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Temperature uniformity mapping in a high pressure high temperature reactor using a temperature sensitive indicator

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ABSTRACT

Recently, the first prototype ovomucoid-based pressure–temperature–time indicator (pTTI) for high pressure high temperature (HPHT) processing was described. However, for temperature uniformity mapping of high pressure (HP) vessels under HPHT sterilization conditions, this prototype needs to be optimized. To this end, this work aimed at the development of an ovomucoid-based indicator with combined pressure temperature dependent inactivation kinetics and a sufficient pressure temperature stability relevant for commercial HPHT sterilization. After varying buffer type and the pH at ambient pressure and temperature (pH_i), an indicator based on 1 g/L ovomucoid in 0.1 M MES-NaOH buffer pH_i 6.2 was selected. The inactivation behavior of this indicator system is characterized by pressure temperature dependent (combined Arrhenius–Eyring) first-order kinetics in the processing domain relevant for HPHT sterilization. This indicator showed good integrating properties under isobaric–isothermal and dynamic pressure temperature conditions.

In a temperature uniformity study of a vertically oriented, pilot-scale HPHT vessel, pTTI readouts at different coordinates illustrated low and high temperature zones. As the inactivation of spores under HPHT is clearly positively temperature dependent, the food safety objective has to be verified in the former sampling zone.

1. Introduction

1.1. High temperature short time treatment of conductively heating products

Enhancing microbial safety and extending the shelf-life of high moisture content food products is generally performed by relatively slow thermal processes (de Heij et al., 2005). The high temperatures needed, whether or not in combination with long residence times, result in a decrease in food quality (e.g. texture, nutritional value). The high temperature short time (HTST) principle has been introduced as a basis for the optimization of thermal sterilization. Today, liquid foods, whose temperature can be increased and decreased rapidly by exploiting convection phenomena, are successfully flash heated and cooled in industry resulting in high residual quality products after processing (e.g. UHT milk) (Holdsworth, 2009).

Recently, high pressure high temperature (HPHT) treatment (500–800 MPa; 80–120 °C; 1–10 min) of food products has been put forward as a worthy alternative for the optimization of sterilizing conductively heating (i.e. solid) food products (de Heij et al., 2003; Heinz and Knorr, 2005; Barbosa-Canovas and Juliano, 2008). In HPHT processing, compression and decompression of compressible materials causes respectively rapid heating and cooling of food products, since a temperature change is linked to every pressure change (Barbosa-Canovas and Rodriguez, 2005). As pressures can be generated fast, this phenomenon can create reduced process times (see HTST principle) (de Heij et al., 2005; Heinz and Knorr, 2005; Juliano et al., 2009a). The improved quality of conductively heating products after a HPHT treatment in comparison to their equivalently conventionally treated counterparts has been discussed (Matser et al., 2004; de Heij et al., 2005; Juliano et al., 2007; De Roeck et al., 2008, 2009; Leadley et al., 2008). In this context, a HPHT process is sometimes termed a 'pressure-assisted thermal process' (PATP) (Barbosa-Canovas and Juliano, 2008).

Starting from room temperature, using only compression heating, the food product temperature cannot be raised to the point where inactivation of spores under high pressure (HP) is feasible. Therefore, a preheating step to a well-defined initial temperature

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(T_i) (e.g. T_i = 90 °C) needs to precede the actual HPHT treatment. When pressurization starts from this T_i, due to compression heating, the temperature of the product reaches the process temperature (T_p) (e.g. T_p = 121 °C for water-like components assuming adiabatic conditions during pressure build-up to 600 MPa). In practice, a HPHT process is a three-step process: (i) preheating at atmospheric pressure; (ii) actual HPHT treatment; (iii) further cooling at atmospheric pressure.

1.2. Hurdles in high pressure high temperature processing implementation

In contrast to UHT-applications, to date, no commercial application of HPHT processing is available. In the literature, different hurdles have been reported: (i) insufficient insight of the temperature distribution in a HPHT reactor (Knoerzer et al., 2007; Juliano et al., 2009a) and poor understanding of the process uniformity of a HPHT treatment (Denys et al., 2000; Juliano et al., 2009b); (ii) HPHT food products are subjected to the 'novel food regulation' (EC 258/97) (Howlett et al., 2003); (iii) no industrial-scale HPHT unit has been built that incorporates all individual HPHT processing steps. In the following section, hurdle (i) will be focused on.

