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Determination, analysis and prediction of the volumetric behavior of milk at high pressure

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ABSTRACT

High pressure preservation technologies are consolidating in the food industry as an interesting alternative to traditional thermal processes. Process modeling contributes to its progress and requires the input of food properties like density for calculations. The dependency on pressure of these properties is indispensable but it is rarely available in the literature. The sector of dairy products is an important target for the development of novel foods by high pressure treatments (both high hydrostatic pressure processing and ultra-high pressure homogenization). Thus, the main objective of this research was to characterize the volumetric properties of raw whole milk and skim milk. A variable-volume piezometer with a solid-piston volumeter was employed for this purpose. Density, specific volume, isothermal compressibility and thermal expansion coefficient were determined between 0 and 60 °C under pressures up to 350 MPa; at atmospheric pressure, measurements cover temperatures up to 90 °C. Results show that milk solutes and fats, although present in low quantities in milk compared to water, have an influence which is worthy of consideration on milk volumetric properties. Irregularities appear from 200 MPa in the dependencies on temperature of the studied milk properties. From a composition-based model, it is highlighted that milk solutes' specific volume behavior is inverted around 55 °C and that milk fats' compressibility goes through a maximum around 30 °C. The composition-based model is further developped for the calculation of milk properties as a function of pressure at different temperatures; prediction errors are below 2%. Useful data and equations for high pressure processing simulation are provided together with an original view on the combined effects of pressure and temperature on milk solutes and fats.

1. Introduction

Consumers look for safe, nutritional added value and extended shelf-life foods. In this way, many beneficial effects of both hydrostatic and dynamic high pressure on dairy products have been observed (Rastogi, Raghavarao, Balasubramaniam, Niranjan, & Knorr, 2007). The microorganisms in milk are inactivated within few minutes up to 1 h when pressure is between 200 and 400 MPa (Mussa & Ramaswamy, 1997). Antigenicity of milk proteins can be reduced by high pressure processing (HPP) (Chicón, Belloque, Alonso, Martín-Álvarez, & López-Fandiño, 2008; Peñas, Snel, Floris, Préstamo, & Gomez, 2006). Yogurts made from high pressure treated skim milk show an improved creamy texture compared to traditional yogurts (Ciron, Gee, Kelly, & Auty, 2012; Udabage et al., 2010). Cheeses manufactured from pressurized milk reach higher production yields compared to cheeses prepared using thermally-treated milk; besides, the syneresis observed after storage is lower in the first case (Guamis, Trujillo, Sendra, Buffa, & Saldo, 2000; Zamora, Ferragut, Jaramillo, Guamis, & Trujillo, 2007). Thus, high pressure treatments of milk are potentially attractive for the dairy industry. In fact, some dairy products treated at high hydrostatic pressure have already started to appear in the market. These are cheese- and mayonnaise-based sandwich fillings (in Spain since 2007), yogurt dressings (in the USA since 2007), colostrum beverage (in New Zealand since 2008), culinary preparations (in the Netherlands since 2009), and cheese snacks (in the UK since 2010). In order to further develop the high pressure applications in the dairy sector and to optimize processes, several hurdles should be overcome. One of them is the lack of knowledge about the thermophysical properties of dairy products in the high pressure domain since they are indispensable for engineering purposes as explained below.

High pressure homogenizers have been recently developed to reach higher pressures than those conventionally used to date with the aim of improving food quality and safety. Ultra-high-pressure homogenizers are now able to stabilize milk, emulsions and other liquid foods using pressures that can reach up to 400 MPa. A critical point of such equipment is the pressure valve. Its design and the materials used for its fabrication have to resist against liquid pass under extreme conditions: the liquid food (e. g. milk, emulsions, juices) is pressurized throughout the valve gap and shortly heated due to frictional forces. While pressure is set *a priori*, the extent of heating of the food around the valve is more difficult to know. The knowledge of the thermophysical properties of

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milk at high pressure should help in estimating at which temperature both the valve and the milk are submitted. They could be used for accurate modeling studies of fluid flow in the valve and therefore to optimize the choice of pressure valve characteristics. Additionally, for a given valve, process performances are linked to the physico-chemical characteristics of the fluid passing through the homogenizer, like density and viscosity (Dumay et al., 2012). Thus the behavior of these properties with both pressure and temperature seems to be important for the control of the resulting product characteristics.

Besides, high hydrostatic pressure processes are evolving toward their use in combination with low or high temperatures. This allows for microbial inactivation while natural characteristics of foods are usually better preserved than with traditional thermal processing. During the pressurization step of the process, adiabatic heat gives rise to temperature gradients likely to produce non-uniform microbial inactivation. This important concern has recently been examined by Grauwet et al. (2012). One of the main strategies to solve this problem is to obtain temperature uniformity mapping by computational thermal fluid dynamics modeling and simulation of temperature fields in the high pressure vessel. These authors show the potential of such strategy and underline that the absence of knowledge about the combined effect of temperature and pressure on food properties is an important limitation to get accurate predictions.

Heat, mass and momentum transfer equations for HPP simulation require their input not only as a function of pressure but also as a function of temperature. The experimental determination of food thermophysical properties under pressure is seldom performed, probably because of its laborious character. The measuring devices have to be technically adapted to the high pressure domain and are often prototypes. The corresponding methodologies for their implementation have to be developed to reduce uncertainties in the results. The necessary scanning of a wide range of pressure and temperature conditions is highly time-consuming. Therefore, in the absence of data at high pressure, the thermophysical properties of foods are usually approximated.

