

Water Distribution in Brine Salted Cod (*Gadus morhua*) and Salmon (*Salmo salar*): A Low-Field ^1H NMR Study

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Low-field (LF) ^1H NMR T_2 relaxation measurements were used to study changes in water distribution in lean (Atlantic cod) and fatty (Atlantic salmon) fish during salting in 15% NaCl and 25% NaCl brines. The NMR data were treated by PCA, continuous distribution analysis, and biexponential fitting and compared with physicochemical data. Two main water pools were observed in unsalted fish, T_{21} , with relaxation times in the range 20–100 ms, and T_{22} , with relaxation times in the range 100–300 ms. Pronounced changes in T_2 relaxation data were observed during salting, revealing changes in the water properties. Salting in 15% brine lead to a shift toward longer relaxation times, reflecting increased water mobility, whereas, salting in saturated brines had the opposite effect. Water mobility changes were observed earlier in the salting process for cod compared to salmon. Good linear correlations ($F \leq 0.05$) were found between T_2 parameters and water holding capacity, centrifugation loss, water activity, and salt content in the liquid phase for all fish groups. Fillet pH and total weight changes correlated linearly with T_2 parameters for some of the fish groups.

KEYWORDS: NMR; T_2 relaxation; water mobility; Atlantic salmon; Atlantic cod; muscle; brine salting

INTRODUCTION

In the food processing industry, there is an increasing interest for rapid noninvasive and nondestructive methods. By using such techniques, extensive preparation procedures such as mincing, extraction, or dilution can be avoided (*1*). Nuclear magnetic resonance (NMR) techniques are among those most capable of performing such a task.

Low-field (LF) ^1H NMR can measure proton relaxation and is therefore a technique for investigating changes in water and fat mobility and muscle structure during processing. In LF ^1H NMR studies, proton relaxation is described by the relaxation time constants T_1 (longitudinal) and T_2 (transversal). After an NMR pulse has been applied, protons reach their equilibrium state at different times dependent on their neighboring molecules. T_2 measurements are faster compared to T_1 and are therefore considered a better indicator of molecular mobility in solidlike materials.

More than three decades ago, it was found that the T_2 relaxation decay in muscle tissue is multiexponential. This indicates the existence of different water populations or water “pools” in the muscle tissue (*2, 3*). In recent years, many other

LF NMR T_2 relaxation time investigations have supported this theory for both fish and meat (*4–10*). The distribution of water in the muscle may be described as divided into three compartments. The “first” compartment is often named “bound water”, which contains less than 10% of the water in the muscle. This water is very closely bound to proteins, has reduced mobility, and cannot easily move to other compartments. In LF NMR studies, this water has been reported to relax in the area 1–10 ms, and it has been referred to as the T_{2b} relaxation component (*5*). The amount of bound water changes very little, if at all, in postrigor muscle (*11*), and it is very resistant to freezing and heating (*12*). The “second” compartment, often called “entrapped water”, is the most affected by the rigor process and the conversion of muscle to meat. This water can eventually escape as drip loss (*11*). In earlier LF NMR studies on fish and meat, this water, reflected by T_{21} , have been found to relax in the range 10–100 ms (*4, 5, 10*). The “third” compartment, called “free water”, is mainly held by weak surface forces and is not readily seen in prerigor meat but can gradually develop during and after the rigor process (*12*). The free water is thought to be described by the relaxation component T_{22} , which has relaxation times in the range 100–400 ms (*4, 5, 10*). Together, the “entrapped” and the “free” water describe approximately 90% of the water in muscle (*13*).

LF NMR has been used to study in situ changes of water distribution occurring in foods during storage and processing (*1*). Using T_2 relaxation time measurements, several populations

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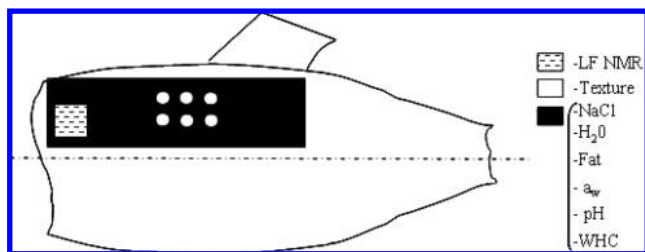
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Table 1. Weight and Length of Guttled Fish and Fillets, and Fillet Thickness of Salmon and Cod Used for Brine Salting^a

	salmon (whole, gutted)		cod (head off, gutted)	
	batch 1	batch 2	batch 1	batch 2
brine solution (w/w NaCl) (%)	15	25	15	25
guttled weight ^b (kg)	3.5 (0.3)	3.8 (0.1)	2.1 (0.3)	1.7 (0.2)
fillet weight ^c (g)	1139 (113)	1397 (69)	831 (165)	712 (104)
fillet length ^c (cm)	45.9 (2.6)	45.9 (0.9)	43.4 (3.3)	46.9 (2.8)
fillet thickness ^c (cm)	3.0 (0.1)	3.0 (0.2)	2.7 (0.2)	2.6 (0.2)

^a Values are given as mean (SD). ^b $n = 8$ for all groups. ^c $n = 16$ for all groups.

**Figure 1.** Sampling locations for various analyses of cod and salmon fillets. For details, see text.

of water were defined in fresh (9, 14) frozen, thawed, chilled, and processed (7, 15–19) fish muscle and mince. In addition, fat and water content, as well as the water holding capacity in intact fish muscle, have been determined (20).

