



Reveal the internal moisture changes of white gourd during hot air-drying process using low-field NMR

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Abstract

The moisture content, distribution and state changes of White gourd were studied during hot air-drying process using Low-field nuclear magnetic resonance (LF-NMR). The transverse relaxation time (T_2) inversion spectrum of White gourd was measured at different temperatures during drying process, and the characteristics of internal moisture state and change were analyzed using the LF-NMR. The results showed that T_2 was changed and the moisture of liquidity was reduced during drying process. In addition, the moisture with high degree of freedom changed to the moisture with low degree of freedom during drying. The amplitude of total NMR signal and the moisture content of drying base were a linear relationship during drying. The kinetics of the percentage of bound water and free water were based on exponential function and polynomial function respectively with drying time and different drying temperatures.

Keywords: white gourd; hot air drying; moisture; Low-Field Nuclear Magnetic Resonance (LF-NMR); transverse relaxation time (T_2).

Practical Application: LF-NMR was used to determine the changes of water content, distribution and state of wax gourd in the process of reheating and drying, which provided a technical basis for the drying process research and actual production control of white gourd.

1 Introduction

White gourd (*Benincasa hispida* (Thunb.) Cogn.) belongs to the Cucurbitaceae, which was widely distributed in the tropical, subtropical, and temperate areas of Asia. It has antioxidant, anti-obesity, anti-ulcer, reducing blood pressure and many other effect (Zaini et al., 2011). White gourd, a natural product of high quality, was used as medicine as well as food for a long time and has more and more consumers (Wang et al., 2022). In China, currently the White gourd was mainly used for fresh food. White gourd was one of the principal vegetables in summer and fall with high yield, easy growth and good economic returning characteristics.

Low field nuclear magnetic (LF-NMR) utilized the hydrogen nucleus spin relaxation characteristics in the magnetic field (Su et al., 2022). It could be used for investigating the moisture distribution changing and the moisture transferring in the sample utilizing the changes of relaxation time (Hansen et al., 2010; Aursand et al., 2008). Recently, LF-NMR has been used in the area of food science extensively because it was a simple, quick, and non-destructive means (Balthazar et al., 2021). For example, LF-NMR has been successfully applied in understanding of the moisture state and its existing in the process of freezing-thawing cycles from meat (Molina-García et al., 2004), applied in understanding of the moisture mobility and distribution in the process of vacuum cooling of bean products Guan (Guan et al., 2011), applied in understanding of the water relaxation and diffusion during the ripening of banana (Raffo et al., 2005), also

applied in understanding of the variation of moisture state and its existing during the grain drying process (Hwang et al., 2009; Ghosh et al., 2006) and the characterization of water movement during bread storage and for investigating the forms of water and its changing rule for other kinds of food (Hills & Nott, 1999; Micklander et al., 2008). However, it has not been widely applied in White gourd drying.

The moisture content of White gourd was more than 90%, which resulting in perishable and losing economic values. Dehydration drying was a kind of method which could effectively control moisture content and extend the preservation period. By controlling the water mobility and reducing the moisture content, it could prevent product from spoilage and deterioration (Labuza & Hyman, 1998). The characteristic of water distribution and migration during the process of hot air drying of White gourd was studied, which had important meaning for industrialization and preserving.

During the drying process of fruits and vegetables, a series of physical and chemical changes occurred. In general, the form of moisture in fruits and vegetables has three states: free water, immobilized water and bound water (Vicente et al., 2012). The transversal relaxation time (T_2) inversion graphs was measured using the Carr-Purcell-Meiboom-Gill sequence (CPMG). Using the T_2 inversion graphs at different state of water could be divided, which allowed to analyze of quantitative samples.

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In this manuscript, T_2 inversion graphs of White gourd during drying process were studied by the LF-NMR. And the changes of moisture content, moisture distribution and moisture state in White gourd were analyzed under different temperatures. This study provided a theoretical foundation for the further study on the industrialization and the storage of White gourd.

