LWT - Food Science and Technology 51 (2013) 218–224

Contents lists available at SciVerse ScienceDirect

LWT - Food Science and Technology

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

Development of a new salmon salting–smoking method and process monitoring by impedance spectroscopy

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ARTICLE INFO

Article history:
Received 12 March 2012
Received in revised form
18 September 2012
Accepted 25 September 2012

Keywords:
Salmon
Vacuum salting–smoking
Impedance spectroscopy
Process monitoring

ABSTRACT

In this work two objectives were proposed: (i) to optimize a new salmon salting–smoking method using vacuum packaging and (ii) to evaluate the application of impedance spectroscopy (IS) to the on-line monitoring of the process. Different processing conditions were evaluated (4 smoke flavoring (SF) salt concentrations, 3 salting times, salting in vacuum or in air). Physico-chemical analyses and IS measurements were performed with three different sensors during the process. Salting with 16 g SF salt/100 g fish in vacuum packaging provided smoked salmon similar to products currently available on the market. This new method has the advantages of reducing processing times and waste. IS measurements were carried out by three different electrodes. The most appropriate sensor for process monitoring was a needle electrode, with which robust prediction models for NaCl content, moisture and a_w during the salting–smoking process were obtained. The results showed the potential of IS as a rapid on-line monitoring method of the salmon salting–smoking process.

1. Introduction

Smoking is one of the oldest methods of fish preservation. The preservative effect of smoking is due to a combination of different factors, including the addition of salt, partial dehydration of the tissues that occurs throughout the different stages of the process, as well as the preservative action of the smoke components. The smoking process slows down the biological processes and oxidative damage and gives the final product sensory characteristics highly appreciated by consumers.

Improvements in the smoking process, including the reduction in processing times and the amount of brine wastes or the improvement of the hygienic quality would be of interest to this sector. To obtain high-quality smoked salmon with a long shelf-life, optimization of the various stages that constitute the smoking process is essential. The salting step is especially critical. A salting process in which the exact amount of salt to be absorbed by the fish would be directly dosed, combined with vacuum packaging, could be an alternative to these techniques. With this new method both brine wastes and contamination would be reduced, since the lack of oxygen in vacuum packaging would delay microbial growth and lipid oxidation. The main disadvantage that could present this method is the growth of anaerobic microorganisms, such as *Clostridium botulinum*. Smoke flavoring salt could also be used and would provide salt and a smoky flavor to the product in a single stage, so that the total processing time would be significantly shortened.

It is well-known that certain physico-chemical parameters, such as a_w or salt content, directly affect the shelf-life of smoked salmon. However, some studies have found high variability of these parameters within the same fish product (Cornu et al., 2006; Espe, Kiessling, Lunestad, Torrissen, & Røra, 2004; Fuentes, Fernández-Segovia, Barat, & Serra, 2010a), which have implications for consumers' safety and also for the sensory characteristics of the product. This is due to the fact that smoking processes are standardized for a certain fish species, without taking into account the effects of the initial characteristics (fat, moisture, fish size, freshness, etc.) of the raw material (Barat et al., 2006). In this regard, the development of rapid non-destructive methods for on-line monitoring of the process, in order to detect when the product has reached optimum moisture, salt and/or a_w values would be of interest to producers.

Electronic sensors based on impedance spectroscopy (IS) could help to meet this objective. The relationship between sodium chloride content and impedance measurements has already been

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http://dx.doi.org/10.1016/j.lwt.2012.09.025

demonstrated (Guerrero et al., 2004; Karásková et al., 2011). In the IS technique an electrical sinusoidal stimulus is applied to the electrodes in order to measure the impedance of the sample at different frequencies. The module and phase of the impedance can vary significantly according to the charges present (free ions), types of microstructure and electrolytes, as well as texture, geometry and the electrodes used (Masot, 2010).

In this work two objectives were proposed. The first was to optimize a new salting–smoking method for salmon using vacuum packaging. The second was to evaluate the application of impedance spectroscopy in the on-line monitoring of the salting–smoking process.

2. Materials and methods

2.1. Sample preparation

Fillets of Atlantic salmon from a Norwegian farm (Hallvard Leroy AS) of commercial size 1.4–1.8 kg was used as raw material. The fillets were purchased in a local supermarket and transported to the laboratory under refrigeration.

Fourteen salmon fillets were employed for the complete test (8 for the first phase and 6 for the second). The fillets were cut transversally into 4 cm portions, obtaining 6 or 7 samples per fillet. Each sample was weighed.

Smoke flavoring (SF) salt (Salinera Española, SA) was used in the salting–smoking stage. Its composition included 50% refined salt, white sugar, baking soda, smoke flavoring and anti-caking agent (E-536).