In fish salting processes, the properties of the water have great influence on the total fish quality. Therefore, it is of importance to develop basic knowledge about the water properties in muscle tissue. Numerous studies have been performed on the salting of foods by using standard physicochemical methods. Investigations have been performed on the stability, degradation, or denaturation of muscle proteins (21–25), effects of additives and salting procedure (26–28), process modeling and mass transfer kinetics (29–35), as well as raw material quality (36, 37). However, only a few LF ¹H NMR studies have been done on salting of fish and meat (8, 38–41).

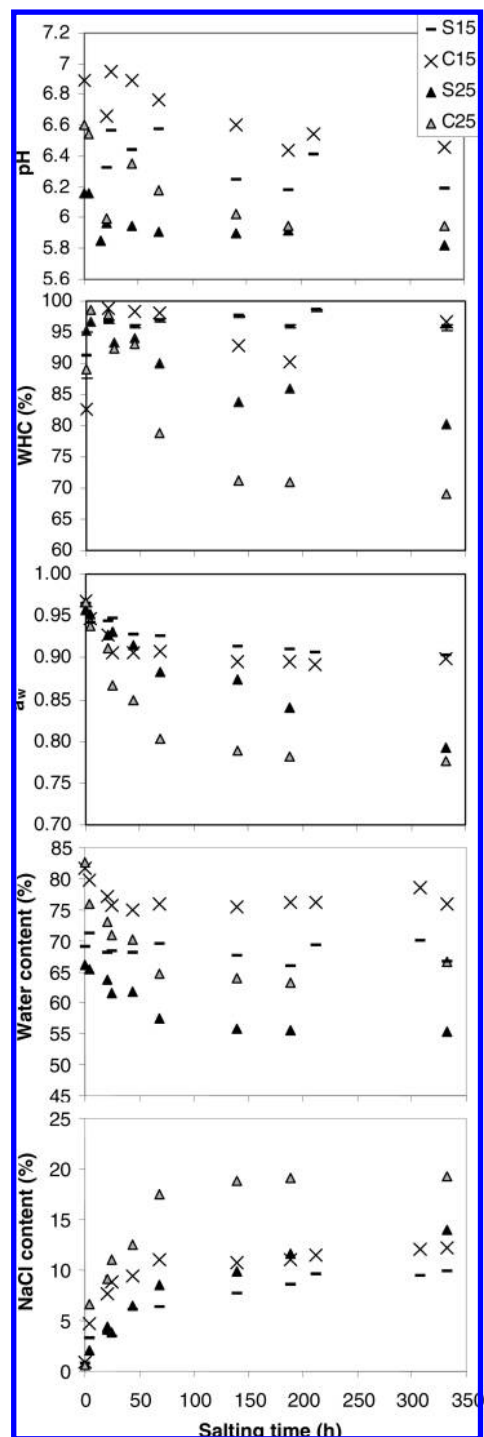
The aim of this study was to contribute to a further understanding of the water dynamics occurring during salting of lean (cod) and fatty (salmon) fish in 15 and 25% brines, through T_2 relaxation measurements. In addition, NMR data were correlated to physicochemical analyses.

MATERIALS AND METHODS

Fish. Atlantic salmon (*Salmo salar*) were commercially farmed (SalMar AS) near Frøya in Central Norway. The fish were slaughtered in July 2004 (batch 1, $n = 8$) and September 2004 (batch 2, $n = 8$) and processed in the plant according to standard routines. On the same day as they were slaughtered, the fish were transported on ice in Styrofoam boxes to our laboratory, where they arrived about 6–8 h post mortem. On arrival, the mean core temperature was close to 0 °C.

Atlantic cod (*Gadus morhua*) were gill netted in the Trondheimsfjord, Central Norway, in July (batch 1, $n = 8$) and September (batch 2, $n = 8$) 2004. On board, the fish were killed, bled, gutted, deheaded, and placed on ice. The fish were landed and transported to our laboratory on ice in Styrofoam boxes. On arrival, about 24–30 h post mortem, the mean core temperature was close to 0 °C.

All fish were held on ice in a cold room (4 °C) for 3 days. All fish were in a postrigor state at the start of the experiment. Before brine salting, 3–4 days post mortem, both species were filleted, with skin on. The fillets were individually weighed, washed, and tagged. The

**Figure 2.** Fillet pH, water holding capacity, water activity, water, and salt content of cod (C) and salmon (S) fillets salted in 15% (C15 and S15) and 25% (C25 and S25) brines throughout the salting time. Each point represents the average of 3 parallel measurements.

general appearance of the fillets was considered as good. The average weight and length of gutted fish and fillets as well as fillet thicknesses are shown in **Table 1**.

Salting and Sampling. Salmon and cod fillets were salted for 14 days at 4 ± 1 °C in closed plastic tanks using 15% (batch 1) or 25% (batch 2) NaCl (w/w) solutions at a fish/brine ratio of 1:3. Ordinary commercial refined salt (Jozo salt, Akzo Nobel Salt, Göteborg, Sweden) and distilled water were used to make the brines.

The fillet total weight changes (Δ weight) ($n = 5$) were measured after brine drainage for 2 min on a grid (eq 1):

$$\Delta \text{weight}_t = \left(\frac{\text{weight}_t - \text{weight}_0}{\text{weight}_0} \right) \quad (1)$$

where weight_t and weight_0 are the cod and salmon weights at sampling time t and 0, respectively. One fillet of each species was sampled randomly from the brine after 0, 4, 20, 25, 44, 68, 140, 188, and 332 h. Samples for different analyses were taken as shown in **Figure 1**.

