Food Control 121 (2021) 107613

Contents lists available at ScienceDirect

Food Control

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

Desalting of dried salt-cured cod (*Gadus morhua* L.) without water renewal - 3D imaging of volume change

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ARTICLE INFO

Keywords:
Dried salt-cured cod
Desalting
Stirring
Weight
Volume
Water content
Salt content
Microbial growth

ABSTRACT

Desalting of dried salt-cured cod without water renewal with stirring every 12 h, and in stagnant water, was explored. Products of about 450 g were desalted for 96 h, followed by refrigerated storage in 120 h. During the desalting and subsequent storage, the change in weight, volume, salt-, and water content was calculated. Two different methods for calculation were applied, namely calculating the changes time by time, and by calculating the kinetic constants, k_1 and k_2, which express the total mass variation by pressure gradients, and the overall pseudo-diffusional and hydrodynamic mechanisms, respectively. A positive correlation between the change in weight, volume, and water content was observed, and these variables correlated negatively with the change in salt content. By calculating changes time by time, a significant difference in weight and salt content of the surface between the two desalting regimens was observed only after 96 h. The kinetic constants did, however, show a significant difference between the two desalting regimens for weight, salt, and water. A positive correlation between the volume and weight was observed irrespective of the desalting regimens. This indicates a future potential in applying an improved 3D imaging as an indicator of the weight changes during the desalting process.

1. Introduction

In the North Atlantic countries, salting and drying of cod and other fish species have been going on for centuries. Salted and dried fish products are mainly produced in Norway, Iceland, Faroe Islands, Portugal, Canada, Russia, and China, with the main markets in Latin America, Africa, the USA, and southern Europa.

Salted fish must be rehydrated, i. e,. desalted before use. During the desalting, the protein matrix is rehydrated while the salt content decreases to levels making the product ready for further preparation and consumption. Traditionally, the desalting process has been performed in households by immersion of the products in tap water for at least 24 h (Barat, Rodríguez-Barona, Andrés, & Ibáñez, 2004). To satisfy consumer requirements for ready-to-prepare food products, industrial desalting has evolved (Kurlansky, 2011). A variety of desalting procedures has been explored to control and optimize the process. For example, desalting with different ratios between water and product (Muñoz-Guerrero, Gutiérrez, Vidal-Brotons, Barat, Gras, & Alcaina, 2010), different product thickness (Oliveira et al., 2016), desalting at different water temperatures (Oliveira et al., 2014), and desalting using technology to minimize the total desalting time applying tumbling and injection technologies (Bjørkevoll et al., 2004).

Salt affects the water holding capacity (WHC) of muscle proteins and thereby, the quality of the desalted fish product. During the desalting process, the WHC of the protein matrix increases as the myofibrils undergo a substantial swelling in the presence of salt. In the case of the rabbit *psoas* muscle, it has been shown that the myofibrils swell to about twice their original volume in a salt solution and that the maximum WHC occurred when the salt concentration was more than 4.6% on a wet basis (Offer & Trinick, 1983). The desalting process contributes to substantial changes in weight, salt, and water content of the product and this has been thoroughly described (Barat, Rodríguez-Barona, Andrés, & Ibáñez, 2004; Barat, Rodríguez-Barona, Andrés, & Visquert, 2004; Barat et al., 2006). Another study showed that mass transfer was not influenced by the stirring rate (Andrés et al., 2005). In this experiment, the total desalting time was only 8 h, due to small sample size (4 × 2 × 1 cm), and the stirring was performed continuously at rates ranging from 60 to 500 rpm.

It has been shown that desalting without water renewal resulted in a higher yield when compared to traditional desalting with water renewal (Barat, Rodríguez-Barona, Castelló, Andrés, & Fito, 2004). No water renewal implies a lower water requirement during the desalting process,

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https://doi.org/10.1016/j.foodcont.2020.107613
Received 2 June 2020; Received in revised form 11 August 2020; Accepted 7 September 2020
Available online 9 September 2020

and thereby a lower wastewater volume. This aligns well with the requirements in minimizing the use of natural resources (https://sustainabledevelopment.un.org).

Nowadays, the industrial desalting process of dried salt-cured cod is managed by controlling the time. A fixed desalting time is not necessarily sufficient as geometry and product weight varies. Thus, a method to control this process have been developed (Santos, Mesquita, Galvis-Sánchez, Delgadillo, & Rangel, 2011). The method is a sequential injection analysis for real-time turbidimetric determination of salt concentration in the desalting water.

Concurrently to the changes in salt concentration of the desalting water, the products increase in weight due to the water uptake, resulting in volume expansion. Previously, measurements of volume have been applied in the development of machines for weight estimation before cutting the fish into portions (https://marel.com/products-solutions/i-cut-130-portioncutter-fish/) and for estimation of volume and size for fish and fish products (Marelec Food Technologies, Nieuwpoort, Belgium). To our knowledge, analyses of volume during the desalting process have not been reported.

In this study, the aim has been to 1) explore if stirring influences the change in weight, volume, water-, and salt content in products of about 450 g, and 2) if 3D imaging for volume measurement can be applied as an indicator of the desalting process.

2. Material and methods

2.1. Raw material

In 2017, the cod was captured using a long line. Onboard, the fish was gutted, headed, and frozen. After landing and frozen storage of one month, the cod was thawed, split, salted, dried, and cut. In this study, products from the loin region with an average weight of 459 (±62) g and the dimensions (length x width x thickness) of 6.8 (±0.3) x 18.5 (±1.4) x 5.1 (±0.6) cm were used. Before the desalting, the products were stored at 4 °C for one year.

2.2. Desalting procedure

Two different desalting regimens were performed in two separate containers of 556 L (Sæplast Iceland ehf, Dalvik, Iceland) in 96 h. In container no. 1, the stirring was performed every 12 h (12 h stirring regimen), while in container no. 2, the desalting was performed in stagnant water as a control.