2.2. Experimental design

2.2.1. Phase I: optimization of salting–smoking process and selection of impedance spectroscopy electrode

This phase of the study had two different aims. The first was to establish the appropriate salting conditions (correct amount of smoke flavoring salt, processing time and type of packaging) to obtain smoked salmon with similar characteristics to currently marketed products (60–63 g H_2O/100 g, 3.5–3.8 g NaCl/100 g, $a_w = 0.963–0.965$) (Cardinal et al., 2004; Fuentes et al., 2010a) while generating the minimum of brine waste. The smoked salmon obtained by this new process is intended to be distributed vacuum packaged under refrigeration. The second objective was to select the most suitable electrode to monitor the salting–smoking process.

A total of 48 portions of fresh salmon obtained as described above were randomly divided into 4 batches. Each batch was submitted to a salting–smoking process under different conditions (Fig. 1). Four concentrations of smoke flavoring salt were studied: 4, 6, 8 and 16 g SF salt/100 g fresh salmon. These concentrations were selected from previous studies (Fuentes, Pérez, Fernández-Segovia, & Barat, 2011). The weight of SF salt was spread over the fish muscle surface and the samples were individually placed inside plastic bags. Each batch was subdivided into 2 further groups, one was packaged in air and the other in vacuum (Fig. 1). Three processing times were also studied (12, 18 and 24 h). The salting–smoking process was carried out at 4 °C. At the end of the processing time, the samples were placed in saturated brine under constant stirring for 30 s to remove any traces of SF salt attached to the surface. Finally, the samples were dried with absorbent paper and re-weighed.

Two samples were used for each condition ($n = 2$).

Analysis of moisture, pH, NaCl content and a_w were carried out on the fresh salmon and the smoked samples at different times during the study. Impedance spectroscopy measurements were also carried out using the 3 different sensors (double electrode, arrow electrode and a coaxial needle) described below.

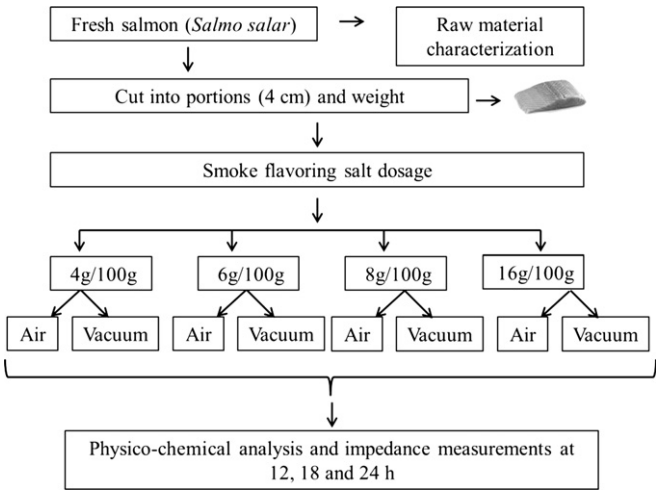

Fig. 1. Experimental design of Phase I.

2.2.2. Phase II: monitoring of salting–smoking process using impedance spectroscopy

The objective of this second phase of the study was to evaluate the application of impedance spectroscopy to monitoring the salmon salting–smoking process.

The salting–smoking conditions that provided smoked salmon similar to currently available products were selected from the results obtained in the previous phase. This part of the study was repeated with 3 batches of fish consisting of 12 samples per batch. Each batch was purchased at intervals of 1 week.

The samples were salted–smoked with 16 g SF salt/100 g fresh salmon in vacuum packaging for 25 h at 4 °C. Analyses were carried out at 5 h-intervals, after rinsing the samples in brine and drying as described above. Physico-chemical analyses were carried out (moisture content, pH, NaCl content and a_w) as well as impedance spectroscopy measurements with the electrode selected in Phase I (needle electrode). Two samples were used ($n = 2$ in each batch) for each salting time, including time 0, corresponding to fresh salmon.

2.3. Analytical determinations

The physico-chemical analysis and impedance spectroscopy measurements were performed in the center of each fillet. The analyses were done in triplicate on each sample, except for pH, which was measured in quintuplicate.