Drying was a process of heating wet materials to vaporize water (Moloto et al., 2021). Due to the high moisture content of White gourd, water and heat were transmitted mutually during drying process (Datta, 2007), which led to a series of physical, biological, and chemical reactions. Furthermore, the drying process caused the shrinkage of the materials, the hardening of the surface of white gourds, and the degradation of the available nutrients, which led to a decline in product quality. Also, drying caused uneven water distribution of the interior materials which affected the quality of the final products and energy consumption. Therefore, understanding the interior water distribution in the process of drying and transmitting is important and significant for producing high quality products and also was an effective method to reducing energy consumption.

2 Materials and methods

2.1 Sample preparation

Fresh White gourd purchased from a market in Henan Province was washed, peeled, seeded and cut into cuboids at $0.5 \text{ cm} \times 0.5 \text{ cm} \times 2 \text{ cm}$. The White gourd cuboids were spread in a single layer in a blast drying oven. The hot air drying of White gourd was carried out at different temperatures ($50 \text{ }^\circ\text{C}$, $60 \text{ }^\circ\text{C}$, $70 \text{ }^\circ\text{C}$, $80 \text{ }^\circ\text{C}$ and $90 \text{ }^\circ\text{C}$). The samples were taken out from the oven and T_2 was analyzed at predetermined time intervals of 15 min continuously throughout the drying process until the moisture content reduced to 5%. To ensure the maximal consistency and reliability of the data, the shape of sample must have the consistent of the weight.

2.2 NMR T_2 measurements

T_2 measurements were performed on NMR Analyzing system (PO001, Niumag Corporation, Shanghai, China) with a resonance frequency for protons of 22.7 MHz at $32 \pm 0.02 \text{ }^\circ\text{C}$ (Li et al., 2012). All the samples were accurately weighed, wrapped with plastic film and placed into a cylindrical glass tube (18 mm in diameter). The T_2 was measured using the Carr-Purcell-Meiboom-Gill sequence (CPMG) with the following parameters: P90 = 18 μs ; P180 = 35 μs ; τ = 450 μs ; TD = 2700090; SW = 200 KHz; D3 = 60 μs ; TR = 10000 ms; RG1 = 20; RG2 = 3; NS = 4; Echo Count = 15000.

2.3 Determination of the moisture content of drying base

The White gourd cubes were spread in a single layer in a blast drying oven dried at $50 \text{ }^\circ\text{C}$, $60 \text{ }^\circ\text{C}$, $70 \text{ }^\circ\text{C}$, $80 \text{ }^\circ\text{C}$ and $90 \text{ }^\circ\text{C}$, respectively. The samples were taken out from the oven and the weight was analyzed at predetermined time intervals of 15 min continuously throughout the drying process until the moisture content reduced to 5%. The moisture content of drying base was measured according to $105 \text{ }^\circ\text{C}$ oven drying method:

The moisture content of drying base (Equation 1) (Kekkonen et al., 2014).

$$(\omega) = (m_t - m_g) / m_g \quad (1)$$

Where m_t and m_g represent the material absolute dry mass and the material dry mass at time t, respectively.

2.4 Statistical analysis

All the tests were run in triplicate. Data were reported as mean \pm standard deviation. The data were analyzed using the General Linear Models procedure of Statistics 17.0 software package. Analysis of variance (ANOVA) was performed to determine the significance of the main effects. Significant differences ($p < 0.05$) between means were identified using Tukey procedures.

