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Ice content and temperature determination from ultrasonic measurements in partially frozen foods

C. Aparicio, L. Otero, B. Guignon, A.D. Molina-García, P.D. Sanz *

Department of Engineering, Instituto del Frío (CSIC), José Antonio Novais 10, 28040 Madrid, Spain

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Abstract

The knowledge of the ice content of frozen systems is of the utmost interest for monitoring and controlling frozen food production and storage. It is also useful if freezing is an undesirable process. Although there is a well known relation between temperature and ice content, this is of little practical help since the easily measurable superficial temperature is not suitable for this calculation: information on the spatial temperature distribution is required. The speed of sound depends strongly on the physical state of media, briskly varying from water to ice. It is also a function of temperature. An easy and fully feasible method to determine a representative overall temperature of food and the ice content by measuring the speed of sound through a given sample thickness is here reported. The method is quick and suitable for online monitoring of frozen, freezing and thawing systems, and it can be adapted to a large variety of containers, geometrical situations and water contents. Data from model and real food systems are presented.

Keywords: Ultrasound; Speed of sound; Frozen food; Freezing; Ice content; Non-invasive measurements; Temperature determination; Hake

1. Introduction

Ice content, the percentage of the water in a system that is in solid form, is one of the most important parameters of food when freezing is involved or feared. It is also one of the more elusive and difficult to measure data. Frozen systems at any temperature are known to have only a fraction of their water content as ice, while the rest remains in liquid state. Only completely pure water attains complete freezing at temperatures below its freezing-point when the whole of the molecules are crystallized into ice. The rest of systems are more or less complex solutions of one or several solutes. The freezing temperature of these solutions is reduced by the presence of solute, following well known dependences (Clapeyron Equation). But this freezing temperature so determined is only an initial freezing-point. As crystalline ice is formed, the solute molecules or ions are expelled from its lattice and the solute concentration gradually increases. Consequently, the equilibrium temperature decreases as the ice content grows.

Most frozen foods are stored at temperatures about −18/−20 °C. At this temperature a significant amount of water remains in liquid state, as cryo-concentrated solution, and it enables many undesirable processes, such as microbial growth, enzymatic and chemical reactions, migrations and ice crystal size growth. In this relation, it must be taken into account that only free water can undergo state transitions such as ice crystallization (Chen 1985; Heldman 1974; Schwartzberg 1976). Only at temperatures below the glass transition the remaining liquid water molecules are unable to move and be a vehicle for these processes, often related to food spoilage. Other frozen storage procedures keep food for shorter time periods at higher temperatures, i.e., −8 °C "superchilling" and "partial freezing" (Fik et al. 1988; Magnussen et al. 2007). In these conditions a larger amount of water should remain in liquid state.

Freezing at any temperature is a relatively slow process, and, as food portions are often large, during a considerable

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* Corresponding author. Fax: +34 915493627.
E-mail address: psanz@if.csic.es (P.D. Sanz).

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time period there is an evolving gradient of temperature and changing ice fraction. During this period, the named mechanisms of food spoilage are especially active, and they are strongly dependent of liquid water content. The same can be said about thawing; so, the knowledge of the ice and liquid water fractions is of importance to monitor and control these processes. Also, in other occasions, the formation of ice is not desired (defence of crop plants against freezing, monitoring of correct refrigeration...). In all these cases, a method for determining the ice content would be valuable.

Ice content can be measured by a variety of methods, differential scanning calorimetry being the more precise, although it is destructive for the sample and impossible to be used for online monitoring. Nuclear magnetic resonance can also be employed, but it is technically complex and expensive for its use in food storage and processing applications.

Nowadays, ultrasound is currently used to evaluate different quality attributes in food (fruit ripeness, microbial contamination in liquid food, gelation point, among others) mainly due to the advantages of this technique. It may be implemented online and non-invasively and so, food quality can be evaluated in a safe, hygienic and economic way. The speed of sound longitudinal waves in water is a smooth function of temperature both in liquid water and in ice but, in the phase change region, the speed of sound increases dramatically when water gets transformed into ice. This is a well known consequence of the state change, as sound propagation in solids is faster than in liquids. This fact, together with the additivity of the speed values over different media regions in the path of the waves is the base of the procedure proposed in this paper, in which the overall speed of sound is employed to determine the solid and liquid water content of a sample.

Ideally, many foods can be described in a simplified way as aqueous solutions with small amounts of other non-soluble components such as structural polymers and lipids. This would allow for a prediction of the speed of sound through a food sample, calculated as a combination of the speeds in the liquid and solid fractions, considered the temperature of the system. Nevertheless this should be only a crude description of the phenomena of sound propagation on foods and similar systems. This is due to several facts not accounted in the simplified model, basically the difficulty or impossibility to describe accurately the composition of food and its spatial distribution. Liquid water contains a large number of diverse solutes and these can have a non-negligible influence in the speed of sound in the solution. Also the non-soluble components will have a contribution not easily calculable to the overall speed. Finally, the interphases between different food component regions and ice crystals would have a contribution difficult to account for *a priori*. For this reason, it is more advisable to absorb most of these effects by resorting to calibration employing samples as similar as possible to the product under consideration and relate known ice content values to measured speed of sound. As it will be seen, calibration with a simple solution model is enough for the determination of the ice fraction in high water content foods.

Different authors have performed ultrasound experiments to study the propagation of ultrasonic waves through partially frozen foods and the relationship between ultrasonic properties and ice content (Miles, 1974; Lee et al. 2004; Sigfusson et al. 2004; Gülseren and Coupland 2007; Carcione et al. 2007). Miles's (1974) method requires the previous knowledge of the value of the speed of sound through the sample at 0°C and its water fraction. Lee et al. (2004) measured the ultrasonic velocity and attenuation in partially frozen orange juice over a wide temperature range. The authors related these properties to the ice content determined by nuclear magnetic resonance. Sigfusson et al. (2004) proposed the pulse-echo technique as a method to locate the position of the ice front during freezing processes. Gülseren and Coupland (2007) measured the speed of sound in solutions of sucrose and glycerol and in orange juice as a function of temperature. They obtained an empirical model that relates the speed of sound to temperature and composition in unfrozen solutions. These authors also suggested a single linear relation for relating the change in velocity on freezing to the amount of ice formed. Recently, Carcione et al. (2007) successfully used a poroelastic model to describe the propagation of ultrasonic waves through orange juice, which is subjected to a freezing process. They used the Kelvin's model to obtain the amount of unfrozen water in the juice as a function of temperature and the Biot's poroelastic theory (Biot, 1962) to calculate the ultrasonic properties of orange juice as a function of temperature, below the eutectic point. This theory about propagation of elastic waves in porous media is widely used in the literature (Leclaire et al. 1994; Daher et al. 1997, among others) and considers the two-phase material as a continuum and thus the macroscopic variables follow the laws of continuum mechanics. Basically, the theory assumes that anelastic effects arise from viscous interactions between the fluid and the solid and unifies the treatment of the mechanics of deformation and acoustic propagation in porous media.

The method described in this paper allows the online, continuous and non-invasive determination of a representative overall temperature and the corresponding ice content of frozen food with high water content. No calibration procedure is required as the method just takes advantage (Aparicio et al. 2007) of the behaviour of simple solutions used as food model in similar conditions.

2. Materials and methods

2.1. Samples

A set of NaCl aqueous solutions (1%, 2%, 3% and 4% *w/w*) (Panreac Química SA, Barcelona, Spain) has been employed as a simplified model for high water content food. The rest of the measurements were performed on a