2.3.1. Physico-chemical analyses

Moisture content was determined according to the AOAC method 950.46 (1997). The pH measurements were carried out using a digital pH-meter micropH 2001 (Crison Instruments, S.A., Barcelona, Spain) with puncture electrode (Crison 5231) in five different locations on the sample. Water activity (a_w) was measured in minced samples with a fast water activity-meter (GBX scientific FA-st/1, Cédex, France). Sodium chloride content was determined according to the procedure described by Fuentes, Fernández-Segovia, Barat, and Serra (2010b) using an automatic Sherwood Chloride Analyzer Model 926 (Sherwood Scientific Ltd., Cambridge, UK). Changes in total mass, water and sodium chloride during the salting process were estimated by Eqs. (1)–(3):

$$\Delta M_t^0 = \left(\frac{M_t^o - M_0^o}{M_0^o}\right) \quad (1)$$

$$\Delta M_t^W = \left(\frac{M_t^o \cdot x_t^w - M_0^o \cdot x_0^w}{M_0^o}\right) \quad (2)$$

$$\Delta M_t^{NaCl} = \left(\frac{M_t^o \cdot x_t^{NaCl} - M_0^o \cdot x_0^{NaCl}}{M_0^o}\right) \quad (3)$$

(M_t^o and M_0^o are the salmon weight, x_t^w and x_0^w are the water weight fractions in the salmon, and x_t^{NaCl} and x_0^{NaCl} are the NaCl weight fraction in the salmon, at sampling times t and 0, respectively).

Sodium chloride concentration referred to the fish liquid phase (Z^{NaCl}) was estimated from the determinations of weight fractions of water (x^w) and sodium chloride (x^{NaCl}) according to Eq. (4), thus considering that nearly all the sodium chloride and water were free in the salmon muscle.

$$Z^{NaCl} = \left(\frac{x^{NaCl}}{x^w + x^{NaCl}}\right) \quad (4)$$

2.3.2. Impedance spectroscopy measurements

A low-cost, flexible, light, non-destructive measurement system was developed by the Instituto de Reconocimiento Molecular y Desarrollo Tecnológico (IDM) at the Universitat Politècnica de València (UPV) (Masot et al., 2010). This impedance spectroscopy measurement system applies an electric signal to food products and measures the response in a frequency sweep between 1 Hz and 1 MHz.

Since the electrical response depends on the type of electrode used, three different electrodes were tested in this study. One was a double electrode (DE) composed of two stainless steel needles 1.5 cm long and 1 mm in diameter, separated by a distance of 1 cm in a non-conductive frame. This design keeps the separation between both needles constant during measurements.

The second electrode, known as the Arrowhead (AH), was designed using thick-film technology, which uses high-resolution screen-printing methods to deposit pastes or inks of different electrical characteristics (conductive, resistive and dielectric) on an insulating substrate, in order to form an electronic circuit. This electrode is designed with a pointed end to help it to penetrate through the sample.

The third sensor (needle electrode) consisted of a hollow needle with an internal isolated wire, so that a two-electrode system is configured. The external part of the needle is made of stainless steel and acts as the outer electrode. The internal wire is also made of stainless steel and acts as the inner electrode. Both electrodes are separated by dielectric material (epoxy resin). The hollow needle (TECAN 53156, Oxford-FEDELEC) has an outer diameter of 0.46 mm.

The impedance measurements were taken by inserting the sensors into the sample perpendicular to the muscle fibers of the fish. The penetration depth of the electrodes was constant in all the analyses (1.5 cm). All measurements were carried out at room temperature.

2.4. Statistical analysis

Data are reported as mean ± standard deviation. One-way ANOVA was conducted for each physico-chemical parameter evaluated in Phase II, to determine whether there were significant differences between the salting–smoking times. Statistical treatment of the data was performed using the Statgraphics Centurion XVI (Manugistics Inc., Rockville, MD, USA).

In order to assess the feasibility of the impedance spectroscopy technique to discriminate between different moisture, NaCl contents, and/or a_w levels, three Principal Component Analyses (PCAs) were carried out with data obtained from the DE, AH and needle electrodes. PCAs were performed using impedance module and phase data obtained by the equipment in the frequency range established for each sensor. A PCA was conducted in the same way for the needle electrode with data obtained in Phase II. Partial Least Squares (PLS) were also carried out to create predictive models of each physico-chemical parameter evaluated from the IS measurements. PLS prediction models were created using a set of experimental data (calibration set). The model was then validated with a new set of experimental data (validation set). All multivariate analyses were performed using MATLAB ® PLS Tool-box (Eigenvector Research, Inc.).

3. Results and discussion

3.1. Phase I: optimization of the salting–smoking process and selection of the impedance spectroscopy electrode

3.1.1. Physico-chemical analyses

Moisture, pH, a_w and sodium chloride content values of raw material ($t = 0$) and smoked salmon are shown in Table 1. The values obtained for the raw material are similar to those reported by other authors for fresh salmon (Fuentes, Fernández-Segovia, Masot, Alcañiz, & Barat, 2010; Gallart-Jornet et al., 2007).

In all the experimental conditions the salting–smoking process caused a significant reduction in the water content and a_w values, as well as an increase in the NaCl concentration, as compared with fresh salmon. Reducing the a_w values lengthens smoked salmon shelf-life. These changes are due to dehydration and NaCl absorption into the muscle. It should be noted that the samples with higher sodium chloride levels showed a slight decrease in pH values, due to the higher ionic strength of the internal solution in fish muscle cells, as described by Leroi and Joffraud (2000).

