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Highlights:

-The ultrasonic time of flight (TOF) decreased progressively during salting.

-Online monitoring of dry salting is reliable using ultrasound pulse-echo technique.

-The TOF variation during salting was related to the salt gain.

-Classification using ultrasound of loins and hams according to salt gain is viable.

NON-INVASIVE ULTRASONIC TECHNOLOGY FOR CONTINUOUS MONITORING OF PORK LOIN AND HAM DRY SALTING

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ABSTRACT

Online ultrasound measurements were taken using pulse-echo mode in loins (*Longissimus dorsi*) and hams at different salting times (up to 30 days). From the time-domain ultrasonic wave, the time of flight (TOF) was computed as well as its variation between two signals (ΔTOF). A progressive decrease in TOF during dry salting was found, which was linked to the salt gain, water loss and the reduction in sample thickness. Predictive models based on the ultrasonic parameters (ΔTOF and initial time of flight, TOF_0) correctly classified 85% of the loins and 90% of the hams into 3 groups of salt content (low/medium/high). The results obtained confirm that the use of the ultrasonic pulse-echo technique is of great potential in the non-destructive monitoring of the dry salting in pork loins and hams, as well as in the prediction of the salt gain for classification purposes.

Keywords: Pork meat, Curing, Ultrasound, Non-destructive technology, Process control

1. INTRODUCTION

Dry salting is the traditional technique used in meat products with anatomical integrity, such as hams or loins, in order to obtain dry-cured products. In the dry-cured loin and ham industries, an accurate control of the dry salting process is especially complex because of the high degree of heterogeneity in the meat pieces (pH, weight, fat content and moisture, size of the piece…), the effect of the pre-treatments on the product (skin-trimming of the fresh ham or the freezing/thawing processes) and the influence of the different process variables (temperature, relative humidity, position in the salt pile, size of the coarse salt…) (Costa-Corredor, Muñoz, Arnau, & Gou, 2010; García-Gil, Muñoz, Santos-Garcés, Arnau, & Gou, 2014; Fulladosa, Muñoz, Serra, Arnau, & Gou, 2015). All of these factors lead to there being a highly variable salt content in the batches of dry cured hams and loins. This salt content variability is of great concern to the meat industry as an excessive amount of salt produces a too salty taste (Ruusunen & Puolanne, 2005), but an insufficient amount may cause sensory defects, such as pastiness and softness (Albarracín, Sánchez, Grau, & Barat, 2011) or microbiological problems (Desmond, 2006). Thus, the meat industry demands non-destructive quality control techniques that allow the salt content to be determined after the salting stage for quality control purposes.

Nowadays, the feasibility of using several non-destructive technologies (X-Ray, NMR, ultrasound...) to determine the salt content in meat products has been tested. In this regard, Fulladosa, Muñoz, Serra, Arnau, and Gou (2015) predicted the salt content in bone-in hams after salting by using an X-Ray inspector. Similarly, Manzocco et al. (2013) proposed predictive models with which to estimate the salt content in ham muscles by using magnetic resonance imaging (MRI). In this study, image analysis was carried out in different stages of dry-cured ham processing (before salting, after salting and at different times during resting, maturing and ageing). Recently, ultrasound has also been applied to predict the salt content in brine salted pork meat (De Prados, García-Pérez, & Benedito, 2015) and in dry-salted hams (De Prados, García-Pérez, & Benedito, 2016), by taking ultrasonic measurements before and after salting. An accurate prediction of the salt content after the salting may allow the products to be classified according to the different levels of salt, which can be used for the purposes of optimizing the subsequent processing stages. However, the measurement after salting does not permit the correction of the variability in the batch salt content by reducing the number of over-salted or insufficiently salted pieces. Thereby, the use of non-destructive quality control techniques to monitor the salt gain during the dry salting process is gaining importance in the meat industry as a means of meeting the target salt content in each piece. In this context, ultrasound has been used by De Prados, García-Pérez, and Benedito (2016) to monitor the dry salting process of *Longissimus dorsi* and *Biceps Femoris* pork muscles. In this study, the ultrasonic velocity was measured online during salting by the ultrasonic through-transmission mode. That mode is characterized by the use of two transducers in direct contact with two opposite and parallel sides of the sample, which makes its implementation in a ham or loin salting pile very complicated. Otherwise, the ultrasonic equipment could be greatly simplified by using the pulse-echo mode, where a single transducer can be placed on one side of the sample, simultaneously acting as emitter and receiver (Mulet, Benedito, Bon, & Sanjuan, 1999; Awad, Moharram, Shaltout, Asker, & Youssef, 2012). The use of one transducer located in the ham’s base during dry salting would facilitate the ultrasonic implementation, reducing the cost and the impact of the ultrasonic measurement on the mass transfers (salt and water).

The pulse-echo mode is most commonly used to detect internal defects in metallic materials, but it has also been applied in food characterization. Thus, the pulse-echo mode has been used to determine the ripeness of avocados (Gaete-Garretón, Vargas, León, & Pettorino, 2005), to detect anomalies in the internal structure of Mahon cheese

(Benedito, Cárcel, Gisbert, & Mulet, 2001) or bone fragments in deboned chicken breasts (Correia, Mittal, & Basir, 2008) and to measure the sugar content and viscosity of reconstituted orange juice (Kuo, Sheng, & Ting, 2008). Another application of the pulse-echo mode consisted in the monitoring of the cooling and freezing processes in several food products (gelatin, chicken, salmon, beef and yoghurt) (Sigfusson, Ziegler, & Coupland, 2001 and 2004). However, no studies have been found so far in the literature regarding either the online ultrasonic monitoring of the salting process in meat products or the salt gain assessment by means of the pulse-echo mode. Moreover, the ultrasonic monitoring of salting in structural and compositional complex meat pieces, such as whole hams, has not been addressed elsewhere.

The objective of this paper was to investigate the feasibility of applying ultrasound in the pulse-echo mode for both the online monitoring of dry salting in loins and hams and the prediction of the final salt gain.

