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Study of the dynamic states of water and effects of high-pressure homogenization on water distribution in tofu by using low-field nuclear magnetic resonance

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ABSTRACT

Low-field nuclear magnetic resonance was used to study two traditional types of tofu. The size of diffusive domains was estimated based on diffusive exchange model. T_2 parameters were used to monitor changes in the microstructure and water distribution under different high pressure homogenization (HPH) conditions (50, 100, and 150 MPa). The T_{21} relaxation time significantly decreased with increasing pressure, and the tofu prepared using HPH-pretreated soymilk showed a compact protein network structure compared with the control sample. From the perspective of water distribution and water-holding capacity, 50 MPa was the appropriate pressure for pretreatment of soymilk. Significant correlations were further detected among T_{21} relaxation time, water-holding capacity, and particle size. Therefore, T_{21} relaxation time could be used as the optimal indicator to reflect changes in the microstructure and water distribution in tofu.

Industrial relevance: The morphology and water of tofu play important roles in its textural properties and sensory attributes. So a rapid and economic monitoring method for quality control of industrial manufacture of tofu is desperately needed. In the present study, time domain nuclear magnetic resonance was introduced to obtain more insights in the effects of ultra high pressure homogenization pretreatments of soymilk on morphology and water distribution of final tofu matrix. In conjunction with results of present study, LF-NMR was suggested to be a good tool for study and monitoring of tofu making.

1. Introduction

Tofu or soybean curd is a traditional Chinese food prepared by curdling fresh hot soymilk with a coagulant. The history of tofu can be traced back to the Han dynasty 2000 years ago, and the two traditional types of tofu include northern and southern tofu, which were named after the places where they are popular in China. The main difference between northern and southern tofu is the type of coagulant used for their processing; the former is induced by magnesium chloride, and the latter by calcium sulfate. Despite wide consumption of tofu by Chinese, processing of tofu in China remains largely dependent on workers experience. As tofu is a gel-like protein matrix containing a high amount of water, a rapid method must be established to study the dynamic states of water and the morphology of tofu to improve its production.

High pressure homogenization is a continuous and non-thermal processing technique that is widely applied in the food industry (Kaushik, Kaur, Rao, & Mishra, 2014; Poliseli-Scope, Hernández-Herrero, Guamis, & Ferragut, 2014; Sevenich et al., 2013). The homogenization pressures used in the food industry range from 20 MPa to 350 MPa (Cruz et al., 2007). HPH is a widely used pretreatment method for soymilk to achieve fine stable emulsion for further processing. The application of HPH may change the physical and chemical properties of soymilk; such properties include particle reduction, protein denaturation, and enzyme inactivation (Cruz et al., 2007; Keerati-U-Rai & Cooredig, 2009; Wang, Zhou, & Chen, 2008). HPH pretreatment also influenced the shelf life, sensory attributes, and physical properties of soymilk and soymilk-derived products, such as soy yogurt (Ferragut, Cruz, Trujillo, Guamis, & Capellas, 2009; Poliseli-Scope et al., 2014; Smith, Mendonca, & Jung, 2009). The potential of HPH to improve the textural properties of tofu has been reported. Tofu prepared using HPH-pretreated soymilk presents a developed microstructure and satisfactory textural properties (Liu, Chien, & Kuo, 2013).

Low-field nuclear magnetic resonance (LF-NMR), a rapid, non-destructive, and low-cost technique, has gained attention in the field of food science. The two important parameters in LF-NMR research are spin–lattice relaxation (T_1) and spin–spin relaxation (T_2), which represent two features of proton relaxation (Cheigh, Wee, & Chung, 2011). T_2 is frequently used for analysis because it exhibits more evident changes in relaxation time and is more sensitive to multi-phase environment than T_1 (Trout, 1988). LF-NMR can be successfully applied in quality control of food products, such as cheese, meat, and various

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plant tissues (Bertram, Dønstrup, Karlsson, & Andersen, 2002; Hansen et al., 2010; Métaisa, Camberta, Riaublancb, & Mariette, 2006). In general, T_2 relaxation times can be used as indicators to monitor water mobility because water fractions in different microenvironments exhibit different T_2 relaxation properties (Bertram, Karlsson, et al., 2001). The LF-NMR signals can also reflect the states of fat in complex food matrix, such as ice cream (Mariette & Lucas, 2005). Most studies revealed that food matrices usually present multi-exponential relaxation behavior (Hager, Bosmans, & Delcour, 2014; Hansen et al., 2010; Métaisa et al., 2006). In a previous work, three water fractions were detected through LF-NMR using T_2 measurements and the assignments of these three water fractions were proposed based on the microstructure of tofu (Li et al., 2014). In the present work, the model of diffusive exchange established by Hill and Belton (Hills, Takacs, & Belton, 1990; Belton & Hills, 1987; Belton, Hills, & Raimbaud) was introduced to elucidate the dynamic states of water and the morphology of the two types of tofu. In relation to the microstructure of tofu, the influence of HPH pretreatment of soymilk on the water distribution in the final tofu matrix was also discussed. The potential use of different T_2 parameters in monitoring changes in water distribution at different HPH treatments was further investigated.

2. Materials and methods

2.1. Materials

Calcium sulfate ($CaSO_4$) and magnesium chloride ($MgCl_2$) were obtained from local food ingredient suppliers. All chemicals were of analytical grade. Commercial soybeans from northeast China were provided by the Key Laboratory of Biology and Genetic Improvement of Soybean, Ministry of Agriculture (Nanjing, P. R. China).