In both types of packaging, the highest SF salt dosages (8 and 16 g SF salt/100 g fish) caused the largest increase in NaCl content, with the consequent reduction of a_w values as processing time advanced. However, for the 4 and 6% SF salt doses, the magnitude of

Table 1
Physico-chemical parameters of raw material ($S = 0$, $t = 0$) and salmon submitted to salting–smoking with different smoke flavoring (SF) salt doses (S) (g SF salt/100 g fresh salmon), types of packaging (P) and processing times (t). Mean values ± SD ($n = 2$).

S	P	t (h)	Moisture (g H_2O/100 g)	pH	a_w	NaCl (g NaCl/100 g)
0		0	70.39 ± 1.47	6.13 ± 0.02	0.992 ± 0.002	0.00
4	Air	12	67.34 ± 0.13	6.10 ± 0.01	0.980 ± 0.000	1.84 ± 0.09
		18	65.91 ± 0.18	6.08 ± 0.08	0.978 ± 0.000	1.71 ± 0.07
		24	67.81 ± 0.24	6.07 ± 0.06	0.972 ± 0.002	2.03 ± 0.04
	Vacuum	12	67.03 ± 0.12	6.09 ± 0.04	0.979 ± 0.001	1.78 ± 0.05
		18	67.13 ± 0.29	6.09 ± 0.02	0.980 ± 0.001	1.79 ± 0.01
		24	68.03 ± 0.01	6.09 ± 0.03	0.980 ± 0.000	1.68 ± 0.01
6	Air	12	66.11 ± 0.21	6.12 ± 0.02	0.978 ± 0.001	1.77 ± 0.06
		18	65.13 ± 0.30	6.10 ± 0.03	0.977 ± 0.000	2.04 ± 0.01
		24	65.70 ± 0.17	5.99 ± 0.02	0.978 ± 0.001	2.54 ± 0.08
	Vacuum	12	65.52 ± 0.02	6.08 ± 0.04	0.978 ± 0.001	2.02 ± 0.02
		18	66.95 ± 0.18	6.15 ± 0.05	0.982 ± 0.001	1.67 ± 0.00
		24	67.23 ± 0.80	6.06 ± 0.05	0.976 ± 0.000	2.01 ± 0.02
8	Air	12	65.65 ± 0.20	6.14 ± 0.04	0.977 ± 0.000	2.16 ± 0.10
		18	65.52 ± 1.22	6.08 ± 0.02	0.976 ± 0.000	2.14 ± 0.05
		24	64.69 ± 0.35	6.10 ± 0.04	0.971 ± 0.000	2.58 ± 0.03
	Vacuum	12	65.15 ± 0.28	6.13 ± 0.03	0.978 ± 0.001	1.98 ± 0.09
		18	65.57 ± 0.17	6.16 ± 0.05	0.977 ± 0.000	2.13 ± 0.16
		24	64.20 ± 0.03	6.05 ± 0.02	0.965 ± 0.001	3.01 ± 0.00
16	Air	12	62.68 ± 0.28	6.15 ± 0.06	0.976 ± 0.002	1.79 ± 0.05
		18	62.34 ± 0.88	6.17 ± 0.02	0.978 ± 0.000	1.90 ± 0.14
		24	59.91 ± 0.06	5.99 ± 0.05	0.968 ± 0.001	3.40 ± 0.10
	Vacuum	12	60.53 ± 0.67	6.12 ± 0.06	0.969 ± 0.002	2.35 ± 0.22
		18	60.99 ± 0.26	6.01 ± 0.03	0.967 ± 0.000	3.12 ± 0.25
		24	62.45 ± 0.39	5.96 ± 0.01	0.963 ± 0.002	3.62 ± 0.02

these changes was smaller in samples packaged in air, being practically negligible in salmon processed in vacuum packaging (Table 1). This could be explained by the fact that in these last cases at 12 h almost all the SF salt dose had been absorbed, so that the changes during the rest of the processing time were minimal.

Regarding the type of packaging, vacuum packaging caused faster sodium chloride absorption and dehydration of the salmon than air packaging. This effect was only observed for the highest SF salt dose (16 g/100 g fish), with minimal differences between the two types of packaging for the rest of the studied dosages, since these low amounts of salt are easily dissolved and absorbed in the first hours of processing in both types of packaging.

Of all the conditions studied, only those samples salted-smoked with 16 g SF salt/100 g of fresh salmon for 24 h in vacuum reached the levels of moisture, NaCl and a_w, previously established (60–63 g H_2O/100 g, 3.5–3.8 g NaCl/100 g, a_w = 0.963–0.965). These were consequently the salting–smoking conditions selected for Phase II.