Initially, a net was put at the bottom in each container. The nets enabled an efficient removal of the products, drainage, and sampling during the desalting period. In advance, the nets were sterilized at 120 °C to minimize the risk of contamination of the products. In total, 49 samples were included in the study, with 24 and 25 in container no. 1 and 2, respectively. The volume of the dried salt-cured cod was 431 (±66) and 478 (±65) cm^3, with a weight of 445 (±61) and 493 (±53) g, in container no. 1 and 2, respectively. Each sample was ID tagged (Floy Tag & mgf. Inc., Seattle, WA, USA) using a pistol (Floy Tag) before being evenly distributed in one layer with the skin side facing down on the container bottom. Fresh water was then added to the two containers obtaining a 9:1 water to product ratio.

During the desalting, the containers were closed with a lid to minimize the evaporation and the risk of cross-contamination. To ensure a constant temperature during the desalting, the containers were placed in a room at 4 °C. The temperature was logged every 10 min using K-type thermocouples connected to data loggers (model 175H1, Testo Ltd., Hampshire, UK).

2.3. Sampling and analyses

Sampling was performed both before ($t = 0$) and during the desalting in water, as well as during the subsequent storage at 4 °C in a climate chamber (Binder GmbH, Tuttlingen, Germany). The total time of desalting and subsequent 4 °C storage was 216 h. During the desalting period (96 h), the products were sampled every 24 h. In the following storage at 4 °C, a sampling after 24, 72, and 120 h, corresponding to a total desalting and storage time of 120, 168, and 216 h, was performed. During the desalting period, the nets were gently withdrawn from the containers allowing drainage in about 10 min. The weight was registered, followed by 3D imaging of all products. Then, three products were randomly sampled from each container for analyses of the salt- and water content. Before putting the net with the remaining products back into the water, stirring of the water in container no. 1 was performed in 10 min using a spade. The stirring procedure was repeated every 12 h.

Before the removal of the products and the stirring, the desalting water was analyzed every 24 h for salt content using a refractometer (ATAGO Master-Sur/NM, Tokyo, Japan). The samples were obtained from the center of each container at three different depths. In detail, the samplings were performed at about 4 cm corresponding to just above the products, in the middle of the water depth, and about 2 cm below the water surface.

After 96 h, the remaining 37 samples were single packed in plastic bags (Finnvacuum Sverige AB, Bandhagen, Sweeden), semi-closed, and stored in an additional 120 h at 4 °C in the climate chamber. During the desalting period, the sampled products were analyzed with respect to weight, volume, salt-, and water content. In the following storage in the climate chamber, microbial analyses were also included with samplings after 24, 72, and 120 h. Besides, drip loss as a function of the weight after desalting in water (96 h) was calculated at the corresponding sampling points. The salt- and water contents were analyzed from a) the surface (5 mm thickness), and b) from the core of the product. Muscle samples from the core were obtained by cutting the product in the middle along the spine, splitting the product into two halves. Then, the muscle sample of about 1 cm^3 was obtained from the core from one of the halves. Samples for microbial analyses were obtained from the surface only.

2.4. Salt- and water content of the products

The salt content was determined using sample homogenization according to Volhard method (AOAC, 1995). The water content was measured by oven drying at 105 °C for 24 h (Cohen, 1971). All analytical determinations were performed in two replicates for each sample.

2.5. 3D imaging

To estimate the volume, 3D imaging was performed with a Gocator 2370 camera (LMI Technologies BV, Kerkrade, The Netherlands). The camera was mounted 53 cm above a flat conveyor belt, with the laser line pointing downwards perpendicular to the conveyor belt, and the camera looking down at an angle relative to the laser line (Fig. 1). The image resolution was 0.3, 0.5, and 0.0022 mm across the belt (x), along the belt (y), and in the vertical (z) directions, respectively.

Due to the rough surface structure and the sharp sample surfaces, some artifacts were observed in the height measurements. To correct for these effects, some preprocessing steps were performed before the volume was calculated (Fig. 2). Initially, the sample was segmented out based on a height threshold, then large objects, not the sample, was removed from the segmentation before a dilate/erode operation was varied out to fix and preserve sample surfaces. Finally, small "noise" objects were removed to finalize the segmentation process (Fig. 2, lower left side). Secondly, missing data from the height measurement, within the identified sample, was reconstructed based on surrounding data. The last step was then to smooth the height data with a 13 × 13 median operator (Fig. 2, lower right side). Finally, the volume was calculated as follows.

$$V = \sum_{x,y} Height(x,y)\Delta x \Delta y \quad (1)$$

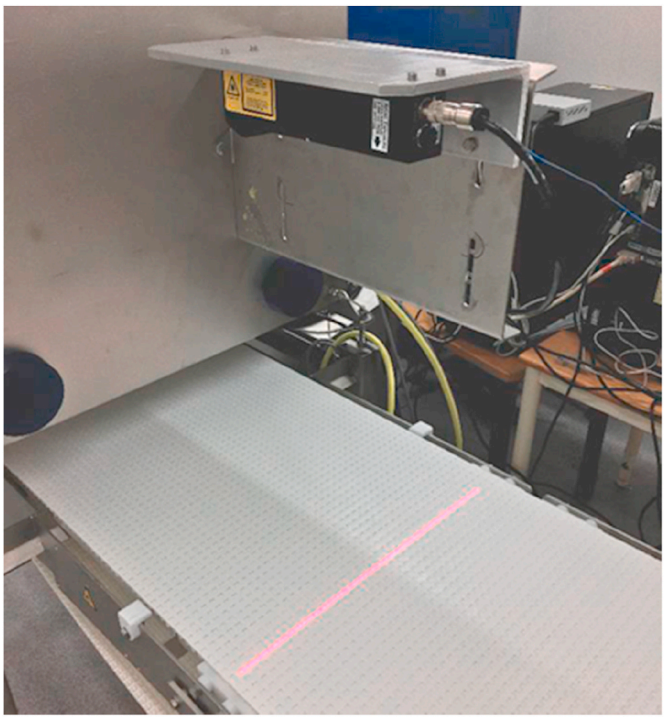

Fig. 1. 3D camera mounted above conveyor belt.

where Δx and Δy are the spatial resolutions across the belt and along the belt, respectively.