NMR Sample Preparation and Analyses. For the LF ^1H NMR measurements, one cube ($3 \times 3 \times 2 \text{ cm}^3$) was excised from each fillet (**Figure 1**) and a 1 cm thick layer closest to the fillet surface was thereafter divided into three equal subsamples (dimensions: $1 \times 1 \times 3 \text{ cm}^3$; weight: approximately 2 g) and placed in NMR tubes (diameter 10 mm). The tubes were immediately placed in ice and kept there for about 30 min before they were equilibrated to 4 °C in a thermostatted water bath (Julabo labortechnik GmbH, Germany) before analysis.

T_2 relaxation measurements were performed using a LF NMR analyzer minispec mq 20 (Bruker Optik GmbH, Ettlingen, Germany) with a magnetic field strength of 0.47 T corresponding to a proton resonance frequency of 20 MHz. The instrument was equipped with a 10 mm temperature-variable probe. A built-in heating element was connected to the temperature control unit (BVT3000, Bruker Optik GmbH). The temperature in the probe was regulated to 4 °C by blowing compressed air through the sample holder. T_2 was measured using the Carr-Purcell-Meiboom-Gill pulse sequence (CPMG) (42, 43). The T_2 measurements were performed with a time delay between the 90° and 180° pulses (τ) of 150 μs . Only even echoes were sampled. Data from 3000 echoes were acquired from 16 scan repetitions. The repetition time between two succeeding scans was set to 2 s.

The NMR T_2 relaxation data were analyzed by three different methods: (1) A continuous distribution of exponentials related to water and fat protons located in different muscle compartments was fitted for all CPMG curves by use of the CONTIN algorithm (44) after normalizing the raw data by setting the first sampled echo to a value of 1 and scaling the rest of the echo-train thereafter. This analysis gave a plot of the distributed relaxation amplitudes.

(2) A multivariate data analysis was performed for all raw T_2 relaxation (CPMG) curves. These curves were normalized in the same way as described for the continuous distributed curves; however, in

this case, the first sampled echo was set to a value of 100. The first 800 data points were used for the principal component analysis (PCA) (45) using an in-house made program written in Visual Basic. In the data matrix, each row represented a single fish sample, and each column represented a signal amplitude from an echo in the CPMG echo train. Four principal components were used. The input matrix was not mean-centered.

(3) Biexponential fitting analysis of T_2 relaxation data was performed by fitting the absolute value of the CPMG as shown in eq 2, using the SigmaPlot (version 9.0, Systat Software, Inc., 2004), as reported by Erikson et al. and Lambelet et al. (8, 16):

$$\text{signal} = A_{21}e^{-t/T_{21}} + A_{22}e^{-t/T_{22}} \quad (2)$$

where T_{21} and T_{22} were the relaxation components and A_1 and A_2 were the corresponding amplitudes. T_{21} populations were calculated as $(A_{21}/(A_{21} + A_{22})) \times 100$. Three samples were taken from the same fish and were averaged at each sampling point.

Physicochemical Methods. The textural properties of the fillets were measured by double compression using a Texture Analyzer TA.XT2 (Stable Micro Systems, Surrey, U.K.) equipped with a flat-ended cylindrical plunger (12 mm diameter), by a modification of the method described by Einen and Thomassen and Hultmann and Rustad (46, 47). The probe was pressed into the fillets at a constant speed of 1 mm s⁻¹ until it had reached 60% of the initial sample height. The holding time between the two compressions was 5 s. The texture profile curve was recorded continuously (Texture Profile Analysis (TPA)). The maximum force of the first compression in the force-time curve (F_{hardness}) was recorded and calculated as described by Bourne (48). Each fillet was measured 6 times at different locations (**Figure 1**), and the average is reported here.

Once the determination of texture was finished and the samples for LF NMR (and MRI, published elsewhere (35)) had been excised, the fillet sample was minced and used for chemical analyses. The salt content in the brine and in the fillets was measured in triplicate using the Dichromat II salt analyzer (PCL Control Instruments, Leicester, U.K.). The salt content in the liquid phase, (x^{NaCl}), was estimated from determinations of weight fractions of water ($x^{\text{H}_2\text{O}}$) and NaCl (x^{NaCl}), as shown in eq 3 (31, 49).