3.1.2. Impedance spectroscopy

Impedance spectroscopy was used to detect changes in the salmon muscle during the salting–smoking process. The impedance measurements of fresh salmon were compared with those of samples submitted to salting–smoking under the different conditions described above. In this phase, 3 different electrodes (DE, AH and needle) were studied as described in Section 2.3.2 on Materials and methods.

Impedance spectroscopy equipment generate 100 values for each measurement, corresponding to the modules and phases of the 50 frequencies analyzed. A Principal Component Analysis (PCA) was conducted for each electrode to determine whether impedance spectroscopy could discriminate between the different samples. The impedance data used in this analysis were from the samples processed for 24 h, since the highest differences in moisture, NaCl content and a_w, from the 4 SF salt levels were obtained for this time.

The results of the PCAs carried out on the DE, AH and needle electrodes are shown in Fig. 2a–c, respectively.

In all cases, a clear separation of the raw material (0%) from the rest of the samples was observed. The ED electrode could also discriminate samples processed with 16 g SF salt/100 g fish, while no discrimination was observed for the rest of the samples (Fig. 2a). For the AH electrode, all samples subjected to the salting–smoking process were overlapped (Fig. 2b). The needle electrode showed 4 clusters: fresh salmon (0%), samples with 16% SF salt, samples with 8% SF salt and a fourth group with samples with 6% and 4% SF salt (Fig. 2c). The best sensor in discriminating the different levels of moisture, NaCl contents and/or a_w was therefore the needle electrode and was consequently selected for the next phase of the study. These results confirm the importance of the measuring sensor design (electrode geometry and characteristics).

3.2. Phase II: monitoring of salting–smoking process using impedance spectroscopy

3.2.1. Physico-chemical analyses

Values of moisture, pH and a_w of salmon submitted to the salting–smoking process (16% SF salt dosage, vacuum packaging) for 25 h are shown in Table 2. Moisture content progressively decreased during the salting process, with a higher rate at the beginning of the process, mainly due to the presence of salt crystals on the surface. The use of solid salt causes greater dehydration in the product at the beginning of the process. The water exiting from the muscle is needed to dissolve the salt on the surface and form brine at the interface, which enables the salt to later penetrate into the fish muscle.