2.6. Desalting kinetics

To obtain an overview of the effect of the two desalting regimens, and the subsequent 4 °C storage, the relative mass changes (ΔM_t^0) for the two groups were calculated from time $t = 0$:

$$\Delta M_t^0 = \frac{M_t^0 - M_0^0}{M_0^0} \tag{2}$$

where M_t^0 is the weight at sampling time t (Barat et al., 2006). Furthermore, the relative volume change (ΔM_t^V) was calculated as:

$$\Delta M_t^V = \frac{V_t^0 - V_0^0}{V_0^0} \tag{3}$$

where V_t^0 is the volume at sampling time t.

Also, the changes in water and salt content (ΔM_t^w and ΔM_t^{NaCl}) according to the weight of the samples (M_t^0) were calculated as:

$$\Delta M_t^w = \frac{M_t^0 x_t^w - M_0^0 x_0^w}{M_0^0} \tag{4}$$

$$\Delta M_t^{NaCl} = \frac{M_t^0 x_t^{NaCl} - M_0^0 x_0^{NaCl}}{M_0^0} \tag{5}$$

were x_t^w and x_t^{NaCl} are the weight fraction at time t for water and NaCl, respectively (Barat et al., 2006). In this work, we have studied the change in water and salt measured in the core and surface of the samples in relation to the total sample weight.

The changes in all variables throughout the first 96 h were fitted to the experimental data, supposing that the changes in weight, volume, salt-, and water content were related to the square root of time, and assuming pseudo-diffusional transportation (Fito & Chiralt, 1997).

$$\Delta M_t^i = k_1 + k_2\sqrt{t} \tag{6}$$

At the start of the desalting process, the k_1 could be connected to the total mass variation by pressure gradients, while k_2 represents the overall pseudo-diffusional and hydrodynamic mechanism on the sample weight change, and t refers to sampling time (Barat, Rodríguez-Barona, Andrés, & Ibáñez, 2004).

In addition, density during the desalting period was calculated by dividing the sample weight (g) with the volume (cm^3).

2.7. Microbial analysis

Muscle samples for microbial analyses of the products surface were obtained after 24, 72, and 120 h during the 4 °C storage. Every sampling, three replicates were obtained from each container. The muscle sample of about 5 g was cut from the products surface and transferred to a stomacher bag with filter (Seward Medical Ltd., London, UK.). The sampling was followed by a 1:5 dilution with sterile saline water 2% w/v NaCl and 0.1% w/v peptone (Difco Laboratories, Sparks, MD, USA). The stomacher bag was then pummeled for 2 min in a Lab Blender 400 Stomacher (Seward Medical Ltd.). Total viable psychrophilic counts were obtained after serial decimal dilutions on tryptone soy agar plates (TSA) (Merck KGaA, Darmstadt, Germany) added 2% NaCl. After incubation at 20 °C in 3–5 days, the enumeration was performed.

2.8. Statistical analysis

The differences in the data obtained from the two desalting regimens at each sampling time were explored, performing Two-Sample t-Test using SYSTAT 13 for Windows version 13.2 (Systat Software Inc., San Jose, CA, USA). In addition, analysis of variance was carried out to determine significant differences in the data between all sampling times for each variable.

To estimate the constants k_1 and k_2 for each data set and to calculate the related statistics, a software solution was developed using IDL 8.7.3 (Harris Geospatial Solutions, Bracknell, United Kingdom). The k_1 and k_2 were calculated, minimizing the quadratic error. The standard error for k_1 and k_2, and the model fit (standard error and R^2) was estimated for each response variable and desalting regimens.

All data were analyzed by performing Principal Component Analysis (PCA) using The Unscrambler version 10.3 (CAMO Process AS, Oslo, Norway). The PCA-analysis was used to identify any differentiation in the data obtained during different desalting regimens. Prior to the PCA, all variables were weighted by 1/STDEV in order to standardize the data. All analyses were performed at 5% probability level ($p = 0.05$).

3. Results and discussion

3.1. Desalting conditions

The desalting was performed at a weight water and product ratio of 9:1. During the daily withdrawal of three products from each container, this ratio changed. To obtain a constant 9:1 ratio, a calculated volume of the desalting water was removed from the containers after each sampling.

The salinity of the water measured just above the products was 2.1 and 3.1% in container no. 1 and 2 after 24 h of desalting time, respectively. In water samples obtained from the middle and 2 cm below the surface after 24 h of desalting time, the salinity was 1.5 and 0.4% in container no. 1 and 2, respectively. After 96 h, the salinity was about 2.3% irrespective of the location of the sampling or desalting regimen. An equalization of the salinity after 24 h desalting is partly assumed to be due to the increasing salt content of the water, and partly by the slight mixing of the water in container no. 2 as a result of the withdrawal of the samples in the net.

The average water temperature during the desalting period in water and the climate chamber was 4.18 (±0.17) and 3.82 (±0.73) °C,