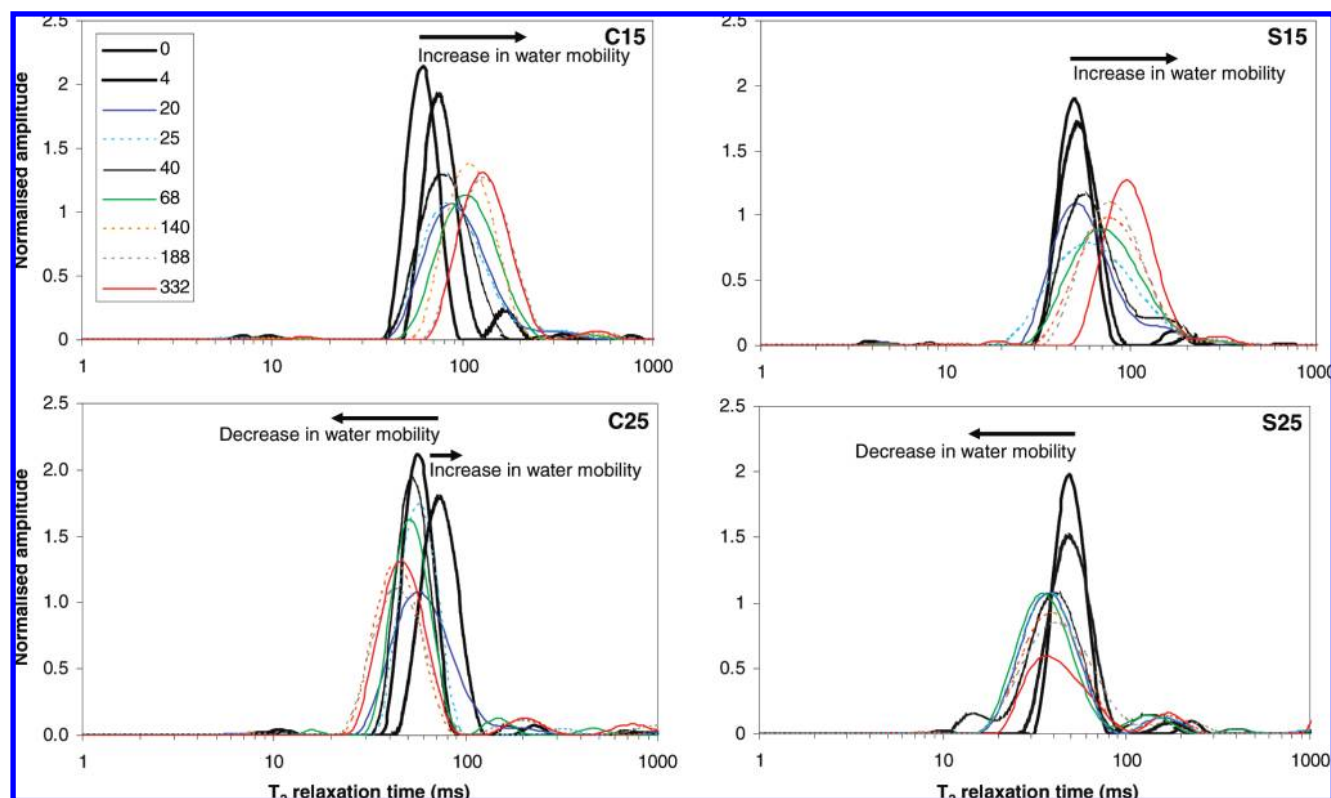


Figure 3. Continuous T_2 relaxation time spectra of cod (C) and salmon (S) fillets brine salted at 15% (C15 and S15) and 25% (C25 and S25) brines along the salting time. Each curve represents the average of 3 parallel measurements.

$$z^{NaCl} = \left(\frac{x^{NaCl}}{x^{H_2O} + x^{NaCl}} \right) \quad (3)$$

The moisture content ($n = 3$) was determined by drying three parallel samples of 5 g of mince at 105 °C for 24 h (50). In salmon, the total amount of fat was determined by the Blich and Dyer method (51) as modified by Hardy and Key (52).

Water activity (a_w) ($n = 2$) was determined by reading each sample 3 times by CX-2 AQUALab, (Decagon Devices Inc., Pullman, WA).

Fillet pH was determined in a suspension made by mixing 5 g of mince with 50 mL of distilled water, using a PHM210 pH-meter connected to a Mettler Toledo inlab 413 electrode, (METERLab, Copenhagen, Denmark).

Water holding capacity (WHC) and centrifugation loss was determined on minced muscle by low-speed centrifugation as described by Eide et al. (53) with a centrifugation force of 210g. The WHC is expressed as the percentage of water retained in the mince after