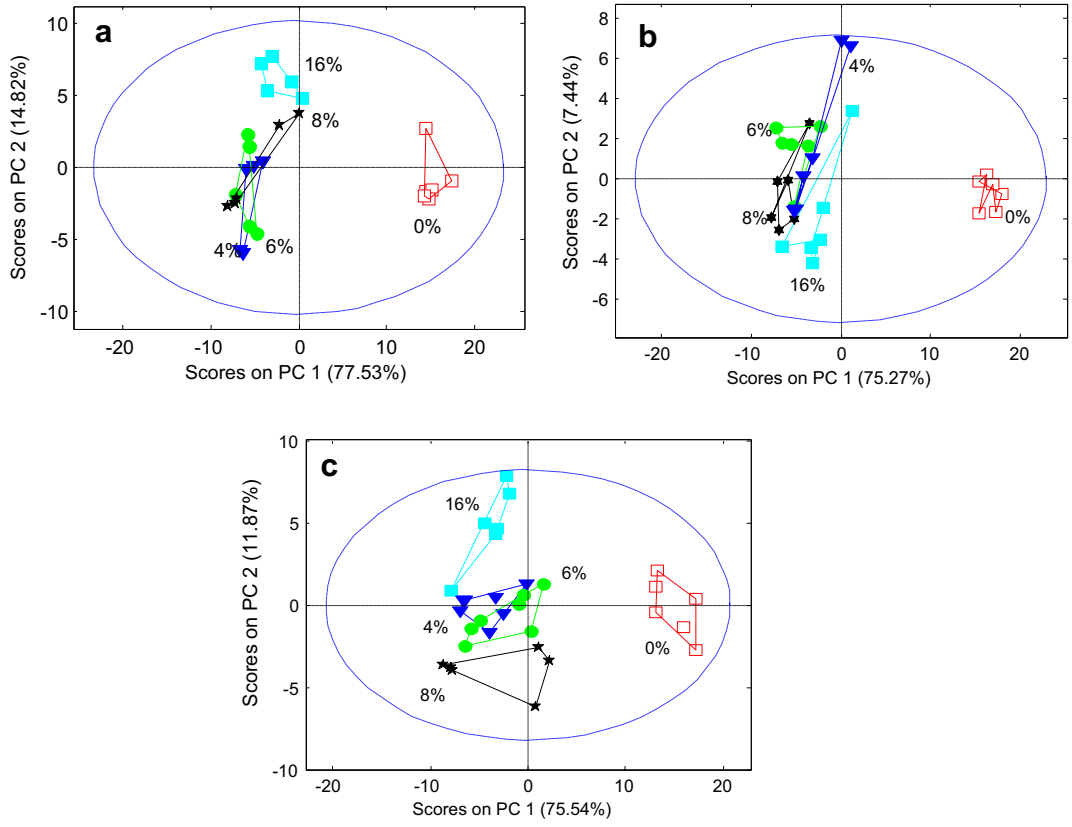

Fig. 2. Principal component analysis (PCA) performed on the impedance spectroscopy measurements of samples in air and in vacuum with different smoke flavoring salt dosages (0 (□), 4 (▼), 6 (●), 8 (★) and 16 (■) g SF/100 g) for 24 h. (a) Double electrode, (b) Arrowhead electrode and (c) Needle electrode.

Table 2
Physico-chemical parameters of raw material (t = 0) and salmon submitted to salting–smoking process (16% smoke flavoring salt dosage, vacuum packaging) for different processing times. Mean values ± SD (n = 6).

t (h)	Moisture (g H_2O/100 g)	pH	a_w
0	70.97 ± 2.64^{a}	6.08 ± 0.04^{ab}	0.991 ± 0.002^{a}
5	66.45 ± 2.56^{b}	6.13 ± 0.04^{b}	0.972 ± 0.002^{b}
10	64.90 ± 1.94^{bc}	6.11 ± 0.07^{b}	0.968 ± 0.001^{b}
15	63.91 ± 2.32^{bc}	6.10 ± 0.07^{b}	0.963 ± 0.004^{c}
20	63.85 ± 2.84^{cb}	6.08 ± 0.02^{ab}	0.957 ± 0.008^{d}
25	62.42 ± 2.30^{c}	6.03 ± 0.06^{a}	0.957 ± 0.002^{d}
α	***	ns	***

Same letters in the same column indicate homogeneous group membership. Significance level (α): ns no significant difference; ***$p < 0.001$.

The pH values showed a slight drop with processing time, although the differences were not significant. The a_w values decreased throughout the salting–smoking time, due to muscle dehydration and salt penetration.

Fig. 3 shows the evolution of sodium chloride content (x^{NaCl}, X^{NaCl} and Z^{NaCl}) during the salting–smoking process. The NaCl content of the samples increased progressively with processing time. The highest increase was observed in the case of sodium chloride concentration expressed on a dry basis (X^{NaCl}), because of the solute incorporation in fish muscle and osmotic dehydration that occurs during the process.

Total weight, water weight and sodium chloride weight changes are shown in Fig. 4. Moisture and sodium chloride variations showed opposing behavior throughout the salting, as mentioned above (Fig. 4).

Total weight changes could be considered a combination of both weight changes (water and NaCl). However, protein denaturation due to salt action would also contribute to the weight loss, as different authors have shown (Barat, Rodriguez-Barona, Andrés, & Fito, 2003; Ismail & Wootton, 1982). It can be assumed that there is pseudo-diffusional transport due to the strong dependence between the weight changes and the square root of time, as has been pointed out by other authors (Barat et al., 2006; Fuentes, Barat, Fernández-Segovia, & Serra, 2008; Gallart-Jornet et al., 2007).

It should be noted that samples reached the selected NaCl and a_w levels after 15 h of processing, a shorter time than in the preliminary study (Phase I). This is due to the salting process depending directly on the composition and initial quality of the raw material (Barat et al., 2006). Different batches of fresh salmon were used in Phases I and II, so that the differences in the raw material could have been the cause of the differences found in the process kinetics.

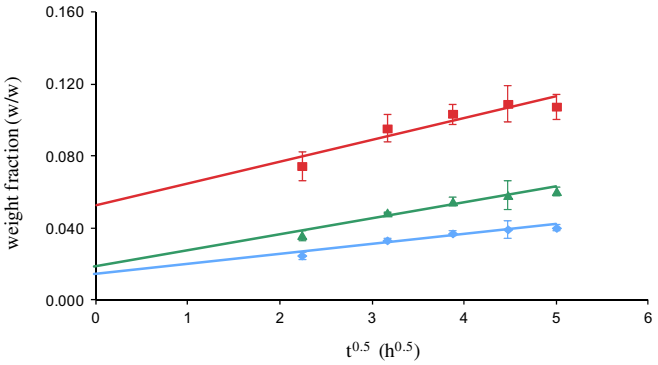

Fig. 3. NaCl weight fraction (x^{NaCl} (◆), $y = 0.005x + 0.014$, $R^2 = 0.933$), NaCl concentration on a dry basis (X^{NaCl} (■), $y = 0.012x + 0.053$, $R^2 = 0.875$) and NaCl weight fraction in the liquid phase of salmon (Z^{NaCl} (▲), $y = 0.009x \pm 0.019$, $R^2 = 0.951$) *versus* square root of the processing time ($t^{0.5}$).

