



Caking characteristics and sensory attributes of ramen soup powder evaluated using a low-resolution proton NMR technique

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ABSTRACT

A low-resolution proton nuclear magnetic resonance (NMR) technique was used to characterize the quality of ramen soup powder during storage. The caking behavior of ramen soup powders was observed during 20 weeks of storage at different temperatures (30, 37, 45, and 55 °C) by monitoring changes in spin–spin relaxation times (T_2) using a CPMG (Carr–Purcell–Meiboom–Gill) pulse sequence. Water in ramen soup powder was classified into two fractions based on two spin–spin relaxation times, T_{21} and T_{22} , and their respective proton intensities, A_1 and A_2 . Increases in T_{21} and T_{22} were observed during storage at 37, 45, and 55 °C, which indicated that the molecular mobility of water in ramen soup powder increased. The changes in A_1 and A_2 suggested that there was a redistribution of water from a lesser “bound” water state to more mobile state during storage. T_{21} and A_1 were critical factors for explaining the caking characteristic of ramen soup powder during storage. Also, the results showed that sensory attributes of ramen soup powder deteriorated during storage, indicating that caking could accompany changes in organoleptic qualities and that these changes could be detected by monitoring changes in NMR parameters.

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1. Introduction

Caking of powdered products occurs due to water redistribution or absorption during processing and storage. Unwanted moisture redistribution in these materials may cause undesirable changes in quality and long-term stability. Several processes take place within the powders before caking actually occurs. Inter-particle bridging, the initial stage of caking, results from surface mobilization by water, agglomeration results from the consolidation of bridges, and compaction results from the thickening of inter-particle bridges, which produces caking (Aguilera, del Valle, & Karel 1995; Chung et al. 2000). There are many factors that influence the rate of caking, including chemical composition, moisture content, relative humidity, environment temperature, and water activity of the food system (Chen & Wang 2006; Walker et al. 1998). The water activity is a measure of the “availability” of water. Water activity has been a principal guideline for the safety and quality control of food, biological, and pharmaceutical products (Schwimmer 1980; Troller 1980). The “availability” of water refers to how “freely” water molecules can participate in reactions or how easily water molecules diffuse to the reaction sites to participate in the reactions (Lai & Schmidt 1990). The relationship between water activity and deteriorative chemical reactions and other factors determining quality has been investigated (Nelson & Labuza 1994; Schwimmer 1980; Troller 1980). An increased water activity that causes caking of foods not only

increases lipid oxidation, enzymatic activity, and microbial growth, but also results in a deterioration of sensory qualities. The Maillard reaction is one of the chemical reactions causing the quality deterioration of powder during the processing and storage of foods (Malec, Pereyra Gonzales, Naranjo, & Vigo 2002). Lea and Hannan (1949) found that the reaction rate of non-enzymatic browning in milk powder was increased as the water activity increased up to 0.6–0.7. The onset of the Maillard reaction spoils the appearance and flavor of powdered food. Stapelfeldt, Nielsen, and Skibsted (1997) have reported that the change in the color of milk powder with storage temperature was smaller at lower water activities, and increased with the water activity and storage temperature. Also, the off-flavor from the Maillard reaction was increased with the storage temperature. Consequently, higher temperature and water activity expedite non-enzymatic browning and adversely affect the sensory quality. In particular, a poor organoleptic quality increases consumer doubts about food safety and reduces their confidence in food products (Castro, Motizuki, Murai, Chiu, & Silva 2006; Chen & Wang 2006; Chung et al. 2000).

Nuclear magnetic resonance (NMR) spectroscopy provides a rapid, sensitive, noninvasive, and nondestructive determination of not only the quantity of water present, but also the structure and dynamic characteristics of water in foods (Borompichaichartkul, Moran, Srzednicki, & Price 2005; Richardson & Steinberg 1987). The ^1H nucleus, especially the proton, is the most commonly used nucleus in NMR techniques (Pitombo & Lima 2003). There are two relaxation parameters that can be detected by NMR techniques. The relaxation behavior is commonly characterized by the spin–spin (T_2) and spin–

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lattice (T_1) relaxation times. Relaxation times are analyzed to determine changes in molecular mobility (Kim & Cornillon 2001). Our previous studies using NMR techniques (Chung & Ruan 2002a; Chung et al. 2000, 2001, 2003; Wee, Choi, & Chung 2007) have shown that caking of food powders is closely related to their physical state transition and molecular mobility as measured by NMR techniques, and the resistance or sensitivity of food powders to caking varies with their state transition and molecular mobility properties. The studies also demonstrated that the rate of caking is mainly governed by $\Delta T = T - T_g$, where ΔT is the difference between the storage temperature (T) and the glass transition temperature (T_g), and this equation indicates that the product will cake at different rates, depending on how far above the storage temperature is from the T_g . The NMR technique can also be used to determine the quantitative relationship between storage temperature or period and the molecular mobility for each powder (Chung & Ruan 2002b).