2. MATERIALS AND METHODS

2.1 FRESH MEAT SAMPLING

Ten fresh hams and twenty loins (*Longissimus dorsi*) from *Large White* breed pigs were obtained in a local market. Loin and ham pieces were selected with a pH range of between 5.3 and 5.8. In loins, the subcutaneous fat and external connective tissue were removed and samples of 20.0±0.5cm in length (l) and with an average weight of 1.0±0.1kg (wg) were obtained, keeping the original width (z) and thickness (e) of the muscle.

2.2 DRY SALTING PROCESS

Dry salting experiments in loins and hams were carried out at 2±1ºC by covering the sample with 6kg or 15kg of coarse salt, respectively (NaCl moisturized at 10% w/w)

(Figure 1). The fresh samples and coarse salt were previously stored for 24h at 2ºC for tempering purposes. The thickness (e) was measured before and after dry salting in the loins and hams and the thickness reduction (Δe) was calculated. In the case of loins, four replicates were carried out for each salting time (6, 12, 24, 48 and 72h), while in that of hams, one was used for each salting time (4, 7, 10, 11, 12, 14, 15, 16, 20 and 30 days).

2.3 ULTRASOUND EXPERIMENTAL SET-UP

Figure 1 shows the experimental set-up used for the ultrasonic measurements during the dry salting of loins and hams. The experimental set-up consisted of four narrow-band piezoelectric transducers of 1MHz and 0.5" crystal diameter, two of them type TA (TA_1 and TA_2) (A303S model, Panametrics, Waltham, MA, USA) and the other two type TB (TB_1 and TB_2) (A103S-RM model, Panametrics, Waltham, MA, USA). The pulse generation and reception (5058PR, Panametrics, Waltham, MA, USA for loins and 5077PR, Panametrics, Waltham, MA, USA for hams) was multiplexed to the transducers using a digital input/output device (NI 6501, National Instruments, Austin, TX, USA) and a high-speed digitizer (PXI/PCI-5112, National Instruments, Austin, TX, USA) installed in a PC (Figure 1). The multiplexation unit allowed the signal from the pulser to reach the first transducer every 1h. The ultrasonic signal generated in this transducer crossed the sample, was reflected in the meat/salt interface and returned to the same transducer, sent to the receiver through the multiplexation device and digitized by the oscilloscope (Figure 1). This operation was sequentially repeated in the rest of the transducers by the action of the multiplexation unit.

The sample was placed on a layer of salt in direct contact with the four transducers (TA_1, TA_2, TB_1 and TB_2, which corresponds to the four ultrasonic measurement points) inside a plastic container (30x25x15cm for loins and 120x35x20cm for hams) (Figure 1). Afterwards, two type-K thermocouples were located in the salt and the sample, respectively; and the rest of the salt was added (total salt amount of 2kg for loins and 5kg for hams) until the sample was entirely covered. In the case of hams, a part of the skin in the cushion zone (177±14cm^2) was removed, which coincides with the region where the transducers were placed (Figure 1) and 1mL of water was added on the transducers' surfaces in order to guarantee the acoustic matching, thus, improving the signal intensity. The ultrasonic measurements were taken by the pulse-echo mode at intervals of 1h in the central part of the loins and in the cushion zone of the hams (Figure 1).

The time of flight represents the time which elapses between the pulser sending the signal to the transducer acting as emitter, until the signal crosses the sample twice and is detected in the transducer acting as receiver. The time of flight variation (ΔTOF) between two ultrasonic signals was the ultrasonic parameter considered for the online monitoring of the loins and hams. For that purpose, the cross correlation method (Leemans & Destain, 2009) was employed to calculate the ΔTOF between ultrasonic signals one hour apart by using a specific software developed in LABVIEW^{TM} 2015 (National Instruments, Austin, TX, USA) and the final ΔTOF for the different salting times was computed. The initial time of flight (TOF$_0$), which corresponds to the raw meat, was calculated through the energy threshold method (Avanesians & Momayez, 2015), using the same software.

2.4 CHEMICAL ANALYSIS

The fat, salt and water contents were determined in the fresh muscles and hams. To this end, a piece (200±50g) was taken from each loin after obtaining the fresh muscle sample for the salting process. In the case of the hams, as the sample integrity cannot be altered before salting, the average values of the fat, salt and water contents were obtained from 30 hams of the same breed purchased from the same supplier.

Once the salting finalized, the excess salt was removed from the surface of the loins and hams and four cylindrical salted samples (64±13g for loins and 172±39g for hams) corresponding to the ultrasonic measurement points (Figure 1), were taken by using a cylindrical cutter (5cm in diameter). Each cylindrical salted sample was ground and homogenized before the analysis. The analyses of the fat and water contents were carried out according to AOAC procedures 991.36 and 950.46, respectively (AOAC, 1997). The salt content was analyzed following the process described by De Prados, García-Pérez, and Benedito (2015 and 2016). All the analyses were performed in triplicate.

The salt (X_S), water (X_W) and fat (X_F) contents of the fresh samples and the salted loin and ham cylinders were expressed as percentages (%) in wet basis (w.b.). The final salt gain (ΔX_S) and the water loss (ΔX_W) were also calculated as an average of the four cylindrical samples from the loins and hams at each salting time.

2.5 STATISTICAL ANALYSIS AND DEVELOPMENT OF PREDICTIVE MODELS

The influence of the salting time on the ΔX_S, ΔX_W, Δe and ΔTOF in loins and hams was evaluated by means of an analysis of variance. Similarly, the analysis of variance was carried out in order to determine the significant influence of the type of transducer (TA and TB) on the final ΔTOF value during salting. Additionally, a multiple regression model was used to evaluate the influence of the salting time, ΔX_S, ΔX_W and Δe on the ΔTOF. In every case, the Statgraphics® Centurion XV (Statpoint Technologies Inc., Warrenton, VA, USA) software was used and a significance level of 95% was fixed.