Some properties of interest for the HPP simulation are density, heat capacity, thermal conductivity, viscosity, and thermal expansion coefficient. In this work, we focus on the volumetric properties of milk: density, specific volume, isothermal compressibility and thermal expansion coefficient. Approximations can only be found for density or specific volume (its reciprocal). In the case of density approximation, a possible choice is the compositional or additive model (Otero, Guignon, Aparicio, & Sanz, 2010):

$$\frac{1}{\rho_{food}(p,T)} = \frac{w_{H_2O}}{\rho_{H_2O}(p,T)} + \frac{w_{solutes}}{\rho_{solutes}(p_{atm},T)} \tag{1}$$

where WH_2O and $w_{solutes}$ represent the water and solute mass fractions, respectively. This model is based on water density (ρH_2O) behavior with pressure and on solute density ($\rho_{solutes}$) at atmospheric pressure. Another possibility is to use the "ratio" model (Hartmann, Delgado, & Szymczyk, 2003):

$$\rho_{food}(p,T) = \frac{\rho_{food}(p_{atm},T)}{\rho_{H_2O}(p_{atm},T)}\rho_{H_2O}(p,T). \tag{2}$$

The last option is to use water density, directly, instead of the corresponding food density. Water properties are easily calculated as a function of temperature and pressure by means of the equation of state developed by the International Association of Properties of Water and Steam (IAPWS-95 formulation). This could be reasonable for food with high moisture content though the uncertainty generated by this assumption has not been assessed in depth since there are almost no data for this. Sensitiveness of predictions to engineering factors such as material properties has begun recently. The influence of compression fluids, carriers and packaging properties on temperature-related microbial inactivation profiles has been studied (Knoerzer & Chapman, 2011; Knoerzer, Buckow, Sanguansri, & Versteeg, 2010). Regarding foods, for orange juice, it was shown that the uncertainty generated by thermophysical property approximations increases with solute concentration and may not be negligible (Guignon, Aparicio, Sanz, & Otero, 2012). More studies are expected in that way.

For all of these reasons, food thermophysical properties under pressure have to be studied further, including also temperature as a variable. Given that water and lipids have very different densities and adiabatic heats, it appears interesting to address their respective contributions in a food system such as milk. Moreover, except lactose, the other main milk components, that is to say proteins and milk fats, undergo modifications under pressure. Lactose is likely not affected by pressure because its structure relies on covalent bounds (which are not disrupted by pressure); caramelization and the browning reaction are inhibited under pressure at 60 °C (Moreno, Villamiel, & Olano, 2003). By contrast, fats and proteins which involve non-covalent interactions are sensitive to pressure. The crystallization of fats is induced by pressure (Costa et al., 2007; Huppertz, Kelly, & Fox, 2002). The conformation of proteins — like casein micelles and whey proteins — changes with pressure. Many authors have already observed that casein micelle size is reduced at high pressure, especially between 150 and 300 MPa (Knudsen & Skibsted, 2010; Orlien, Boserup, & Olsen, 2010) while their size is increased near 40 °C by interactions with pressure-denatured whey proteins. Other authors have reported denaturation of whey proteins above 100 MPa, β-lactoglobulin being the most sensitive to pressure denaturation (López-Fandiño, 2006). Macroscopic properties like density could be influenced by all these physico-chemical modifications. The extent of such effects needs to be quantified before being able to state whether the use of approximations for milk density (and other properties) introduces or not any significant uncertainty in HPP simulation results. Thus the objective of this work was to characterize the contribution of composition to the volumetric behavior of milk with pressure and temperature.

For this purpose, the density of whole and of skim milk was measured under pressures up to 350 MPa and for temperatures between 0 and 60 °C (up to 90 °C at 0.1 MPa). From these measurements, the specific volume, the isothermal compressibility and the thermal expansion coefficient were derived. Then, the experimental data of skim milk were compared with the data for pure water. In the same way, whole milk data were compared with skim milk data. This allows for standing out the role of composition. Finally, a simple composition-based model was developped and tested to predict the volumetric properties of milk as a function of temperature and pressure.

2. Material and methods

2.1. Samples

Raw whole bovine milk was provided by a local dairy (CLESA, Madrid, Spain) with its corresponding compositional analysis (Foss MilkoScan FT120, Foss, Denmark). Experiments were performed over 4 months during summer time. Mean values with standard deviations for composition in mass percentage resulted to be: 3.44 ± 0.14% for fats, 3.06 ± 0.04% for proteins and 4.63 ± 0.03% for lactose. A skimmer (Elecrem, France) was used to obtain samples of skim milk from the initial raw whole milk. Raw whole and skim milk samples were stored on ice at 0 °C in glass bottles and used within 48 h. Milk pH was checked before use and it was obtained on average 6.7 ± 0.1 pH units. The pH-meter (Crison pH-Burette 24 1S, pH 50 21 electrode with temperature sensor C.A.T. 55 31, Crison, Spain) used for that purpose was calibrated prior to these measurements using commercial standard buffers at pH 4 and pH 7.

After this study, homogenized pasteurized semi-skimmed milk (Puleva, Granada) was purchased at a local supermarket and used to test the proposed composition-based model for milk properties' prediction.