Development of a puncture electronic device for electrical conductivity measurements throughout meat salting

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ABSTRACT

Conductivity measurements of food systems are of high interest because they are related with food characteristics such as free water and salt content. Nevertheless, as far as now no devices have been developed for punctual conductivity measurements inside solid foods. The aim of this work was to develop a conductimeter which allows obtaining punctual measurements in different locations of solid foods. The sensor consists of a coaxial needle while an electrical sign controlled by microcontroller is applied. The preliminary results indicate that the obtained response is proportional to the conductivity and the salt content in the zone of measurement of the food, being possible its use for salted food analysis and control.

Keywords:
Food quality
Salted meat
Conductivity sensor
Microcontroller control

1. Introduction

Salting is one of the most ancient techniques used for food preservation. It has mostly been used for meat preservation due to the achieved reduction in water activity and bacteriorstatic effect in the products. Although new preservation methods have been developed salting is still of high importance because highly appreciated products, due to their sensorial characteristics, such as bacon and ham can be obtained.

Nevertheless the measurement of salt content and control of salt gain in the processed products still remains a problem to solve in the food industry due to the destructive nature of the conventional measurement methods. That is why it is of current interest to develop fast and non-destructive methods for the measurement of salt content, and in some cases related properties such as water activity and moisture content. A plausible technique may be measurement of electrical conductivity, in which the relationship with NaCl content in a solution is well known.

Measurement of conductivity in solutions is a well-known technique. Basically, it is carried out using an alternating current with a frequency between 60 Hz and 30 kHz to avoid polarization effects. The most commonly used measuring circuit is the Wheatstone

bridge for alternative current, though there exist other non-invasive methods

In measurement of conductivity, electrolytic conductivity κ (mS) is defined as the conductance G between two electrodes whose area S is equal to $1 \, \mathrm{cm}^2$ and which are placed at a distance I to each other equal to $1 \, \mathrm{cm}$ ($\kappa = G \frac{I}{S}$). Due to the field lines between both electrodes not being uniform, conductivity is more precisely expressed as $\kappa = G \cdot k$, where k is called cell constant and is defined by the ratio k = I/S. Cell constant can be considered invariant for a given cell, provided measurement conditions are invariant too

Measurement of electrical conductivity can be applied in the field of food technology in order to determine the quality of a certain food sample as well as to control industrial processes. The investigation of salt concentration and/or moisture in salted and cured products such as ham is an example of these applications.

In essence, investigation of conductivity in solid foods is similar to that of solutions. The only variation is the measuring cell geometry. Mizhari designed a cell of different form for measuring solids such as potatoes, carrots, etc. The procedure they used consisted in submerging the solid in different saline solutions until one was found whose conductivity did not change on immersion of the food. Then, they assumed that the solid's conductivity was equal to that of the dissolution. Mitchell and de Alwis developed a cell composed by two cylinders with electrodes. The diameter of the cylinders was equal to 10 mm and the sample was placed between

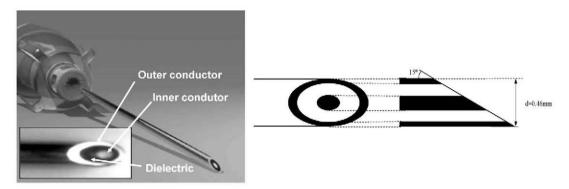


Fig. 1. Concentric measuring cell.

both of them. Shirsat worked on a similar cell consisting of a PVC tube (5 cm long and 2.65 cm wide) with two stainless steel electrodes.

