Food Research International 43 (2010) 893–901

Contents lists available at ScienceDirect

Food Research International

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

Understanding high pressure-induced changes in wheat flour–water suspensions using starch–gluten mixtures as model systems

Katleen J.R. Vallons, Elke K. Arendt *

Department of Food and Nutritional Sciences, National University of Ireland, University College Cork, College Road, Cork, Ireland

ARTICLE INFO

Article history:
Received 29 July 2009
Accepted 15 December 2009

Keywords:
Wheat flour
Wheat starch
Wheat gluten
High pressure
Gelatinisation
Rheological properties

ABSTRACT

The effect of high pressure (HP) on wheat flour–water suspensions was investigated. Suspensions were treated for 10 min at 200–600 MPa. HP-treatment significantly increased the consistency of the flour suspensions, as studied by frequency sweep tests. Temperature sweeps revealed that HP-induced starch gelatinisation, with a sigmoidal-shaped correlation between degree of gelatinisation and treatment pressure. Analysis of protein solubility in different buffers indicated the HP-induced formation of urea-insoluble complexes and/or disulphide bonds. Furthermore, the effects of HP on the isolated components wheat starch and gluten were studied, and starch–gluten mixtures were used as a model system for flour. A negative effect of gluten on the consistency increase of starch suspension was observed. Comparing the rheological parameters of HP-treated wheat flour suspensions to those of starch suspensions, confirmed the weakening effect of gluten. However, the presence of gluten in flour could not fully explain the differences between starch and flour suspension.

1. Introduction

For more than a decade, high pressure (HP) processing has been investigated as an alternative to the traditional thermal processing. HP has advantages over thermal treatment such as better retention of nutritional and functional ingredients in the processed product. It also provides the possibility to produce cereal products with novel textures by altering the structure of biopolymers such as proteins and starch (Stolt, Oinonen, & Autio, 2001). HP has been shown to cause gelatinisation of starch similar to heat-treatment. Douzals, Marechal, Coquille, and Gervais (1996) observed wheat starch gelatinisation occurring in the range of 300–600 MPa. However, HP-treated starch exhibits unique gelatinisation properties. Previous studies (Douzals, Perrier Cornet, Gervais, & Coquille, 1998; Douzals et al., 1996; Selmi, Marion, Perrier Cornet, Douzals, & Gervais, 2000) showed limited granule expansion (water binding), denser gels, lower rates of amylase release and lower initial rates of enzymatic activity of HP-gelatinised starch compared to thermal-treated starch. HP can also irreversibly change the structural and functional properties of proteins, as recently reviewed by Winter (2003). The sensitivity of protein structure to HP is mostly due to weakening of electrostatic and hydrophobic interactions, and to thiol/disulphide exchange reactions. The effects of HP on wheat gluten, gliadins, and glutenin have been extensively described by Apichartsrangkoon, Ledward, Bell, and Brennan (1998), Kieffer, Schurer, Köhler, and Wieser (2007), and Schurer, Kieffer, Wieser, and Köhler (2007). These authors showed that increased HP and heating had strong effects on the rheological properties of gluten and led to an increase of gluten strength. Furthermore, there studies supported the hypothesis by Funtenberger, Dumay, and Cheftel (1997) stating that the SH/S–S interchange reactions occurred via the nucleophilic attack of a disulphide bond by the ionized S^--form of an SH-group.

Previous studies mainly focused on the effects of HP on isolated biopolymers and little information is available concerning the impact of HP on whole flour systems. Gomes, Clark, and Ledward (1998) investigated the effect of HP on amylases in wheat and barley flours and observed a significant increase in activity of starch-degrading enzymes at 400–600 MPa, while the activity was decreased at pressures $\geqslant$600 MPa. Ahmed, Ramaswamy, Ayad, Alli, and Alvarez (2007) studied the effect of HP-treatment of basmati rice slurries and found gelatinisation of starch and denaturation of proteins, and pointed out the important role played by the combination of starch and proteins on the calorimetric behaviour of HP-treated basmati rice slurries. Furthermore, Hüttner, Dal Bello, Poutanen, and Arendt (2009) showed that HP can be used to modify the viscoelastic and structural properties of oat batters and that starch gelatinisation as well as protein structural changes were involved.

The objectives of this study were to investigate the effects of HP on the structural and viscoelastic properties of wheat flour

* Corresponding author. Address: Department of Food and Nutritional Sciences, University College Cork, College Road, Cork, Co. Cork, Ireland. Tel.: +353 21 490 2064; fax: +353 21 427 0213.
E-mail address: e.arendt@ucc.ie (E.K. Arendt).

0963-9969/$ - see front matter Crown Copyright © 2010 Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.foodres.2009.12.010

suspensions. Furthermore, HP-induced gelatinisation and protein structural changes were studied and related to rheological changes. The relative contribution of these two processes to the effects on the complex flour suspensions was determined by using wheat starch–gluten mixtures as model systems for the flour.

2. Materials and methods

2.1. Materials

The wheat flour (Wholefood Wholesalers, Dublin, Ireland) used in this study, was characterised by: 10.9% protein, 0.2% fat, 13.24% moisture, 1.0% ash. The wheat starch and gluten were obtained from Sigma–Aldrich. The flour and starch suspensions (40% w/w) were mixed with a Glutomatic (Perten instruments AB, Sweden). For the wheat starch–gluten mixtures, the dry components were mixed manually before water addition, and subsequently mixed with a glutomatic. The dried gluten was rehydrated with 150% water and left to rest for 15 min at room temperature. The wet gluten was then centrifuged and excess water was removed.

