ABSTRACT

In the dry-cured ham industry, an accurate control of the dry-salting process is especially complex because of the great heterogeneity of the meat pieces and the effect of different operational variables. The main objective of this study was to evaluate the feasibility of using an ultrasound system and methodology, adapted to the industry requirements, for the online monitoring of the ham dry-salting process. For that purpose, hams were dry salted for different times (4, 10, 11, 14, 16 and 30 days) at 2ºC. The cushion zone of the ham was placed over the transducers during salting and ultrasonic signals were taken automatically (5 min interval by using pulse-echo mode. Several methods of signal analysis were considered in order to assess the time of flight (TOF). TOF estimations by means of the energy threshold and cross-correlation methods (between the initial ultrasonic signal and the remaining signals measured during salting and between consecutive signals 5 min apart without interpolation) were affected by the low signal-to-noise ratio and the pulse distortion and were discarded for the online monitoring of ham salting. Otherwise, the cross-correlation method between consecutive signals (5 min apart) with interpolation n=3 (CCM-CS n=3), between non-consecutive signals (1 h apart) (CCM-NCS) and the phase spectrum method (PSM), provided close estimations of the variation of the TOF, which correlated well with the ham salt gain (R^2=0.83 for CCM-CS n=3, 0.93 for CCM-NCS and 0.90 for PSM). Consequently, the use of ultrasonic pulse-echo TOF measurements could be considered as a simple, non-invasive, non-destructive and reliable technique for the industrial monitoring of the ham dry-salting process.

Keywords: *Salting; Ham; Time of flight; Energy threshold; Cross-correlation; Phase spectrum*

1. INTRODUCTION

The online monitoring of food processes allows the physicochemical changes that take place in food matrices during manufacturing to be controlled, in order to achieve the expected organoleptic and safety attributes. Nowadays, several new non-destructive techniques (X-Rays, NIR spectroscopy, ultrasound, etc.) have been developed or adapted for the purposes of measuring a wide range of quality parameters during food processing (Dixit et al., 2017; Perez-Santaescolastica et al., 2019).

In dry-cured ham manufacturing, the dry-salting stage consists of stacking the hams, subcutaneous fat side down, surrounded by coarse salt at 2-4ºC and a relative humidity of 90-95% for several days (Toldra, 2010). At present, the salting time is mainly determined from the average weight of the hams, which have previously been separated into batches of similar weight. The monitoring of the dry-salting process would be of great interest for the meat industry, since the salt gain in a batch of hams varies widely, because of the great heterogeneity of the meat pieces (composition, shape and structure) and the effect of the different operational variables (De Prados et al., 2015). This variable salt gain causes non-uniform behaviour of the hams during the subsequent processing stages, and consequently, the final dry-cured hams from the same batch have heterogeneous sensory properties, affecting their quality.

Low-intensity ultrasound is one of the most promising non-invasive technologies for food process monitoring since it is accurate, fast, easy to implement on-line and relatively inexpensive. In this regard, the ultrasonic measurements have been used to monitor a wide range of food processes, such as the rennet of whole milk during cheese manufacturing (Koc and Ozer, 2008), the alcoholic fermentation in synthetic broths (glucose, fructose and sucrose) and in natural media (must and wort) (Resa et al., 2009), the temperature and the ice content of fish (hake) during freezing (Aparicio et al., 2008), the ripening of tofu (Ting et al. 2009), the quality of oil during frying (Benedito et al., 2002) or the crystallization of palm oil in O/W emulsions (Awad and

Sato, 2002). Recently, De Prados et al. (2016) used the ultrasonic through-transmission technique to monitor the pork meat dry-salting process (*Biceps femoris* and *Longissimus dorsi* muscles). In most of these studies, ultrasonic velocity, calculated from the time of flight (TOF) and the sample thickness, is the parameter calculated from the ultrasonic signal and chosen for the purposes of correlation with compositional and mechanical properties. The accurate estimation of other ultrasonic parameters, such as attenuation, results very complicated in solid foods if coupling materials, such as gels, are not used. The use of coupling materials has some disadvantages, which strongly prevents their application in many cases because it may involve the product's surface contamination, as well as the slowdown of the measurement.

Overall, the energy threshold method is the one most often used to determine the TOF from the ultrasonic signals. However, when the signal in the sample has a low signal-to-noise ratio (SNR) and/or a large pulse distortion, this method can lead to a miscalculation of the TOF, and other approaches, such as the cross-correlation and phase spectrum methods, must be considered (Sachse and Pao, 1978; Leemans and Destain, 2009; Pallav and Hutchins, 2009). Alternatively, when the different echoes overlap in the time domain, amplitude spectrum methods can be used (Pialucha et al., 1989; Gomez Alvarez-Arenas, 2009a and 2009b; Sarabia et al., 2013). Moreover, when the material under study is dispersive, cross-correlation methods can only be applied to sufficiently large tone bursts.

De Prados et al. (2016) used the through-transmission method for the online monitoring of the dry-salting of meat muscles, placing two transducers on the opposite sides of the product. This set-up allows good signal amplitude to be obtained, since the ultrasonic wave has to cross the sample only once. However, this configuration could lead to important drawbacks when implemented for the online monitoring of the dry-salting of hams, since transducers have to be located on both faces of the stacked hams and perfectly aligned during the whole salting process. An alternative configuration might be the use of transducers located at the bottom of the stack of hams and working in the pulse-echo mode. However, using this simple and easy-to-implement online arrangement, the ultrasonic signal has to cross a thick, highly heterogeneous and complex medium composed of skin, bones and different muscles twice, which could hinder the measurement of the TOF due to a more intense attenuation and signal distortion. Therefore, the aim of this study was to investigate the feasibility of using an ultrasound pulse-echo technique, adapted to the industry requirements, for the online monitoring of the dry-salting process of hams and to determine the most adequate method for signal analysis in order to calculate the changes in the TOF during salting.

2. MATERIALS AND METHODS

2.1 SAMPLES AND DRY-SALTING PROCESS

Large White breed hams were purchased in a local market with an average weight of 13.3±4.5 kg and pH 5.7±0.1 (FG2-FiveGo^{TM}, Mettler Toledo, US). The average fat (14.8±4.1 kg/100 kg) and moisture contents (70.5 kg/100 kg) were measured according to AOAC (1997) standard procedures 991.36 and 950.46, respectively. Analytical determinations were carried out in the cushion zone of the ham where ultrasonic experiments were performed. In raw hams, the thickness of the cushion zone was 11.5±0.4 cm and maximum width of raw hams reached 31.6±1.3 cm. The analysis of salting process focused on the cushion zone of the ham, which is the most critical one in terms of salting since it is the thickest part.

All the hams were dry-salted by covering the piece with 15kg of coarse salt (NaCl moisturized at 10% w/w) at 2±1ºC in a cold chamber (AEC330r, Infrico, Spain) in which relative humidity ranged between 80-85%. Raw hams and coarse salt were previously stored for 24h at 2ºC for the purpose of tempering. One ham was used for each salting time considered (4, 10, 11, 14, 16 and 30 days).

2.2 ULTRASOUND EXPERIMENTAL SET-UP

The ultrasonic experimental set-up was designed to develop a reliable, simple and robust methodology aiming to be further applied at industrial level. Ultrasonic measurements were during the dry-salting experiments carried out on hams. The ultrasonic exprimental set-up used (Figure 1) consisted of two narrow-band piezoelectric transducers of 1MHz and 0.5'' crystal diameter (T1 and T2, A303S model, Panametrics, Waltham, MA, USA), a pulser-receiver instrument (5077PR, Panametrics, Waltham, MA, USA), a digital input/output USB 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. Signal digitalization was started by the output trigger signal of the pulser-receiver instrument using an independent channel of the digitizer. The digital input/output device was used as a multiplexer of the excitation pulse and the received signal, so a single pulser/receiver unit was used.

