Study on the mobility of water and its correlation with the spoilage process of salmon (*Salmo solar*) stored at 0 and 4 °C by low-field nuclear magnetic resonance (LF NMR ^1H)

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Abstract Low-field nuclear magnetic resonance (LF NMR) was used to investigate the water mobility of salmon during cold storage and the correlation between texture, freshness, sensory quality and transversal relaxation times (T_2) of salmon were studied. With the increasing of cold storage time, trapped water (T_{22}), sensory, water holding capacity and cooking loss were descended while free water (T_{23}), TVB-N and TBA were increased steadily, that reflected the quality of salmon quality visually. There was a significant correlation between sensory, hardness, TBA, cooking loss, K value and LF NMR parameters. The study showed that LF NMR was sensitive to different storage conditions which may be applied to monitor the quality of fish muscle, when the spoilage mechanism was affected by water properties and muscle structure.

Keywords LF NMR · Water mobility · Quality · Cold storage

Introduction

Fresh Atlantic salmon (*Salmo salar*), belonging to the family Salmonidae, is one of the most traded and consumed fish products worldwide (Carlson and Hites 2005). Salmon is prone to deterioration with limited shelf life during cold storage because of physiochemical, enzymatic and microbial activity, which may lower the market value significantly (Zhou and Ackman 2003). Deterioration of the muscle can changes the protein structure and affect the mobility of water eventually (Sone 2012). Therefore, monitoring the mobility of water might be a prospective way to detect the quality of fish during storage (Gudjónsdóttir et al. 2011a, b).

Water is a significant component in aquatic product, which is essential for chemical and enzymatic reactions and microbial growth. Therefore, it has a direct effect on the stability of quality attributes of food during storage (Carneiro et al. 2015). Water is also the degradation product of muscle proteins. According to the mobility and the link between the molecules and the muscle structure, water can be separated into different groups (Anderson and Rinnan 2002). The low-field nuclear magnetic resonance (LF NMR) technique has been widely applied to gain further insight into the behavior of water molecules in muscle tissues (Pearce et al. 2011). As a non-destructive and non-invasive spectroscopic techniques, LF NMR requires only minimal or no sample preparation, which can investigate changes in the mobility of water in fish muscle during storage by detecting the relaxation of hydrogen protons (Gudjónsdóttir et al. 2011a, b; Sánchez-alonso et al. 2012). LF NMR provides constant relaxation times T_1 (Longitudinal) and T_2 (Transverse) which can reflect the mobility of water molecular (Aursand et al. 2008). T_2 transversal relaxation times measured by LF NMR are

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reported to represent three different populations of water in muscle: strong bound water; bound or hydration water and free or weakly bound water (Bertram et al. 2001). Weakly bound water, hydration water and free water, accounting for 90% of the water found in meat, are the most important variables during the cold storage and have a great significance in the study of food storage (Carneiro et al. 2015). Carneiro et al. (2015) studied the water mobility in muscle of salted sardine on different days of storage, by the use of low-field nuclear magnetic resonance. Physical and chemical analyses revealed a close correlation ($p < 0.05$) between quality parameters and the relaxation data. Previous studies shows that water is closely related to chemical reaction in food, including protein denaturation, fatty acid hydrolysis, enzyme activity change and starch gel, as well as rheological property such as viscoelastic (Horigane et al. 2013). Sánchez-alonso et al. (2014) obtained T_2 transversal relaxation times of hake muscle measured by NMR after different freezing and storage conditions, found that T_2 accurately reflect the quality changes of fish muscle. Shumilina et al. (2015) used high resolution NMR technique to monitor post-mortem changes in salmon fillets during storage at 4 and 0 °C. The data from this study shows that NMR spectroscopy provides the amount of all metabolites necessary for the calculation of the K-index used to express fish freshness, and a good correlation was found between the K-index increase and the formation of the undesired biogenic amines.

Although the quality changes of cold storage fish and water distribution of fish during different storage conditions using LF NMR were studied (Sánchez-alonso et al. 2012, 2014), quality changes is not yet compared with water distribution of salmon. Therefore, there is a great need for a correlation analysis between water distribution and texture, freshness and sensory quality of salmon during cold storage. The study was aimed to investigate the effects of storage temperatures on the quality of salmon examined with regard to physicochemical and spoilage indicators, demonstrate the possibility of NMR spectroscopy to monitor distribution and mobility of water that take place in salmon during cold storage, and find a rapid method to analyze the quality change of salmon.