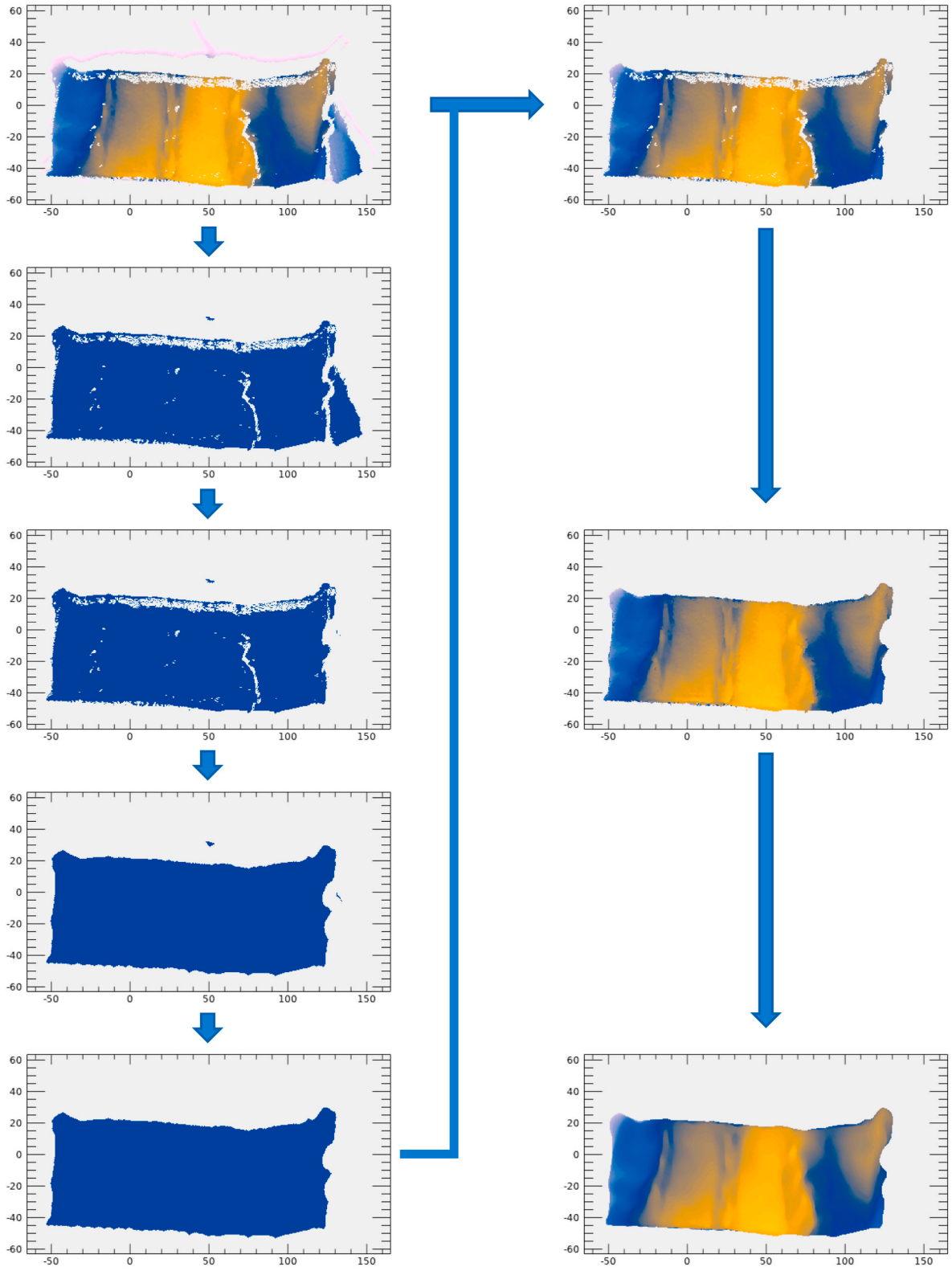

Fig. 2. Left side from top to bottom; 1) Raw height data, 2) segmented with an 8 mm threshold, 3) large object(s), not sample, removed, 4) dilate/erode operation with a 21x21 circular kernel, and 5) the final segmentation after removing small objects. Right side from top to bottom; 1) raw height (top left) restricted by segmentation (lower left), 2) gaps in height data filled based on surrounding data, and 3) final height data after a 13x13 median operation.

respectively.

3.2. Desalting kinetics

During the desalting period, the weight, volume, and water content increased while the salt content decreased (Fig. 3). The changes in

weight, volume, salt-, and water content (Eqs. (2)–(5)) of the products were most predominant in the first 24 h. The changes in salt- and water content occurred earlier and at a higher rate in the products surface (Fig. 3A) than in the core (Fig. 3B). Although not being significant, stirring every 12 h resulted in a lower salt- and higher water content in the products surface after 24 h when compared to desalting in stagnant water (Fig. 3A). During the following desalting, the salt content in the surface was slightly lower in the 12 h stirring regimen compared to the control, i.e., desalting in stagnant water. The weight change did not differ between the two desalting regimens. In the surface samples from the 12 h stirring regimen, the equilibrium of the rehydration on the surface was reached simultaneously with the desalting equilibrium, namely after 48 h (Fig. 3A). The surface equilibrium was defined as the time when the subsequent samplings during the desalting did not differ significantly. On the contrary, products desalted in stagnant water reached the rehydration surface equilibrium after 48 h, while the desalting equilibrium was reached after 72 h. This shows that the time to reach the surface equilibrium for salt was clearly influenced by stirring. Differences between the rehydration and desalting process have also been demonstrated by the group of Barat et al. (2006), and it was explained by changes in the bound water, i.e., the protein hydration water is slower than in the transport of free water.

Samples obtained from the core, reached an equilibrium of rehydration after 72 h, while the corresponding desalting equilibrium was reached after 120 h, independent of the desalting regimens (Fig. 3B). As expected, the product size and weight influenced the rate of salt and water transfer, and that interpreting the effect of the desalting regimens should preferably not be performed on the core samples. Besides, the mass transfer of salt continued after the withdrawal of the product from the desalting water as well. This phenomenon could potentially be utilized in the distribution of the products to avoid loss in shelf life.

Irrespective of the desalting regimen, the changes in the weight and volume of the product followed a similar pattern throughout the trial. During the desalting time, the products weight and volume increased. Then, after the initial 24 h of the 4 °C storage, a noticeable decline in weight and volume was observed. Following, no significant changes in either the products volume or weight occurred during the subsequent 4 °C storage. The decline in both weight and volume was probably due to drip loss of about 4%, which was detected simultaneously with the observed decline. During the remaining 96 h, a drip loss of only 1% was observed. The drip loss was not significantly affected by the desalting regimens.

3.3. Model fit to experimental data

The kinetic constants for the overall mass transfer, k_1 and k_2 were estimated for the two desalting regimens (Eq. (6)) (Table 1). The k_1 constant was significantly different between the two desalting regimens for all parameters except for the change in volume. Similar results were obtained for the k_2 constant. As described by Barat, Rodríguez-Barona, Andrés, & Ibáñez, 2004, the k_1 and k_2 estimates should be based on the total desalting period ($t = 0, 24, 48, 72, 96$). However, when studying the response variables collected from the sample surface, the fit was poor, as visualized for the change in salt content during the 12 h stirring regimen (Fig. 4A, dotted line). However, by excluding time, $t = 0$, from the estimation, a better fit was obtained (Fig. 4A, dashed line). The differences between the fitted models were small for the salt content in the core of the samples with a 12 h stirring regimen (Fig. 4B). Thus, in order to be able to analyze both surface and core samples, the estimates for k_1 and k_2 were estimated by excluding time $t = 0$.