For the purposes of taking the ultrasonic measurements, the two transducers (T1 and T2, Figure 1) were firstly embedded in a layer of 5kg of salt placed in the bottom of a plastic container (120x35x20cm). Secondly, the cushion part of the ham was placed on the layer in direct contact with the transducers' surface and the salt. Coupling materials were not used to improve the contact between the transducer and sample surface. Next, two temperature sensors (type-K thermocouples) were placed both in the salt and on the surface of the sample; and the remaining 10 kg of salt were added until the sample was entirely covered (Figure 1). The ultrasonic measurements were taken by pulse-echo mode at intervals of 5min in the cushion zone of the ham. The signal received from each transducer was digitized (25kpoints at 100Msamples/s, 10% pre-trigger points) and stored for further signal analysis.

2.3 METHODS FOR SIGNAL ANALYSIS

Different signal analysis methods (energy threshold, cross-correlation and phase spectrum) were applied in order to determine which was the most appropriate as a means of calculating the time of flight (TOF) (Povey and Mason, 1998) and so monitoring the ham salting.

In the energy threshold method (ETM), the dead zone (zone A, Figure 2) was discarded, this can be defined as the portion of signal corresponding with the transducer's own vibration and also reflections of the ultrasonic wave on interfaces close to the transducer-ham surface interface. Thus, the TOF was calculated when the wavefront arrived at the transducer (zone B, Figure 2), when the amplitude of the received signal exceeded the established threshold (0.1V) (Sarabia et al., 2013), which was sufficiently above the existing background noise level. This method assumes no signal distortion and a good sound-to-noise ratio (SNR) and has been applied to compute ultrasonic velocity in different meat products (Nowak and Markowsky et al., 2015 and 2016). Novak and Markowsky (2013) reported that similar results are obtained by identifying the maximum amplitude of the ultrasonic signal instead of the wavefront arrival.

Additionally, the cross-correlation method (CCM) (Leemans and Dastain, 2009) was considered. This method calculates the time of flight variation between two similar signals (ΔTOF_{2s}) as follows.

Given two waveforms f(t) and g(t), the cross-correlation (f*g)(t) is defined as:

$$(f * g)(t) = \int_{-\infty}^{\infty} f^*(s)\, g(t+s)ds \qquad (1)$$

where $f^*(s)$ denotes the complex conjugate of $f(s)$. The discrete form of the cross-correlation function, which is the one used in this paper is:

$$(f * g)(t) = \frac{1}{N}\sum_{n=0}^{N-1-k} f_n \, g_{n+k} \qquad (2)$$

where f_n, g_n are the two N-length discrete waveforms

The maximum value of (f*g)(t) determines when the two signals are overlapped and the position of the maximum in the array permitted the calculation of the ΔTOF_{2s}. In order to identify the maximum, the adoption of appropriate interpolation methods may improve the accuracy of the estimation (Svilainis, 2016).

Several approaches were considered to calculate the change in the time of flight from the beginning to the end of the salting experiment (ΔTOF). Firstly, the ΔTOF_{2s} was calculated using the CCM between the initial or reference signal (0h salting, RS) and the signal at the salting time considered (CCM-RS). Secondly, the CCM was performed for each pair of consecutive signals (5min apart) and then the ΔTOF for each salting time was calculated by the addition of the ΔTOF_{2s} between consecutive signals from the beginning of salting until the salting time considered. In this case, two approaches were followed: CCM was performed on the raw signals (no interpolation; CCM-CS n=0) and on interpolated signals (3 samples' interpolation; CCM-CS n=3). Finally, the same procedure was followed, but performing the CCM between the signals that are 1h apart (CCM-NCS). CCM algorithms were programmed in Labview™ 2018 (National Instruments, Austin, TX, USA) using the available functions for cross correlation and interpolation and the signals were standardized to the maximum amplitude. The correction of DC components in the signal was not necessary.

Finally, a phase spectrum method (PSM), which is an adaptation of the method proposed by Sachse and Pao (1978), was used to calculate the ΔTOF_{2s} between consecutive signals each one 5min apart. For that purpose, a square time window of fixed location and length (from 13k to 20k points) was used in every case to select the portion of the B-Scan where the echo coming from the ham back surface appears. The FFT of the signal is a complex number, whose module and phase give rise to the module spectrum and phase spectrum, respectively. The square of the module spectrum provides the energy of the signal at each frequency and the phase spectrum (ϕ) is given by (Koksel et al., 2014 and 2017):

$$\phi = \omega(t - t_0) \qquad (3)$$

where ω is a vector containing the discretized angular frequency values within the signal frequency band, t the time and t_0 the time origin which is, normally, set at the centre of the time window. From the module spectrum of the signal received at the beginning of the salting process, the system frequency band (including the frequency band of pulser, transducers, receiver, analog-to-digital converter and the contribution of the attenuation over the travelled distance), which is defined as the frequency band where the module spectrum is above the threshold given by a peak value of - 6dB (frequency window 0.85-1.04 MHz), was obtained. Given any two signals, the time delay can be calculated from the difference between the phase spectrum of both, within the system frequency band:

$$\phi_1(\omega) = \omega(t_1 - t_0),\ \ \phi_2(\omega) = \omega(t_2 - t_0) \qquad (4)$$

$$\Delta\phi(\omega) = \ \phi_1(\omega) - \ \phi_2(\omega) = \omega(t_1 - t_2) = \Delta t(\omega) = \Delta TOF(\omega) \qquad (5)$$

Therefore, in this case, a time delay is obtained for every frequency value within the system frequency band, which is why this is the only method able to cope with TOF estimations of wideband signals in the presence of dispersion. In order to obtain a single ΔTOF_{2s} estimation, instead of $\Delta TOF(\omega)$, which can be compared with the results obtained by the other procedures, the average value of $\Delta TOF(\omega)$ was assessed:

$$\Delta TOF_{2s} = \frac{\sum_{i=1}^{i=N}(\Delta TOF(\omega_i))}{N} \qquad (6)$$

Where ω_i i=1,N is the angular frequency within the system 6-dB frequency band.

Finally, the ΔTOF for each salting time was calculated by the addition of the ΔTOF_{2s} between consecutive signals from the beginning of salting until the salting time considered.

As well as for CCM, specific software programmed in LABVIEW™ 2015 (National Instruments, Austin, TX, USA) was used in PSM.

2.4 CHEMICAL ANALYSIS

The salt and water content were determined in the salted ham. For that purpose, two cylindrical salted samples (204±21g), which included the ultrasonic measurement zones, were taken by using a cylindrical cutter (5cm in diameter). Each cylindrical salted sample was ground and homogenized before analytical determinations. The water content was determined by oven drying to constant weight at 102ºC following the standard AOAC method, 950.46 (1997). The salt content was analyzed after sample homogenization (1g for fresh samples and 0.5g for salted samples) in 100mL of distilled water using an ULTRATURRAX (T25, IKA Labortechnik, Germany) at 9500rpm for 5min. Supernatant was filtered through membrane filters (45µm) and a 500µl aliquot sample was taken and titrated in Chloride Analyzer equipment (Chloride Meter 926L, Ciba Corning, U.K.) (Carcel et al., 2007). All the analyses were performed in triplicate. As the ham's integrity cannot be altered before salting, the initial average values of salt and water content were obtained from 6 hams of the same breed purchased from the same supplier. The final salt gain (ΔX_S) and water loss (ΔX_W) were also calculated for each salting time.