In this study, the effects of storage conditions (storage period and temperature) on caking were determined by monitoring NMR parameters, and the relationships between caking characteristics measured using an NMR technique and sensory qualities were analyzed.

2. Materials and methods

2.1. Materials and storage conditions

The samples used in this study comprised 10 g of instant ramen soup powder (Ottogi Co., Anyang, Gyeonggi, South Korea). The recipe of ramen soup powder was listed in Table 1. Each sample was packed in an aluminum laminating film (Ottogi Co.) which offers superior barrier to water vapor and total water vapor transmission rate is almost zero during the 20 week-storage period. In order to investigate changes in the caking characteristics of ramen soup powder during storage, the samples were kept for 5 months (20 weeks) in incubators maintained at temperatures of 30, 37, 45, and 55 °C. The water activity of each sample was measured every second week (AW SPRINT TH500, Novasina, Lachen, Schwyz, Switzerland). Water activity measurements were performed at an ambient temperature (25 °C) in triplicate for each sample.

2.2. NMR experiments

The proton relaxation measurements were performed using a low-resolution proton NMR device (minispec mq20, Bruker BioSpin,

Karlsruhe, Germany) operating at a resonance frequency of 20 MHz (^1H) and a temperature of 40 ± 0.1 °C. Duplicate samples stored at the storage temperature were removed from incubators maintained at 30, 37, 45, and 55 °C every week during the 20 weeks storage period. Approximately 1.5 g of ramen soup powder stored at each condition was added to a vial (8 mm in diameter and 45 mm long). The vial was placed in a 9-mm-diameter cylindrical tube, which was then covered with a cap to limit moisture adsorption and loss. The experiments were performed three times for each sample.

The spin–spin relaxation time constant (T_2) was determined using the Carr–Purcell–Meiboom–Gill (CPMG) sequence, comprising a 90° pulse at a specific radio frequency, followed by a series of pulses in 180° phase, with the following parameters: $P90 = 9.42 \mu\text{s}$, $P180 = 19.54 \mu\text{s}$, RD (recycle delay between scans) = 1.50 s, DT (delay between the pulse and data acquisition) = 0.009 ms, and NS (number of scans) = 4. The T_2 experiment was designed to characterize and quantify the physical states of water in ramen soup powders. Two transverse relaxation time constants, T_{21} and T_{22} , and their respective maximum amplitudes, A_1 and A_2 , were measured. The values were estimated from free induction decay (FID) curves obtained by NMR experiments and it was found that total amplitudes (A_T) at a certain time (t) follow a bi-exponential fitting model as following equation:

$$A_T = A_1 \cdot \exp(t / T_{21}) + A_2 \cdot \exp(t / T_{22}).$$

2.3. Sensory evaluation

The samples used in this study comprised the nine types of stored ramen soup powder listed in Table 2. A panel comprising seven females recruited from the Department of Food Science and Engineering at Ewha Womans University (Seoul, South Korea) compared the degrees of caking of the stored ramen soup powders. The sensory attributes considered in the evaluation were brownness, hardness, and off-flavor, which are defined as an intensity of brown color, a softness/hardness as determined by fingers, and any atypical flavor, respectively. All the attributes were evaluated on 15-point category scales labeled with the words “weak” for 1 point and “strong” for 15 points. The samples were first placed in a light box to score brownness, and then they were put in individual booths and the panel members were instructed to push the ramen soup powder with their fingers to assess the hardness. Finally, to evaluate the off-flavor attribute, each ramen soup powder sample was grounded by a mortar and pestle and then about 20 g of the sample was placed in brown vials. The panelists were not permitted to use lotion or perfume, or touch the vials, which could have affected the off-flavor score. All tests were performed in duplicate. Analysis of variance was performed to evaluate the significance of differences among samples. Duncan's multiple range test was performed to examine significant differences ($P < 0.05$) of sensory attributes among samples. The statistical analyses were conducted using SPSS for Windows 12.0 software (SPSS Inc., Chicago, IL, USA).

Table 1
Recipe of ramen soup powder.

Ingredient	Percentage by weight
Garlic powder	1.9
Hydrolyzed animal protein	6.2
Hydrolyzed vegetable protein (HVP)	1.3
Seasoning (HVP + lactose)	3.7
Beef extract powder-3	2.5
Powdered soy sauce	6.2
Seasoning (glucose + corn syrup)	5.0
Onion powder	5.0
Disodium succinate	0.6
Glucose	1.3
Beef extract powder-1	7.5
Beef extract powder-2	2.5
Red pepper	3.7
Corn powder	1.4
Black pepper	1.9
Seasoned red pepper	1.3
Dried green onion	1.4
Salt	28.0
Sucrose	9.3
Monosodium glutamate	9.3
Total	100.0

Table 2
Designations of ramen soup powders used in this study.

Designations	Storage temperature (°C)	Storage period (weeks)
RC	Not stored	Not stored
R30-1	30	10
R30-2	30	20
R37-1	37	10
R37-2	37	20
R45-1	45	10
R45-2	45	20
R55-1	55	10
R55-2	55	20

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