According to Fig. 3, significant differences in weight change, ΔM_t^0, between the two desalting regimens were not detected when analyzing the replicates in every 24 h interval (24, 48, 72, and 96 h). However, k_1 and k_2 for ΔM_t^0 were significantly different when comparing the two desalting regimens (Table 1). This shows that the kinetic parameters reveal differences between the two desalting regimens with a higher k_1 for the 12 h stirring regimen compared to the desalting in stagnant water. This can be explained with a higher total mass variation by pressure gradients early in the desalting process when stirring was applied. On the other hand, k_2, was lower when stirring was performed every 12 h, giving a lower overall pseudo-diffusional and hydrodynamic mechanism for the sample weight change. This is reasonable since both desalting regimens resulted in similar equilibrium levels for the weight change ΔM_t^0.

It appears that the change in volume, ΔM_t^V, also depends on the desalting regimens (Fig. 3). However, there is no significant difference in k_1 and k_2 when comparing the two desalting regimens (Table 1). This is most probably because the change in volume does not appear to be well described by eq. (6) (Table 1, model fit parameters) combined with rather large sample variations. The high variance in estimated volumes can be related to the choice of instrumentation for 3D imaging. As the sample cut surfaces were sharp, a setup with only one camera measuring the sample height was not ideal. The laser line on one side of the sample turned out to be invisible for the camera, making the volume estimates less accurate.

Kinetic constants were also calculated for the changes in water content, ΔM_t^w in samples obtained from both the surface and the core (Table 1). By analyzing the replicates at 24, 48, 72, or 96 h, a significant difference between the two desalting regimens was observed after 96 h in samples obtained from the surface only (Fig. 3A). For both the surface and the core, a significant difference in the development of water change was observed (Table 1, k_1 and k_2). Stirring every 12 h, resulted in a

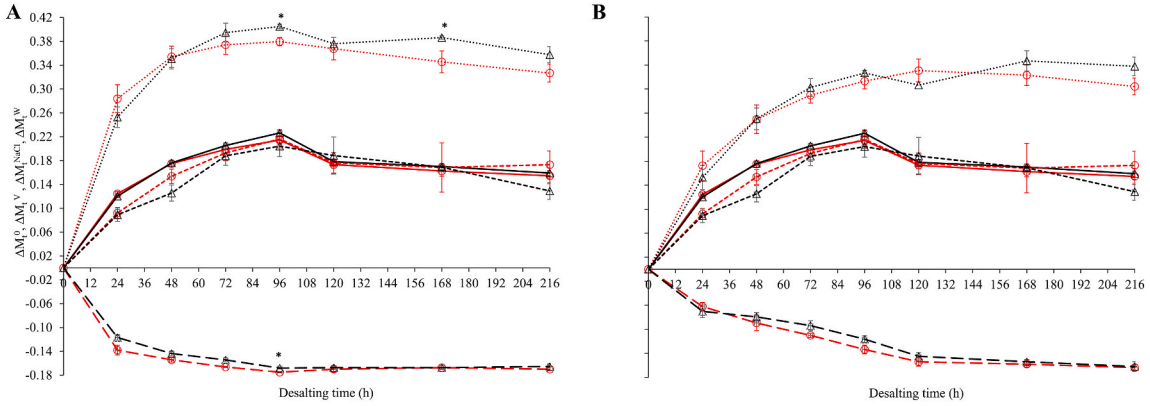

Fig. 3. Changes in weight (—) volume (——), salt (– – –), and water (….) in samples desalted with stirring every 12 h (o), and in stagnant water (Δ). Changes in samples obtained from the surface (A) and from the core (B) throughout the desalting time in water and subsequent storage at 4 °C. Vertical bars show the standard error. Asterix shows significant differences between the desalting regimens at each sampling.

Table 1
Kinetic parameters, k_1 and k_2 for changes in weight, volume, water-, and salt content adjusted to Eq. (6).

	Desalting regimen	Sample location	$k_1 \pm SE^a$	$k_2 \pm SE^a$	Model fit	
					SE^a	R^2
ΔM_t^0	12 h	Whole	0.030 ± 0.004*	0.019 ± 0.001*	0.024	0.677
	Control		0.017 ± 0.003	0.022 ± 0.001	0.018	0.838
ΔM_t^v	12 h	Whole	−0.026 ± 0.012	0.027 ± 0.001	0.045	0.573
	Control		−0.030 ± 0.020	0.026 ± 0.002	0.061	0.376
ΔM_t^w	12 h	Surface	0.200 ± 0.017*	0.020 ± 0.002*	0.030	0.627
	Control		0.112 ± 0.014	0.032 ± 0.002	0.029	0.829
	12 h	Core	0.039 ± 0.019*	0.029 ± 0.002*	0.035	0.771
	Control		−0.013 ± 0.015	0.036 ± 0.002	0.028	0.873
ΔM_t^{NaCl}	12 h	Surface	−0.101 ± 0.006*	−0.008 ± 0.001*	0.007	0.833
	Control		−0.069 ± 0.004	−0.010 ± 0.000	0.007	0.905
	12 h	Core	0.010 ± 0.007*	−0.014 ± 0.001*	0.011	0.889
	Control		−0.021 ± 0.008	−0.009 ± 0.001	0.014	0.645

[a] SE: Standard error.
Note. Asterisks indicate significant differences ($p < 0.05$) between the two desalting regimens within the values of k_1 or k_2 for each variable.
Abbreviations. ΔM_t^0, change in weight; ΔM_t^V, change in volume; ΔM_t^w, change in water content; ΔM_t^{NaCl}, change in salt content.

higher total mass variation by pressure gradients early in the desalting process (k_1), and a lower overall pseudo-diffusional mechanism (k_2). The initial water uptake, as described by k_1, was significantly higher in the sample surface compared to the core samples in both desalting regimens.