1.3. Insight in the temperature distribution in a HPHT reactor and its effect on the process impact uniformity

Pressure, temperature and time are the critical process variables in HPHT processing (Barbosa-Canovas and Juliano, 2008; Ramaswamy et al., 2009; De Roeck et al., 2010). It has been generally acknowledged that high pressure used in HPHT processing can be assumed uniform. In addition, time is fixed as a HPHT process operates under batch conditions. However, securing temperature uniformity in HPHT reactors is not straightforward (Delgado and Hartmann, 2003). Differences in compression heat of pressurized materials in HP reactors (e.g. pressure medium, food components) and the corresponding heat transfer underlie the existence of temperature gradients in HP vessels (Delgado et al., 2008). Since the inactivation kinetics of spores, in view of HPHT sterilization, are clearly positively temperature dependent, it is very likely that temperature non-uniformity under HPHT conditions results in process impact non-uniformity (Margosch et al., 2006; Zhu et al., 2008; Shao et al., 2008). Direct monitoring of the temperature profile at different coordinates in a HPHT reactor seems to be the most straightforward method to gain insight in the temperature distribution in a HPHT reactor. However, this method is technically too complex at pilot or industrial scale. There is a need for another method easily detecting temperature differences under HPHT conditions. Once different temperature zones could be indicated, it would be necessary to increase the understanding on the effect of these different temperature zones on the process impact distribution. This requires either kinetic information of the target attributes under HPHT conditions or direct process impact evaluation on the target attribute (i.e. *in situ* method). Kinetic data of target attributes under HPHT conditions are scarce. As a first step in more removing hurdle (i), this work aimed at the development of a method for easy detection of low and high temperature zones in a HPHT vessel.

1.4. Protein-based pressure–temperature–time indicator (pTTI) for mapping temperature uniformity in a high pressure high temperature reactor

In general, a pTTI can be defined as a small, wireless pressure temperature sensitive device characterized by an easily quantifiable, irreversible response to the HP treatment (Van Loey et al., 2002; Van der Plancken et al., 2008). The potential of two α-amylase-based pTTIs to detect low and high temperature zones in an industrial-scale vessel under HP pasteurization (HP-P) conditions (400–600 MPa; 10–40 °C; 1–15 min) was previously demonstrated (Grauwet et al., 2009, 2010a,c). However, the stability of these α-amylase-based indicators does not allow use under HPHT conditions: the HP-P indicator read-out would be below the detection limit after HPHT treatment.

Recently, the first prototype protein-based pTTI with potential use under HPHT conditions has been described (Grauwet et al., 2010b). A protein system consisting of 1 g/L ovomucoid, a commercially available inhibitor of trypsin, dissolved in a specific solvent environment (0.1 M sodiumphosphate buffer pH 8.0) (OM-PhB8.0) was characterized by a temperature sensitive inactivation under increased pressure (p > 400 MPa) in the HPHT window (i.e. range of pressure–temperature–time conditions necessary for reaching HPHT conditions). The temperature sensitive inactivation under increased pressure suggested the potential of an ovomucoid-based system as a tool to map temperature uniformity under HPHT conditions. However, the prototype developed had some drawbacks: (i) pressure changes (400–700 MPa) did not affect the inactivation rate; (ii) the inactivation window of the system was restricted to mild HPHT conditions (400–700 MPa; T_p 95–110 °C; 0–20 min), in which spores inactivation occurs but is reduced; (iii) the thermal stability of the ovomucoid system at atmospheric pressure was rather limited.

In this work, the first prototype sensor will be optimized using solvent engineering (purposely changing the solvent characteristics in order to reach the desired indicator characteristic) to accomplish the following objectives: (i) obtaining a pressure sensitive inactivation of ovomucoid, without restriction of the temperature sensitivity; (ii) shifting the ovomucoid inactivation window to the HPHT processing window relevant for commercial sterilization; (iii) improving the heat stability of the candidate indicator at atmospheric pressure. The potential of such a pressure temperature sensitive protein-based indicator to map temperature uniformity was experimentally verified.

2. Materials and methods

For all data reported in this work, the same experimental approach was used. First, an ovomucoid-based indicator system was prepared by dissolving ovomucoid in a specific solvent conditions (Section 2.1). Next, this system was treated under particular pressure–temperature–time conditions (Sections 2.2 and 2.3). In a third step, its irreversible read-out upon treatment was quantified (Section 2.4). Finally, data obtained were analyzed (Section 2.5). In the following, materials and methods will be described step-by-step.

2.1. Ovomucoid-based indicator system

Ovomucoid (EC 2329069) is a trypsin inhibitor present in chicken egg white. Type III-O (no ovo-inhibitor) was purchased in dried state from Sigma (lot number 117K7011, Germany). To avoid batch-to-batch differences, for all experiments performed, the same storage solution of ovomucoid was used as described by Grauwet et al. (2010b). From this storage solution (100 g/L in 0.1 M Tris (2-amino-2-hydroxymethyl-propane-1,3-diol)-HCl buffer pH 8.6), just before treatment, the ovomucoid system was prepared by dilution in a given buffer condition to a concentration of 1 g/L. The effect of initial pH (i.e. pH measured at atmospheric pressure and room temperature; pH_i) of a pressure stable MES (2-(*N*-morpholino)ethanesulfonic acid)-NaOH buffer (pH 5.0–7.0) on the inhibitor capacity of ovomucoid was investigated in the context of solvent engineering.