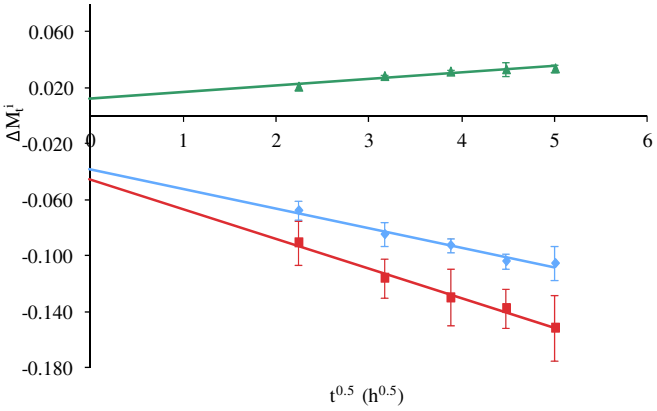

Fig. 4. Total weight changes (ΔM_t^0 (◆), $y = -0.014x - 0.038$, $R^2 = 0.974$), water weight changes (ΔM_t^w (■), $y = -0.021x - 0.046$) ($R^2 = 0.987$) and sodium chloride weight changes (ΔM_t^{NaCl} (▲), $y = 0.005x + 0.012$) ($R^2 = 0.915$) *versus* the square root of the processing time ($t^{0.5}$).

The use of vacuum during processing and distribution of salmon could permit the growth of *C. botulinum* type A, B and E, as well as toxin production. *C. botulinum* type E could grow in smoked salmon under vacuum at temperatures as low as 3.3 °C, if a_w is higher than 0.966. To minimize the risk of *C. botulinum* growth, the exact control of a_w is of outmost importance. This confirms the need for rapid monitoring methods that can be used on-line to determine the end of the salting process.

3.2.2. Impedance spectroscopy

A PCA was used to assess the feasibility of impedance spectroscopy for monitoring the salmon salting–smoking process, with the impedance spectroscopy values obtained at 5 h-intervals during the 25 h process.

Fig. 5 shows the results of the PCA performed with data obtained from the needle electrode according to processing time. The samples can be seen to be clearly separated according to processing time, except for 20 and 25 h, which are overlapped in the same graphic area. These two samples showed similar values for all the physico-chemical parameters studied, which justifies the behavior observed in the PCA.

Since the PCA analysis showed that impedance spectroscopy with the needle electrode could discriminate between different

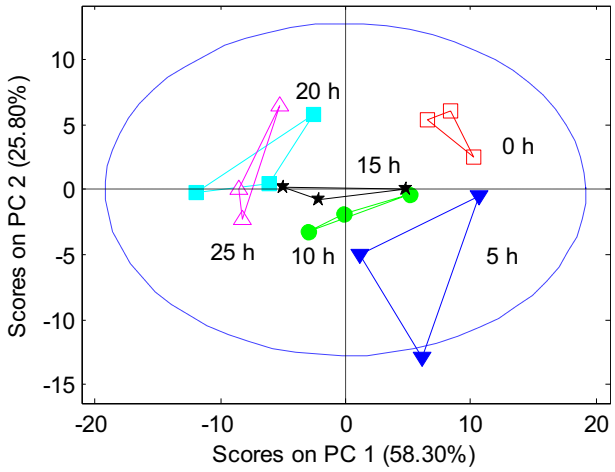

Fig. 5. Principal components analysis (PCA) performed with the impedance spectroscopy measurements (needle electrode) of salmon submitted to the salting–smoking process (16% smoke flavoring salt dosage, vacuum packaging) for different processing times (0 (□), 5 (▼), 10 (●), 15 (★), 20 (■) and 25 (△) h).