3. RESULTS AND DISCUSSION

3.1 ULTRASONIC ONLINE MONITORING OF THE HAM DRY SALTING PROCESS

As an example of the ultrasonic signals obtained, Figure 2 shows the first and last signals of one transducer (T1) in the 11 days of the ham salting trial. On the one hand, the portion of signal received in zone A (dead zone) represents the transducer's own vibration after emission and also reflections of the ultrasonic wave on interfaces close to the transducer surface (for example, from the subcutaneous fat/lean meat interface). On the other hand, zone B includes the reflection of the wave on the meat/salt interface, and thus shows the arrival of the wavefront when it has crossed the whole sample twice. As can be observed in Figure 2, the TOF was shortened from the first to the last day of salting (11 days). This same behaviour, a decrease in the TOF as the salting time progressed, was observed in every experiment carried out (4, 10, 14, 16 and 30 days). In the example of Figure 2, the ultrasonic signal at 11 days was displaced 20.9µs (calculated by the ETM) to the left compared to the signal on day 0, which illustrates the shortening of the TOF and, consequently, the increase in velocity. This behaviour can be explained by the fact that an increase in ultrasonic velocity will occur as a result of either any increase in the material's elastic stiffness or any decrease in the density or both. Since density increases during salting (salt gain and water loss), the increase in velocity could be attributed to the meat's greater elastic stiffness due to the sample contracting and hardening during salting as a consequence of the salt gain and water loss. In this regard, De Prados et al. (2015, 2016) observed an ultrasonic velocity increase in brined and dry-salted *Biceps femoris* and *Longissimus dorsi* muscles, respectively. It has to be mentioned that the TOF reduction was not only ascribed to the increase of the ultrasonic velocity, but also to the ham's thickness reduction, due to the shrinkage caused by the coupled salting-dehydration. Thus, the shortening of the TOF observed during salting could be used to monitor the progress of the salting process and to determine the salt content modification. For that purpose, an accurate calculation of the TOF is required.

3.2 TIME OF FLIGHT CALCULATION BY USING THE ENERGY THRESHOLD METHOD

Figure 3 depicts the TOF evolution of the ultrasonic signals corresponding to the two measurement points (T1 and T2) in the hams dry-salted for 10 and 16 days at 2ºC. Similar behaviour was observed for the two measurement points in the remaining salting times tested (data not shown). Figure 3 shows a downward trend of the TOF during the salting time, which, as previously mentioned, is related to the increase in the meat's solid content and, consequently, to an increase in elastic stiffness. In addition, the initial TOF (TOF_0) and the TOF evolution were different for both T1 and T2, which could be explained by considering the highly heterogeneous nature of the piece of ham from both compositional and structural points of view. Moreover, the different TOF_0 value could also be due to the differences in the ham's thickness.

Taking into account the ultrasonic signal displacement shown in Figure 2 and the compositional changes that take place during salting, a progressive decrease in the TOF during salting could be expected, coupled to the salt gain shown in Table 1. However, every salting experiment demonstrated several abrupt changes in the TOF evolution (Figure 3). These abrupt changes might not be related to compositional variations, as they occurred randomly and appeared upward and downward (Figure 3). In order to find the origin of this fact, the ultrasonic signals taken during salting were analysed. As an example, the ultrasonic signals (non-noise) (zone B, Figure 2) in T2, after 0, 127, 254 and 380h of the ham dry salted for 16 days were plotted in Figure 4.

As observed in Figure 4, the amplitude of the ultrasonic signals fluctuated during salting. The salt gain, water loss, sample contraction and the chemical and structural changes in the protein matrix during salting might be the reason for the amplitude fluctuation in the ultrasonic signal shown in Figure 4. In addition, the transducer-ham contact also may affect the amplitude of the ultrasonic signal. The fluctuations in the signal amplitude mean that the energy threshold method detects the wavefront's arrival at a particular position and, when the signal amplitude decreases or increases over time, the new peak crossing the threshold can be randomly displaced backwards or forwards regardless of the salt gained. signal Different energy thresholds (0.05-1.2V) were evaluated in order to study their influence on the TOF assessment. However, the abrupt changes in the TOF evolution appeared in every case.

When an ultrasonic signal presents a high SNR, despite the amplitude fluctuations, the peak corresponding to the arrival wavefront will always exceed the established energy threshold [13], and thus, the ETM will always locate the arrival wavefront from the same peak, avoiding fluctuations in the TOF calculation. However, the ultrasonic waves found in this study were attenuated after crossing the ham twice, decreasing the SNR, and therefore, the fluctuations in the signal amplitude led to miscalculations of the TOF, resulting in the observed fluctuations in the ∆TOF calculated by the ETM (Figure 3). Therefore, the ETM, which was successfully used by De Prados et al. (2016) for the purposes of monitoring the salting process in LD and BF muscles, was not suitable for application in the low SNR ultrasonic signals obtained during salting of whole hams.

3.3 ΔTOF CALCULATION BY USING THE CROSS-CORRELATION METHOD

An alternative to the ETM for analyzing the ultrasonic signals and calculating the TOF is that of the cross-correlation method (CCM). Leemans et al. (2009) used this method to calculate the TOF and detect foreign bodies in cheese. Similarly, Pallav et al. (2009) analyzed ultrasonic signals using the cross-correlation method to determine the TOF and detect foreign bodies and additives in food. This method compares two ultrasonic signals and calculates the time of flight variation (ΔTOF) between them.

3.3.1 ΔTOF CALCULATION USING THE CROSS-CORRELATION METHOD BETWEEN THE INITIAL ULTRASONIC SIGNAL AND THE REMAINING SIGNALS

In the present section, the ΔTOF_{2s} was calculated between the initial or reference ultrasonic signal (0h-RS) and the remaining signals measured during the dry-salting of hams (CCM-RS). Figure 5 shows the ΔTOF decrease in hams salted for 14 and 30 days at 2ºC. Although the cross-correlation method was not conditioned by the amplitude fluctuations of the wavefront's arrival, abrupt changes in the ΔTOF evolution were also found. The abrupt changes observed in Figure 5 could be explained by the change in the shape of the ultrasonic signal (signal distortion) during salting compared to the reference signal. As an example, Figure 6 shows both the RS and those obtained at 0, 200, 400 and 725h in the ham dry salted for 30 days at 2ºC (T2). In Figure 6, only the zone of the ultrasonic signal corresponding to the reflection of the wave in the sample/salt interface is represented (non-noise, zone B, Figure 2). As can be appreciated, the pulse presents a clear distortion that can be ascribed to wave dispersion (variation of propagation properties with frequency), scattering and multipath propagation. As previously mentioned, this pulse distortion could be linked to the compositional changes (salt gain and water loss), the reduction in thickness and the protein denaturation suffered by the ham during salting.

As an example, the abrupt change observed in Figure 5 for the 14 days' salting trial was analyzed. Figure 7A shows the overlap of RS and the signal before the abrupt change (SBA) obtained by the cross-correlation method (where the maximum of the cross-correlation array is found), while Figure 7B shows the overlap of RS and the signal after the abrupt change (SAA). The maximum value obtained in the cross-correlation between RS-SBA (91.23μs) was quite different to the one obtained in the cross-correlation between RS-SAA (94.04μs), which gives rise to the abrupt change observed in Figure 5.