As expected, the change in salt content, ΔM_t^{NaCl}, was negative. As for the change in water content, a significant difference between desalting regimens was only observed in the surface samples after 96 h desalting (Fig. 3A). Regarding the change of the salt content, a significant difference was observed in both k_1 and k_2 for both desalting regimens (Table 1). This was the case for samples obtained from both the surface and the core. By combining knowledge gathered from the changes in weight, salt, and water content, it is evident that stirring every 12 h speeds up the desalting process on the 'products surface in the initial phase (Table 1).

Estimating the changes during desalting applying Eqs. (2)–(5), a significant difference between the two desalting regimens was observed after 96 h only for change in the water- and salt content (Fig. 3A). However, estimating the kinetic parameters k_1 and k_2 applying Eq. (6) (Table 1), significant differences between the two desalting regimens were observed for weight, water-, and salt content. This illustrates that change in the weight, salt, and water is significantly different between the two desalting regimens, even if there is no significant difference at a specific sampling time. It is noteworthy that these results are based on different estimations, and thus, a direct comparison cannot be performed. Besides, the changes in weight, volume, salt-, and water contents are estimated for both the desalting period and the storage at 4 °C of total 216 h, while the estimation of the kinetic parameters is performed in the desalting period only.

3.4. Microbial growth

The level of psychrophilic at each sampling time (24, 78, and 120 h) did not differ significantly between the two desalting regimens. The number of psychrophilic were in the ranges from log 5.26 (±0.33) to 5.55 (±0.30), 7.25 (±0.09) to 7.75 (±0.10), and 8.40 (±0.10) to 8.77 (±0.30) CFU/g in samples analyzed after 24, 72, and 120 h of refrigerated storage time, respectively.

After 24 h of storage at 4 °C, the microbial growth was in its exponential phase. Although the sensory evaluation was not included in this study, no off-odor was observed when preparing for microbial analysis at this sampling time. After 72 h storage, however, a distinct off-odor was noticed, turning the product sensory unacceptable. Taken into consideration the microbial growth pattern, the shelf life in terms of sensory acceptance was in the range from 24 to 72 h, during the refrigerated storage. In the work of Bjørkevoll et al. (2003), the rehydrated dried salt-cured cod product was considered as sensory unacceptable within 7–10 days when stored at 4 °C. They showed that the dominating bacterium after rehydration belonged to the genus *Psychrobacter*, which originates from the skin mucus of fresh fish. *Psychrobacter* can survive as non-growing cells during the period of salting and drying and to recover during the desalting process (Bjørkevoll et al., 2003). The shelf life of 7–10 days is most probably due to a desalting period of only 24 h due to the low product weight, i.e., 20–30 g.

3.5. Main effects and future perspectives

In order to identify any differentiation amongst samples as well as to explore all possible correlations between the response variables, the data were subjected to a principal component analysis (PCA) (Fig. 5).

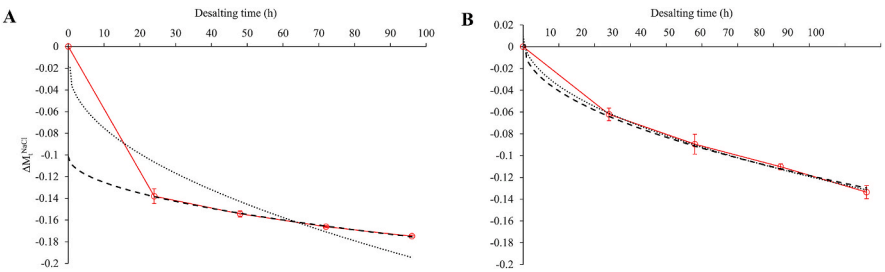

Fig. 4. Changes in salt content in the surface (A) and the core (B) of the product detected during the 12 h stirring regimen. Curve fit without "desalting time 0 h" (—), curve fit with "desalting time 0 h" (….), and curve "actual" (——). Vertical bars show the standard erro.

The variables used in the PCA included both measured and calculated parameters obtained at all samplings during the trial of 216 h. Measured parameters include weight, volume, salt-, and water content, while calculated parameters include data applying Eqs. (2)–(5), and density. The score and correlation loading plots illustrate that the first and the second principal components (PC-1 and PC-2) cumulatively accounted for 88% of the data variance. Along PC-1, 74% of the data variance was explained. This is assumed to be due to the extreme product changes like increased water content and, at the same time, a decrease in salt content during the desalting process.

In the score plot, a distinct pattern in the data followed mainly PC-1, corresponding to the desalting time (Fig. 5A). As expected, no grouping of the samples prior to the desalting was observed. Then, a slight grouping in the data for the two desalting regimens was observed after 24 h desalting. Afterward, this grouping became less clear (Fig. 5A, right side). This indicates that the stirring of water influenced the desalting process, mainly in the first 24 h. This is assumed to be related to the more considerable changes in salt- and water contents in the products surface during the 12 h stirring regimen compared to desalting in stagnant water (Fig. 3A).

According to the correlation loadings plot, a positive correlation between the changes in weight and volume, as well as water content both in the surface and in the core of the products were observed (Fig. 5B, right side). These variables correlated negatively with the salt content and changes in salt content both in surface and core (Fig. 5B, left side). Further, when studying the score and the correlation loading plots simultaneously, it is evident that the products went through remarkable changes in weight, volume, salt-, and water content. This is clearly due to the water uptake, which resulted in a highly rehydrated protein matrix. The decrease in salt content caused increased WHC of the muscle protein and thereby a weight gain. Besides, a change in the products textural and histological properties was assumed to occur according to previously reported observations ((Albarracín, Sánchez, Grau, & Barat, 2011); Barat, Rodríguez-Barona, Andrés, & Ibáñez, 2004 and Barat, Rodríguez-Barona, Andrés & Visquert,2004).