The ultrasonic (ΔTOF and TOF_0) and sample parameters (weight, wg) and salting time (t) were used as independent variables so as to predict the salt gain (ΔX_S) in loins and hams. For that purpose, both the loins and hams were split into two sets; a model set (M) and a validation set (V). The model set (M) included 15 loins and 7 hams, chosen randomly. Their ultrasonic measurement points (n_{ump}=60 for loins and 28 for hams) were used to develop multiple regression models with the Statgraphics® Centurion XV (Statpoint Technologies Inc., Warrenton, VA, USA). The optimal number of independent variables and the interactions in the model were obtained using the Marquardt method. The p-value used to keep the independent variables in the model was 0.05. The validation set included 5 loins (n_{ump}=20) and 3 hams (n_{ump}=12). The overall classification capacity was tested using the optimal model for salt gain prediction. For this purpose, loins and hams and their corresponding ultrasonic measurement points from sets M and V were classified into three different categories. Loins with a salt content of <2.5% w.b. were considered to have a low level of salt, those with a salt content of >4.0% w.b. a high level of salt and the remaining ones to have a moderate level of salt. For hams, three categories were also considered (low <2.0% w.b., medium 2.0-3.0% w.b. and high >3.0% w.b. salt content level). The levels of salt in loins were higher than in hams due to the fact that the loins are not as thick as hams, so actually, they tend to be saltier.

3. RESULTS AND DISCUSSION

3.1 FRESH SAMPLE CHARACTERIZATION

As shown in Table 1, a similar initial salt content (X_S) was observed in loins and hams. However, significant differences ($p<0.05$) were found between the fat (X_F) and water (X_W) contents in fresh loins and hams, the fat content being greater in hams than in loins. The ranges of X_F, X_S, and X_W found in the present study (Table 1) coincide with the ones reported for *Large White* breed loins (*Longissimus dorsi*) and hams in the literature (Cierach & Modzelewska-Kapituła, 2011; Moreiras, Carbajal, Cabrera, & Cuadrado, 2013). Additionally, a high degree of variability was found in the X_F (0.3-6.1% w.b. for loins and 7.5-26.4% w.b. for hams) and X_W (67.9-75.6% w.b. for loin and 56.3-70.2% w.b. for ham) compared to the that observed in the X_S (0.10-0.26% w.b. for loins and 0.16-0.33% for hams), which is especially evident in the case of hams (Table 1). This compositional variability, especially the fat content and its distribution, should be considered when designing the salting process due to the fact that it may affect the mass transfer (salt gain and water lost) during salting (Cierach & Modzelewska-Kapituła, 2011; De Prados, García-Pérez, & Benedito, 2015).

3.2 COMPOSITIONAL CHANGES DURING DRY-SALTING

Figure 2 illustrates the kinetics of salt gain and water loss in loins and hams during dry salting. The ΔX_S values in the present work were slightly lower than the ones reported by other studies. For example, De Prados, García-Pérez, and Benedito (2016) reported a salt gain of between 2.1 and 6.7% w.b. in loins salted from 6 to 48h. When studying hams that had been dry salted for between 2 and 16 days, Fulladosa, Muñoz, Serra, Arnau, and Gou (2015) and Håseth, Sørheim, Høy, and Egelandsdal (2012) found a salt content ranging from 0.8 to 4.8% w.b. and from 1.2 to 4.5% w.b., respectively. This lower salt gain could be explained by considering that other authors reported the salt content as an average of the whole piece. However, in the present study, the salt gain shown represents the average value between the four cylindrical samples corresponding to the four ultrasonic measurement points of the cushion zone of the ham (Figure 1), which is its thickest part and where the salt diffusion takes longer (Toldrá & Wai-Kit, 2008; Håseth, Sørheim, Høy, & Egelandsdal, 2012). On the other hand, the ΔX_S and ΔX_W in loins and hams showed a marked experimental variability, especially the ΔX_W (Figure 2). As previously mentioned, salting is a complex process affected by the different process variables (temperature, size of coarse salt, quantity of salt, position in the salt pile, salting time...) and the high degree of compositional heterogeneity of the meat pieces. In this study, the process variables were accurately controlled and the pieces (loins and hams) were individually salted (not pile salted). Thus, this great variability in the ΔX_S and ΔX_W could be mainly ascribed to the highly heterogeneous nature of the fresh meat pieces in terms of the content and distribution of the water, fat and connective tissue, among other things. This fact is more relevant in hams due to the presence of both the skin and bone and also to that of different muscles with a greater degree of heterogeneity than in single muscles.

3.3 ULTRASONIC MONITORING OF THE DRY SALTING PROCESS

A gradual decrease of the TOF during salting was found. As an example, Figure 3 shows the displacement in the time domain of the ultrasonic signal from the position at time 0 to the one captured after 30 days of salting. As observed, the ultrasonic signal after 30 days is displaced to the left (Figure 3), which illustrates a shortening of the TOF. This could be explained by the fact that ultrasound travels faster in solids, with a high elastic modulus (Benedito, Cárcel, Clemente, & Mulet, 2000), than in liquids (water). Thus, the increase in the solid content during salting, as a result of the salt gain and water loss, leads to an increase in the sample’s ultrasonic velocity, and thereby, a decrease in the TOF. When analyzing different kinds of samples (water, fish, juice and meat), several studies have reported an increase in the ultrasonic velocity in line with an increase in the solid content (Kinsler, Frey, Coppens, & Sanders, 1982; McClements, 1995; Ghaedian, Coupland, Decker, & McClements, 1998; Kuo, Sheng, & Ting, 2008; De Prados, García-Pérez, & Benedito, 2015 and 2016).

Figure 4 illustrates the evolution of the ΔTOF in loins and hams during different salting experiments (24, 72h for loins and 11, 20 days for hams). The same behavior (data not shown) was observed in the remaining experiments (6, 12 and 48h dry salting of loins and 4, 7, 10, 12, 14, 15, 16 and 30 days’ dry salting of hams). As can be appreciated, the ΔTOF decreased gradually due to the above mentioned increase in the solid content of the sample. This result is consistent with the increase in the salt content and the decrease in the water content in loins and hams during dry salting, as observed in Figure 2. The ΔTOF evolution was different for each ultrasonic measurement point corresponding to the different transducers (TA_1, TA_2, TB_1 and TB_2) (Figure 4). This fact could be linked to the heterogeneity of the compositional changes (ΔX_S and ΔX_W) at each ultrasonic measurement point during salting, which was especially evident in the ham salting experiments (Figure 2). Non-significant ($p>0.05$) differences were observed in the ΔTOF evolution between type TA and TB transducers (Figure 4). Therefore, both types of transducers could be considered equivalent for the purposes of monitoring the salting process.

