pressure line. Temperature and pressure were recorded using a data logger (Agilent 34970A, Agilent Technologies Canada Inc. Mississauga, ON) during the HPT process.

Low concentration agar gel (3%, w/w) was used as a food model in this study because it had thermal-properties similar to water but its convective heat transfer was stopped due to the gel network during the freezing and thawing experiment. Agar powder (Becton Dickinson, MD) and distilled water (3%, w/w) were mixed in a flask. The mixture was mixed well using a magnetic stirrer to allow it to be completely dissolved at a temperature close to boiling point. When cooled to about 70 °C, the solution was poured into sample holder and sealed with a rubber stopper (Fig. 2) by retaining a column of 135 mm length and 47.5 mm diameter. Three K-type thermocouples (0.5 mm diameter, Omega, Stamford, CT) were placed in the middle region of the test sample with the tip located along different radial planes: center, midway between the center and the surface (midway) and near the surface (surface) (see T_o, T_m and T_r in Fig. 2). Before the freezing treatment, the prepared samples were aged in a cooler (4 °C) overnight for gel texture maturity.

Fresh Atlantic salmon (about 4 kg) was obtained from Waldman, a special market of fresh fish in Montreal, Canada. The consignment was procured and transported from the East Coast of Canada to Montreal by a refrigerated truck within 24 h of purchase. After de-skinning and filleting in post-rigor condition, the larger salmon pieces were cut into smaller pieces and put into the same cylindrical sample holder as shown in Fig. 2.

The all samples were frozen in a conventional air freezer (about −20 °C). Three pressure levels (100, 150 and 200 MPa) were

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