According to the results obtained in this section, the compositional and structural changes that take place in the meat during salting involve a distortion of the ultrasonic signal and, thereafter, a miscalculation of the ΔTOF when CCM-RS is applied.

3.3.2 ΔTOF CALCULATION USING THE CROSS-CORRELATION METHOD BETWEEN CONSECUTIVE SIGNALS

From the analysis of the ultrasonic signals, it was observed that the distortion in the ultrasonic signal did not happen abruptly between consecutive signals (5min) but progressively during salting. Thus, in order to solve the problems found in the calculation of the ΔTOF using the CCM-RS discussed in the previous section, the cross-correlation method was applied between consecutive (each one 5 min apart) ultrasonic signals (CCM-CS; n=0, no interpolation). Therefore, the ΔTOF_{2s} between each pair of signals was calculated and the relationship between the ΔTOF and the salting time was represented by using the sum of the estimated ΔTOF_{2s}.

Figure 8 shows the ΔTOF evolution, calculated using the CCM-CS n=0, in hams dry salted for 11 and 16 days. As can be observed, the ΔTOF decreased during salting and no abrupt change was found. However, in the ΔTOF of T1 during the 16 days' salting, an anomalous decrease trend was observed (Figure 8), which leads to a very different ΔTOF compared to T2. Initially, a normal decrease was found; however, at around 100 h the rate of decrease fell abruptly. This anomalous behaviour was also observed in T1 during the 30 days' salting (data not shown). Overall, the TOF estimation error is random, positive or negative; thus, it does not accumulate. However, for signals with very small differences between each other (only 5 min apart), i.e. TOF differences in the order of the discretization time in the digitized waveform, a bias in the CCM-CS algorithm that calculates the maximum in the correlation could appear, and lead to an accumulated error.

Thus, in order to minimize the possible error accumulation and to improve the resolution of the CCM for signals that are only very slightly displaced between each other, the sampling frequency could be increased, applying an interpolation method to the acquired signals. A different number of interpolation points (n from 1 to 5) were considered in order to study their influence on the ΔTOF calculation (data not shown), n=3 turning out to be the most appropriate one. Using the CCM-CS n=3, a progressive decrease in ΔTOF was observed for every salting time studied (4, 10, 11, 14, 16 and 30 days). Moreover, the differences between the final average ΔTOF at measurement points T1 and T2 in the 16 and 30 days' salting experiments were drastically reduced when using the CCM-CS n=3 ($\Delta TOF_{avgT1\text{-}T2}$= -11.2±1.3µs for 16 days and -17.3±0.1µs for 30 days) compared to the CCM-CS n=0 ($\Delta TOF_{avgT1\text{-}T2}$= -9.3±2.2µs for 16 days and -13.2±1.1µs for 30 days) (Table 1). Non-significant ($p>0.05$) differences were observed between the final average ΔTOF values using the CCM-CS n=3 and the CCM-CS n=0 for the remaining salting times (4, 10, 11 and 14 days) (Table 1). Therefore, the CCM-CS n=3 might reduce the accumulated error and allow the ΔTOF calculation throughout the whole salting period. As the CCM-CS n=3 involves an increase in the signal processing time, complementary methodologies will be explored in this paper.

3.3.3 ΔTOF CALCULATION USING THE CROSS-CORRELATION METHOD BETWEEN NON-CONSECUTIVE ULTRASONIC SIGNALS

In order to reduce the computing requirements and the analysis time, the cross correlation method between non-consecutive ultrasonic signals (each one 1 h apart) (CCM-NCS) was evaluated. The objective is to increase the difference between the TOF of the two signals while keeping distortion as small as possible. Figure 9 shows the evolution of the ΔTOF, calculated using the CCM-NCS, in hams dry salted for 11 and 16 days (where the anomalous changes in the evolution of the ΔTOF appeared when using the CCM-CS n=0). The evolution of the ΔTOF calculated using CCM-NCS behaved similarly to the ΔTOF calculated using CCM-CS n=3 for every salting time studied (4, 10, 11, 14, 16 and 30 days). Consequently, when using the CCM-NCS, the final average ΔTOF were not significantly ($p>0.05$) different from those obtained using the CCM-CS n=3 (Table 1), except for the ham salted for 16 days. Therefore, the

CCM-NCS could be an alternative method with which to calculate the ΔTOF and to monitor the salting process of hams, reducing the signal analysis processing time and avoiding the miscalculations found when the ETM and the CCM-CS n=0 were used. The accuracy of this method reflects the fact that time interval wave acquisition could be extended from 5 min to 1 hour without affecting the ΔTOF assessment.

3.4 ΔTOF CALCULATION USING THE PHASE SPECTRUM METHOD

Another alternative methodology used in the present study as a means of calculating the ΔTOF was the phase spectrum method (PSM). When calculating the ΔTOF by using the PSM, a similar trend was observed (Figure 10) in the T1 and T2 ΔTOF evolution for the entire salting experiment. Overall, non-significant ($p>0.05$) differences were observed in the final average ΔTOF values obtained using the PSM, the CCM-NCS and the CMM-CS n=3 (Table 1). Therefore, any of these signal analysis methods could be used to calculate ΔTOF and monitor the ham salting process.

3.5 PREDICTION OF THE SALT GAIN THROUGH THE ΔTOF

As mentioned in section 3.1, the shortening of the TOF found during ham salting was influenced by the compositional changes (salt gain and water loss) as well as by the sample contraction and structural changes that take place during salting. When the final ΔTOF value was related with the salt gain (ΔX_S) (Table 1), a great variability was found (data no-shown). In order to take into account the initial sample thickness, which can affect the relationship between the change in the time of flight and the salt gain, the TOF_0 was considered for the estimation of the salt gain. Therefore, the relationship between the salt gain and the $\Delta TOF \cdot TOF_0$ was studied (Table 2). Since in previous sections, the CMM-CS n=3, CCM-NCS and PSM have been shown to be the most convenient ones with which to monitor the salting process, they were the methods chosen to calculate the ΔTOF in every experiment. Using any of these methods to calculate the ΔTOF, a significant ($p<0.05$) relationship between the ΔX_S and the

$\Delta TOF \cdot TOF_0$ was found, showing a high correlation coefficient (R^2=0.83 for the CMM-CS n=3, R^2= 0.93 for the CCM-NCS and R^2=0.90 for the PSM, Table 2). Consequently, the use of ultrasonic pulse-echo measurements, together with any of the three methods evaluated in this work (CMM-CS n=3, CCM-NCS and PSM), could be considered a reliable and effective technique for calculating the ΔTOF and predicting the salt gain during ham dry-salting.

Several studies have shown the relationship between the ultrasonic velocity and the solid content in foodstuffs. In this regard, Valente et al. (2016) showed that the ultrasonic velocity increased along with a rise in the solid content during mango ripening. De Prados et al. (2015; 2016) reported that the ultrasonic velocity rose in pork meat (*Biceps femoris* and *Longissimus dorsi*) during salting. The ultrasonic velocity measurements require the sample's thickness be measured by means of some electronic gage, which could be considered a limitation in an industrial environment where hams are placed in a pile during dry-salting. By contrast, the pulse-echo TOF measurement presents a twofold advantage: the sample thickness does not need measuring and the ultrasonic transducers are only in contact with one of the ham surfaces, making the ultrasonic industrial application easier.

