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# Development of a low-cost non-destructive system for measuring moisture and salt content in smoked fish products

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**Abstract**

In this study, a new method to assess salt content in smoked fish products using impedance spectroscopy has been developed. Two fish species (salmon and cod) from two different brands were analyzed. The analyses carried out were moisture, lipid, and salt contents, as well as $a_w$. Impedance measurements were performed directly in different locations on smoked product by using a coaxial needle. The modulus and phase of the spectra of impedance were acquired between 1 Hz and 1 MHz, for 50 values of frequency. The impedance modulus and phase values were used to predict the studied physico-chemical parameters, by using the PLS method. In general, the highest lipid contents and the lowest moisture values were observed in salmon samples, independently of the brand and the batch analyzed. The results showed a high variability between batches of a same product, regarding to moisture and salt content. Prediction models were established for all the samples studied and also individually for each product. A higher accuracy of prediction was obtained for the PLS models established for each product compared with the model applied for all samples. In general, the highest $R^2$ was obtained for prediction of $a_w$ parameter, followed by moisture content. The best correlations were obtained for smoked cod samples independently of the brand. These results can be explained by the isolating effect of the fat. Due to the differences in the raw material composition, a single model cannot be applied for all the products. A previous characterization of the raw material would improve the accuracy of the developed methodology. These results show the feasibility of the impedance spectroscopy to determine salt and moisture contents.



*Keywords*: Smoked fish; Electrical impedance spectroscopy; Physico-chemical analyses; Quality control.

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## 1. Introduction

The ability of the smoking process to preserve fish is due to the synergistic action of salt incorporation, the preservative effect of smoke compounds, and dehydration. Salt content has a strong influence on product flavour, as well as on decreasing water activity ($a_w$), and consequently on shelf life. In this sense, it has been demonstrated that shelf life of smoked samples processed in the same way and stored under the same conditions can vary in 2-3 weeks depending on their salt concentration in water phase (4.6 or 2.2%, w/w) [1]. For this reason, controlling salt content during fish processing is one of the priorities for the fish industry. As new technologies and products are developed, a fast and non-destructive method for analysing traits and product composition would be useful to optimize unit operations and processes. In general, methods for salt content determination are time-consuming, expensive, destructive for samples, and require skilled personnel; for this reason, industry demands new in situ methods, faster, cheaper, easier, and non-destructive. In this sense, electrochemical impedance spectroscopy (EIS) is one of the techniques that can be applied. EIS measures the dielectric properties of a material as a function of frequency and can be correlated with its physical parameters. EIS has been applied in food technology for different purposes, such as fruit ripening evaluation and some quality parameters of fruit products [2-4], meat quality assessment [5], prediction of intramuscular fat [6], moisture and salt measurements [7, 8], differentiation between freezing methods and number of freezing cycles in fish [9, 10], etc.

The objective of this study was to evaluate the ability of an impedance system to measure moisture, salt and lipid content, as well as $a_w$ values on commercial smoked cod and salmon.

## 2. Materials & Methods

Samples employed in this study were commercial smoked products which were, at distribution point, sliced, vacuum packed, and stored at 4 ºC. Two species of smoked fish, salmon and cod, were used in this study. From each one, 2 brands were analyzed; being used 3 batches of each product (acquiring each one at 1 month-intervals). Each batch was made up of 3 different packages.

### *2.1. Physico-chemical analyses*

Moisture was determined by oven drying until constant weight at 103 ± 2ºC, according to the AOAC method 950.46 [11]. Lipid content was determined by solvent extraction with a SoxtecTM 2055 semiautomated analyser (FOSS Ltd., Warrington, UK) using petroleum ether, according to the AOAC method 991.36 [11]. Sodium chloride content was determined after sample homogenization in distilled water in an Ultraturrax T25 (IKA, Labortechnik, Staufen, Germany) at 9000 rpm for 2 min. Samples were made up to 100 ml and then centrifuged using a Medifriger BL centrifuge (JP Selecta, S.A., Barcelona, Spain), at 500 g for 10 min. The obtained supernatant was filtered and an aliquot of the supernatant was used for the NaCl determination with an automatic Chloride Analyzer (Sherwood Scientific Ltd., Cambridge, UK). Water activity ($a_w$) was measured in minced samples with a fast water activity-meter (GBX scientific FA-st/1, Cédex, France). All the physicochemical analyses were run in triplicate.