Materials and methods

Sample preparation

About 6 kg of Salmon (*S. salar*) were obtained from aquatic products wholesale market of Tongchuan Road. The gutted fish were transported to laboratory in perforated polystyrene boxes with ice in 2 h. After washing in distilled water, the fish were cut and the steaks were divided in

pieces of around 200 g and placed in separate plastic bags. The samples were divided into two groups randomly and stored at 4 and 0 °C, respectively. Sampling was carried out every 2 days.

Sensory evaluation

The sensory evaluation of samples was conducted according to the freshness grade guide (Meilgaard et al. 1999). A ten-member panel, comprised of the graduate students was asked to evaluate the organoleptic quality of the samples based on 5-point hedonic scales in descending order. The panelists were asked to be familiar with the rating scales beforehand. The total scores of four attributes including colour, odour, fleshy and tissue elasticity were added to represent the overall sensory quality. The acceptability was defined as having a sensory score of over 8.

Texture profile analysis

Hardness was performed by the use of a TA.XT Plus Texture Analyser (Stable Micro Systems, Ltd., Godalming, Surrey, UK). The fish sample was cut into small squares (10 mm × 10 mm × 10 mm). Each sample was compressed once using a cylindrical probe of 6 mm in diameter (P/6) on the platform under the following conditions: constant test speed, 1.0 mm/s; sample deformation, 50%. Six measurements were made for each sample in the same batch. The hardness of the fish samples was obtained and expressed as N/g.

Water holding capacity (WHC)

The water holding capacity of fish meat was determined according to the method of Gudjónsdóttir et al. (2013) with some modifications. In brief, the cutted fish meat were weighed precisely (about 2 g, recorded as M_1) and after centrifugation at 5000 rpm at 4 °C for 10 min, the sample was weighed precisely again (recorded as M_2). WHC (%) was calculated by the following formula:

$$\text{WHC (\%)} = \frac{M_1 - M_2}{M_1} \times 100\% \quad (1)$$

Aw

The Aw of each 1 g sample was measured using a water activity meter (Aqualab Series 4TE, Decagon Devices Inc., Pullman, WA, USA) at 23 ± 2 °C according to the method described by Fundo et al. (2015). Each batch was conducted in triplicate.

Cook loss

The raw fish samples were weighed precisely (recorded as C_1) and weighed again after cooking in hot water (80 °C) for 10 min (recorded as C_2) (Zell et al. 2010). The cook loss was calculated as the following formula:

$$\text{Cookloss (\%)} = \frac{C_1 - C_2}{C_1} \times 100\% \quad (2)$$

Thiobarbituric acid-reactive substances (TBARS)

Thiobarbituric acid-reactive substances (TBARS) was determined by the method of Zhu et al. (2016) with some modifications. Briefly, a 5 g muscle sample was dispersed in 25 ml 20% trichloroacetic acid then homogenized for 60 s. After incubation at 0 °C for 1 h, the mixture was centrifuged at 8000 rpm at 4 °C for 10 min. Five ml filtrate was mixed with 0.02 M 2-thiobarbituric acid and heated at 100 °C for 20 min. The TBARS content, expressed as mg malonaldehyde per kg muscle sample, was calculated with the following equation:

$$\text{TBARS (mg/kg)} = A_{532} \times 7.8 \quad (3)$$

where A_{532} is the absorbance at 532 nm.

Total volatile base-nitrogen (TVB-N)

The contents of TVB-N were determined using the FOSS method (2002). Samples were at exact dose of 5 g and massed, and then mixed with 0.5 g of magnesium oxide. The samples were distilled in a Kjelttec8400 apparatus, the distillate was transferred into a 25 g/L H_3BO_4 solution containing methyl red-bromocresol green mixed indicator and titrated with 0.01 M HCl. The results were expressed as mg of TVB-N per 100 g of salmon muscle.