4. CONCLUSIONS

The time of flight was progressively shortened during the ham dry-salting process. The energy threshold and the cross-correlation method between the initial signal and the remaining signals measured during the dry salting of ham were not able to provide reliable results for computing the the variation of the time of flight (ΔTOF). The lack of accuracy was linked to the the change in the signal amplitude, the limited SNR and the signal pulse distortion. The cross-correlation method between consecutive signals (5min apart) with interpolation n=3, between non-consecutive signals (1h apart) and the phase spectrum method provided reliable results, being the most appropriate methods with which to calculate the ΔTOF and monitor the ham salting process. By using any of these methods, the ΔTOF weighted by the initial TOF_0 was significantly ($p<0.05$) correlated to the salt gain in the hams.

Nowadays, quality control in pork ham industry lacks of nondestructive techniques to monitor the dry-salting process, which presents a high variability and complexity due to intrinsic (size, shape, fat content, pH …) and extrinsic properties (air temperature and relative humidity, sample location in the pile layer …) to the ham. Moreover, the high value of the ham pieces hinders the use of conventional destructive techniques to measure the salt content, due to their impact on the process cost. Thereby, the process control of ham dry-salting is largely conditioned by the background and empirical knowledge of industry technicians. Therefore, the industrial application of the ultrasonic experimental technique used in this work, based on laying the ham pieces over ultrasonic transducers and taking pulse-echo measurements, would contribute to a better process and product control. This strategy could be considered as feasible, robust and simple and emerges as a non-destructive and relatively affordable tool to monitor the dry-salting process of hams. The methodologies developed in this work would contribute to an accurate assessment of the variation of time of flight from the ultrasonic signal acquired during salting, which is critical for the estimation of the evolution of the salt gain. Future industrial implementation should address the analysis of the number of samples to be monitored, as well as the development of durable and low-cost electronics for signal generation, acquisition and processing.

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FIGURE CAPTIONS

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

Figure 2. Received signal (B-Scan) in pulse-echo mode for a ham sample at two different stages of salting (0 and 11 days). A: Dead zone. B: Echo coming from ham back surface.

Figure 3. Time of flight (TOF) evolution in the hams dry salted for 10 days (left) and 16 days (right) at 2ºC. TOF calculated using the Energy Threshold Method (ETM). T1 and T2 refer to the two measurement points/transducers.

Figure 4. Change in the amplitude of the ultrasonic signals of T2 in the ham dry-salted for 16 days at 2ºC (0, 127, 254 and 380h).

Figure 5. Time of flight variation (ΔTOF) in the ham dry salted for 14 days (left) and 30 days (right) at 2ºC. ΔTOF calculated using the CCM-RS. T1 and T2 refer to the two measurement points/transducers used. The arrow shows an abrupt change in the ΔTOF evolution. SBA and SAA refer to the signal before and after the abrupt change, respectively.

Figure 6. Signal of T2 in the ham dry-salted for 30 days at 2ºC at different salting times (0, 200, 400 and 725h). Figure 7. Overlapping between the initial signal (RS) and that before (SBA; A) and after (SAA; B) the abrupt change shown in Figure 5, considering the maximum value of the cross-correlation array (transducer T1 in the ham dry salted for 14 days at 2ºC).

Figure 7. Overlapping between the initial signal (RS) and that before (SBA; A) and after (SAA; B) the abrupt change shown in Figure 5, considering the maximum value of the cross-correlation array (transducer T1 in the ham dry salted for 14 days at 2ºC).

Figure 8. Evolution of time of flight variation (ΔTOF) in hams dry-salted for 11 days (left) and 16 days (right) at 2ºC. ΔTOF calculated using the CCM-CS n=0. T1 and T2 refer to the two measurement points/ transducers used.

Figure 9. Evolution of time of flight variation (ΔTOF) in the ham dry-salted for 11 days (left) and 16 days (right) at 2ºC. ΔTOF calculated using the CCM-NCS. T1 and T2 refer to the two measurement points/transducers.

Figure 10. Evolution of time of flight variation (ΔTOF) in hams dry-salted for 4 days (left) and 11 days (right) at 2ºC. ΔTOF calculated using the PSM. T1 and T2 refer to the two measurement points/transducers used.

Table 1. Final average time of flight variation (ΔTOF) calculated using the different signal analysis methods (CCM-CS n=0, CCM-CS n=3, CCM-NCS and PSM) and average salt gain (ΔX_S)

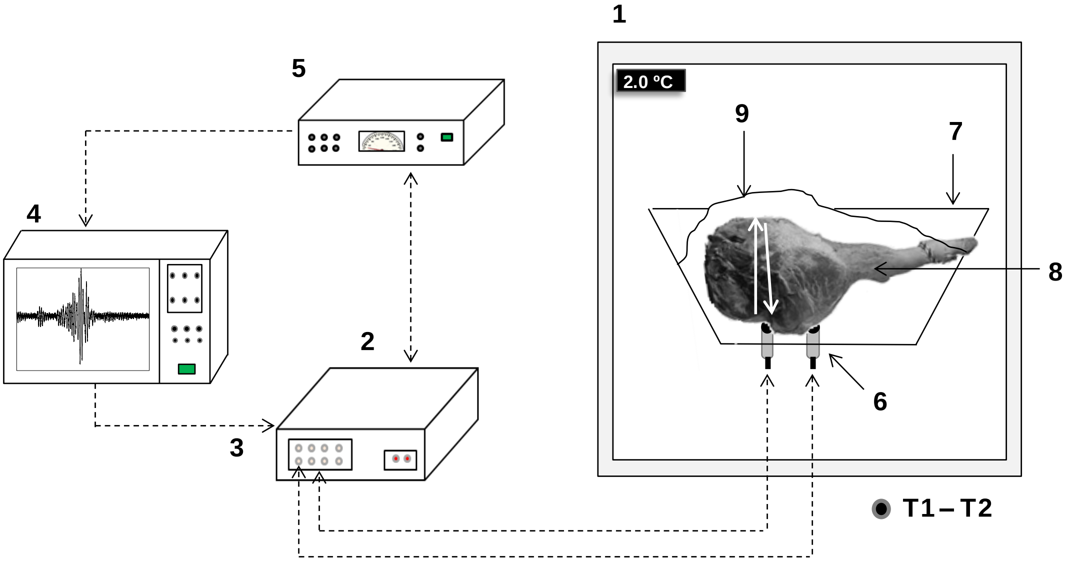
Days	CCM-CS n=0(μs)	CCM-CS n=3(μs)	CCM-NCS(μs)	PSM(μs)	ΔX_S (%w.b.)
4	-6.1±0.8^{a1}	-6.0±1.0^{i1}	-5.2±0.2^{e1}	-4.9±0.2^{m1}	0.71±0.04
10	-6.7±1.8^{ab2}	-7.3±1.2^{j2}	-7.6±1.5^{f2}	-7.0±0.9^{n2}	1.40±0.03
11	-10.6±1.7^{cd3}	-10.7±1.3^{k3}	-10.9±1.1^{g3}	-9.4±0.4^{o3}	2.55±0.21
14	-9.9±0.3^{bcd4}	-10.3±0.3^{k4}	-10.4±0.3^{g4}	-8.1±0.3^{no5}	2.11±0.38
16	-9.3±2.2^{abc6}	-11.2±1.3^{k6}	-14.1±0.1^{h7}	-13.2±0.2^{p67}	3.79±0.13
30	-13.2±1.1^{d9}	-17.3±0.1^{l10}	-17.3±0.2^{i10}	-16.1±0.2^{q10}	3.19±0.34

Mean values and standard deviations between T1 and T2. Different letters in the same column indicate significant ($p<0.05$) differences between salting times and different numbers in the same row significant ($p<0.05$) differences between the methods.
CCM-CS n=0 makes reference to the cross-correlation method between consecutive signals (every 5min) without interpolation, CCM-CS n=3 between consecutive signals (every 5min) with interpolation n=3 and CCM-NCS between non-consecutive signals (every 1h). PMS is the spectrum phase method between signals 5min apart.