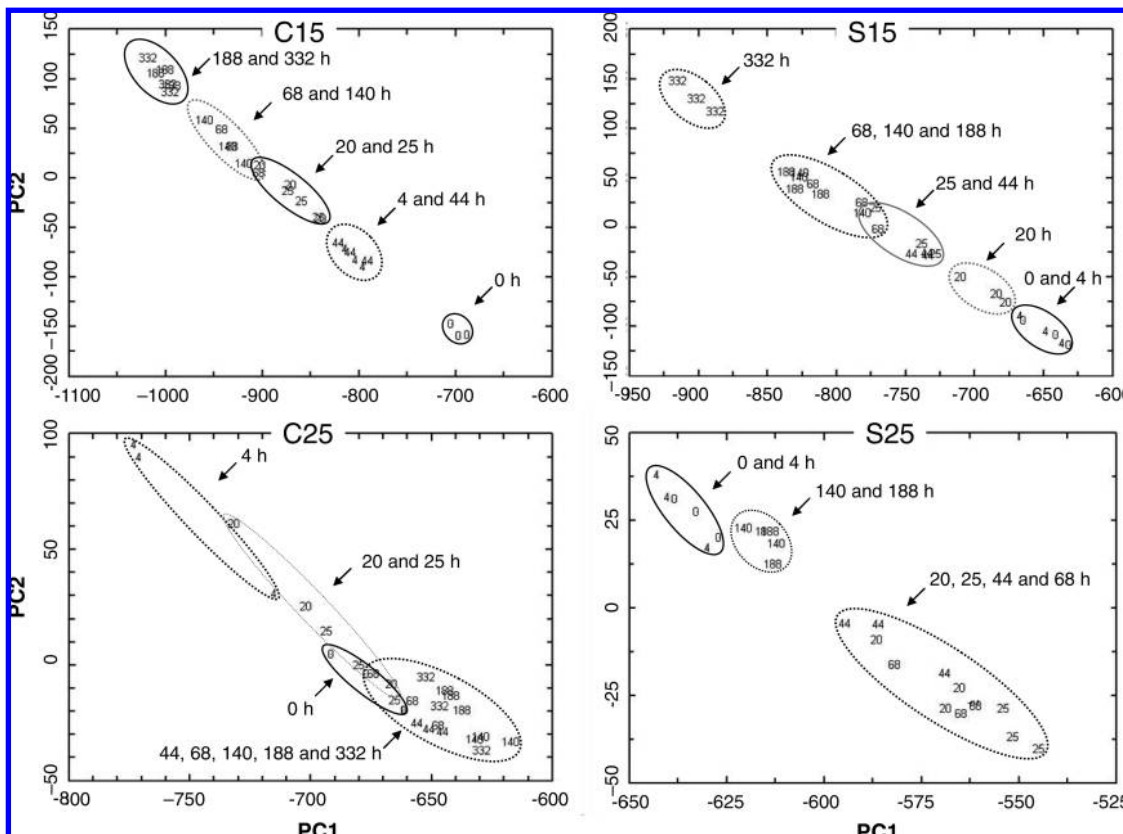


Figure 4. PCA score plots of raw T_2 data (CPMG) obtained from cod (C) and salmon (S) fillets brine salted in 15 and 25% brines (C15, S15, C25, and S25) for 0, 4, 20, 25, 44, 68, 140, 188, and 332 h.

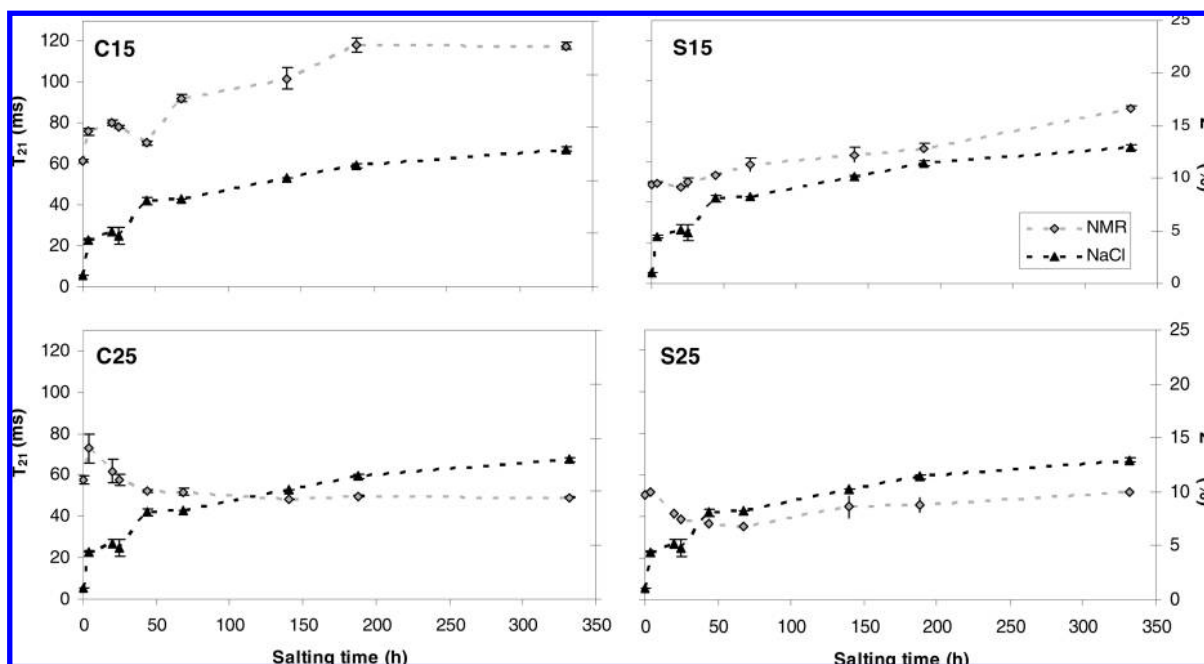


Figure 5. T_{21} relaxation times found by biexponential fitting ($n = 3$) and NaCl content in the liquid phase (z^{NaCl}) ($n = 3$) at different salting times for cod (C) and salmon (S) fillets immersed in 15 and 25% brines. Values are given as mean \pm SD.

Table 2. Levels of Significance for Linear Correlations between of T_2 Relaxation Parameters Found by Biexponential Fitting and Physicochemical Data^a

T_2 parameter	group	WHC	centrifugation loss	a_w	z^{NaCl}	pH	Δ weight
T_{21} (ms)	C15	N.S.	N.S.	b	b	b	b
T_{21} (ms)	S15	N.S.	N.S.	b	b	b	b
T_{21} (ms)	C25	b	b	b	b	N.S.	N.S.
T_{21} (ms)	S25	N.S.	N.S.	N.S.	N.S.	N.S.	N.S.
T_{22} (ms)	C15	N.S.	b	N.S.	N.S.	N.S.	N.S.
T_{22} (ms)	S15	b	N.S.	N.S.	N.S.	N.S.	N.S.
T_{22} (ms)	C25	N.S.	N.S.	N.S.	N.S.	N.S.	N.S.
T_{22} (ms)	S25	N.S.	N.S.	N.S.	N.S.	N.S.	N.S.
T_{21} pop (%)	C15	b	b	N.S.	N.S.	N.S.	N.S.
T_{21} pop (%)	S15	c	b	N.S.	N.S.	N.S.	N.S.
T_{21} pop (%)	C25	b	b	b	b	b	b
T_{21} pop (%)	S25	b	b	b	b	N.S.	N.S.

^aN.S. = Not significant. ^b $F \leq 0.05$. ^c $F \leq 0.001$.