In all above-mentioned cases measurements were non-invasive. Consequently they did not provide information on internal parts of the solid. However, when analyzing salt content in salted and cured products, it would be very interesting to acquire information from inside the solid. For this reason, some alternative non-destructive methods are currently being tested, for instance nuclear magnetic resonance (NMR) X-ray tomography ultrasounds The first two techniques may allow the invesmicrowaves tigation of both salt profiles and their evolution along time Nevertheless, they have relevant drawbacks as their high cost and high equipment size, the need for caring about operator's security and the requirement for tested pieces to pass through the testing machinery. All these drawbacks result in a cost of implementation that is unaffordable for most industrial applications. As for the other two techniques, both microwaves ultrasounds the feasibility of their application to the case of ham testing is still not clear. This is due to both the intrinsic system heterogeneity and the specific salt-concentration profiles

Within the work herein presented, a non-destructive measuring system that allows quantification of conductivity both in the surface and in different depth levels in pieces of meat has been developed. This way, monitoring the evolution of salt concentration in any point can be accomplished. Another potential application of the developed technique would be the measurement of local water activity inside the food product, attending to the relationship of NaCl concentration and water activity observed in published studies

2. Experiment and results

2.1. Measuring cell

The measuring cell consists of a hollow needle inside which a wire is introduced so as to configure a system of two electrodes. The needle is the same as used in medical applications for carrying out electromyography. The needle is made of stainless steel and it acts as the outer electrode in our system. The inside wire is made of steel and it plays the role of inner electrode. Between both electrodes, there is a dielectric material, namely epoxy resin (Fig. 1).

The utilized needle (TECA N53156 of Oxford – FEDELEC) has an outer diameter equal to 0.46 mm and its structure is shown in Fig. 1. In the systems used by Mitchell and de Alwis — and Shirsat sample size affected the measurements. Due to this, cell calibration and computation of its constant k is unnecessary. On the contrary, measurements obtained with the cell herein presented do

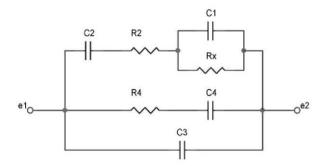


Fig. 2. Equivalent model for a cell having two electrodes.

not depend on sample dimensions, so computation of cell constant is compulsory, as it was in the case of solutions.

Cell characteristics have been determined using reference KCl solutions whose concentrations were 0.5×10^{-3} , 1×10^{-3} , 5×10^{-3} , 0.01, 0.025, 0.05, 0.1, 0.25, 0.5 and 1 M. Conductivity for each case has been checked with a commercial conductivity meter (Crison GLP31), cells whose constants where k = 0.1 (Crison 5295) and k = 10 (Crison 5298) and a temperature compensator (Crison 5531).

Cell geometry is another relevant aspect for its characterization. Oehme proposes an equivalent model for two-electrode cells that is depicted in Fig. 2. Within this model, Rx is the resistance of cell in contact with the food sample; C1 is a parasitic capacity that appears when working in high frequencies; C2 and R2 account for cell polarization effects that can be minimized through either reduced working frequency or appropriate cell material selection; C3 is the capacity of wires used for measuring and, last, C4 and R4 are defined by cell form and dimensions and they are usually neglected for commercial cells.

In the case of our concentric cell, both R4 and C4 must be considered, since on the one hand the contact surface is planar and, on the other, the whole structure is cylindrical. The fact that the contact surface being planar implies a disperse distribution of electric field lines. More over, such dispersion is greatly influenced by the conductivity of the material between the electrodes; the lower the conductivity, the greater the dispersion (see Fig. 3). The cylindrical form of the needle causes the appearance of a cylindrical capacitor

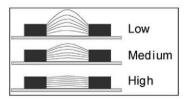


Fig. 3. Planar electrodes with different conductivity values in dielectric material.

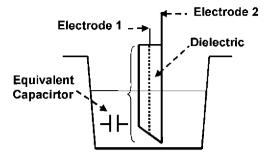


Fig. 4. Equivalent capacitor due to cell geometry.

with a thin dielectric material (epoxy) having a high permittivity; this is illustrated in Fig. 4.

Contribution of C1, C2 and R2 to the behaviour of the whole system can be minimized if working frequency is appropriately selected [4]. On the contrary, the effect of both R4 and C4 should be considered for this concentric cell. Consequently, the measured conductance is not proportional to the conductance of the measured piece and the relation between them has to be found prior to food measurement.