Table 2. Linear regression models between the salt gain (ΔX_S) and the ultrasonic parameter ($\Delta TOF \cdot TOF_0$) calculated using the different signal analysis methods (CCM-CS n=3, CCM-NCS and PSM).

Analysis method	Equation	R^2
CCM-CS n=3	$\Delta Xs = -0.0032\ \Delta TOF \cdot TOF_0\ -0.039$	0.84
CCM-NCS	$\Delta Xs = -0.0028\ \Delta TOF \cdot TOF_0\ -0.129$	0.93
PSM	$\Delta Xs = -0.0029\ \Delta TOF \cdot TOF_0\ -0.032$	0.90

CCM-CS n=3 makes reference to the cross-correlation method between consecutive signals (each 5min) with interpolation n=3 and CCM-NCS between non-consecutive signals (each 1h). PMS is the spectrum phase method between signals separated 5min.



1. Cold Chamber 2. Multiplexer 3. USB 4. Data Acquisition Card-PC 5. Pulser-Receiver
6. Transducers 7. Container 8. Ham 9. Salt

FIGURE 1

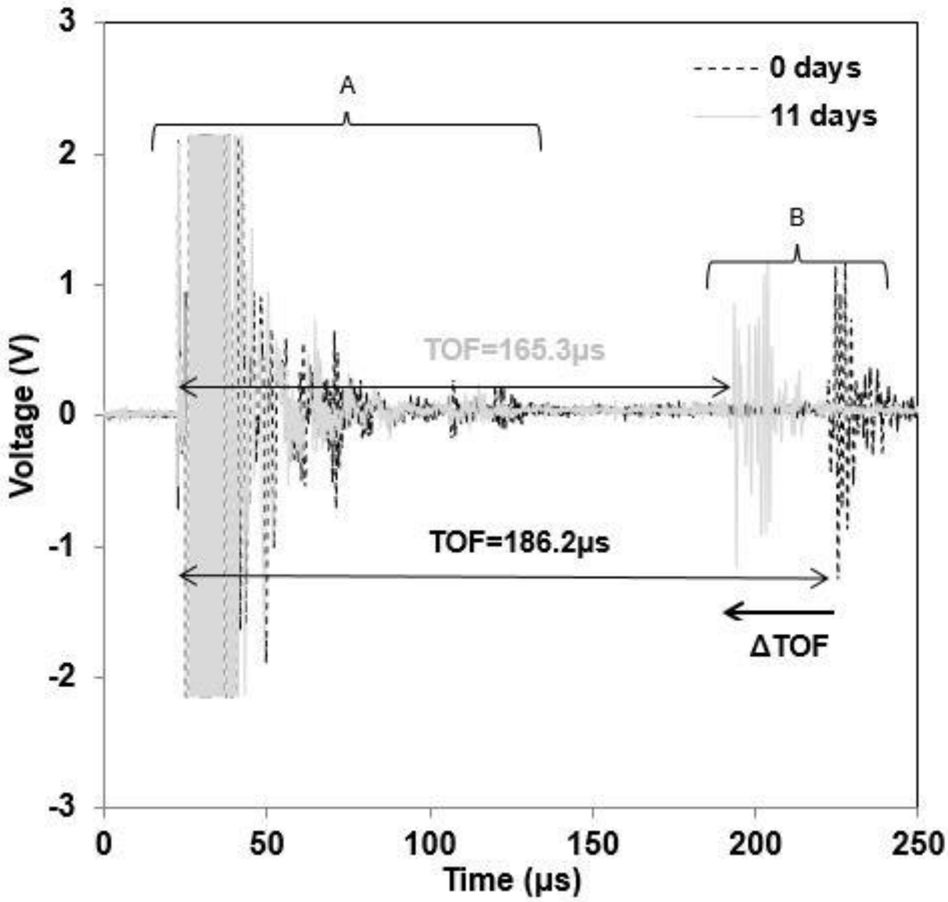
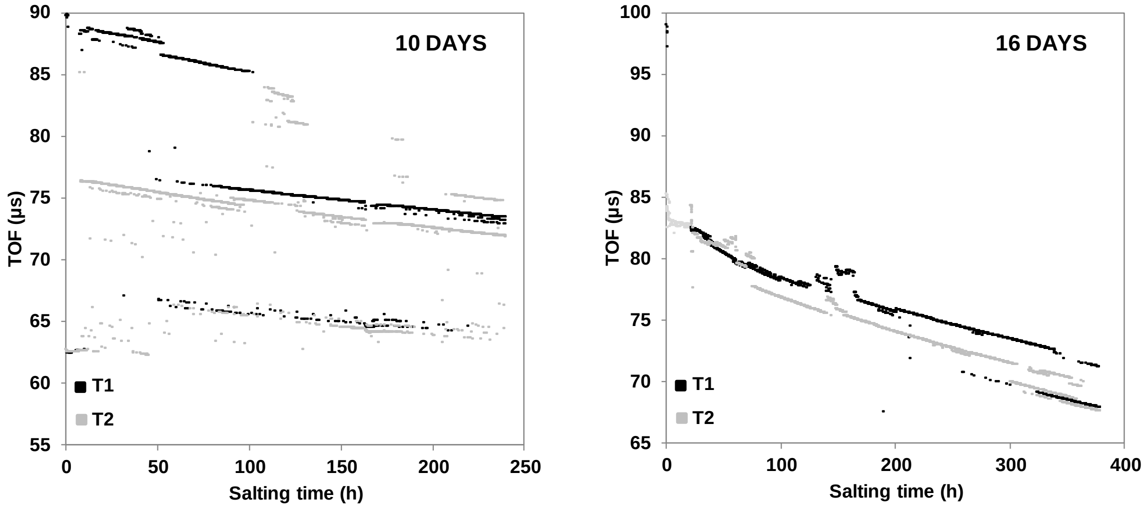