An unexpected, fast decrease in the ΔTOF was observed during the first few hours of dry salting (Figure 4) for both loins and hams, which was not coherent with the kinetics for salt and water diffusion (Figure 2). Similar behavior was observed in the evolution of the ultrasonic velocity in *Longissimus dorsi* and *Biceps femoris* during dry salting (De Prados, García-Pérez, & Benedito, 2016). De Prados, García-Pérez, and Benedito (2016) associated this behavior with the formation of a salt solution between the transducers and the meat, due to the initial extraction of water from the external meat layers, which could partly explain the important decrease in the ΔTOF during the first 3h, as may be observed in Figure 4. Additionally, the fast initial ΔTOF could also be associated with the textural changes that take place on the meat surface due to the effect of the salt in contact with the sample. Thus, a test was conducted in order to prove this hypothesis. Two loins (1kg) were salted for 1 and 3h, respectively and the hardness, characterized as the maximum penetration force (N), was evaluated in each loin before and after salting. Penetration test was conducted with a 6mm flat cylinder probe, a crosshead speed of 1mm/s and strain of 20% (penetration distance of 10mm). The results showed that, after 1 and 3h, the salted loins were significantly ($p<0.05$) harder (14.7±1.8N at 1h and 21.4±3.4N at 3h) than the fresh ones (8.6±4.0N). Ruiz-Ramírez, Arnau, Serra, and Gou (2005) related the increase in hardness with the increase in the salt content in the dry cured pork muscles (*Biceps femoris* and *Semimembranosus*). This increase in hardness was linked to the compaction of the myofibrillar structure due to the salt content and an inhibitory effect of salt on the calpains activity (Ruiz-Ramírez, Arnau, Serra, & Gou, 2005; Lorenzo, Fonseca, Gómez, & Domínguez, 2015). Thus, the salt gain and water loss that takes place in the external meat layers during the first few hours gives rise to a great increase in the salt concentration in these layers, leading to a rapid surface textural increase which would explain the fast decrease in the ∆TOF.

The TOF and velocity measurements have been used to monitor other food processes. Thus, Sigfusson, Ziegler, and Coupland (2001 and 2004) monitored the cooling and freezing process in different food products by measuring the TOF by means of the pulse-echo mode. Recently, De Prados, García-Pérez, and Benedito (2016) monitored the meat dry salting process online. In that work, the ultrasonic velocity was measured online in *Longissimus dorsi* and *Biceps femoris* by using the through-transmission mode, where the sample’s thickness had to be measured and two transducers were in direct contact with two opposite and parallel sides of the sample (De Prados, García-Pérez, & Benedito, 2016). This fact complicates the implementation of ultrasonic technology in the meat industry where pile salting is used. On the contrary, in the present study, the ultrasonic measurements were taken in loins and hams by using the pulse-echo mode, which is characterized by the use of a single transducer and the sample’s thickness does not have to be measured. The use of a single transducer on the loin or ham’s base during pile salting would simplify the industrial implementation, reduce the cost of the device and minimize the impact of the ultrasonic measurement on the mass transfers (salt and water).

Therefore, the results reported in this section confirm that the ΔTOF measured by using the pulse-echo mode could be a useful ultrasonic parameter for the purposes of performing the online monitoring of the salting process in individual loins (average thickness 5.0cm), as well as in more complex and thicker meat pieces, such as hams (average thickness 15.7cm). Additionally, the ultrasonic pulse-echo technique represents a significant improvement for the industrial application of the system compared to the ultrasonic through-transmission technique due to the fact that it avoids the need to measure the sample thickness and the number of transducers required is reduced.

3.4 INFLUENCE OF THE DRY SALTING ON THE TIME OF FLIGHT

Table 2 shows the thickness reduction (Δe), salt gain (ΔX_S), water loss (ΔX_W) and time of flight variation (ΔTOF) in loins and hams during dry salting at 2ºC. As appreciated in Table 2, the longer the salting time, the higher the ΔTOF. Thus, the ΔTOF increased in loins from -2.7µs (6h) to -7.0µs (72h) and in hams from -5.5µs (4 days) to -17.6µs (30 days) (Table 2). As previously mentioned, and as observed in Table 2, the ΔTOF during salting was related to the ΔX_S and ΔX_W in the samples (loins and hams). Overall, the higher the ΔX_S and ΔX_W, the higher the ΔTOF. In addition, water loss during salting leads to meat shrinkage (García-Gil, Muñoz, Santos-Garcés, Arnau, & Gou, 2014), and thus, to thickness reduction (Δe) (Table 2), which could also contribute to shortening the time of flight. Despite the fact that the ΔX_W and Δe factors affect ΔTOF, the multiple regression model used showed that both factors were statistically non-significant ($p>0.05$) on the ΔTOF prediction, which can be attributed to the fact that the magnitude of the individual effect of these factors can be masked by their highly variable nature in the salting process. Thus, according to the statistical analysis, the ΔTOF is mainly related to the salt gain in the sample during salting.

Previous results have shown a similar relationship between the ΔX_S and the change of the ultrasonic velocity regardless of the sample nature (formulated samples from ground pork meat, pork muscles or water solution) (De Prados et al., 2015). Consequently, as velocity of ultrasound in a sample is the ratio between the sample thickness (e) and the TOF, the ΔX_S must be related not only to the ΔTOF, but also to the initial sample thickness and the thickness change (Δe). As far as in the present work the effect of Δe on ΔX_S has been found to be negligible, the models for predicting

ΔX_S should consider both ΔTOF and the initial e. As an example of the need of considering the initial sample thickness, it can be observed that the ΔTOF was -3.3µs in loins (e_{avg}= 5.0cm) for a ΔX_S of 2.5% w.b. after 12h of salting, while the ΔTOF was -11.2µs in hams (e_{avg}=15.7cm) for a similar ΔX_S (2.2% w.b.) after 14 days of salting. However, when the ΔTOF/e was computed for these two cases, a similar value was found for loins (-0.7µs/cm) and hams (-0.8µs/cm). Since measuring the thickness before salting could be complex on an industrial scale, the initial time of flight (TOF_0), related to the sample thickness and velocity of sound in the raw meat, could be used.