Besides, a positive correlation between the weight and volume of the products was observed (Fig. 5B). It tends that the development in these variables followed PC-2, although with only 14% of variation explained. The products density was estimated to discover a potential relationship with the other variables. A negative correlation between the density and the weight and volume of the product was observed. This is in accordance with previous observations reported by Barat, Rodríguez-Barona, Andrés & Visquert, (2004).

The correlation between the changes in weight and volume during the desalting time, as well as changes in density, shows that 3D imaging can be applied to indicate the progress of the desalting process. As only one camera was used in the volume measurements, a considerable variation in the volume data was obtained, resulting in a non-significance between the two desalting regimens (Table 1). It is assumed that improvements in terms of a more sophisticated 3D-imaging setup with two cameras and one laser line to avoid "invisible" areas would lead to less variation. By implementing an improved 3D imaging system, a verification of the data in this study should be performed. Succeeding with this, an easy, fast, real-time, and non-destructive method could be implemented to monitor the desalting process. In large scale desalting process facilities, on-line volume measurements covering large volumes would be beneficial in terms of saved costs.

In this study, a desalting time of 96 h was required to obtain a salt level of about 2%. The long desalting time is strongly coherent with the product weight of about 450 g. The distance from the surface to the core defines the time to obtain a low and even salt concentration. The products tested in this experiment were large, resulting in significant differences in salt content between the surface and the core. A desalting time of 96 h resulted in limited remaining shelf life. From an industrial and a marketing point of view, this is unfavorable as it leads to an increased risk of economic loss due to severe microbial growth in a short time after the rehydration. To prolong the shelf life, freezing of the desalted product right after the desalting process is an alternative. Besides, frozen desalted products are more flexible in terms of distribution

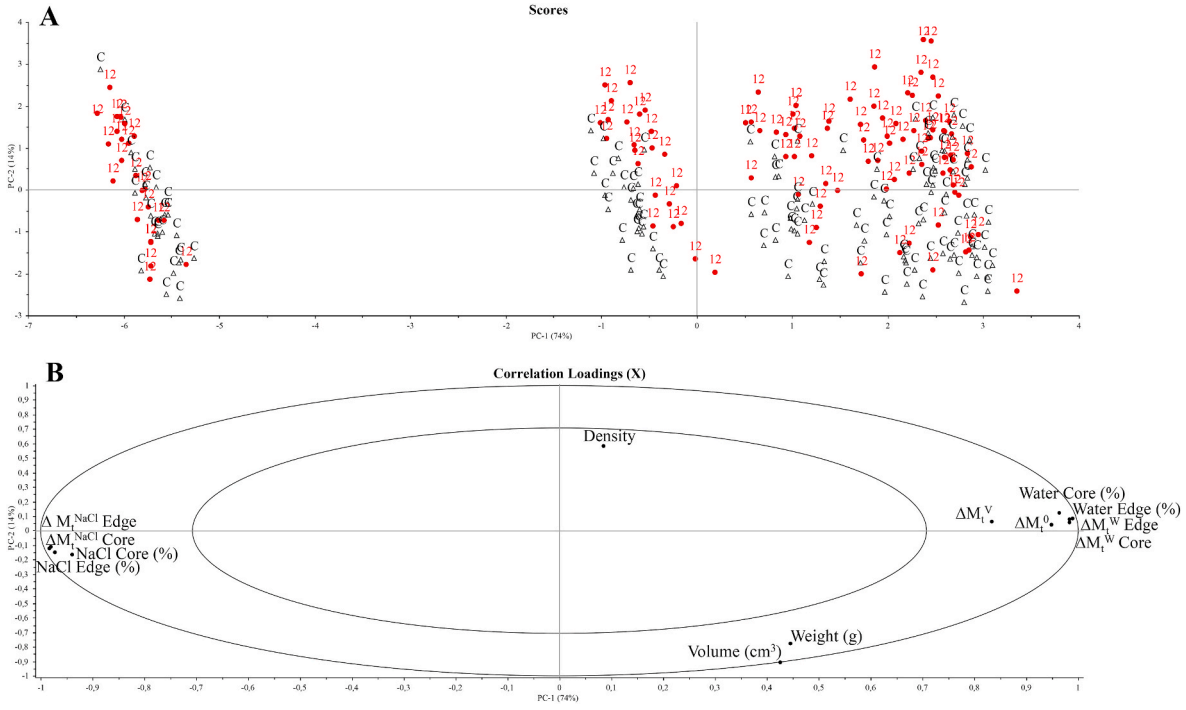

Fig. 5. Score plot (A) with observations grouped by the two desalting regimens, 12 h stirring regimens (12) and desalting in stagnant water (C). The correlation loading plot (B) show the correlation between the response variables Weight (g), ΔM_t^0, Volume (cm^3), ΔM_t^V, NaCl Edge %, NaCl Core %, ΔM_t^{NaCl} Edge, ΔM_t^{NaCl} Core, Water Edge %, Water Core %, ΔM_t^w Edge, ΔM_t^w Core, and Density. The outer and the inner ellipse indicate 100 and 50 % explained variance, respectively.

systems. From a consumer's point of view, frozen products are more adaptable compared to the purchase of fresh desalted products with a rather short shelf life. In case the freezing of the desalted products is not an option, smaller products than used in this experiment are recommended. Smaller products require a shorter desalting time, leading to shelf life of more than 24–72 h when stored at 4 °C.

4. Conclusion

A positive correlation between the change in weight, volume, and water content was observed, and these variables correlated negatively with the change in salt content. The changes of all these variables were estimated by applying two methods, a) by comparing the values time by time, and b) by calculating kinetic constants for both the initial and the overall desalting process. By comparing the values time by time, a significant difference in weight and salt content of the surface between the two desalting regimens were observed only after 96 h desalting. The kinetic constants, however, did show a significant difference between the two desalting regimens for all variables except for the volume, which was calculated by the 3D images.

The 3D imaging was not optimized, resulting in a high variation in the volume data. By implementing a more sophisticated 3D imaging setup, a lower variation in the volume data is expected. The positive correlation between the volume and weight indicates a future potential in applying 3D imaging as an indicator of the weight of the products during the desalting process.