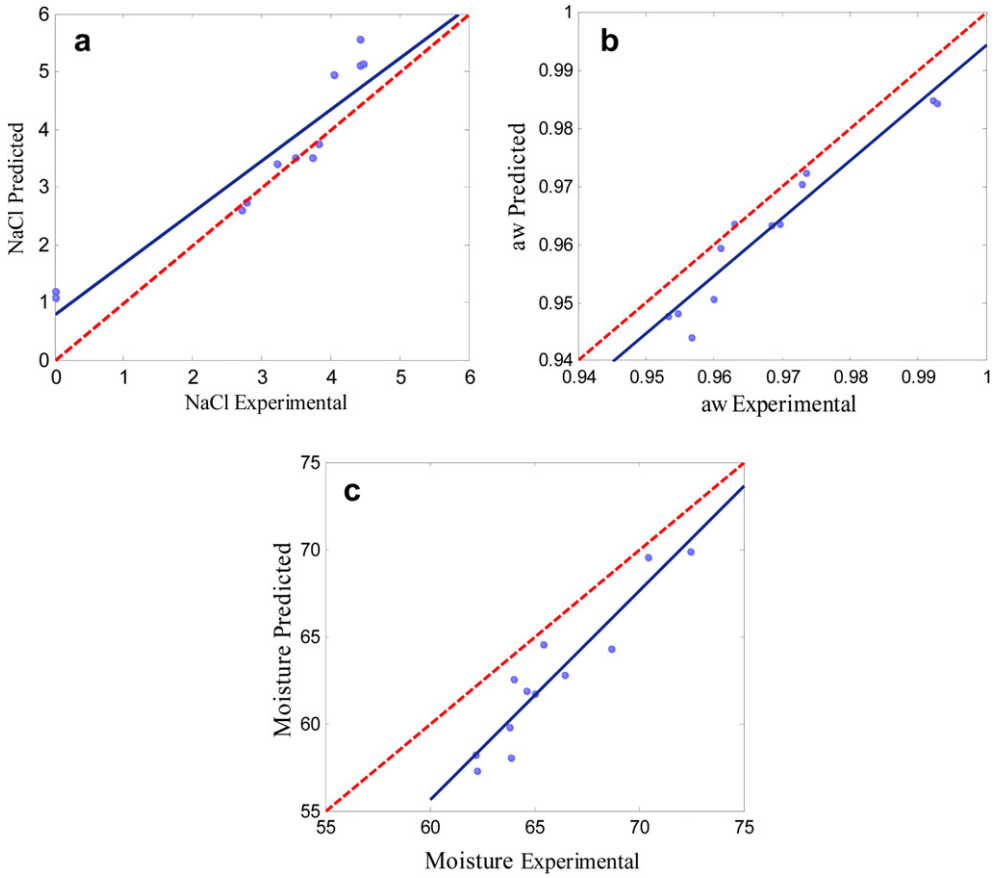

Fig. 6. Predicted *versus* experimental values by the PLS statistical model (—) and ideal behavior (---). (a) NaCl, (b) a_w and (c) moisture.

levels of moisture, NaCl and/or a_w, a statistical tool (PLS) was used to predict the values of these parameters from the measurements of the impedance device. In this way, statistical models were established for all the parameters except for pH, whose evolution was not significant throughout the processing time.

Sodium chloride, a_w and moisture experimental values *versus* values predicted by the PLS statistical models are shown in Fig. 6a–c respectively.

In all cases, the predicted values successfully fitted to the experimental values (RMSEP values of 0.685 for NaCl, 0.006 for a_w and 3.579 for moisture), especially in the case of a_w, for which the intercept was near to 0 and the slope near to 1. These results agree with other studies on the use of impedance spectroscopy in the characterization of commercial smoked salmon and cod, in which the best predictions were obtained for a_w (Karásková et al., 2011).

The results obtained from the PLS confirm the potential of impedance spectroscopy with needle electrode for monitoring the salmon salting–smoking process.

4. Conclusions

The results obtained from the physico-chemical analyses showed that packaging under vacuum speeded up the process of NaCl absorption and dehydration in salmon, although this effect was observed only for the highest dosage of smoke flavoring salt (16 g/100 g). The optimum processing conditions to obtain a similar product to the currently available smoked salmon on the market were 16 g SF salt/100 g salmon in vacuum packaging. This new method has the advantages of reducing processing times and waste. Further sensory evaluation and shelf-life studies should now be carried out to determine whether the sensory characteristics of smoked salmon obtained by the new method are comparable to currently marketed products and whether it has a similar or longer shelf-life.

Of the three electrodes used (double, arrowhead and needle electrode) in the IS measurements, the needle electrode was found to be the most appropriate for process monitoring. The increase in NaCl content and the reduction in moisture and a_w values with 16 g SF salt/100 g fish for 25 h in vacuum were detected by the EI technique using the needle electrode. This sensor was able to obtain robust NaCl content, moisture and a_w prediction models during the process. The best of these was the a_w prediction model, which is particularly interesting because of the relationship between this parameter and the shelf-life of smoked products. The results therefore showed that IS is a rapid on-line monitoring method for the salmon salting–smoking process and could provide an important tool to obtain products of uniform quality.

Acknowledgments

The authors gratefully acknowledge the financial support for the work reported here received from the Generalitat Valenciana (GV/2011/098) and the Universitat Politècnica de València (UPV) (PAID-06-09-2940). A. Fuentes would like to thank the Campus de Excelencia Internacional at the UPV for its support. The proof-reading of this paper was funded by the UPV, Spain.

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