3.5 PREDICTIVE MODELS FOR SALT GAIN AND CLASSIFICATION OF LOINS AND HAMS

Since the ΔTOF is related to the salt content and the TOF_0 is related to the initial thickness, both ultrasonic parameters (ΔTOF and TOF_0) were used as factors with which to develop predictive models for the salt gain. The ultrasonic measurement points of the M set were used to develop the predictive models as mentioned in section 2.5. Eq.1 and Eq.2 show the best models obtained for the salt gain estimation in loins and hams by combining the two ultrasonic variables (ΔTOF and TOF_0) from the pulse-echo measurements. The R^2 and RMSE were 0.787 and 0.73% for loins (Eq.1) and 0.774 and 0.57% for hams (Eq.2). Additionally, other predictive models were established, including two variables which can be easily measured at industrial level: one regarding the sample (the initial weight, wg) and the other one related to the salting process (the salting time, t) (Eq.3 for loin and Eq.4 for ham). The inclusion of t and wg provided additional information that improved the predictive models, obtaining a reduced model error (RMSE=0.45% for loins using Eq.3 and 0.43% for hams using Eq. 4) and an increase in the determination coefficient (R^2= 0.923 for loins and 0.891 for hams). Similar results were obtained by Fulladosa, Muñoz, Serra, Arnau, and Gou (2015) and Håseth et al. (2008) using X-Rays. In these studies, the salt prediction was more accurate than in the present analysis, the RMSE being 0.30% for hams of different breeds (Fulladosa, Muñoz, Serra, Arnau, & Gou, 2015) and 0.20-0.40% for ground pork *Semimembranosus* muscles (Håseth et al., 2008).

LOIN $$\Delta X_S = 3.524 - 0.0039 \cdot TOF_0^2 - 0.0255 \cdot TOF_0 \cdot \Delta TOF \qquad \text{Eq.1}$$

HAM $$\Delta X_S = 0.253 - 0.0003 \cdot TOF_0^2 - 0.0125 \cdot \Delta TOF_0 - 0.0066 \cdot TOF_0 \cdot \Delta TOF \qquad \text{Eq.2}$$

LOIN $$\begin{aligned} \Delta X_S &= 1.568 + 0.143 \cdot t - 0.0010 \cdot t^2 - 0.0414 \cdot \Delta TOF^2 + 0.0431 \cdot t \cdot wg \\ &- 0.0034 \cdot t \cdot TOF_0 - 0.0107 \cdot t \cdot \Delta TOF \end{aligned} \qquad \text{Eq.3}$$

HAM $$\begin{aligned} \Delta X_S &= -39.051 + 0.140 \cdot t - 0.00003 \cdot t^2 + 3.550 \cdot wg - 0.00004 \cdot TOF_0^2 - 0.0120 \cdot t \cdot wg \\ &+ 0.0001 \cdot t \cdot TOF_0 + 0.00004 \cdot t \cdot \Delta TOF \end{aligned} \qquad \text{Eq.4}$$

where ΔX_S is the salt gain (% w.b.), t the salting time (h), wg the initial sample weight (kg), TOF_0 the initial time of flight (μs) and ΔTOF the time of flight variation (μs).

The usefulness of ultrasound as a reliable method of classifying loins and hams according to the different levels of salt gain was tested by using the best predictive models (Eq.1, Eq.2, Eq.3 and Eq.4). For that purpose, the validation (V) and model (M) sets of the whole loins and hams and each ultrasonic measurement point were classified into three different categories of salt gain (ΔX_S), as mentioned in section 2.5. Similar percentages of correctly classified samples (CC) at the ultrasonic measurement points (79% in loins and 75% in hams) and in the whole pieces (85% in loins and 90% in hams) were computed (Table 3) by using only the ultrasonic parameters (Eq.1 and 2). On the other hand, the classification improved by using Eqs.3 and 4, especially in the case of loins (Table 3). In this regard, the percentage of CC ultrasonic measurement points increased from 79% to 86% for loins and from 75% to 78% for hams. In the case of whole loins, the percentage of CC samples increased from 85% to 95% whereas no improvement was found for whole hams.

4. CONCLUSIONS

The gradual shortening of the time of flight during the dry salting of loins and hams was mainly related to the salt gain. 85% of the loins and 90% of the hams were correctly classified by using predictive models based on the ultrasonic parameters. A slight improvement in the percentage of correctly classified samples was achieved in loins (95%) with the inclusion of the sample's weight and salting time in the predictive model. Therefore, the ultrasound pulse-echo mode could be a useful technique for continuous dry salting monitoring, as well as for the salt gain prediction for classification purposes. In addition, the pulse-echo technique is characterized by the use of a single transducer on one side of the sample, which facilitates the industrial implementation of this non-destructive technique compared to the through-transmission mode.