K value

Nucleotide degradation was determined using high performance liquid chromatography (HPLC, SHIMADZU, LC-2010C HT, Kyoto, Japan) according to the method described by Kamalakanth and Ginson (2011) with slight modifications. Five kilograms of salmon meat was homogenized with 10 ml of 10% perchloric acid at 0 °C for 1 min and then centrifuged at 10,000 g for 15 min at 4 °C. The supernatant obtained from two-times centrifugation by adding 5% perchloric acid were poured into a flask, and the pH of the supernatant was adjusted to 6.5 using 10 M and 1.0 M KOH. After the final volume adjusted to 50 ml with distilled water, the solution was filtrated through a 0.45 μm membrane before use. Compounds were identified and quantified based on the

commercially obtained standards (ATP, ADP, AMP, IMP, Hx, and HxR). The K value was calculated as follows:

$$\text{K value (\%)} = \frac{Hx + HxR}{ATP + ADP + AMP + IMP + Hx + HxR} \times 100\% \quad (4)$$

NMR measurements

The T_2 relaxation times measurement

A LF NMR analyser minispec PQ 001 (Niumag, Ltd., Shanghai, China) with a proton resonance frequency of 21 MHz was used for the measurement. It was performed according to Sánchez-Alonso et al. (2012) as follows. Samples (about 10 g) cut from salmon fillets were placed in NMR tubes (60 mm in diameter). The measurements of the transverse relaxation time (T_2) were carried out using the Carr-Purcell-Meiboom-Gill pulse sequence (CPMG). For each measurement 4 scans were acquired at an interval of 2 s with a total of 8000 echoes.

Proton magnetic resonance imaging ($^1\text{H-MRI}$)

Nuclear magnetic resonance spectroscopy was performed by the method of Heijden et al. (2009) with slight modifications, using a LF NMR spectroscopy analyser MR 60 (Niumag, Ltd., Shanghai, China). The fish samples (about 10 g) were cut from salmon fillets and then placed in NMR tubes (60 mm in diameter). Proton density images were measured using the Multiple-Spin-Echo (MSE). The spectroscopy was performed with a repetition time of 500 ms and a echo time of 18.2 ms. Imaging planes were chosen through Larmor's relation as follows:

$$\nu = \gamma B_0 / 2\pi \quad (5)$$

where γ is the ratio between magnetic moment and moment of momentum, B_0 is the number of the intensity of external magnetic fields.

By regulation of noise-signal ratio and the image sharpness, spectroscopy images were obtained.

Data analysis

At least three measurements for per fish samples were performed. The mean and standard deviation of data was performed in Microsoft Excel 2007 (Microsoft Cor., Redmond, USA). Line charts, scatter plots and bar graphs were drew by origin8.5 (Origin Lab, Northampton, USA). The relationship between each sensory, freshness and texture analysis and the transverse relaxation parameter was examined using SPSS 10.0 (SPSS Inc., Chicago, USA.) by

Pearson correlations. Statistical significance was set as $p < 0.05$.

Results and discussion

Changes of sensory quality

Figure 1a shows the sensory scores of samples stored at 0 and 4 °C. The changes of fish properties, such as proteins and lipids that contribute to microbiological and chemical variations (Ventanas et al. 2006), had a great effect on colour, odour, fleshy and tissue elasticity of the samples, which led to a decline in sensory evaluation during storage. Sensory scores of 9.46 for 0 °C and below 8 for 4 °C was observed for the samples on 8th day of storage, while the scores of 0 °C below 8 until day 12. Significant differences ($p < 0.01$) were detected in sensory scores between the fish stored at 0 °C and the fish stored at 4 °C.

Physicochemical analysis

Hardness is the peak force (kg) sensed on the first curve cycle. As shown in Fig. 1b, the hardness value declined progressively during the process, this was in accordance with earlier studies (Li et al. 2011). The hardness of salmon stored at 4 °C was lower than those stored at 0 °C ($p < 0.05$).

Figure 1c shows the steady increase in TBA in storage period. Thiobarbituric acid index was used to evaluate the degree of lipid oxidation, which mainly comes from the degradation of polyunsaturated fatty acid in fish (Fuentes et al. 2013). The degradation products from lipid oxidation contribute to the off-flavor development in fresh salmon during refrigerated storage. The values of salmon increased from 0.1326 for 0 °C to 0.88335 and from 0.1326 to 1.24215 for 4 °C after 10 days. Comparisons between the different storage conditions showed that 0 °C was more effective than 4 °C to inhibit lipid oxidation, in agreement with Zhu et al. (2016) on catfish fillets.

