Journal of Food Engineering xxx (2017) 1–7

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Low-field nuclear magnetic resonance for online determination of water content during sausage fermentation

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ARTICLE INFO

Article history:
Received 26 October 2016
Received in revised form
16 May 2017
Accepted 22 May 2017
Available online xxx

Keywords:
Sausage
Water content
Low-field nuclear magnetic resonance
Relaxation time

ABSTRACT

Traditional fermented sausages containing 0%, 25%, 50%, and 75% fat are produced by natural fermentation. Water in sausages was investigated by low-field nuclear magnetic resonance (LF-NMR). T_1-weighted phantom magnetic resonance images visually showed spatial inhomogeneity in water content. Center of intensity was brighter than its edges, indicating that water distribution in sausages was inhomogeneous because of vaporization. Low-fat samples showed remarkable spatial inhomogeneity in water content. T_2 populations, which were referred to as free water (T_{23}) and immobilized water (T_{22}), shifted toward short relaxation times and gradually merged into one population during fermentation. Relative area percentage of immobilized water (M_{22}) increased, whereas that of free water (M_{23}) decreased. Approximately 95% of water was present as free water and immobilized water. Water content of sausages showed the strongest linear correlation ($R^2 > 0.92$) with T_2 area. The linear model showed good accuracy and precision but overestimated water content. Reliability of model decreased as fat content increased because of inhomogeneity in water content of low-fat samples. Therefore, water content can be measured based on sample weight, fat content, and T_2 area in a nondestructive manner and regardless of sample size.

1. Introduction

Fermented sausages are appreciated by consumers for their special flavor and texture characteristics. Traditional fermented sausages are produced in natural drying systems or industrial dryers for long fermentation periods (Ikonić et al., 2013; Soyer et al., 2005). Reduction in water content occurs during this process. Water affects stability and shelf-life of products because of its involvement in microbiological growth and enzymatic activities (Fernández et al., 2000). Water also influences sensory qualities, such as texture and juiciness, that are attributed to fermented sausages (Muguerza et al., 2001; Olivares et al., 2010). Thus, water content is considered the primary quality indicator of fermented sausages. Given such importance of water, water mobility and content in fermented sausages must be monitored to control product quality (Collell et al., 2012; Olivares et al., 2010).

Several methods were developed to determine water content of foods. Traditionally, water content is measured by oven-drying known volumes of samples. However, this method is time-consuming and destructive and is thus unacceptable for online systems. Several nondestructive methods, such as near-infrared spectroscopy (Collell et al., 2012), microwave, and Raman spectroscopy (Damez and Clerjon, 2013), can measure water content without requiring any contact with samples. However, these technologies perform unstably in monitoring water on production lines. Thus, a suitable and nondestructive method is needed to monitor online water quality in food production.

Low-field nuclear magnetic resonance (LF-NMR) is an excellent noninvasive tool for analyzing water in food because of several advantages, such as non-requirement of sample preparation, being nondestructive, low cost, and rapidity. Relaxation times were measured to obtain information on strongly bound water, bound or hydration water, and weakly bound water in food (Damez and Clerjon, 2013; Pearce et al., 2011). Proton spin–lattice relaxation time (T_1) and spin–spin relaxation time (T_2) were used to measure certain water characteristics, such as water mobility (da Silva et al.,

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http://dx.doi.org/10.1016/j.jfoodeng.2017.05.021

2016), water distribution (Straadt et al., 2007), water activity (Katika et al., 2015), water-holding capacity (Gudjónsdóttir et al., 2011), water compartmentalization (Møller et al., 2010), and heterogeneity (Damez and Clerjon, 2013; Gudjónsdóttir et al., 2015). NMR is widely used in detecting water distribution, mobility, and content in beef (Pereira and Colnago, 2012), pork (Sørland et al., 2004), and fish (Gudjónsdóttir et al., 2015). Such studies are extremely important in understanding water characteristics of fresh meat. However, few studies were conducted to demonstrate application of NMR in fermentation. Limited information describe correlation between LF-NMR parameters and water content of fermented food.

The present study aimed to evaluate capability of LF-NMR in monitoring water content during sausage fermentation. Suitable LF-NMR parameters were explored to predict water content.

2. Materials and methods

2.1. Sausage preparation

Lean pork and pork fat were purchased from a supermarket (Sushi Group, Jiangsu, China). Natural-salt hog casings were obtained from Far-Eastern Casing Co. (Hebei, China). Lean pork and pork fat were minced (MG815S, Chulux Co., Shenzhen, China) to a particle size of 5 mm and mixed with salt (2%, w/w), sugar (2%, w/w), and wine (1%, v/w). Mixtures were stuffed in hog casings with a diameter of 2.2 cm and length of 10 cm. Sausages were fermented in an incubator (LHS150SC, Hengke Co., Shanghai, China) at 4 °C for 10 days.