3 Results and discussion

3.1 The relationship between NMR total signal amplitude and the moisture content of drying base

Figure 1 was the T_2 inversion graphs of fresh White gourd. According to the LF-NMR, White gourd T_2 spectrum had three peaks after inversion. The three peaks were characterized by three distinct water populations: T_{21} (0.1 ~ 10 ms), T_{22} (10 ~ 100 ms), and T_{23} (100 ~ 1000 ms), corresponding to the three states of water (bound water, immobilized water and free water, respectively) in White gourd. Previous research has shown that the T_2 relaxation time was positively related to the water mobility (Vicente et al., 2012; Straadt et al., 2008). Characteristics of the current peaks were similar to the previous research findings. The lower the T_2 was, the lower the freedom of moisture had, which was more tightly bound. On the other hand, the higher the T_2 was, the more freedom of moisture had, which was more easily removed. Moreover, the changes of the proportion of T_{21} , T_{22} and T_{23} peak area indirectly reflected the moisture content in different states (Pearce et al., 2011), which could be expressed by S_{21} , S_{22} , S_{23} , respectively.

The total signal amplitude of NMR had a positive correlation with the number of hydrogen proton in the sample (Lu & Seetharaman, 2013). Therefore, the signal amplitude could indirectly represent the relative amount of the moisture content during the drying

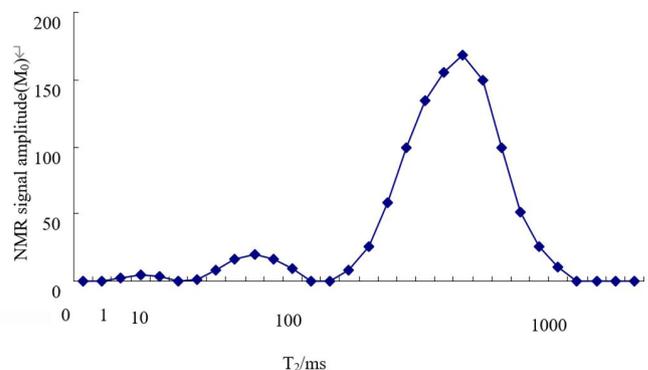


Figure 1. NMR inversion spectrum of T_2 of White gourd.

process. In the current research, the corresponding moisture content of drying base of White gourd at different drying stages and the total signal amplitude of NMR were linearly fitted with each other. The fitting curve, which demonstrates the moisture content of drying base in White gourd linearly corresponded to the total NMR signal. The result was consistent with potato tubers (Hansen et al., 2010). The regression equation by SPSS 13.0 software was $y = 30.399x - 4.986$, and the corresponding coefficient of R^2 was 0.9937, with a significant level α smaller than 0.01.

3.2 Changes of white gourd moisture state and content at different drying temperatures and the drying kinetics of white gourd

The changes of white gourd moisture state and content at different drying temperatures

During the drying process of White gourd, the proportion of different states of water peak area presented a dynamic balance. Illustrated that the percentage of the bound water peak area changed with different drying temperatures during the drying process of White gourd (Figure 2a). Because the bound water

was a stable chemical substance present in fruit and vegetable, which was difficult to evaporate (Tang & Li, 2013; Colosimo et al., 2020). From Figure 2, with the extending of drying time, the percentage of bound water in the total moisture content was exponentially increased. In the work of Li et al. (2014), the free water also dropped significantly, the reason for increasing bound water in the current study was that the free water was evaporated gradually and the total content of moisture decreased with the extension of drying time, resulting in the percentage of bound water content continuously increasing with the increasing speed slower in the beginning and later faster. When the dry temperature was 50 °C, 60 °C, 70 °C, 80 °C and 90 °C, respectively for 240 min, 210 min, 180 min, 165 min and 135 min most of the moisture content was bound water.

The proportions of immobilized water peak area of the dried White gourd at different drying temperatures were shown in Figure 2b. Immobilized water was a kind of moisture, which adsorbed onto the surface of colloidal in plant tissues. Before the free water was evaporated, the immobilized water was more stable. The current results showed that the percentage of immobilized water content increased firstly, and then decreased with the extension of drying time. With the extension of the drying time,

