Experimental system for investigating processes of shock freezing of meat

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Abstract. Shock freezing affords reduction of freezing time, production of much smaller ice crystals that preserves the tissue structure and nutritional value of the product. The advantage of using ultrasonic bulk waves for monitoring the freezing process is the ability to trace the dynamics of changes in the properties of the meat tissue directly using ultrasound velocity, attenuation and the waveform shape. The purpose of the study was to create an experimental freezing system with follow up ultrasound propagation parameters in meat specimens during shock freezing. The samples were frozen by the electric pipe-freezing unit Frigo 2F-Zero of REMS with the temperature control by a calibrated thermocouple. Ultrasonic signals were recorded continuously by a custom-made ultrasonic setup in the through transmission mode using a pair of 2.5 MHz transducers mounted on a calliper-based probe. The following trends were observed during the freezing stages. Ultrasound velocity decreased in lean meat and increased in fatty meat with cooling at temperatures above zero. Rapid drop of ultrasonic signals at temperatures below zero associated with the beginning of the crystallization process and the presence of both liquid and crystal components. Ultrasonic signals increased as the samples were completely frozen and ultrasound velocity increased to values close to those in icy bodies. Differences in ultrasonic parameters on a time scale during freezing were revealed for lean and fatty meat samples demonstrating a possibility to investigate specific freezing regimes for different types of meat.

Key words: meat products, shock freezing, ultrasonic testing.

INTRODUCTION

Shock freezing (SF) has become the most popular method of freezing meat products, possessing a range of advantages. The SF process is based on the principle of micro-crystallization. Rapid temperature reduction to -25 °C over 0.5–2.0 hours reduces the freezing time of the product by 3–5 times compared to traditional freezing (James et al., 2015; Zhang et al., 2018). In this case, smaller ice crystals are formed, helping to preserve the tissue structure inherent in fresh products and preventing the development of certain types of bacteria (Kiani & Sun, 2011, Syamaladevi et al., 2012). The overall weight loss due to moisture evaporation during SF is 0.8%, significantly lower than in traditional slower technologies. These features ensure the preservation of the taste and

nutritional value of the product (Petzold & Aguilera, 2009¸ Zhang et al., 2018) The processing time in SF depends on the initial temperature of the raw material, the thickness of the frozen batch of products, and the power of the freezing chamber. In some studies, (Kiani & Sun, 2011; Purnell et al., 2017), it is noted that SF can lead to cell wall ruptures due to the formation of large ice crystals. This is explained by the fact that rapid cooling (to -30 °C and below) creates a significant temperature difference between the external and internal surfaces of the cell, leading to cell rupture. Therefore, methods and tools that allow for the control of process parameters and the diagnosis of changes in products are crucial, contributing to improvements in freezing technology. In recent years, numerous studies have been conducted on combined SF methods, involving additional influences such as ultrasound (Zhang et al., 2018) and electromagnetic fields (Purnell et al., 2017; Mironovs et al., 2023).

To understand the SF process and the possibility of its control, it is important to stabilize the temperature during periods of freezing and thawing. Equally important is the use of an effective and accessible means of monitoring changes in product properties in these processes. An urgent demand of customers for high-quality meat promotes automatic, real-time inspection and quality control in meat production. It stimulated development of objective, accurate and rapid non-destructive detection and evaluation techniques, including those based on artificial intelligence. The key technologies of non-destructive detection for meat quality involve ultrasonic technology, machine (computer) vision technology, near-infrared spectroscopy technology, hyperspectral technology, Raman spectra technology, and electronic nose/tongue (Shi et al., 2021). Among the control methods, the following are known: bioelectric spectroscopy and low-temperature microscopy (Martino & Zaritzky, 1988, Pliquett, 2010). It is of great interest to develop a fast and effective method for monitoring the freezing and thawing process, as well as changes occurring in products and materials. This is especially important when conducting research work. These parameters exhibit different characteristics in watery and fatty tissues (Egelandsdal et al., 2019). The temporal regimes of SF and the effects of external factors can also have varying impacts on meat depending on its fat content (Chen et al., 2017).

