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Characterization of water state and distribution in textured soybean protein using DSC and NMR

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ABSTRACT

To explore the role of water during extrusion cooking and the scientific basis for demarcating low-intermediate moisture and high-moisture extrusion technique, soybean protein isolate (SPI) was processed using a pilot-scale twin-screw extruder under 28–60% moisture content and 140–160 °C cooking temperature. The state of water in textured soybean protein (TSP) with different treatments were analyzed by using differential scanning calorimetry (DSC) and low field nuclear magnetic resonance (LF-NMR) together. The distribution of water in TSP was characterized by using magnetic resonance imaging (MRI). The results showed that there were at least two categories of water with different states or mobility in TSP, and the results of water state and critical water content in TSP measured by DSC and NMR were consistent. The distribution of water in TSP was homogeneous, and the intensity of distribution increased markedly with total water content increasing.

1. Introduction

Extrusion, as a continuous efficient cooking, mixing and forming process, has been used increasingly to produce breakfast cereals, baby foods, flat breads, snacks, meat and cheese analogues and modified starches, etc. (Harper, 1978; Ding et al., 2006). The extrusion process involves a major contribution from the water in the system. Water performs several essential functions in the thermoplastic extrusion process, such as being a lubricant, plasticizer and reaction reagent, etc., reducing glass transition temperature to ensure dough melt and to reduce viscosity, finally causing different conversion ratios from mechanical energy into melt dough (Tolstoguzov, 1993; Chen et al., 2010). The difference in water mobility and distribution during the extrusion process could result in the different extent of raw material mixing and the physicochemical reaction, finally causing various extrudate with different structures and configurations. Therefore, it is imperative to investigate the role of water during extrusion cooking process for controlling the extrusion process and producing ideal extrudate.

High-moisture extrusion is an emerging and promising technique for transforming vegetable proteins into palatable meat-like products. It has numerous advantages over traditional low-intermediate moisture extrusion. Feed moisture content is the main basis for differentiating high-moisture extrusion from low-intermediate moisture extrusion. Usually, the extrusion operation with feed moisture content 10–30% is considered as low-intermediate moisture extrusion; the extrusion operation with feed moisture content more than 40% is called high-moisture extrusion (Cheftel et al., 1992; Akdogan, 1999; Liu and Hsieh, 2008). However, in existing literature there has not yet been related report to give out the scientific basis to justify the boundary between high- and low-intermediate moisture regimes being located between 30% and 40%.

Differential scanning calorimetry (DSC) and nuclear magnetic resonance (NMR) spectroscopy are two conventional techniques to characterize the state, mobility and distribution of water in polymer systems, and have been widely used in previous reports (Hayashi et al., 1992; Liu et al., 2000; Tananuwong and Reid, 2004; Mateusa et al., 2007). In the present paper, we used DSC and NMR techniques to investigate the change of water states and distribution in textured soybean protein (TSP) extruded at different feed moisture contents and cooking temperatures, and further to explore the role of water during extrusion cooking and the scientific basis of moisture boundary for demarcating low-intermediate moisture extrusion and high-moisture extrusion.

2. Materials and methods

2.1. Raw Material

Soy protein isolate (SPI) was obtained from Yuwang Group Ltd. (Shandong Province, China). The approximate composition of raw

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materials were as follows: water content 8.15%, fat content 0.06% (dry base), ash content 4.81% (dry base), total protein content (N × 6.25) 92.68% (dry base), nitrogen solubility index 63.92%.

2.2. Extrudate preparation for DSC and NMR measurement

Extrusion was carried out in a pilot-scale, co-rotating, intermeshing, twin-screw extruder typed DSE-25 (Brabender GmbH & Co., Germany) with the following operation parameters: feed speed 20 g/min, screw speed 160 rpm, moisture content 28–60% (wet base), cooking temperature from feeding zone to die zone 80–110-*X*-135–80 °C (*X* was set at 140, 150 and 160 °C, respectively). When the extruder reached steady state, samples for each treatment were collected and immediately put into airtight plastic bags for later DSC and NMR analysis. The DSC and NMR were determined within 3 h from extrusion.

2.3. Differential scanning calorimetry (DSC)

A differential scanning calorimeter (model DSC-Q200, TA Instruments, USA) was utilized to measure the freezable water in the heat treated samples. A slice subsample of 5–10 mg from each treatment was encapsulated into an aluminium pan, and cooled from 303 K to 233 K at 5 K min^{-1} using liquid nitrogen, then heated to 313 K at 5 K min^{-1}.