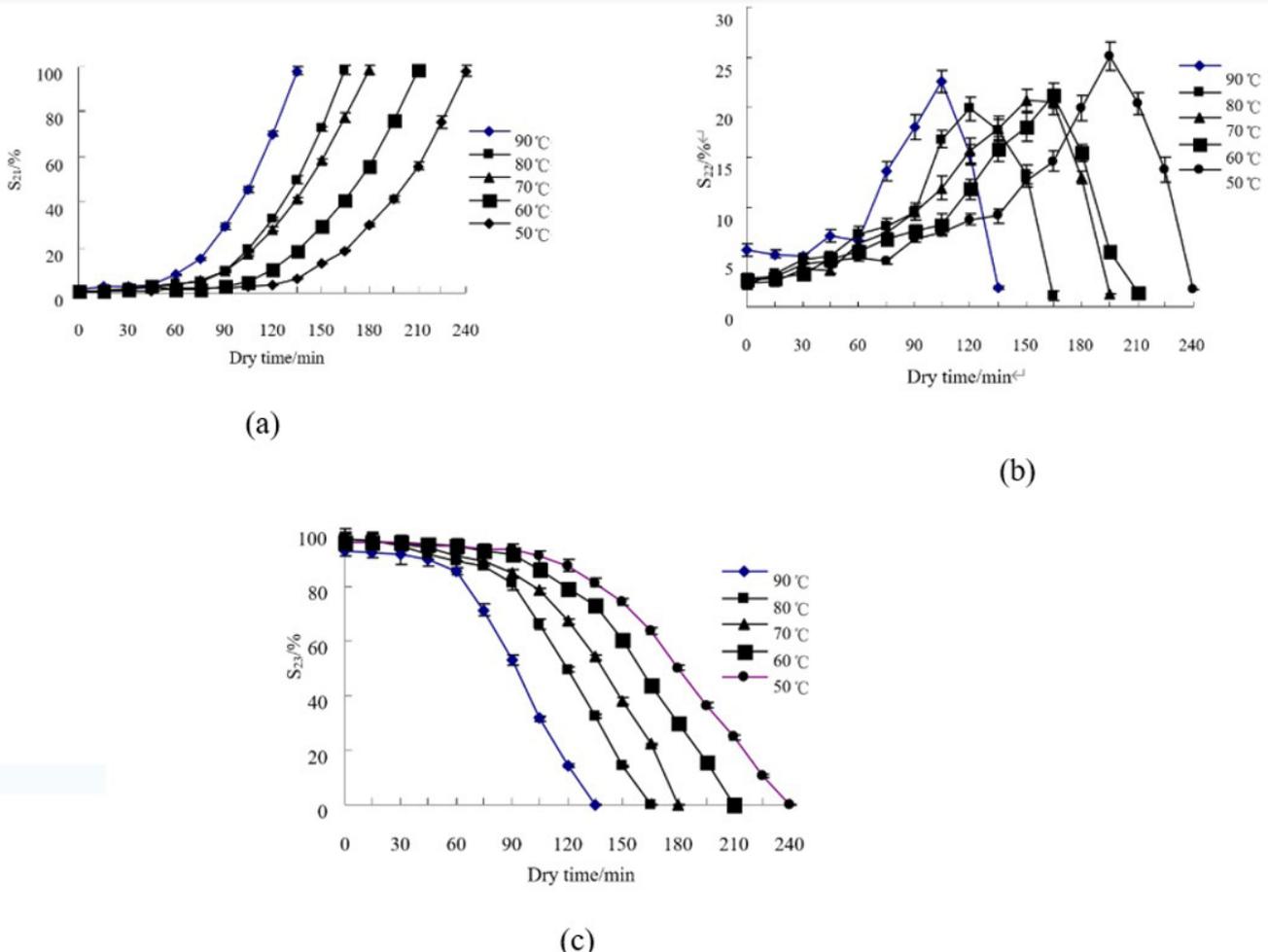


Figure 2. The proportions of different water states [S_{21} (a), S_{22} (b) and S_{23} (c)] of white gourd during drying at different temperatures.

the concentration of carbohydrates increased and as a result the transition from free water to immobilized water occurred. On the other hand, since a series of biochemical reactions occurred inside the material Zhang (Zhang et al., 2012), the migration of moisture through material assumed to be permeable and mainly of bound water and immobilized water by the action of pressure gradient, causing the proportion of immobilized water increased. However, after most of the free water was removed, a large amount of immobilized water rapidly evaporated and only a small part of immobilized water transformed to bound water, leading to immobilized water content significantly increased.