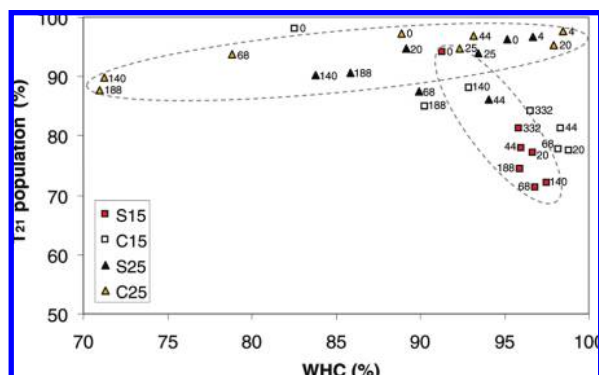


Figure 6. Water holding capacity (WHC) of cod (C15 and C25) and salmon (S15 and S25) plotted against the T_{21} population obtained by biexponential fitting.

centrifugation for 5 min. The analyses were run in quadruplicate. The centrifugation loss was calculated from the same analysis and is expressed as total sample weight lost after centrifugation.

Partial Least Square Regression. Partial least-squares regression, PLSR, was implemented using Unscrambler (version 9.2, CAMO process AS, Oslo, Norway) to describe the relationship between the NMR data and the compositional variables. Signal amplitudes from selected variables of the distributed NMR T_2 relaxation data from all analyzed samples were chosen as X and the corresponding physicochemical data (hardness, centrifugation loss, z^{NaCl} (salt in the liquid phase), a_w , Δ weight (total weight changes), and pH) was chosen as Y.

RESULTS AND DISCUSSION

Physicochemical Properties. In **Figure 2**, a summary of the observed changes in pH, WHC, a_w , water content, and NaCl content during salting of cod and salmon in 15% (C15 and S15) and 25% (C25 and S25) brine are given. Different behavior depending on fish species and brine concentration is clearly observed. The fillet pH decreased in all groups during the first two days of salting. The decrease was most pronounced in the 25% brine salted fillets. In general, fish salted in 15% brine had a higher pH than fish salted in 25% brine, and the pH in the salmon was lower than in cod throughout the salting process. A similar drop in pH was observed by Erikson et al. (8) who studied unsalted cod and cod brine salted in 16.9% NaCl for 43 h. However, Erikson et al. (8) observed a pronounced effect on pH (a drop from 6.8 to 6.5) after only 43 h of salting. At the very beginning of the salting process, the WHC increased in all fish groups. The fillets salted in the 25% brine exhibited the lowest WHC after salting. Throughout the salting process, WHC was generally lower in cod than in salmon. The a_w and the water content dropped in all groups, and the changes were most pronounced in fish salted in 25% NaCl due to higher dehydra-

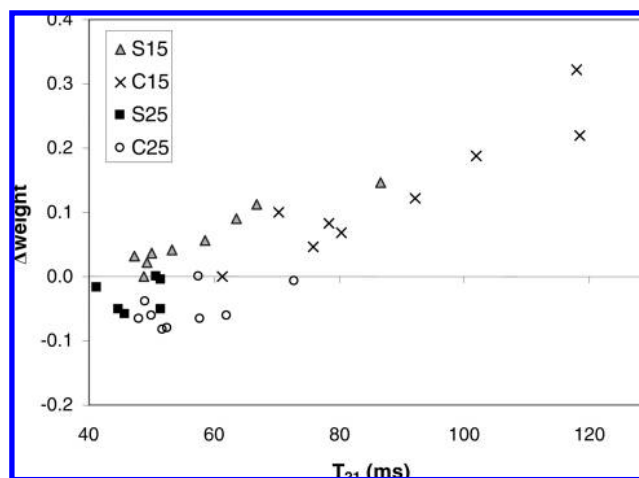


Figure 7. T_{21} relaxation time constants found by biexponential fitting versus total weight changes for cod and salmon during salting in 15 and 25% brines.

tion. During the first two days of salting, the NaCl uptake was the same for fillets immersed in both brines. The NaCl gain was generally higher for the cod than for the salmon. A more detailed description of the physicochemical data is given by Gallart-Jornet et al. (35).

Low-Field (LF) ^1H NMR. Having established the physicochemical properties of the fillets throughout the salting process, we wanted to study NMR proton relaxation measurements at the same sampling times on the same fillets. Different methods were used to analyze the T_2 relaxation data: continuous distributed NMR relaxation curves, multivariate data analysis (PCA), and biexponential fitting.

Continuous T_2 Distributions. **Figure 3** shows the distributed T_2 relaxation time spectra of cod (C15, C25) and salmon (S15, S25) fillets salted for 0, 4, 20, 25, 44, 68, 140, 188, and 332 h in 15 and 25% brines. Before salting, there were two main populations centered at 20–100 ms (T_{21}) and 100–300 ms (T_{22}). These findings are in agreement with earlier studies of pork (4, 5, 7, 40) and fish (8, 10, 17, 18, 20). The water populations changed within the range 20–300 (T_{21}) and 100–1000 (T_{22}) during salting, depending on both fish species and brine concentration. The main T_2 relaxation components (T_{21} and T_{22}) commonly describe more than 90% of the water in muscle (13). After salting, T_{21} , which is thought to correspond to intramyofibrillar water (5), relaxed in the range 20–150 ms. T_{22} , considered to reflect extra-myofibrillar water (5) and water and fat in salmon (10), relaxed in the range 150–1000 ms.