2.2. Measuring system

The signal employed both for cell calibration and for food measurement is a bipolar square wave without any DC component. The measured magnitude is the voltage across the cell at certain moments $t_{\rm x}$. This is accomplished through the use of a circuit in which V_1 and V_2 are the digital outputs of a microcontroller, $V_{\rm x}$ is an analogue input, and $R_{\rm p}$ is a reference resistance for current limitation (see Fig. 5).

As shown in Fig. 5, voltage is measured at two time instants t_1 and t_2 , and the value of $G_{\rm x}$ for both time instants can be obtained using

$$G_{\rm X} = \frac{I_{R_{\rm X}}}{V_{G_{\rm X}}} = \frac{(V_1 - V_{\rm X})/R_{\rm p}}{V_{\rm X} - V_2} = \frac{1}{R_{\rm p}} \cdot \frac{V_1 - V_{\rm X}}{V_{\rm X} - V_2}$$
(1)

The variation of measured conductance referred to a reference system with $R_{\rm p}$ = 100 Ω and frequency equal to 200 kHz is depicted in Fig. 6.

The measuring system records the conductance and, through mathematical adjustment, it computes the conductivity. The adjustment procedure differentiates between two distinct intervals: above and below 1 mS. This way, errors for low conductivity values are minimized. A plot of the adjustment function is shown in Fig. 7.

The task of the microcontroller is to generate test signals, to measure voltages, hence calculating conductance values and to find conductivity from such values. The final result must be subsequently corrected to suppress the effect of temperature. This is

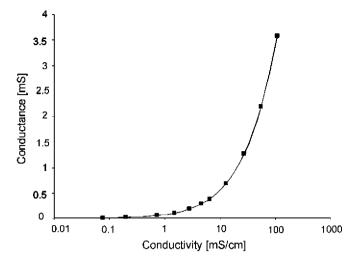


Fig. 6. Variation of measured conductance.

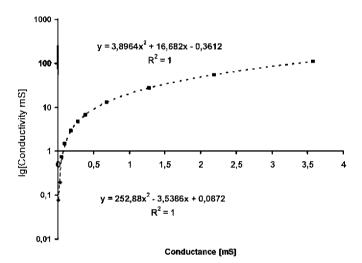


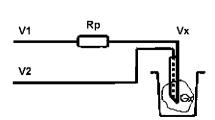
Fig. 7. Adjustment function for conductivity computation.

done considering the following equation:

$$\kappa_{25} = \frac{\kappa_{\rm m}}{1 + \alpha \cdot (t_{\rm m} - 25)} \tag{2}$$

where κ_{25} = conductivity at 25 °C, $\kappa_{\rm m}$ = conductivity at temperature $t_{\rm m}$, and α = constant that depends on the dissolved salts.

The electronic measuring system has been implemented with a PIC18F876 microcontroller which has a 10-bit analogue-to digital (A/D) converter port. The square signal is generated through a digital output port. Signal variation at port's pins is between 0 and 5 V, but the electrode gets a variation between -5 and 5 V. Voltage measurement is through the A/D port with a resolution



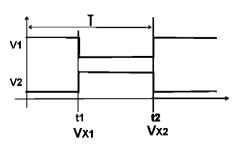


Fig. 5. Measuring circuit schema.

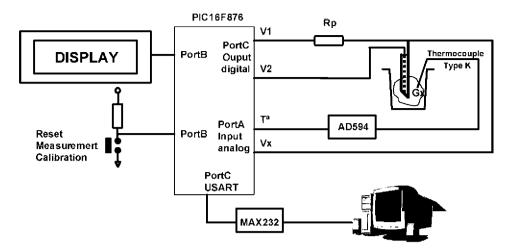


Fig. 8. Schema of the whole measuring system.

equal to 4.8×10^{-3} V for a reference value of 5 V. Temperature is measured with a type-K thermocouple and an amplifier and cold junction compensator (AD594 from Analog Devices). Fig. 8 shows the electronic schema of the system.