Overall, the choice of method for evaluating the changes during the two desalting regimens clearly influences the information in terms of the detailed level of mass transfer. In our study, significant differences between the two desalting regimens were obtained for changes in salt, water, and weight when calculating the kinetic constants. This shows that there is a significant difference between the two desalting regimens regarding how the weight, salt-, and water content develops during the desalting period.

Declaration of competing interestCOI

The authors declare no conflicts of interest.

CRediT authorship contribution statement

Grete Lorentzen: Conceptualization, Funding acquisition, Project administration, Supervision, Methodology, Validation, Investigation, Resources, Visualization, Writing - original draft, Writing - review & editing. **Tatiana N. Ageeva:** Conceptualization, Methodology, Validation, Investigation, Resources, Visualization, Writing - original draft, Writing - review & editing. **Karsten Heia:** Methodology, Validation, Investigation, Resources, Visualization, Writing - original draft, Writing - review & editing.

Acknowledgement

The results presented are deliverables from the project "CoPro" partly funded by the Norwegian Research Council (project no. 256467) and Norwegian Seafood Research Fund (project no. 901182). We want to thank Ingebrigt Bjørkevoll, Møreforsking, for valuable help.

References

Albarracín, W., Sánchez, I. C., Grau, R., & Barat, J. M. (2011). Salt in food processing; usage and reduction: A review. *International Journal of Food Science and Technology, 46*(7), 1329–1336. https://doi.org/10.1111/j.1365-2621.2010.02492.x.

Andrés, A., Rodríguez-Barona, S., & Barat, J. M. (2005). Analysis of some cod-desalting process variables. *Journal of Food Engineering, 70*(1), 67–72. https://doi.org/10.1016/j.jfoodeng.2004.09.014.

AOAC. (1995). Method 937.09-Salt (chlorine as sodium chloride) in seafood. In P. Cunniff (Ed.), *Official methods of analysis of AOAC international (16th ed.). 3rd revision*. Gaithersburg, MD, USA: Association of Official Analytical Chemists (AOAC International).

Barat, J. M., Gallart-Jornet, L., Andrés, A., Akse, L., Carlehög, M., & Skjerdal, O. T. (2006). Influence of cod freshness on the salting, drying and desalting stages. *Journal of Food Engineering, 73*(1), 9–19. https://doi.org/10.1016/j.jfoodeng.2004.12.023.

Barat, J. M., Rodríguez-Barona, S., Andrés, A., & Ibáñez, J. B. (2004b). Modeling of the cod desalting operation. *Journal of Food Science, 69*(4), FEP183–FEP189. https://doi.org/10.1111/j.1365-2621.2004.tb06345.x.

Barat, J. M., Rodríguez-Barona, S., Andrés, A., & Visquert, M. (2004c). Mass transfer analysis during the cod desalting process. *Food Research International, 37*(3), 203–208. https://doi.org/10.1016/j.foodres.2003.11.001.

Barat, J. M., Rodríguez-Barona, S., Castelló, M., Andrés, A., & Fito, P. (2004a). Cod desalting process as affected by water management. *Journal of Food Engineering, 61*(3), 353–357. https://doi.org/10.1016/s0260-8774(03)00140-7.

Bjørkevoll, I., Olsen, J.-V., & Olsen, R. L. (2004). Rehydration of salt-cured cod using injection and tumbling technologies. *Food Research International, 37*(10), 925–931. https://doi.org/10.1016/j.foodres.2004.06.002.

Bjørkevoll, I., Olsen, R. L., & Skjerdal, O. T. (2003). Origin and spoilage potential of the microbiota dominating genus Psychrobacter in sterile rehydrated salt-cured and dried salt-cured cod (*Gadus morhua*). *International Journal of Food Microbiology, 84*(2), 175–187. https://doi.org/10.1016/s0168-1605(02)00418-x.

Cohen, E. H. (1971). Comparison of the official AOAC method with rapid methods for the analysis of moisture in meats. *Journal of AOAC International, 54*(6), 1432–1435. https://doi.org/10.1093/jaoac/54.6.1432.

Fito, P., & Chiralt, A. (1997). Osmotic dehydration: An approach to the modeling of solid food—liquid operations. *Food Engineering, 2000*, 231–252. https://doi.org/10.1007/978-1-4615-6057-9, 13.

Kurlansky, M. (2011). *Cod: A biography of the fish that change the world*. Canada: Vintage.

Muñoz-Guerrero, H., Gutiérrez, M. del R., Vidal-Brotons, D., Barat, J. M., Gras, M. L., & Alcaina, M. I. (2010). Environmental management of the residual brine of cod desalting. Quantification of mass transfer phenomena and determination of some parameters on the residual brine important for its treatment by membrane technology. *Journal of Food Engineering, 99*(4), 424–429. https://doi.org/10.1016/j.jfoodeng.2009.09.023.

Offer, G., & Trinick, J. (1983). On the mechanism of water holding in meat: The swelling and shrinking of myofibrils. *Meat Science, 8*(4), 245–281. https://doi.org/10.1016/0309-1740(83)90013-x.

Oliveira, H., Gonçalves, A., Nunes, M. L., Vaz-Pires, P., & Costa, R. (2014). Quality changes during cod (*Gadus morhua*) desalting at different temperatures. *Journal of the Science of Food and Agriculture, 95*(13), 2632–2640. https://doi.org/10.1002/jsfa.6995.

Oliveira, H., Gonçalves, A., Nunes, M. L., Vaz-Pires, P., & Costa, R. (2016). Influence of temperature and fish thickness on the mass transfer kinetics during the cod (*Gadus morhua*) desalting process. *Journal of the Science of Food and Agriculture, 96*(13), 4457–4464. https://doi.org/10.1002/jsfa.7657.

Santos, I. C., Mesquita, R. B. R., Galvis-Sánchez, A. C., Delgadillo, I., & Rangel, A. O. S. S. (2011). Development of a turbidimetric sequential injection system to monitor the codfish desalting process. *Food Analytical Methods, 5*(2), 287–295. https://doi.org/10.1007/s12161-011-9238-9.