TVB-N is widely used as an index to assess the quality and shelflife of fish and fish products (Fernández-Segovia et al. 2012; Rizo et al. 2015). Figure 1d displays the changes in TVB-N values of salmon during different storage conditions. At the very beginning of the storage, the TVB-N values of fresh fillet were 14.32 mg/100 g. The values of TVB-N increased steadily in 0 and 4 °C samples. The TVB-N content of the 0 °C samples increased by an average of 3.49 mg per day and that of the 4 °C samples by 6.53 mg per day. In contract, the TVB-N values of 4 °C fillets reached 45.99 mg/100 g by day 10, which exceeded the maximum acceptable value (30 mg/100 g) (Gökoglu

and Cengiz 2004). Similar findings were also found in sardine whose TVB-N value changed from 13.2 mg/100 g to 64.8 mg/100 g during 10 days of refrigerated storage at 4 °C (Gökoğlu et al. 1998). Its increase is related to the activity of spoilage bacteria and endogenous enzymes (Kykkidou et al. 2009; Fuentes et al. 2011). The action of such enzymes results in the formation of compounds including ammonia, monoethylamine, dimethylamine and trimethylamine, which give fish a characteristic off-flavour (Liu et al. 2013). Therefore, the low values observed for fillets stored at 0 °C indicated a good quality.

It shows from Fig. 1e that the WHC decreased with storage time at 0 and 4 °C. It dropped from initial value of 86.64% to about 73.89% on 14th day of storage for salmon stored at 0 °C, and that of the 4 °C by 72.41% on 10th day of storage. The WHC and sensory quality decreased, reflecting a decrease in the water-protein interactions during refrigerated storage (Offer and Knight 1988; Jensen and Jørgensen 1997).

Water activity is the ability to bound water to muscle, may be defined as the ratio of steam partial pressure of solutions to vapor pressure of pure water (Wu 1995). The effect of storage temperature on water activity is revealed in Fig. 1f. The water activity of samples continues to decrease. Although water content of samples varied little during the storage (31.29–36.37%), compared to the samples stored at 0 °C, water activity of the fillets stored at 4 °C reduced more significantly.

Cook loss is an important index to reflect the water holding and oil holding capacity of fish muscle (Sheard et al. 1999). Storage temperature has a significant impact on cook loss of samples (Fig. 1g), the decline of cook loss of salmon fillets might be contributed to the oxidation of protein and lipid. Overall, the cooking loss increased with the storage time, and samples stored at 4 °C were higher than those stored at 0 °C. The statistical analysis showed differences between samples stored at two temperatures with a significance ($p < 0.05$).

K value is a value that reflects the degree of nucleotide degradation, and can directly reflect the freshness of fish, and its increase is mainly due to the sharp degradation of IMP into HxR and Hx (Zhu et al. 2016). The changes in the K value of samples under different storage conditions are shown in Fig. 1h. Correlation analysis showed that there are significant differences ($p < 0.01$) between two batches. There has been consistent growth in K value of salmon fillets during storage, the K value of samples stored at 4 °C increased faster than samples at 0 °C, which demonstrate the effectiveness of storage temperature to maintain the freshness of fish, in accordance with earlier studies (Zhang et al. 2016).

Fig. 1 Changes in the values of **a** sensory, **b** hardness, **c** TBA, **d** TVB-N, **e** WHC, **f** water activity, **g** cooking loss, **h** K value of salmon fillets storage at 0 °C (filled square) and 4 °C (filled circle), mean $\pm$ standard error

Fig. 2 Variation in T_2 of salmon stored at 0 °C (a) and 4 °C (b)

Fig. 3 Mean relaxation time T_{22} (a) and T_{23} (c) and the corresponding amplitude A_{22} (b) and A_{23} (d) of LF NMR T_2 relaxation data. Samples were stored at 0 °C (white) and 4 °C (grey)