FIGURE 2



10 DAYS
TOF (µs)
Salting time (h)
T1
T2
16 DAYS
TOF (µs)
Salting time (h)
T1
T2

FIGURE 3

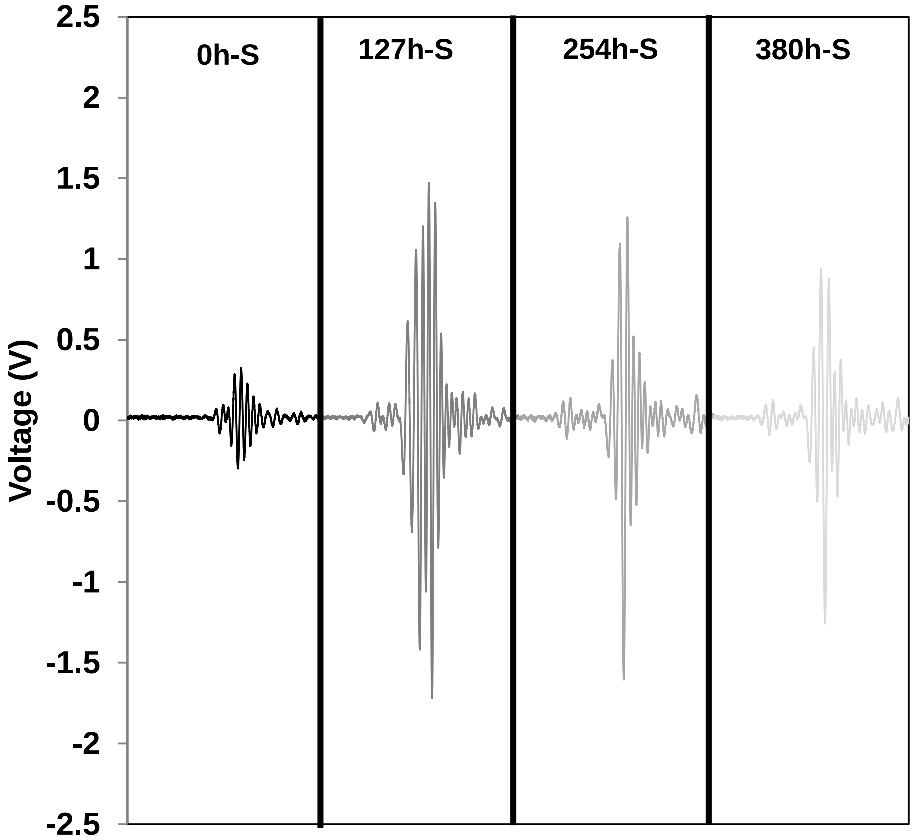


FIGURE 4

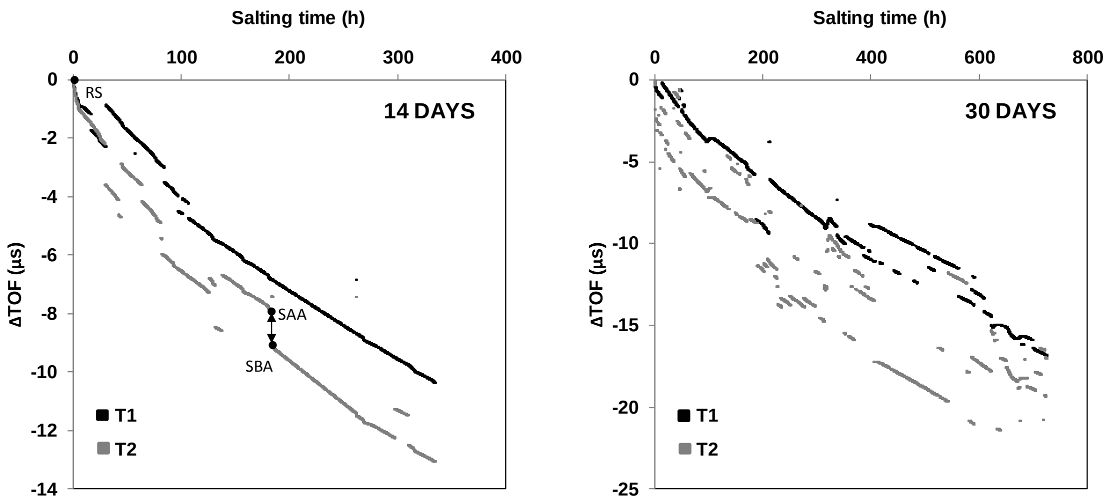

Salting time (h)
14 DAYS
ΔTOF (µs)
RS
SAA
SBA
T1
T2
Salting time (h)
30 DAYS
ΔTOF (µs)
T1
T2