2.2. High pressure-treatment

Samples were vacuum-packed in polyethylene bags (100 × 150 mm; Miller Pack Ltd., Finglas, Dublin 11, Ireland). Packed samples were vacuum-packed again to prevent contact between pressurisation fluid and the suspension. The HP-treatment was performed using a Stansted Fluid Power Iso-lab 900 High Pressure Food Processor (Stansted Fluid Power, Stansted, Essex, UK) as described by Huppertz, Fox, and Kelly (2004). The samples were treated at pressures of 200–600 MPa. Pressure was increased at a rate of 300 MPa/min, maintained at the desired pressure for 10 min and released at a rate of 300 MPa/min. The temperature of the vessel of the pressure unit was thermostatically controlled at 20 °C throughout treatment. Due to compressive heating, increases in the temperature of the processing fluid by up to a maximum of 12 °C at 600 MPa, were observed; increases in the temperature of the processing fluid were transient, and the set temperature ±1 °C was reattained at the end of the treatment.

2.3. Rheological measurements

The rheological measurements of the HP-treated samples were carried out by a rotational rheometer (Physica MCR 301, Anton Paar GmbH, Stuttgart, Germany) using a parallel plate geometry (50 mm diameter) with sanded surface probe to prevent slippage. The temperature, initially set at 30 °C, was regulated by a circulating water bath and pelletier control system. After loading, the sample was trimmed and left to rest for 5 min. The tests performed on the samples were:

(1) An amplitude sweep (γ = 0.001–100%) at a constant frequency (10 Hz) to determine the limits of the linear viscoelastic range (data not shown).
(2) A frequency sweep (ω = 50–1 Hz) at a constant deformation within the linear viscoelastic range. The storage modulus ($G^{"}$), the loss modulus ($G^{"}$), the complex modulus (G^{*}) and the damping factor (tanδ) were monitored. Each test was performed at least in triplicate.
(3) A temperature sweep at a constant deformation (within the linear viscoelastic range) and a constant frequency (10 Hz). For this test, the sample perimeter was covered with a thin layer of petroleum jelly to prevent dehydration. After equilibration at the initial temperature (30 °C) for 5 min, the samples were heated at a rate of 7.8 °C/min to the final temperature of 95 °C. After a holding time of 5 min at this temperature, the samples were cooled at a rate of 7.8 °C/min to the initial temperature at which they were held for another 5 min. The complex modulus (G^{*}) was monitored at 10 s intervals. Changes in G^{*} of the suspensions were evaluated in terms of onset (T_o) and endset (T_e) gelatinisation temperatures. Furthermore, onset (G^{*}_o) and endset (G^{*}_e) complex moduli were extracted from the pasting profiles. The breakdown was calculated as the decrease (%) between G^{*}_e and the complex modulus at the end of the holding phase at 95 °C. The setback was calculated as the increase (%) between the complex modulus at the end of the holding phase at 95 °C and the final complex modulus at the end of the holding phase at 30 °C. Each test was performed at least in triplicate.

2.4. Protein solubility

The solubility of proteins in control and HP-treated samples was determined according to published methods (Alamprese, Iametti, Rossi, & Bergonzi, 2005) by suspending 50 mg of sample in 1 ml of 50 mM sodium dihydrogen phosphate buffer, 0.1 M NaCl, pH 7.0 (P-buffer) followed by ultra-sonification for 60 min. Where indicated, 6 M urea (PU-buffer) and 0.1 M dithiothreitol (DTT, PUD-buffer) were added to the buffer. The amount of soluble proteins was determined spectrophotometrically (Bradford, 1976) on the supernatans obtained from centrifugation of the extracts at 10,000*g* for 20 min at 15 °C, using bovine serum albumin as a standard. The results are the mean values of at least triplicates.

2.5. Lab-on-a-Chip capillary electrophoresis

The profile of proteins extracted in the different buffers was investigated using capillary gel electrophoresis. Proteins in the range 5 to 80 kDa were separated using the Protein 80 + LabChip in the Agilent 2100 Bioanalyser (Agilent Technologies, Palo Alto, CA), as previously described by Klose, Schehl, and Arendt (2008). All measurements were performed at least in triplicate.

2.6. Scanning Electron Microscopy (SEM)

Freeze-dried samples were mounted on aluminium stubs covered with double-sided carbon tape and sputter-coated with gold in a vacuum evaporator. Prepared samples were viewed in a JEOL 5510 scanning electron microscope (JEOL, Tokyo, Japan) at 3–5 kV using 10 mm working distance.

2.7. Statistical analysis

In order to assess the differences between samples at different measurement points, a two sided *t*-test for independent samples (Statistica 7.0 StatSoft, Inc., USA) with a significance level of 0.05 was used.

3. Results

3.1. Frequency sweep

Frequency sweeps under dynamic conditions (i.e. in the linear viscoelastic range) were performed to study the effect of HP on the viscoelastic properties of control and HP-treated samples. The rheological parameters at angular frequency = 7.84 Hz (tenth measuring point) of wheat flour, starch and gluten suspensions are shown in Table 1. HP-treatment significantly increased both the