Although ultrasound velocity and attenuation in meat demonstrate correlations with physicochemical properties such as lipid content and moisture (Jiménez et al., 2023), their behaviour during meat freezing is less studied. Attempts to provide a scientific basis for ultrasonic assessment of meat freezing started decades ago comparing changes of ultrasound velocity with changes of temperature and calorimetric parameters (Miles & Cutting, 1974). Speed of sound or ultrasound velocity determined from time-of-flight measurements was used to measure progressive water crystallization during freezing of food (Sigfusson et al., 2004). Ice formation in plant tissues, particularly, xylem was assessed by ultrasound propagation velocity and attenuation measured with an ultrasonic emission analysis system that helped monitoring stress intensities and estimate physiological effects in situ (Charrier et al., 2014). Ultrasonic setup for monitoring thawing of food products operated at low kilohertz frequencies demonstrated significant delays of ultrasonic signals different for meat, fish and vegetables during thawing to a room temperature (Jha et al., 2023).

The study aimed to develop an operational device for SF and instrumentation for assessing temporal and temperature parameters. Ultrasonic testing was introduced as a monitoring tool of volumetric properties of meat specimens during freezing, where

changes of ultrasonic waveforms and derived changes of ultrasound velocity and intensity of ultrasonic responses were used as quantitative indications of the process. To demonstrate sensitivity to freezing peculiarities in different meat species, lean and fatty pork specimens were chosen as the observation objects.

MATERIALS AND METHODS

The study was conducted in the Laboratory of Powder materials of the Biomedical Engineering and Nanotechnologies Institute of the Riga Technical University. The experimental setup is depicted in Fig. 1. The freezing was provided by a REMS Frigo 2 F-Zero refrigeration system as the cold base with a low power consumption (generally not exceeding 500 watts) (REMS 131012 - Frigo 2 F-Zero Electric Pipe Freezing Set, 2024). Freezing was conducted in a capsule, housing the sample and placed between two freezing claws. Plastic, glass, and aluminum capsules were employed in the study. For monitoring the freezing process, emitting and receiving ultrasonic transducers were attached to the sample. The refrigeration system utilized R-290 refrigerant, high-purity propane, classified as A3 refrigerant, cooling the freezing claws to -30 °C. Temperature was recorded using a thermocouple connected to a DT890C+ multimeter. As temperature decreased, the time at which each one-degree temperature change occurred was recorded using a C++ CLI software application developed for the experiment.

The freezing of the material in the capsule was carried out until a temperature of -25 °C was reached, with particular emphasis on reaching -18 °C. Considering the temperature difference between the surface and the centre of the sample, the thermocouple was placed in the middle at a depth of 10 mm. To investigate the SH process, it was also necessary to maintain a constant temperature at a certain level of freezing. In this regard, a circuit was used to turn off and turn on the supply of refrigerant to the system.
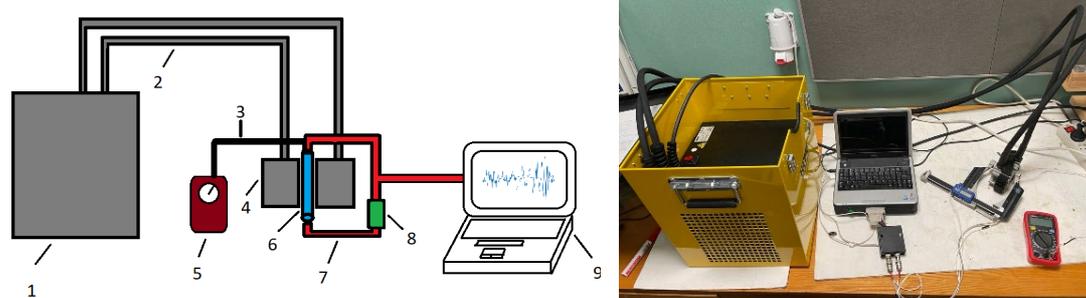

Figure 1. Diagram (a) and general view (b) of the setup for studying SF process: 1 – REMS F-Zero refrigeration system; 2 – pipes with refrigerant; 3 – thermocouple; 4 – freezing claws; 5 – multimeter; 6 – capsule with a meat sample inside; 7 – consoles with ultrasonic transducers; 8 – digital calliper; 9 – ultrasonic measurement unit with display.