Both salting time and brine concentration had a pronounced effect on the T_2 distributions. Longer salting times were

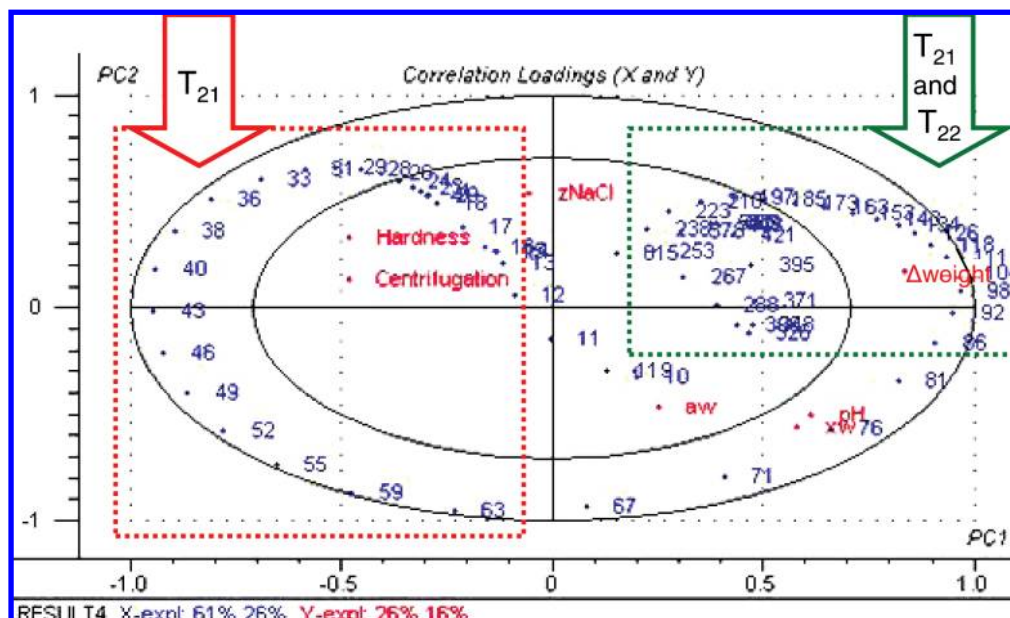


Figure 8. PLSR2 loading plot with selected variables of the signal amplitudes of continuous distributed T_2 data as X (given in relaxation times (ms) marked in blue) and selected physicochemical variables as Y (marked in red). The inner and outer circle represents 50 and 100% explained variance. All data from both fish species are included.

characterized by a broadening of T_{21} population in both brines compared to unsalted fillets. This was accompanied by a decrease in the T_{21} signal amplitudes as the salting process went on. This is in accordance with data presented by Wu et al. (40) where pork meat was salted at 3, 6, and 9% NaCl for 48 h. In addition, we observed a general tendency toward longer T_{21} relaxation times in fillets salted at 15% NaCl (C15 and S15), which corresponds to an increase in water mobility. In general, the opposite behavior was observed in the saturated brines (C25 and S25). However, it is interesting that the C25 at the beginning of the salting process (4 h) exhibited a slight increase in water mobility, whereas in S25, this trend was less pronounced. The large differences in fillet fat contents of the two species have probably had a pronounced effect here (35, 54). The water mobility increase at the very beginning of the cod salting process might be explained by protein swelling (55), which consequently lead to an increase in WHC at the beginning of the salting process (35) (see **Figure 2**).

There were no visible differences in distribution of water mobility between fish species comparing the continuous distributed curves, but there was a certain tendency that the salmon exhibited slightly broader T_{21} distributions than cod in both brines. The changes occurred more slowly in salmon. This could be explained both by the differences in muscle composition and the lower salt uptake in salmon muscle compared to cod muscle (35, 56) and that fat “protects” the proteins in a way that reduces the effect (swelling or denaturing) of salt uptake.

Multivariate Data Analysis of Raw T_2 Relaxation Data.

Another approach for studying the mobility changes was by principal component analysis (PCA) on the entire raw T_2 data set.

Figure 4 represents the PCA score plots of the raw relaxation curves for both species at both brine concentrations and all salting times. In all cases, a clustering along the salting time was observed. This confirmed the shifts in water mobility and water population seen in **Figure 3**. In the 15% brines, the changes in water distribution followed a continuous trend as a function of salting time from unsalted fillets up to 332 h of

salting. In the saturated brines, this trend was not continuous, indicating more complex changes in water mobility during salting compared to 15% brine.

In cod, the water mobility changes followed the same tendency in both brines at the first stage of the salting process (4 h), and considerable changes took place. In salmon, however, only small changes were observed. This indicates that salt had a greater influence on cod tissue than on salmon: that is, the driving forces were weaker in salmon than in cod. This was reflected in the PCA score plots, as more consistent changes in water mobility were observed in salmon than in cod throughout the salting process. Considering the whole salting process, the changes were not as marked in the 15% brine as in the 25% brine.

Biexponential Fitting of T_2 Relaxation Curves. The third approach of analyzing the data was by biexponential fitting of the raw T_2 relaxation curves. This simple and robust method has traditionally been used to interpret T_2 relaxation data and is therefore suitable for comparison with earlier studies. However, one should remember that this form of data processing forces the curve to fit to two exponents. A triexponential fitting of the data set seemed to “overfit” the data by splitting T_{21} instead of determining the faster relaxing component, referred to as T_{2b} . The calculated T_{21} relaxation times are shown in **Figure 5**. The T_{21} relaxation times both for unsalted cod (61 and 58 ms) and salmon (49 and 51 ms) correspond well with previously reported values. For cod, values of 65 ms (16), 50 ms (14), and 45 ms (8) have been reported, whereas for salmon, a T_{21} relaxation time of 50 ms was found (18). Our cod fillets salted in 15% brine had similar T_{21} relaxation times ($T_{21,C15,44h} = 70$ ms, $T_{21,C25,44h} = 53$ ms) as previously reported values ($T_{21,salted} = 69$ ms) in salted cod (16% NaCl, 43 h) (8).