The free water had fluidity of water solution, and was easily evaporated (Figure 2c) (Hatakeyama & Hatakeyama, 2017). Fresh White gourd contained a large amount of free water. During the drying process, the higher the air temperature was, the more the heat transferred, and the faster water evaporated, thus the free water content decreased significantly. As seen from Figure 2, with the extension of drying time at different drying temperature conditions, S_{23} (free water) showed a decreasing trend, and the initial stage of drying down at a slower speed, faster rate of decline later to completely removal free water, The time of 240 min, 210 min, 180 min, 165 min and 135 min were needed under the drying temperature of 50 °C, 60 °C, 70 °C, 80 °C and 90 °C, respectively.

Kinetic models and parameters for white gourd moisture state and content at different drying temperatures

According to the proportion of different water states peak area (S_{21} and S_{23}) within the dried White gourd at different drying temperatures, the dynamic model of S_{21} and S_{23} with drying time could be established. It was found that the variation trend of S_{21} was a typical exponential form, and the variation of S_{23} was a typical polynomial form. By using the Origin 9.0 software, the curves of S_{21} and S_{23} were fitted, and the regression equations and parameters of S_{21} and S_{23} with different drying time were obtained. The interrelation parameters of S_{21} and S_{23} under different drying temperatures were shown in Table 1.

From Table 1 the kinetics models of S_{21} and S_{23} with the drying time grown were accorded with exponential function and polynomial function, respectively, at different drying temperatures. The coefficient of determination (R^2) of the

regression equation of S_{21} and S_{23} with different drying time was more than 0.95, which indicated that the regression equation has higher fitting precision.

3.3 Changes of T_2 at different drying conditions

T_2 of the bound water (T_{21}) at different drying conditions

Different drying conditions combined with the trend of White gourd transverse relaxation time was shown in Table 2. Stood for the T_2 of the bound water (T_{21}), the immobilized water (T_{22}) and the free water (T_{23}) in the White gourd dried at 50 °C, 60 °C, 70 °C, 80 °C and 90 °C, respectively (Table 2). In the whole process of drying, with longer drying time under different temperature conditions, T_{21} value decreased. In addition, the results at 50 °C dried for 2.5~3.0 h, and at 60 °C dried for 1.0~1.5 h illustrated that the changes of the bound water mobility were significantly different ($p < 0.05$). This phenomenon seemed to be similar to some previous observations (Lin et al., 2006; Mortensen et al., 2005) which showed faster relaxation times (T_{21} and T_{22}) for potato samples with high drying matter content than samples with low drying matter content.

The reason was that during the drying process, the internal temperature of the material formed a gradient. Consequently, some bound water which had a relatively large fluidity transferred to the immobilized water. Furthermore, with the extending drying time, the nutrient and enzyme in dry matter were decomposed, so that the partial bound water that changed to immobilized water caused the T_{21} to gradually decrease. Meanwhile, the drying temperature had a certain extent effect on the T_2 of the bound water (T_{21}) in the material. The higher the temperature, the faster the decomposition of the material, the more severely the water migration, which caused degree of freedom of the bound water to increase rapidly.

T_2 of the immobilized water (T_{22}) at different drying conditions

Illustrated the T_2 characteristics of immobilized water (T_{22}) at different drying conditions (Table 3). At the same drying temperature, T_{22} decreased significantly with the extension of drying time. However, T_{22} changed differently with increasing drying temperature but at the same drying time. The reason was that a portion of immobilize water was more tightly integrated

Table 1. The moisture states and moisture content kinetic models for white gourd at different drying temperatures.