Study of the water distribution by LF NMR analysis

Figure 2a and b showed the T_2 transverse relaxation time distribution of fillets from salmon stored at 0 and 4 °C. Three water populations were observed in Fig. 2, in accordance with previous research (Aursand et al. 2008). The population named as T_{21} with the shortest relaxation time (0.1–1 ms), was the immobile water tightly associated

with macromolecules, the water represented by T_{22} component with a relaxation time centered at 28–86 ms was considered to be trapped within the protein-dense myofibrillar network, and the T_{23} component with the relaxation time about 114–7564 ms was due to the loose water in the extramyofibrillar space. The variations in water mobility may be due to variations in free energy of hydrogen bond between water and macromolecules in food (Andersen and

Fig. 4 Magnetic resonance imaging (MRI) of salmon for storing at 0 and 4 °C

Rinnan 2002). With the increasing storage time, the value of T_{22} decreased while T_{23} increased, the variations are obvious and in agreement with previous study (Ren et al. 2015).

The changes of T_{22} , A_{22} , T_{23} , and A_{23} of salmon stored at different temperatures are shown in Fig. 3a, b, c and d, respectively. No differences could be found in T_{22} time constants between salmon fillets stored at 0 and 4 °C whereas the A_{22} population tended to be lower in the samples during cold storage, accounting for 70–95% of the signal. The A_{22} of samples during storage at 4 °C reduced faster than samples at 0 °C. The T_{23} time of samples stored at 4 °C fluctuated during storage, while samples stored at 0 °C first increased and then declined constantly and its relative abundance increased, accounting for 2–27% of the signal, indicating the alteration of strong bound water to free water in salmon muscle. Changes in the values of T_2 components may suggest the spoilage of the chilled meat, this may be evidenced by a slight increase in the value of A_{23} , which was related to an amount of free water.

Magnetic resonance imaging (MRI) permits visual observations of the spatial, internal morphological

organization and molecular distribution of water in high water content its food matrix. The intensity of the signal in the various zones of the sample may be directly proportional to water molecule content. Specifically, darker areas in an image may mean that there are less water/protons. A high content of ions and or contrast agent, etc. enhances relaxation, causing the area to become darker (Butz et al. 2005). As can be seen from MRI of salmon (Fig. 4), the brightness of samples changed from bright yellow to dark blue during storage, which indicated that a decline in water content in fish muscle decreased. Refrigerated storage induced changes in images in all samples, For the samples stored at 0 °C, a slight variation with images was found in the first 4 days, then fish was not until the 14th day that the fish deteriorated seriously and spoilage area radiated out to surface. From day 2, the fillets stored at 4 °C began to degrade and the color of meat in MRI image became bluer and darker than samples stored at 0 °C, indicating the loss of trapped water and loose water. This decrease of brightness may be contributed to the degradation and denaturalization of muscle protein. The results highlighted that the water distribution of salmon fillets was correlated with the

Table 1 Linear regression analyses of samples stored at 0 °C between the relaxation times and amplitudes of the populations of water and texture, freshness and sensory quality

T ₂	R	R ²	<i>p</i>
Correlation with TVB-N			
T ₂₁	− 0.45171	0.204042	ns
T ₂₂	− 0.74985	0.562275	<i>p</i> < 0.01
T ₂₃	0.82429	0.679454	<i>p</i> < 0.01
Correlation with water activity			
T ₂₁	0.27313	0.0746	ns
T ₂₂	0.58421	0.341301	ns
T ₂₃	− 0.62569	0.391488	ns
Correlation with cooking loss			
T ₂₁	− 0.17866	0.031919	ns
T ₂₂	− 0.95825	0.918243	<i>p</i> < 0.05
T ₂₃	0.82429	0.679454	<i>p</i> < 0.05
Correlation with TBA			
T ₂₁	− 0.28964	0.083891	ns
T ₂₂	− 0.95547	0.912923	<i>p</i> < 0.05
T ₂₃	0.99037	0.980833	<i>p</i> < 0.05
Correlation with WHC			
T ₂₁	0.44305	0.196293	ns
T ₂₂	0.73829	0.545072	<i>p</i> < 0.01
T ₂₃	− 0.81123	0.658094	<i>p</i> < 0.01
Correlation with hardness			
T ₂₁	0.28053	0.078697	ns
T ₂₂	0.92516	0.855921	<i>p</i> < 0.01
T ₂₃	− 0.95898	0.919643	<i>p</i> < 0.01
Correlation with sensory			
T ₂₁	0.47063	0.221493	ns
T ₂₂	0.83676	0.700167	<i>p</i> < 0.01
T ₂₃	− 0.91282	0.83324	<i>p</i> < 0.01
Correlation with K value			
T ₂₁	− 0.3798	0.144248	ns
T ₂₂	− 0.88812	0.788757	<i>p</i> < 0.01
T ₂₃	0.94375	0.890664	<i>p</i> < 0.01