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Figure captions

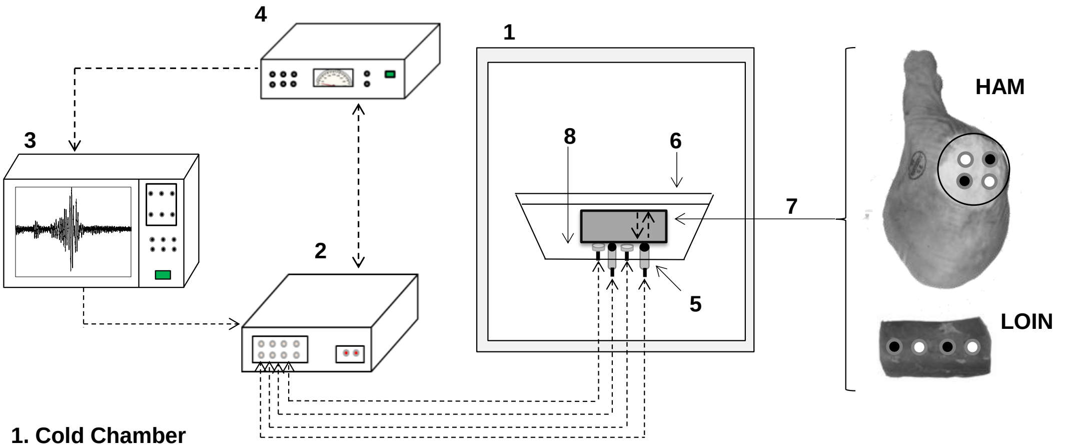
Figure 1. Experimental set-up for online ultrasonic measurements in loins and hams during dry salting.

Figure 2. Experimental kinetics of salt gain (ΔX_S) and water loss (ΔX_W) in loins and hams during dry salting at 2ºC.

Figure 3. Variation of the time of flight (ΔTOF) between the first and last ultrasonic signals captured in a ham dry salted for 30 days at 2ºC.

Figure 4. Time of flight variation (ΔTOF) in loins (**A** and **B**) and hams (**C** and **D**) during dry salting (24-72h for loins and 11-20 days for hams) at 2ºC. Each series corresponds to a different ultrasonic measurement point (TA_1, TA_2, TB_1 and TB_2).

Figure 1



1. Cold Chamber
2. Multiplexation device
3. Data Acquisition Card-PC
4. Pulser-Receiver
5. Transducers TA and TB
6. Container
7. Sample (LOIN and HAM)
8. Salt

TA_1 - TA_2 ●
Ultrasonic measurement points
TB_1 - TB_2 ○

Figure 2

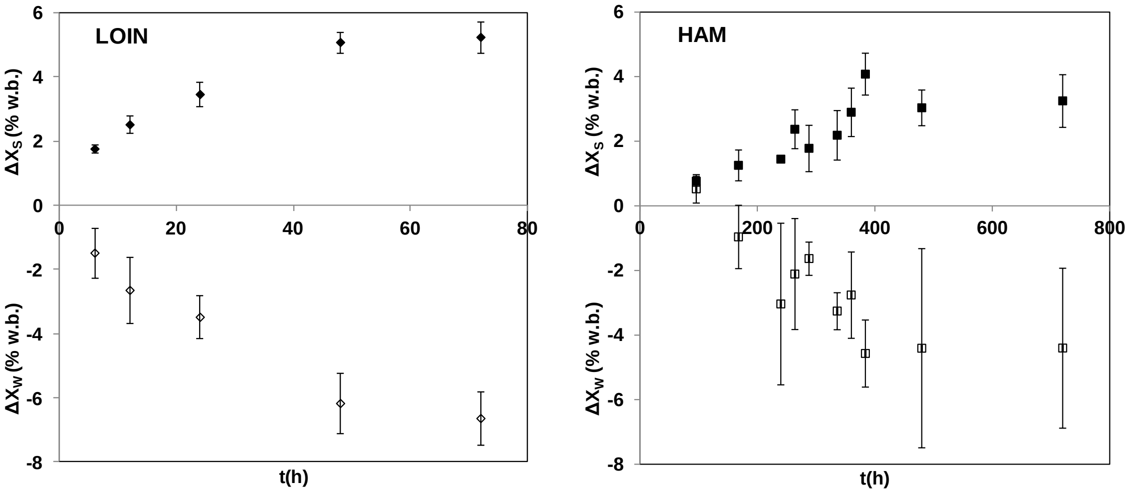

LOIN
6
4
2
0
-2
-4
-6
-8
0
20
40
60
80
ΔX_S (% w.b.)
ΔX_W (% w.b.)
t(h)
HAM
6
4
2
0
-2
-4
-6
-8
0
200
400
600
800
ΔX_S (% w.b.)
ΔX_W (% w.b.)
t(h)

Figure 3

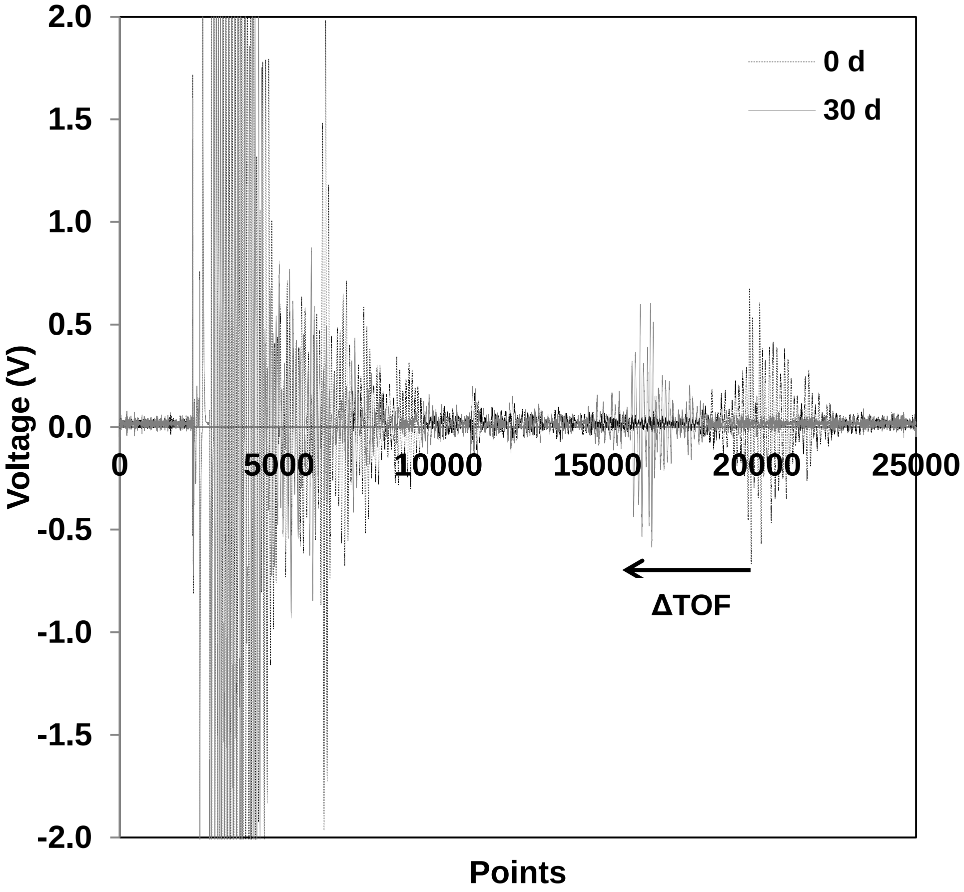

2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2.0
Voltage (V)
0
5000
10000
15000
20000
25000
Points
0 d
30 d
ΔTOF