### *2.2 Impedance spectroscopy*

Impedance measurements were performed with a system developed by the research institute, Instituto de Reconocimiento Molecular y Desarrollo Tecnológico (IDM) of the Universidad Politécnica de Valencia (UPV). The electronic system had been developed to measure impedance of complex matrixes using a coaxial needle. The equipment is able to generate sinusoidal signals with a frequency in the range of 1 Hz to 1 MHz and amplitude up to 1 Vpp. The measuring sensor consists of a hollow needle. An isolated wire is placed inside the needle, so as a two electrodes system is configured. The external part of the needle is made of stainless steel and it acts as the outer electrode in our system. The inside wire is also made of stainless steel and it plays the role of inner electrode. Between both electrodes, there is a dielectric material, namely an epoxy resin. The applied needle (TECAN 53156, Oxford-FEDELEC) has an outer diameter of 0.46 mm.

*2.3 Data analysis*

Differences in physicochemical parameters between batches of each smoked fish were checked by an analysis of variance (ANOVA), considering each attribute as dependent variable, and purchase batch as the factor. The level of significance setting was $p < 0.05$. The spectroscopy impedance data were employed to predict moisture, lipid, and salt content, as well as $a_w$ in smoked fish using Partial Least Square (PLS) regression. All samples for modelling were divided into a calibration set with full cross-validation (66% of the samples) and a prediction set (33% of the samples). The square of the correlation coefficient ($R^2$) and PRESS (RMSE) were used to indicate the models quality. In all cases, the significant level used was 5%. Statistical treatment of the data was performed using the Statgraphics Centurion XV (StatPoint Technologies, Inc., Warrenton, VA, USA).

## 3. Results & Discussion

The results of the physico-chemical analyses carried out for each batch of the smoked products are summarized in Table 1.

Table 1. Physico-chemical parameters of smoked fish products

| | Batch | Moisture (g/100 g) | NaCl (g /100 g) | Lipid (g/100 g) | $a_w$ |
|---|---|---|---|---|---|
| *Cod A* | I | 70.48±0.86$^a$ | 4.34±0.17$^a$ | 0.25±0.15$^a$ | 0.952±0.004$^a$ |
| | II | 72.29±1.44$^b$ | 3.53±0,52$^b$ | 0.09±0.07$^b$ | 0.965±0.007$^b$ |
| | III | 69.06±0.53$^a$ | 2.98±0.67$^b$ | 0.10±0.04$^b$ | 0.965±0.003$^b$ |
| | | *** | *** | ** | *** |
| *Cod B* | I | 69.15±1.62$^a$ | 6.55±1.64$^a$ | 1.60±0.19$^a$ | 0.935±0.002$^a$ |
| | II | 71.90±0.62$^b$ | 5.23±0.50$^b$ | 2.18±0.43$^b$ | 0.954±0.002$^b$ |
| | III | 72.10±0.18$^b$ | 5.32±0.55$^b$ | 1.59±0.14$^a$ | 0.952±0.003$^b$ |
| | | *** | * | *** | ** |
| *Salmon A* | I | 56.71±0.86$^a$ | 4.15±0.65$^a$ | 14.82±1.93$^a$ | 0.946±0.008$^a$ |
| | II | 57.82±2.67$^a$ | 3.34±0.19$^b$ | 15.04±3.50$^a$ | 0.961±0.002$^c$ |
| | III | 61.97±1.17$^b$ | 3.55±0.16$^b$ | 10.18±0.97$^b$ | 0.956±0.002$^b$ |
| | | *** | *** | *** | *** |
| *Salmon B* | I | 57.51±2.09$^{ab}$ | 3.72±0.17$^a$ | 15.20±2.31$^a$ | 0.949±0.006$^a$ |
| | II | 58.32±1.39$^a$ | 3.83±0.42$^a$ | 14.60±1.50$^a$ | 0.950±0.005$^b$ |
| | III | 56.75±0.40$^b$ | 3.39±0.21$^a$ | 16.24±1.61$^a$ | 0.952±0.004$^{ab}$ |
| | | * | ns | ns | ns |

Mean ± SD from the three batches, n = 9. *** $p < 0.001$; ** $p < 0.01$; * $p < 0.05$; ns, non significant

For all the studied physico-chemical parameters, significant differences between the three batches of each product were found, except for Salmon B. This variability has been also observed in other studies carried out with different smoked fish products [14], and it could be due to the lack of control during the salting processes. These differences affect product sensory features, as well as its shelf life. A correlation between NaCl content or $a_w$ and shelf life in products that have been manufactured by different industries would be unreliable, because of a great number of factors such as packaging material, production method, initial microbial load, number of freezing and thawing cycles, and quality of the raw material [15]. However, this could be useful to compare products processed under identical conditions. In this sense, Truelstrup Hansen at al. [1] reported an increase in the shelf life of smoked salmon when salt levels increased, as above mentioned. These findings support the importance of ensuring homogeneous salt contents for all the batches of a same sample, which would allow a reliable shelf life to be established for the product.