The system may be either autonomous or PC-controlled through a serial port. For this second option, the measurement control functions were programmed in Visual Basic. Such program also allows visualizing and storing measurements in MS-Excel format for further processing. System calibration is also controlled from the PC and it is done by "in situ" re-programming of the microcontroller making use of a resident program ("boot-loader").

2.3. Measurement

Measurements have been carried out using reference resistors of $10, 10^2, 10^3, 10^4$ and $10^5 \Omega$ and with signal frequencies ranging from 10^2 to 10^4 Hz. Results indicate that the optimum setup corresponds to the pair $R_{\rm p}$ = 100Ω and f = 2.5 kHz.

The above-described system has been used for measuring salted meat. The first set of experiments was carried out with minced pork meat. A defined amount of salt was added to the minced meat and homogenized, storing the samples in a refrigerator at 4 °C for 6 days to ensure a uniform salt distribution in the sample. A range of samples was prepared with an increasing quantity of salt, with a maximum NaCl concentration in the liquid phase (z^{NaCl}) of 7%. z^{NaCl} was determined assuming that all the NaCl and water in the sample are free, forming a brine embedded in the protein matrix of the meat $(z^{\text{NaCl}} = x^{\text{NaCl}} / (x^{\text{NaCl}} + x^{\text{w}}))$ Fig. 9 shows the obtained conductivity values (black points) as a function of salt concentration in the liquid phase (z^{NaCl}) together with values corresponding to brines measured with a commercial conductance-meter whose concentrations were within the same interval (continuous line in

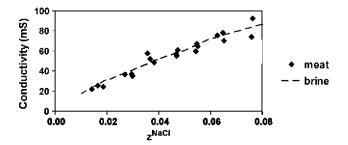


Fig. 9. Measured conductivity as a function of salt concentration.

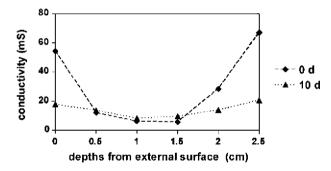


Fig. 10. Conductivity as a function of depth from surface the day salting ends (0 d) and 10 days after (10 d).

Fig. 9). The most remarkable aspects of the graph are, firstly, that conductivity is directly related to the salt concentration in liquid phase of the meat by an adjusted equation of second order (Eq. (3)) and, secondly, that the obtained values are very similar to those of brine with the same NaCl concentration.

$$\mu \text{ (mS)} = -5823.6 \cdot (z^{\text{NaCl}})^2 + 1504.1 \cdot (z^{\text{NaCl}}) + 0.726$$

$$r^2 = 0.95$$
(3)

Additional experiments were made so as to test the possibility of finding salt concentration profiles in whole meat pieces after a salting process. Fig. 10 depicts the conductivity measurements for a 2.5-cm-wide salted pork fillet obtained from a fresh ham piece. Measurements were taken in 0.5 cm depth increments just at the end of the salting process (0 d) (dry salting during 4 h at 4 $^{\circ}$ C) and after 10 days resting at 4 $^{\circ}$ C. It can be appreciated how the designed device allows us to obtain conductivity profiles and their evolution and, related to them, salt concentration profiles. Resulting graphs have the typical aspect of this kind of processes in which most of the salt is located near the meat surface at the end of the salting step and tend to be more homogenously throughout the post-salting time, hence equalizing inner and outer values

3. Conclusions

Preliminary results, obtained with both minced and whole meat, allow being confident about the feasibility of the herein presented device for investigating salt concentration and other closely related parameters in both meat and fish products ($a_{\rm w}$ and/or moisture). The advantages of the described system as compared to other non-destructive techniques would be the reduction in the

application cost, possibility of remote PC-control through wireless networks, applicability in non-destructive applications such as on-line testing, due to the small diameter (d = 0.46 mm) of the needle.

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