Figure 4

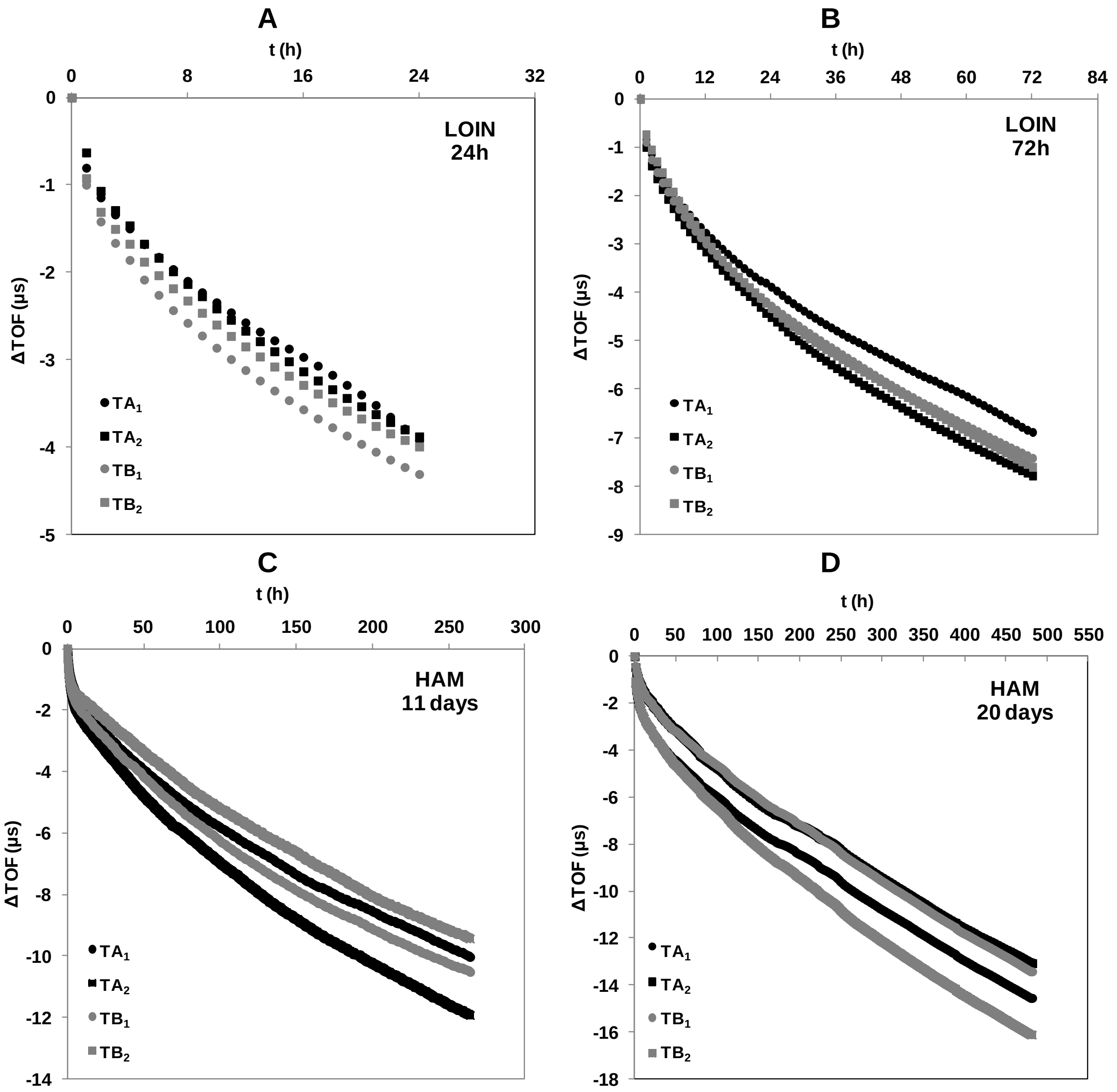

A
t (h)
0 8 16 24 32
0 -1 -2 -3 -4 -5
ΔTOF (µs)
LOIN
24h
TA1
TA2
TB1
TB2
B
t (h)
0 12 24 36 48 60 72 84
0 -1 -2 -3 -4 -5 -6 -7 -8 -9
ΔTOF (µs)
LOIN
72h
TA1
TA2
TB1
TB2
C
t (h)
0 50 100 150 200 250 300
0 -2 -4 -6 -8 -10 -12 -14
ΔTOF (µs)
HAM
11 days
TA1
TA2
TB1
TB2
D
t (h)
0 50 100 150 200 250 300 350 400 450 500 550
0 -2 -4 -6 -8 -10 -12 -14 -16 -18
ΔTOF (µs)
HAM
20 days
TA1
TA2
TB1
TB2

Table 1

Table 1. Fat (X_F), water (X_W) and salt content (X_S), thickness (e), width (z), length (l) and weight (wg) of the fresh loins and hams*.