R regression coefficient, *p* significance level, *ns* no significant

Table 2 Linear regression analyses of samples stored at 4 °C between the relaxation times and amplitudes of the populations of water and texture, freshness and sensory quality

T ₂	R	R ²	<i>p</i>
Correlation with TVB-N			
T ₂₁	0.50068	0.25068	ns
T ₂₂	− 0.92687	0.859088	<i>p</i> < 0.01
T ₂₃	0.84553	0.714921	<i>p</i> < 0.05
Correlation with water activity			
T ₂₁	− 0.21962	0.048233	ns
T ₂₂	0.81246	0.660091	<i>p</i> < 0.05
T ₂₃	− 0.78921	0.622852	ns
Correlation with cooking loss			
T ₂₁	0.26647	0.071006	ns
T ₂₂	− 0.92229	0.850619	<i>p</i> < 0.01
T ₂₃	0.9597	0.921024	<i>p</i> < 0.01
Correlation with TBA			
T ₂₁	0.14018	0.01965	ns
T ₂₂	− 0.92704	0.859403	<i>p</i> < 0.01
T ₂₃	0.92471	0.855089	<i>p</i> < 0.01
Correlation with WHC			
T ₂₁	0.03376	0.00114	ns
T ₂₂	0.86274	0.74432	<i>p</i> < 0.05
T ₂₃	− 0.89655	0.803802	<i>p</i> < 0.05
Correlation with hardness			
T ₂₁	− 0.23405	0.054779	ns
T ₂₂	0.94477	0.89259	<i>p</i> < 0.01
T ₂₃	− 0.92238	0.850785	<i>p</i> < 0.01
Correlation with sensory			
T ₂₁	− 0.00615	0.000038	ns
T ₂₂	0.96857	0.938128	<i>p</i> < 0.01
T ₂₃	− 0.99686	0.99373	<i>p</i> < 0.01
Correlation with K value			
T ₂₁	0.01289	0.000166	ns
T ₂₂	− 0.9616	0.924675	<i>p</i> < 0.01
T ₂₃	0.98822	0.976579	<i>p</i> < 0.01

R regression coefficient, *p* significance level, *ns* no significant

quality changes, and greater water motobility was observed when the salmon stored at 4 °C than samples stored at 0 °C.

Relationship between LF NMR analysis, texture, freshness and sensory quality

Renou et al. (1985) firstly revealed that there was a relationship between T₁ and T₂ and traditional quality characteristics of pork meat, and later studies have confirmed a relationship between WHC, cook loss and LF NMR measurements (Gudjónsdóttir et al. 2011a, b). However,

Tables 1 and 2 indicates that T₂₁ did not correlate with quality parameters well. T₂₂ and T₂₃ of salmon stored at 0 °C were not only significantly correlated (*p* < 0.05) with cook loss and TBA, but also correlated well (*p* < 0.01) with WHC, K value. For samples stored at 4 °C, strong correlations between the NMR parameters (T₂₂ and T₂₃) and quality indicators including WHC, TVB-N, sensory scores, hardness, cook loss, TBA and K value were also found. No correlations were found between T₂ and water activity.

Conclusion

The present study revealed the relationship between LF-NMR parameters and traditional quality indicators including sensory scores, hardness, TBA, TVB-N, WHC, water activity, cook loss, and K-value. The results showed that LF NMR T_2 transverse relaxation time was significantly affected by storage conditions and period of salmon fillet during storage, and showed a strong correlation with the parameters related with water, such as cook loss and WHC, as well as quality indicators including sensory quality, hardness, TBA and TVB-N. The study highlighted the feasibility of application of proton spectroscopy (^1H) by low-field NMR to monitor salmon quality deterioration during storage, which is associated with water mobility.

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