Differences among impedance spectra were observed among the smoked products (Fig. 1). Since the relationship between NaCl content and impedance data has been widely studied by different authors [16, 17], it could be expected that the differences observed could be related to different salt content among samples. This correlation is expected since the conductance is a function of the ion content of samples, and in fact impedance measurements are related to the ions capability of movement under the influence of an electrical field [8].

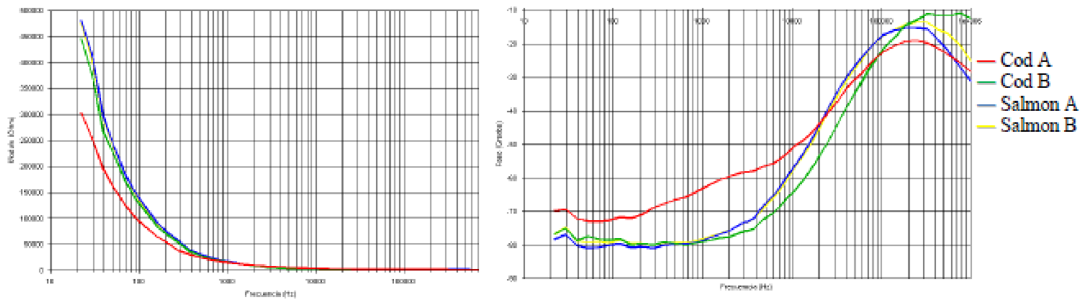


Fig. 1. Module and phase impedance spectra of smoked fish products

In this study, impedance data were used to obtained predictive models for water, NaCl, lipid content and $a_w$ in smoked fish using PLS method. When all samples were analyzed together, poor correlations between electrical measurements and the studied physico-chemical parameters were obtained (data not shown). The impedance of biological tissues, and in particular animal tissue, depends on numerous factors, such as muscle structure and composition, tenderness, or ageing. Moreover, in the case of smoked products, processing conditions, background of animal, or storage time and conditions could also affect product impedance measurements. For this reason, in this study PLS models were tested for each product separately; in this way, the effect of other parameters than the studied would be minimized.

The results of PLS models for calibration and validation set of samples are summarized in Table 2. The samples in calibration and validation groups were comparable in their physico-chemical composition, which are indicated by the similar range of values of each group. In general, higher coefficients of determination ($R^2$) were obtained in cod samples for all the studied parameters. This fact could be explained by the higher lipid content observed in smoked salmon, since fat is an electrical insulator and, therefore, influences the impedance of tissues. Despite of the possible interference of fat in impedance measurement, these results could be considered satisfactory.

Table 2. Parameters of the PLS models for calibration and validation set for moisture, salt and lipid content, and $a_w$ values for each smoked product.

| | Calibration data | | | | Validation data | | | |
|---|---|---|---|---|---|---|---|---|
| | [a]PRESS | [b]$R^2$ | [c]LV | [d]Range | [a]PRESS | [b]$R^2$ | [c]LV | [d]Range |
| *Cod A* | | | | | | | | |
| moisture | 64.95 | 62.21 | 8 | 67.68-73.58 | 19.26 | 73.54 | 7 | 67.68-73.57 |
| salt | 14.67 | 56.71 | 4 | 2.45-4.58 | 4.28 | 66.33 | 6 | 2.49-4.58 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| lipid | 0.42 | 51.17 | 5 | 0.01-0.50 | 0.137 | 64.04 | 5 | 0.1-0.50 |
| $a_w$ | 0.0002 | 75.91 | 6 | 0.952-0.740 | 0.0002 | 75.13 | 6 | 0.951-0.974 |
| | Calibration data | | | | Validation data | | | |
| | [a]PRESS | [b]$R^2$ | [c]LV | [d]Range | [a]PRESS | [b]$R^2$ | [c]LV | [d]Range |
| *Cod B* | | | | | | | | |
| moisture | 16.11 | 91.02 | 9 | 67.31-72.95 | 13.20 | 85.13 | 5 | 67.31-72.96 |
| salt | 22.39 | 69.72 | 7 | 4.51-8.57 | 12.71 | 68.55 | 5 | 4.51-8.58 |
| lipid | 4.73 | 53.43 | 1 | 1.27-2.94 | 1.79 | 53.74 | 9 | 1.26-2.95 |
| $a_w$ | 0.002 | 77.60 | 8 | 0.914-0.960 | 0.001 | 72.09 | 5 | 0.914-0.960 |
| *Salmon A* | | | | | | | | |
| moisture | 239.01 | 52.64 | 8 | 54.02-63.63 | 85.76 | 56.44 | 4 | 54.02-63.01 |
| salt | 8.35 | 48.41 | 8 | 3.05-4.98 | 1.65 | 76.05 | 5 | 3.05-4.98 |
| lipid | 352.08 | 42.62 | 8 | 9.01-19.97 | 116.79 | 49.28 | 4 | 5.93-6.09 |
| $a_w$ | 0.0002 | 62.96 | 5 | 0.937-0.965 | 0.0003 | 82.32 | 5 | 0.937-0.964 |
| *Salmon B* | | | | | | | | |
| moisture | 78.98 | 43.94 | 10 | 54.99-60.06 | 16.96 | 63.72 | 8 | 54.89-60.10 |
| salt | 2.99 | 41.91 | 4 | 3.15-4.49 | 0.85 | 59.95 | 8 | 3.33-4.41 |
| lipid | 188.04 | 15.40 | 10 | 12.25-18.98 | 63.23 | 35.12 | 10 | 12.34-18.90 |
| $a_w$ | 0.0007 | 60.09 | 6 | 0.947-0.960 | 0.0002 | 70.13 | 5 | 0.943-0.959 |