Moisture state	Regression equation	Drying temperature/°C	Parameters			Adjusted R ²
			a	b	c	
S_{21}	$y = ab^x$	90	1.199	1.03378		0.97679
		80	0.5591	1.03281		0.97486
		70	0.787	1.02816		0.97074
		60	0.42804	1.0269		0.96037
		50	0.23204	1.0261		0.94475
S_{23}	$y = ax^2 + bx + c$	90	-0.00685	0.19278	93.25145	0.98523
		80	-0.00506	0.24147	94.89962	0.99098
		70	-0.00441	0.29404	93.10879	0.99229
		60	-0.00353	0.30078	92.36529	0.9935
		50	-0.00278	0.27246	92.11099	0.99194

Table 2. Effect of T_{21} (ms) of white gourd dried at different conditions.

Drying time (h)	Drying temperature/°C				
	50	60	70	80	90
0,5	9.79 ± 0.28 ^{aA}	9.35 ± 0.28 ^{aB}	9.45 ± 0.27 ^{aB}	9.39 ± 0.24 ^{aB}	8.95 ± 0.23 ^{aC}
1	6.90 ± 0.38 ^{bA}	6.49 ± 0.19 ^{bA}	6.19 ± 0.19 ^{bC}	5.62 ± 0.07 ^{bB}	6.92 ± 0.07 ^{bA}
1,5	5.65 ± 0.27 ^{cB}	6.44 ± 0.16 ^{bA}	3.70 ± 0.20 ^{cC}	3.48 ± 0.18 ^{cC}	2.74 ± 0.22 ^{cD}
2	5.08 ± 0.19 ^{dA}	5.32 ± 0.14 ^{cA}	3.21 ± 0.11 ^{dB}	2.46 ± 0.17 ^{dC}	1.53 ± 0.12 ^{dC}
2,5	4.64 ± 0.06 ^{eA}	4.48 ± 0.11 ^{dA}	2.68 ± 0.24 ^{eB}	1.68 ± 0.27 ^{eC}	
3	4.44 ± 0.20 ^{eA}	3.64 ± 0.18 ^{eB}	1.66 ± 0.16 ^{fC}		
3,5	2.40 ± 0.16 ^f	1.79 ± 0.17 ^f			
4	1.05 ± 0.05 ^g				

Different lowercase letters in the same column indicate significant differences ($p < 0.05$); different capital letters in the same row indicate significant differences ($p < 0.05$).

Table 3. Effect of T_{22} (ms) of white gourd dried at different conditions on white gourd.

Drying time (h)	Drying temperature/°C				
	50	60	70	80	90
0,5	47.46 ± 1.42 ^{cC}	49.17 ± 0.68 ^{aBC}	42.13 ± 0.45 ^{dD}	52.32 ± 1.83 ^{aAB}	51.16 ± 0.27 ^{aA}
1	38.55 ± 0.92 ^{bB}	40.53 ± 0.40 ^{bA}	38.09 ± 0.58 ^{bB}	40.28 ± 0.44 ^{bA}	38.45 ± 0.52 ^{bB}
1,5	34.42 ± 1.35 ^{cB}	36.24 ± 0.39 ^{cA}	33.21 ± 0.54 ^{cB}	31.80 ± 0.41 ^{cC}	21.67 ± 0.37 ^{cD}
2	31.26 ± 0.33 ^{dB}	33.14 ± 0.99 ^{dA}	30.46 ± 1.16 ^{dB}	23.40 ± 0.62 ^{dC}	13.29 ± 0.64 ^{dD}
2,5	30.64 ± 0.31 ^{dA}	30.95 ± 1.37 ^{eA}	28.58 ± 1.68 ^{eA}	12.28 ± 0.40 ^{eB}	
3	26.16 ± 0.39 ^{eA}	20.98 ± 0.89 ^{fB}	7.41 ± 0.80 ^{fC}		
3,5	19.48 ± 0.58 ^f	11.36 ± 1.22 ^g			
4	8.45 ± 0.35 ^g				

Different lowercase letters in the same column indicate significant differences ($p < 0.05$); different capital letters in the same row indicate significant differences ($p < 0.05$).