Comparison of NMR and Physicochemical Data. The NMR data obtained by biexponential fitting were linearly correlated with WHC, centrifugation loss, water activity, salt content in the liquid phase, fillet pH, and total weight changes. Good linear correlations were found, and a summary of the significance levels is shown in **Table 2**.

The salt content and the T_{21} relaxation time constant changed in the same pattern during salting for both lean and fatty fish salted in 15% brines, **Figure 5**. In saturated brines, the opposite development in T_{21} relaxation times were seen, and changes were most pronounced during the first two days of salting. Salting in stronger brines or dry salting (25% NaCl in the performed experiment) is known to decrease WHC and total fillet weight (often referred to as "salting out"), whereas salting in more diluted brines (15% NaCl in the performed experiment) is known to increase WHC and total fillet weight often referred to as "salting in" (31, 34). Our T_{21} relaxation time values might reflect this opposite behavior of fillets salted in 15% and saturated brines. A calculation was done to compensate for the fat content in salmon (eq 3). The linear regression analysis between T_2 values and salt in the liquid phase gave correlation for both lean and fatty fish in 15% and saturated brines (**Table 2**).

Figure 6 shows the WHC plotted against the T_{21} populations. A decrease in T_{21} population was seen as WHC changed in all groups, revealing a pronounced change in muscle water distribution. The largest changes in T_{21} population were seen for the fillets salted in 15% brines. A decrease in T_{21} population indicates that the water mobility was increasing. Furthermore, it might denote that a larger amount of the total muscle water content was located as free water. These findings can be explained by the uptake of brine and coupled swelling of myofibrils (12). The changes in water distribution were less pronounced in the 25% brine salted fillets. The largest changes in WHC were found for fillets salted in 25% brine. As proteins were presumably more denatured in C25 and S25 than in C15 and C25 (12, 37), this could be a possible explanation of the observed trend. When proteins are denatured, the water is likely to flow more freely. This was reflected in our data as a decrease in T_{21} populations and an inverse increase in the T_{22} population. In unsalted pork meat, Bertram et al. (4) found a linear correlation between WHC and the T_{21} population ($R_{T_{21} \text{ pop}} = 0.84$). This corresponds well with our observations. Good linear correlations ($F \leq 0.05$) were found between WHC and the T_{21} population for cod and salmon fillets salted in 15% brine and for cod fillets salted in saturated brine (**Table 2**). Furthermore, Bertram et al. (4) reported a good correlation between WHC and the T_{22} time constant ($R = -0.77$). In our data, this was only found for the S15 group ($R = 0.90$).

Figure 7 shows the T_{21} relaxation time constant plotted against changes in fillet weight (Δ weight). For the 15% brine, an increase in water mobility was observed as the fillets gained weight. For the saturated brine, both water mobility and weight decreased. These results support the assumption that T_2 relaxation measurements can actually detect the salting in and salting out effects. Changes were most pronounced in the 15% brines, where both T_{21} relaxation times and Δ weight increased significantly during salting. Good linear correlations ($F \leq 0.05$) were obtained between total weight changes and T_2 parameters for the 15% brine salted samples (**Table 2**).

To further investigate the correlation between changes in water distribution and changes in the physicochemical variables, a PLSR was performed on all samples. **Figure 8** shows the correlation loading plot (PLS1 and PLS2) of the PLSR with the signal amplitude from selected variables of the distributed NMR T_2 relaxation data from all analyzed samples as X and the corresponding physicochemical data (fillet hardness, centrifugation loss, z^{NaCl} , a_w , Δ weight, and fillet pH) as Y. The cross validated explained variances were 61%/26% for X and 26%/16% for Y by PLS1 and PLS2, respectively. Higher

principal components were studied, but they did not reveal further information. The hardness and centrifugation loss were closely associated with T_{21} relaxation times centered at 12–55 ms, which reflect relatively low water mobility. Such low relaxation times were mainly observed in the fillets salted in 25% brine. The longer T_{21} component centered at 71–100 ms, mainly observed in 15% brine salted fillets, was closely associated with Δ weight, z^{NaCl} , water content (xw), pH, and a_w (**Figure 2**).

The present study revealed differences in water mobility when fatty (Atlantic salmon) and lean (Atlantic cod) fish were salted in 15 and 25% brines. Salting in 15% brines lead to increased water mobility, whereas salting in saturated brines had the opposite effect. Possible explanations to the observed changes are the salting in and salting out mechanisms for 15% brines and saturated brines, respectively. In general, the observed changes in water mobility occurred more rapidly in cod than in salmon. This can be explained by the differences in fat content, as fat is known to act as a diffusion barrier (35). The comparison of T_2 relaxation data with physicochemical analyses revealed good linear correlations ($F \leq 0.05$) with WHC, centrifugation loss, a_w , and z^{NaCl} for both cod and salmon fillets salted in both brines. Good correlations were also found between T_2 relaxation data and the fillet pH as well as the Δ weight for some of the fish groups. T_2 relaxation proved to be a sensitive tool to observe water mobility changes in fish muscle during salting and offers a useful alternative for investigating water properties during fish processing.

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