According to the method of Yoshida et al. (1992), the total amount of freezable water (W_f) in extrudate was calculated from the ratio of melting enthalpy at 0 °C for per gram of sample and pure water. The amount of non-freezable water (W_{nf}) in the extrudate was defined as $W_{nf} = W_c - W_f$, where W_c is the total water content of samples, obtained by oven-heating method. Two duplicate DSC measurements were made for each treatment and averaged.

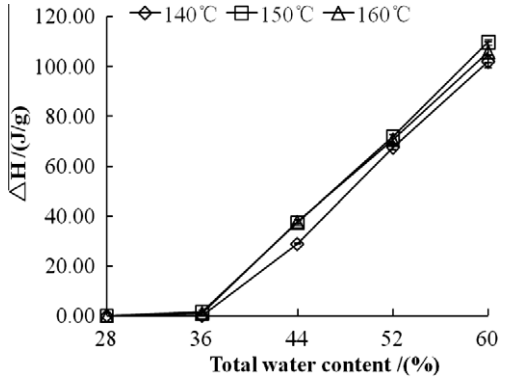

Fig. 1. Relationship between change of enthalpy and total water content for TSP extruded at 140, 150 and 160 °C cooking temperature and 28%, 36%, 44%, 52% and 60% moisture content.

2.4. Nuclear magnetic resonance (NMR)

A low field pulsed NMI 20-Analyst (Shanghai Niumag Corporation, China) with 22.6 MHz was used in the experiment. Approximately 2 g of strip sample was placed in a 15 mm glass tube and inserted in the NMR probe. Carr–Purcell–Meiboom–Gill (CPMG) sequences were employed to measure spin–spin relaxation time, T_2. Typical pulse parameters were as follows: dwell time = 4 μs, echo time = 240.00 μs, recycle time = 600 ms, echo count = 350, scan repetitions = 32. Each measurement was performed in duplicate.

The 2D proton density image from transverse section of extrudate with different moisture contents was performed after T_2 determination. Typical pulse parameters were as follows: echo time = 14 ms, recycle time = 1000 ms, scan repetitions = 32. The size of image was 384 × 128.

2.5. Statistical analysis

The data of each treatment was analyzed for statistical significance using analysis of variance (ANOVA) function and for statistical interrelation using analysis of correlation (CORR) procedure in Statistical Analysis Software (SAS) V 8.01 (SAS Institute, USA). Duncan's multiple range test at 1% and 5% level was used to identify the significant difference of each treatment (Yuan and Zhou, 2000). *T* test was used to identify the significance of correlation coefficient. Scatter diagrams were plotted by Microsoft Office Excel 2007.

3. Results and discussion

3.1. The state of water in TSP measured by DSC

The effect of total water content on change of enthalpy freezable and non-freezable water content of TSP are shown in Figs. 1 and 2. According to the intercept of the regression line at zero freezable water (Fig. 2A), the critical quantity of freezable water in TSP was calculated as 35.2–37.6% (wet base). Below this critical quantity of freezable water, there was no melting endothermic peak (Fig. 1), which indicated that water molecules were bonded tightly by hydrophilic group and present in non-freezable form in the TSP. However, above this critical quantity the changes of enthalpy and freezable water content increased almost linearly with a further increase in total water content (Figs. 1 and 2A), and the

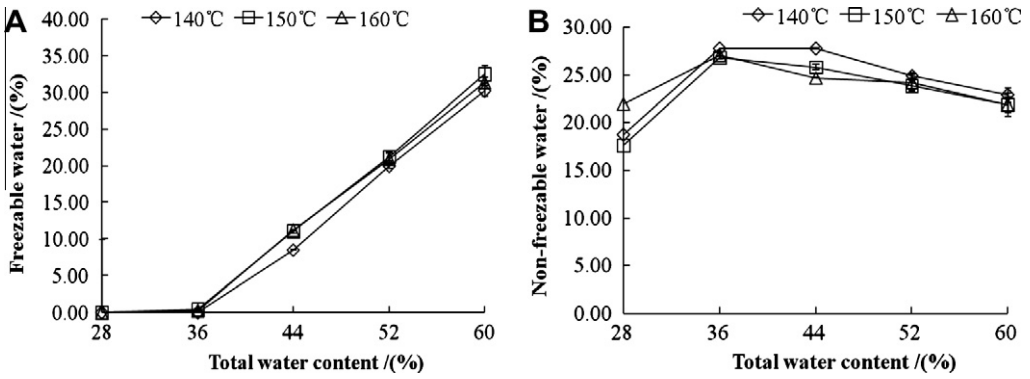

Fig. 2. Freezable and non-freezable water content as a function of its total water content for TSP extruded at 140, 150 and 160 °C cooking temperature and 28%, 36%, 44%, 52% and 60% moisture content.