	LOINS	**HAMS**
X_F (% w.b.)	2.4 ± 1.9^{a}	$14.0 \pm 3.7^{b*}$
X_W (% w.b.)	72.7 ± 2.1^{c}	$65.5 \pm 2.9^{d*}$
X_S (% w.b.)	0.18 ± 0.05^{e}	$0.23 \pm 0.05^{e*}$
e (cm)	5.0 ± 0.8^{f}	15.7 ± 0.6^{g}
z (cm)	11.0 ± 0.8^{j}	27.8 ± 2.8^{i}
l (cm)	20.0 ± 2.0^{j}	49.4 ± 6.0^{k}
wg (kg)	1.0 ± 0.1^{l}	10.3 ± 0.9^{m}

Mean values and standard deviations.

Different letters in the same row indicate significant ($p < 0.05$) differences between loins and hams.

* Average values and standard deviations of 30 fresh *Large White* breed hams .

Table 2

Table 2. Salt gain (ΔX$_S$), water loss (ΔX$_W$), thickness reduction (Δe) and time of flight variation (ΔTOF) in loins and hams during dry salting at 2ºC.

SAMPLE	TIME	Δe(cm)	ΔX$_S$ (% w.b.)	ΔX$_W$ (% w.b.)	ΔTOF (μs)
LOIN	**6h**	-0.23±0.58^{a}	1.8±0.2^{a}	-1.5±1.5^{a}	-2.7±0.8^{a}
	12h	-0.47±0.99^{a}	2.5±0.5^{b}	-2.6±1.9^{b}	-3.3±0.6^{b}
	24h	-0.24±0.45^{a}	3.5±0.7^{c}	-3.5±1.2^{b}	-4.6±1.0^{c}
	48h	-1.15±0.59^{a}	5.1±0.6^{d}	-6.2±1.8^{c}	-6.0±0.7^{d}
	72h	-0.45±0.48^{a}	5.3±0.9^{d}	-6.6±1.6^{c}	-7.0±0.9^{e}
HAM	**4 days**	-1.71±0.49^{abc}	0.8±0.1^{a}	0.5±0.3^{a}	-5.5±0.5^{a}
	7 days	-1.36±0.27^{bc}	1.3±0.3^{ab}	-1.0±0.6^{b}	-8.8±0.8^{bc}
	10 days	-1.28±1.88^{bc}	1.4±0.1^{b}	-3.0±1.5^{de}	-8.0±1.0^{b}
	11 days	-1.17±0.63^{bc}	2.4±0.4^{de}	-2.1±1.1^{bcd}	-10.3±1.3^{cd}
	12 days	-0.40±0.50^{c}	1.8±0.5^{bc}	-1.6±0.3^{bc}	-11.0±1.5^{d}
	14 days	-0.93±0.49^{bc}	2.2±0.5^{cd}	-3.3±0.4^{de}	-11.2±1.0^{d}
	15 days	-2.88±1.31^{a}	2.9±0.5^{ef}	-2.8±0.8^{cd}	-13.1±1.5^{e}
	16 days	-2.18±0.89^{ab}	4.1±0.4^{g}	-4.6±0.7^{f}	-14.2±0.4^{e}
	20 days	-5.15±1.24^{d}	3.0±0.3^{f}	-4.4±1.9^{ef}	-14.3±1.4^{e}
	30 days	-4.75±1.27^{d}	3.2±0.5^{f}	-4.4±1.6^{ef}	-17.6±0.5^{f}

Average values and standard deviations.

Table 3

Table 3. Classification of loins and hams according to different levels of salt gain (ΔX_S) by using the best predictive models (Eqs.1 and 3 for loins and Eqs.2 and 4 for hams).

			Ultrasonic measurement points					**Whole loins**				
			CC for different levels of ΔX_S (% w.b.)					**CC for different levels of ΔX_S (% w.b.)**				
			n_{UMP}	**<2.5**	**2.5-4.0**	**>4.0**	**TOTAL**	**n_S**	**<2.5**	**2.5-4.0**	**>4.0**	**TOTAL**
LOINS	**Eq.1**	**M**	60	11/19 (58%)	15/17 (88%)	23/24 (96%)	63/80 (**79%**)	15	3/4 (75%)	4/5 (80%)	6/6 (100%)	17/20 (**85%**)
		V	20	4/5 (80%)	4/6 (67%)	6/9 (67%)		5	1/1 (100%)	1/1 (100%)	2/3 (67%)	
	Eq.3	**M**	60	16/19 (84%)	14/17 (82%)	24/24 (100%)	69/80 (**86%**)	15	4/4 (100%)	5/5 (100%)	6/6 (100%)	19/20 (**95%**)
		V	20	5/5 (100%)	4/6 (67%)	6/9 (67%)		5	1/1 (100%)	1/1 (100%)	2/3 (67%)	

			Ultrasonic measurement points					**Whole hams**				
			CC for different levels of ΔX_S (% w.b.)					**CC for different levels of ΔX_S (% w.b.)**				
			n_{UMP}	**<2.0**	**2.0-3.0**	**>3.0**	**TOTAL**	**n_S**	**<2.0**	**2.0-3.0**	**>3.0**	**TOTAL**
HAMS	**Eq.2**	**M**	28	9/12 (75%)	6/9 (67%)	6/7 (86%)	30/40 (**75%**)	7	2/3 (67%)	2/2 (100%)	2/2 (100%)	9/10 (**90%**)
		V	12	4/6 (67%)	3/4 (75%)	2/2 (100%)		3	1/1 (100%)	1/1 (100%)	1/1 (100%)	
	Eq.4	**M**	28	10/12 (83%)	5/9 (56%)	7/7 (100%)	31/40 (**78%**)	7	2/3 (67%)	2/2 (100%)	2/2 (100%)	9/10 (**90%**)
		V	12	5/6 (83%)	2/4 (50%)	2/2 (100%)		3	1/1 (100%)	1/1 (100%)	1/1 (100%)	

M and V refer to the model and validation set, respectively.

n_{UMP} and n_S are the number of ultrasonic measurement points (UMP) and samples (S) in each set.

CC represents the correctly classified samples and is expressed as the percentaje of correctly classified n_{UMP} or n_S (in parenthesis/brackets) and as the ratio between the correctly classified and total n_{UMP} or n_S for each level of salt gain.