Table 4. Effect of T_{23} (ms) of white gourd dried at different conditions on white gourd.

Drying time (h)	Drying temperature/°C				
	50	60	70	80	90
0,5	853.16 ± 10.34 ^{aB}	931.64 ± 8.37 ^{aA}	943.82 ± 13.03 ^{aA}	745.69 ± 8.46 ^{aD}	813.80 ± 2.72 ^{aC}
1	752.22 ± 5.52 ^{bC}	921.51 ± 3.44 ^{aA}	762.89 ± 3.33 ^{bB}	655.56 ± 3.34 ^{bE}	672.59 ± 6.65 ^{bD}
1,5	731.53 ± 1.84 ^{cB}	903.29 ± 3.91 ^{bA}	706.62 ± 3.47 ^{cC}	611.39 ± 6.35 ^{cD}	286.01 ± 7.24 ^{cE}
2	686.27 ± 4.71 ^{dB}	807.00 ± 7.30 ^{cA}	335.07 ± 5.57 ^{dC}	255.19 ± 2.40 ^{dD}	156.61 ± 6.45 ^{dE}
2,5	647.57 ± 6.70 ^{eB}	674.34 ± 8.53 ^{dA}	249.16 ± 8.58 ^{eC}	149.80 ± 5.23 ^{eD}	
3	442.03 ± 4.08 ^{fB}	479.66 ± 3.65 ^{eA}	144.64 ± 3.20 ^{fC}		
3,5	348.29 ± 6.44 ^g				

Different lowercase letters in the same column indicate significant differences ($p < 0.05$); different capital letters in the same row indicate significant differences ($p < 0.05$).

with colloid and its freedom reduced in the drying process 2005; Tananuwong (Mortensen et al., 2005; Tananuwong & Reid, 2004). In addition, most of the immobilized water which migrated into free water was removed.

T_2 of the free water (T_{23}) at different drying conditions

Influences of T_2 of free water (T_{23}) on White gourd were shown in Table 4. The results showed that T_{23} decreased with the extension of drying at the same drying temperature. The mobility of the free water changed significantly ($p < 0.05$), which agreed well with the meat drying research (Li et al., 2014). The results indicated that the mobility of free water decreased in the drying process, with some free water converted to immobilized water.

During the drying process, the mobility of free water was poor, and the mobility was not enhanced. There were some differences in T_{23} at the same drying time and drying temperature. The higher the drying temperature, the faster the water loss, the worse the mobility of the proton, the shorter the relaxation time, and the worse the free flow.

4 Conclusions

In this study, the internal moisture changes of White gourd during hot air drying were analyzed by LF-NMR. The T_2 inversion spectrum and its corresponding data were obtained by CPMG. And the proportion of three water states and the relaxation time under different drying temperatures were analyzed. The results showed that during the hot air drying of White gourd

the moisture content of drying base was significantly correlated with total signal amplitude of NMR. The changes of S_{21} and S_{23} with drying time at different drying temperatures were fitted by exponential function and polynomial function, respectively. The dynamic models had correlation coefficient greater than 0.95, thus having high fitting precision. T_2 decreased significantly and the fluidity of water reduced with extension drying time at various drying temperatures, indicating the internal moisture content in White gourd with high degree of freedom converted to the moisture content with low degree of freedom.

Based on the dynamic model of the moisture variety, the moisture distribution and moisture content of White gourd could be accurately predicted at various drying temperatures during hot air drying. The models allowed real-time monitor of the content of free water and bound water contained in White gourd by using the drying time. The models also provided a theoretical basis for the optimal design of White gourd hot air-drying technique and process control. Moreover, the moisture migration mechanism of White gourd during drying was revealed, which could offer help to further study on the storage of the product.

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