[a]PRESS: predictive residual sum of squares, [b]$R^2$: coefficient of determination, [c]LV: latent variables, [d]Range: range of values for each set of samples.

In all the analyzed products, the lowest $R^2$ was obtained for predicting lipid content, being lower in smoked salmon. This poor correlation could be due to the insulating effect of fat, as above mentioned. It is well-known that a higher lipid content lead to a higher resistance which could counteract the increase of conductance that appears as a function of ion content. The resistance also depends on the electrode characteristics; in this sense, to predict lipid content in fatty fish the use of electrodes with different geometries could be more appropriate.

It has to be noted that, in general, coefficients of determination obtained for $a_w$ were higher than those obtained for other physico-chemical parameters. This fact could be especially relevant, since $a_w$ values are closely related to microbial spoilage. A reliable prediction of $a_w$ values could be employed as an useful tool for determining smoked product shelf life. It could be considered that a higher accuracy of prediction would be achieved in case of a deeper knowledge of the raw material, since impedance measurements are highly influenced by muscle composition and structure. In this case, raw material composition, muscle pH, fish freshness, post-mortem time, etc., could be incorporated in the model as co-variables in order to obtain more efficient prediction models.

Fig. 2 shows plots predicted *vs* values of $a_w$ measured, by using the obtained models (validation set). Prediction for $a_w$ parameter ranged from 0.701 to 0.820, for smoked salmon B and A, respectively.

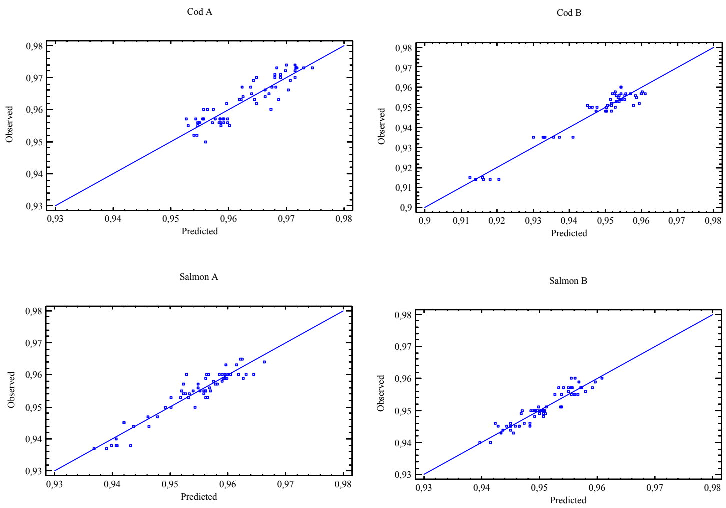


Fig. 2. Scatter plot of observed *vs* predicted $a_w$ values using PLS models in smoked cod (A and B) and salmon (A and B) for the validation set.

## 4. Conclusion

The system designed to performed punctual impedance measurements provide a non-destructive and rapid method to predict moisture and salt content, as well as aw values in smoked fish products. Concerning lipid prediction, results are not sufficient to validate the method; however, these results could be improved using a measuring sensor designed with other materials and/or geometry.

These results are promising for the development of low-cost, rapid, non-destructive and easy-to-use instrumentation for quality control in the smoking fish industry. Further studies should be carried out to measure salt content during fish processing in order to develop a new technique to control salt distribution in samples through the entire salting processes.

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