2.2. Soymilk preparation and HPH pretreatment

Soybeans were rinsed and soaked overnight with deionized water in a ratio of 1:3 w/w for 12 h at 20 °C. After hydration, the soybeans were rinsed again and placed into a household soymilk maker BL-747 (Deer Co., Ltd., Guangdong, China) and ground with deionized water at a ratio of 1:10 w/w. After a cycle was completed, okara (tofu residues) was removed by filtering the soymilk through a strainer and passing twice through a double-layer cheesecloth (0.15 mm). The soymilk was then refrigerated until further use. A dynamic high-pressure homogenizer NS 1001L2K (GEA Niro Soavi Co., Ltd., Parma, Italy) was used to homogenize raw soymilk. The soymilk was passed through a specifically designed valve with an adjustable gap, called the homogenizing valve, at high pressures to disperse protein and fat particles. The temperature of the soymilk increased after homogenization particularly at high pressures (higher than 50 MPa). A cooling cycle system was then installed at the outlet of the discharging pipe to prevent heat aggregation of the soybean protein and ensure optimal homogenization effect. The obtained final temperature of soymilk was lower than10°C.

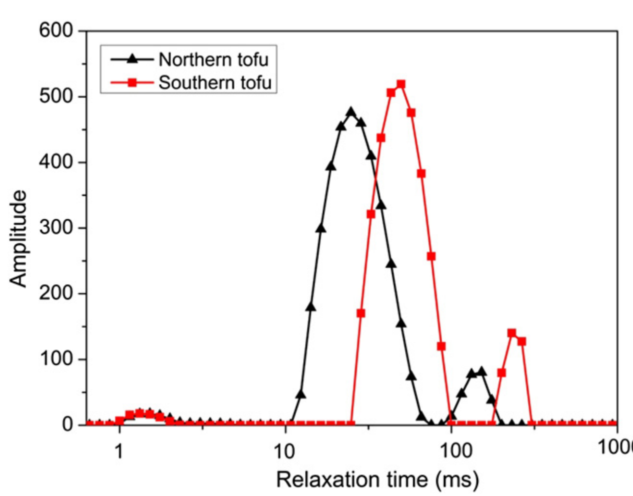

Fig. 1. LF-NMR T_2 relaxation curves of southern and northern tofu.

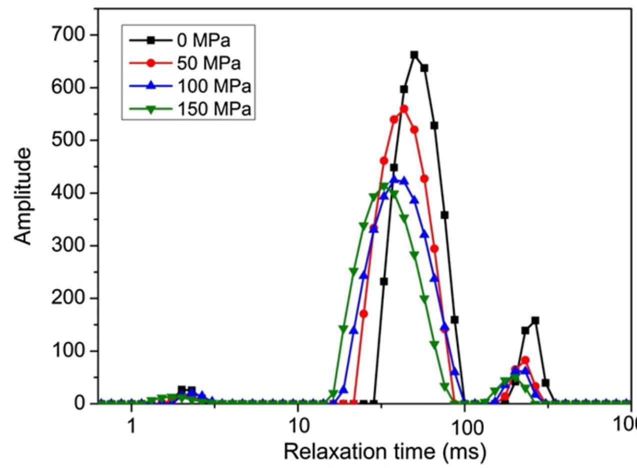

Fig. 2. LF-NMR T_2 relaxation curves of southern tofu prepared using HPH-pretreated soymilk at different pressures.

2.3. Tofu preparation

Two liters of soymilk was heated to boiling point in a WK2102T electromagnetic oven (Media Co., Ltd., Foshan, China), stirred for 3 min, and then cooled to 90 °C. Two types of divalent salt coagulants were dispersed at a concentration of 3% (w/w) by using hot deionized water (90 °C). The prepared coagulant dispersions were immediately added into the soymilk to obtain a final concentration of 15 mM. During addition, the mixture was continuously stirred with a stainless steel spoon for 10 s. The soymilk was decanted into a water bath and preserved at 90 °C for 20 min to allow the coagulation of the soybean protein. The mixture was then transferred into a wooden mold and pressed at 0.01 kg/cm^2 for 30 min to exclude whey and obtain tofu curd. The tofu curds were stored in airtight containers at 4 °C before analysis.

2.4. LF-NMR

LF-NMR was performed on a 22.4 MHz NMR Analyzer PQ001 (Niumag Co., Ltd., Shanghai, China). After storage at 4 °C for 24 h, the tofu samples were thermostated to 30 °C in a water bath for 20 min. Approximately 2 g of different tofu samples were obtained from the middle of the tofu curd and placed in 18-mm NMR glass tube. The tube was inserted into an NMR-probe. T_2 relaxation time was measured using Carr–Purcell–Meiboom–Gill (CPMG) sequence with a τ-value of 150 μs (time between 90° and 180° pulse). Data from 3,000 echoes were acquired at 16 scan repetitions to prevent imperfect pulse settings. The repetition time between two successive scans was 3 s, and the measurements were performed at 30 °C.

MultiExp Inv Analysis software (Niumag Co., Ltd., Shanghai, China) was applied for data analysis to obtain distributed exponential curve fitting. The continuous distribution of exponentials for a CPMG experiment could be defined using the following equation:

$$R_{mag}(t) = \sum_{j=1}^{n} P_{2j} \exp\left(\frac{-t}{T_{2j}}\right) + \mathrm{L} \qquad (1)$$

where R_{mag} is the residual magnetization as a function of the acquisition time t, P_{2j} and T_{2j} are the spin–spin relaxation amplitude and time,