FIGURE 5

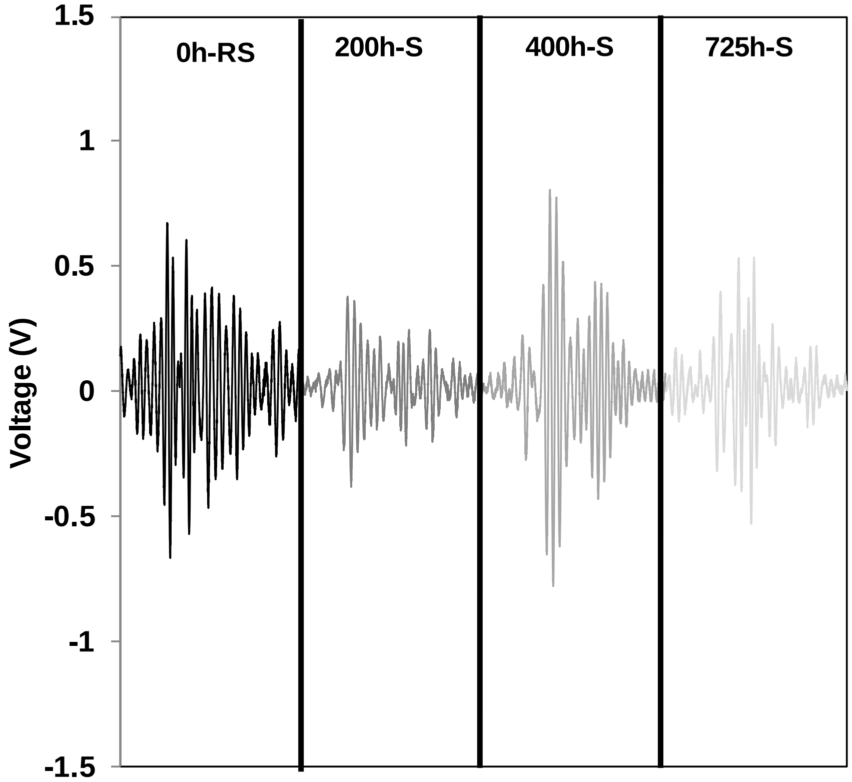


FIGURE 6

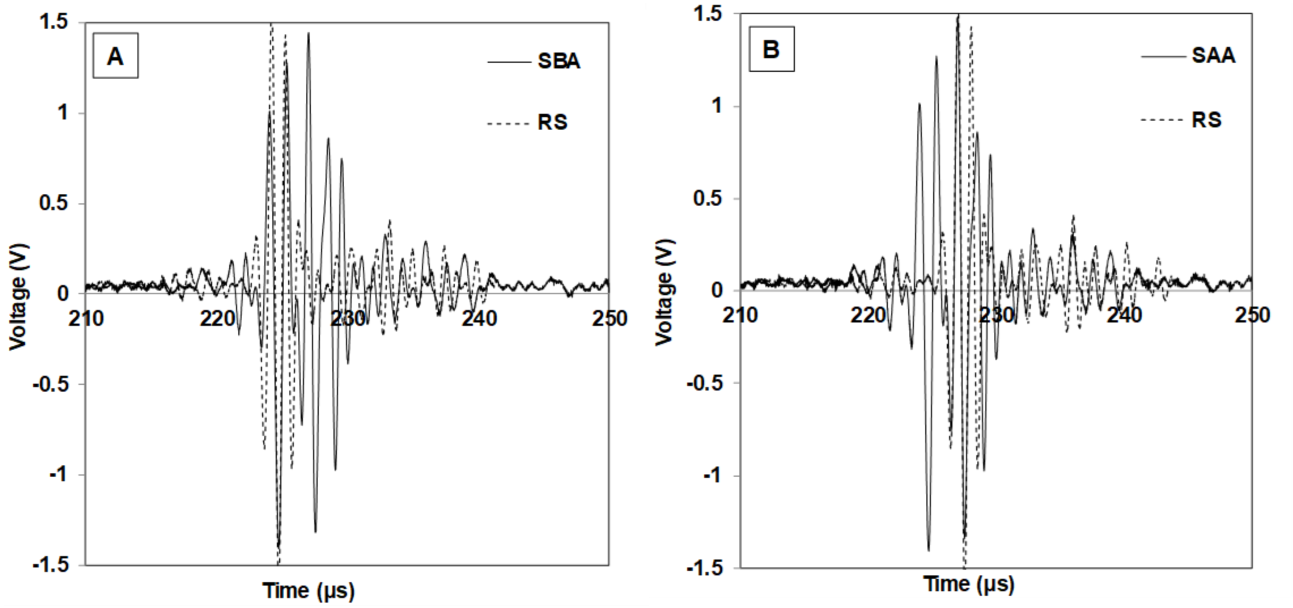


FIGURE 7

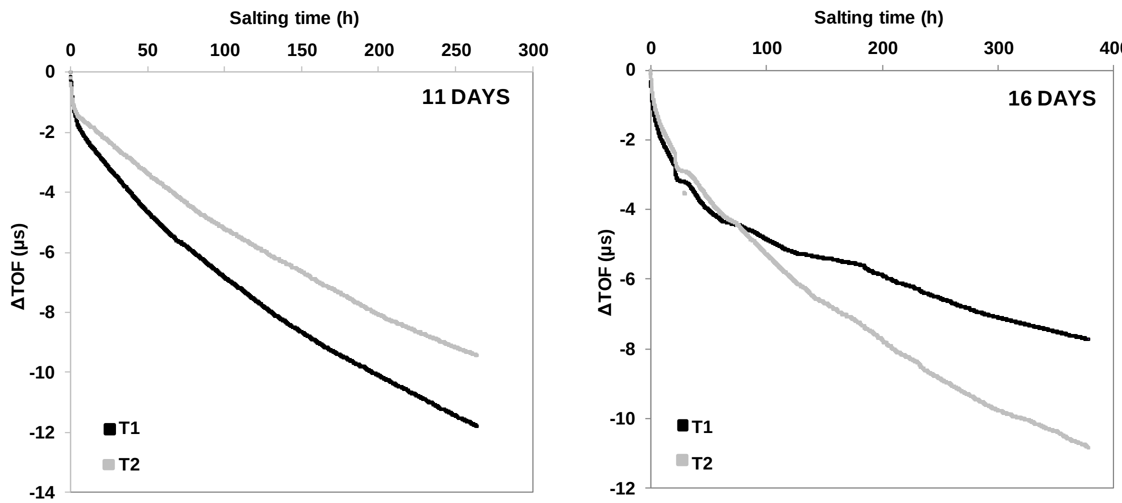

Salting time (h)
0
50
100
150
200
250
300
0
-2
-4
-6
-8
-10
-12
-14
ΔTOF (µs)
11 DAYS
T1
T2
Salting time (h)
0
100
200
300
400
0
-2
-4
-6
-8
-10
-12
ΔTOF (µs)
16 DAYS
T1
T2

FIGURE 8

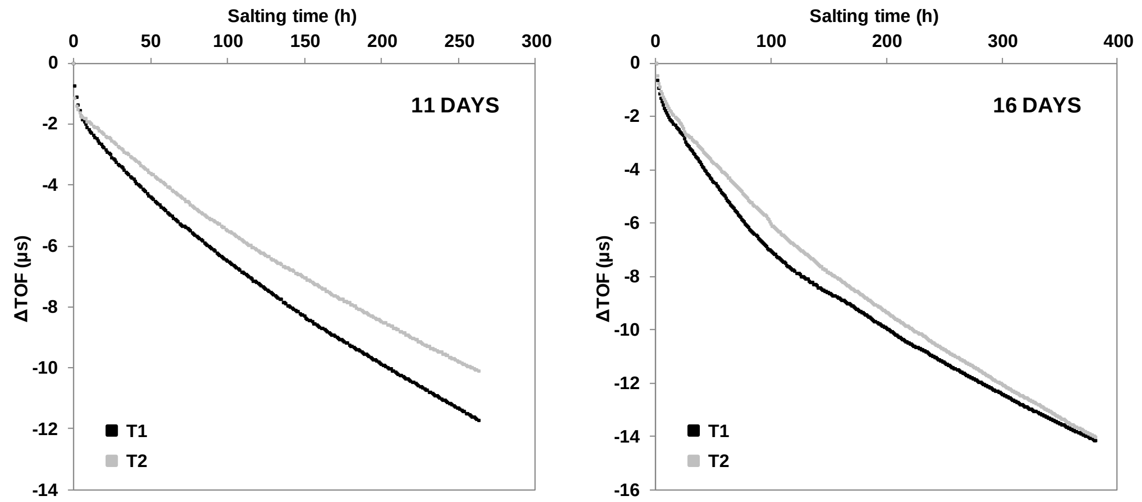

Salting time (h)
0
50
100
150
200
250
300
0
-2
-4
-6
-8
-10
-12
-14
ΔTOF (µs)
11 DAYS
T1
T2
Salting time (h)
0
100
200
300
400
0
-2
-4
-6
-8
-10
-12
-14
-16
ΔTOF (µs)
16 DAYS
T1
T2

FIGURE 9

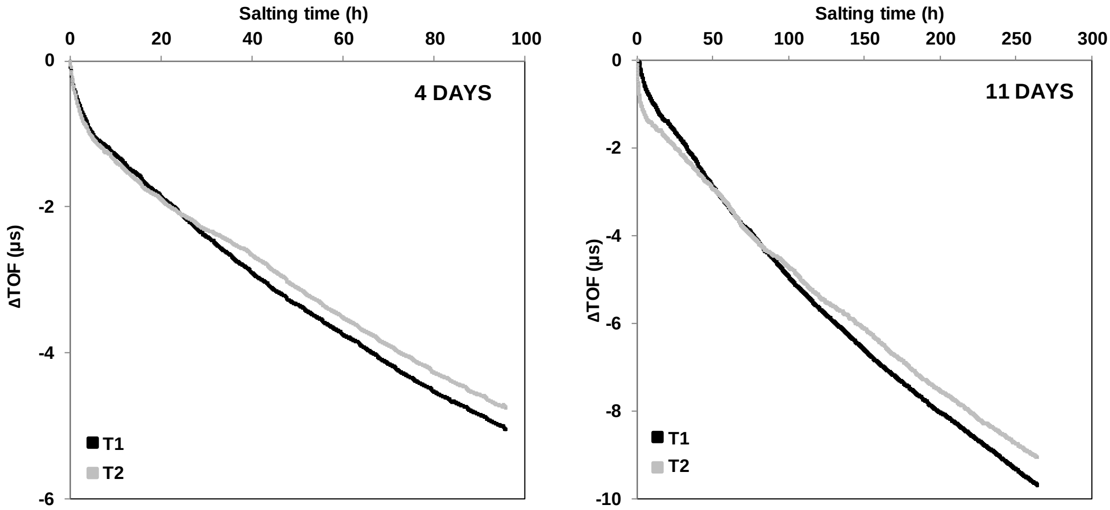

Salting time (h)
0
20
40
60
80
100
0
-2
-4
-6
ΔTOF (µs)
4 DAYS
T1
T2
Salting time (h)
0
50
100
150
200
250
300
0
-2
-4
-6
-8
-10
ΔTOF (µs)
11 DAYS
T1
T2

FIGURE 10