2.2. NMR imaging analysis

NMR imaging measurements were acquired as proton density images using a NMI20-Analyst instrument (Niumag Co., Shanghai, China) on a 0.5 T magnet. Flip and refocus angles measured 90° and 180°, respectively. Three MR imaging (MRI) slices were analyzed in each sample with the following parameters: slice width = 3.0 mm, slice gap = 2 mm, repetition time = 500 ms, echo time (TE) = 20 ms, read size = 256, and phase size = 192.

2.3. LF-NMR measurements

LF-NMR measurement was performed on a 22.4 MHz NMR analyzer (PQ001, Niumag Co., Shanghai, China). Approximately 5 g of sausage samples were accurately weighed every two days and placed in a 2.5 cm-diameter glass tube inserted into the NMR probe. In this study, T_2 was measured using the Carr–Purcell–Meiboom–Gill (CPMG) sequence. Typical pulse parameters were as follows: spectral width = 100 kHz, spectrometer frequency = 22 MHz, time domain = 107,396, radio frequency delay time = 0.16 ms, pulse width at 90° (P_1) = 8.20 μs, pulse width at 180° (P_2) = 16.8 μs, waiting time = 4000 ms, number of scans = 8, number of echoes = 5,000, and TE = 0.215 ms. Relaxation measurements were performed at optimal operating temperature of 32 °C.

2.4. Determination of water content

After NMR T_2 relaxation measurements, water content of samples was determined by oven-drying 5 g of sausage samples at 105 °C for 12 h. Samples were weighed before and after drying. Water content was calculated as weight change percentage.

2.5. Statistical analyses

Statistical analyses were performed with one-way analysis of variance and Duncan's multiple range tests. Differences with p values < 0.05 were considered statistically significant.

The relationship between water content and LF-NMR parameters was analyzed statistically using general linear model (Equation (1)) procedure in OriginPro 8.5 (OriginLab, Northampton, MA, USA).

$$y = a + b*x. \tag{1}$$

Coefficient of multiple determination (R^2), bias factor (B_f), and

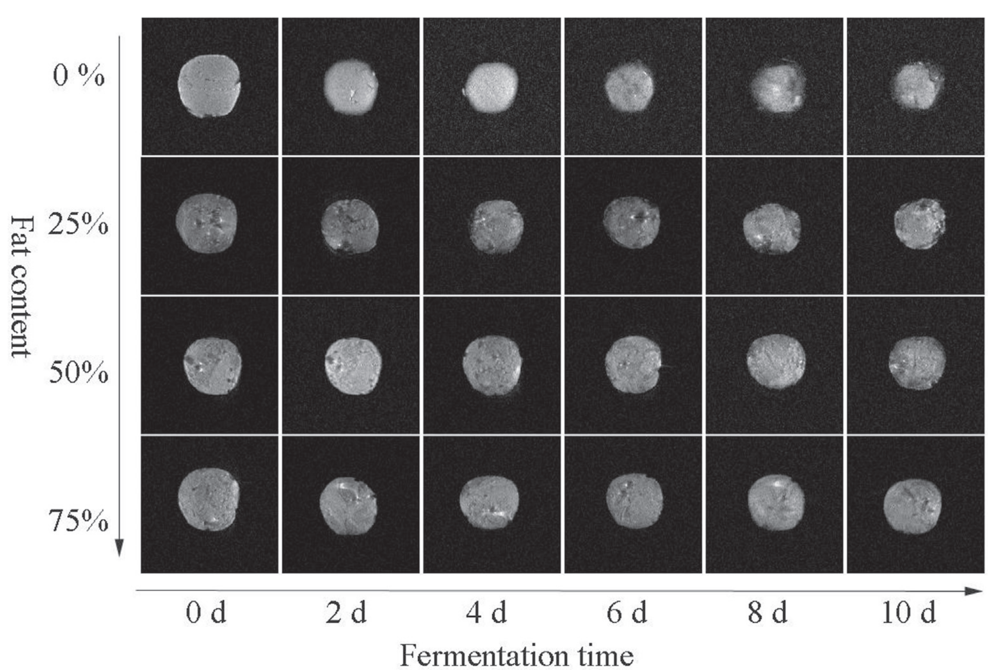

Fig. 1. NMR images of sausages measured during sausage fermentation.