Recording of ultrasonic signals during the cooling and freezing process was carried out in real time in the pitch-catch mode using a pair of ultrasonic transducers positioned opposite each other along the length of the meat specimen. A custom-made ultrasonic acquisition unit was used with the following specifications: a programmable waveform

140 V p-t-p for ultrasound excitation; input gain 40 dB; and 30 MHz 10-bit analogue-to-digital converter for signals digitizing. Ultrasonic transducers applied Ø10×1 mm PZT disks as emitting and receiving piezoelectric elements. The excitation waveform was a two-period sine pulse at the carrier frequency 2.5 MHz under a half-period sine envelope. The acoustic base was measured by a modified digital calliper with a resolution of 0.01 mm, on the consoles of which the ultrasonic transducers were stiffly mounted.

Fresh pork tissue meat in slightly chilled condition was obtained from a local butcher's shop. To demonstrate differences in SF processes for lean and fatty tissues, the specimens were taken from different locations of the loin department of the pig carcass. The examined samples had a volume of about 40 cm^3. Fat content in the lean samples was about 3–5% and, in the fatty samples, it was in the range 35–40%. Fat content was estimated from surface images of the samples applying image segmentation software, where fat and muscle tissue were separated by a RGB colour recognition algorithm.

RESULTS AND DISCUSSION

To evaluate SF process, the parameters such as freezing and thawing rates should be controlled. Three stages were identified during the freezing of water: (from +20 °C to -5 °C), the freezing rate was 0.25 °C min^{-1}, (from -5 °C to -15 °C), the freezing rate was 1.0 °C min^{-1}, (from -15 °C to -25 °C), the freezing rate was 3.0 °C min^{-1}. During water freezing, inertial cooling phenomena were observed. At lower temperature levels (-5 °C), the inertia was insignificant, approximately 0.5–0.6 °C min^{-1}. With further cooling, inertia increased, reaching 2–4 °C min^{-1}. This phenomenon is attributed to the design of the cooling claws containing the refrigerant.
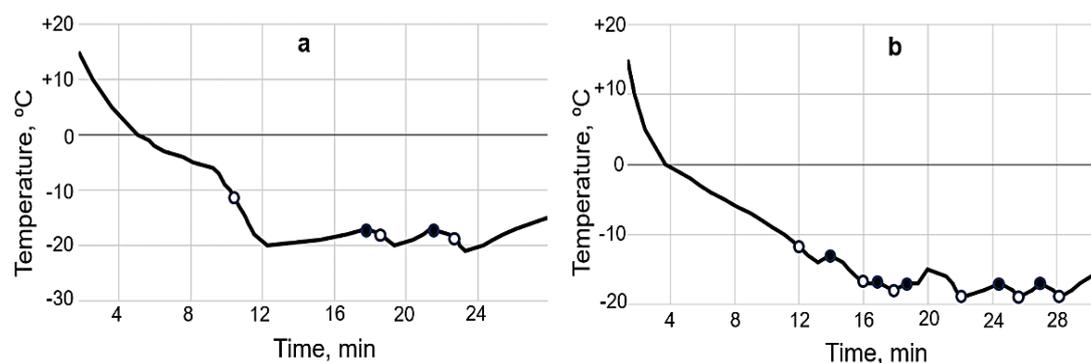

Figure 2. Temperature-time graphs for SF process in water (a) and fatty meat sample (b) showing temperature stabilization control in the range from -15 °C to 20 °C. White circles denote moments of switching off refrigeration system, dark circles – switching on.

Investigation of temperature effects with a feedback from ultrasonic monitoring were continued on lean and fatty pork meat samples. Transformations of ultrasonic signals in lean and fatty tissues during cooling and freezing are shown in Fig. 3. In the unfrozen state, when specimens were cooled to 0 °C, ultrasonic signals preserved its intact shape with a high signal-to-noise ratio and the prevalence of the main frequency component at 2.5 MHz in both types of meat. In the frozen state, the initial signals are

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distorted in both tissues with decreased amplitudes and spectral shifts towards the prevalence of low frequency components. These changes are especially pronounced in fatty meat, where even the low frequency signal is highly distinguishable on the noise background (Fig. 3, b). This occurs due to the difference in the acoustic impedances between fat and ice components.
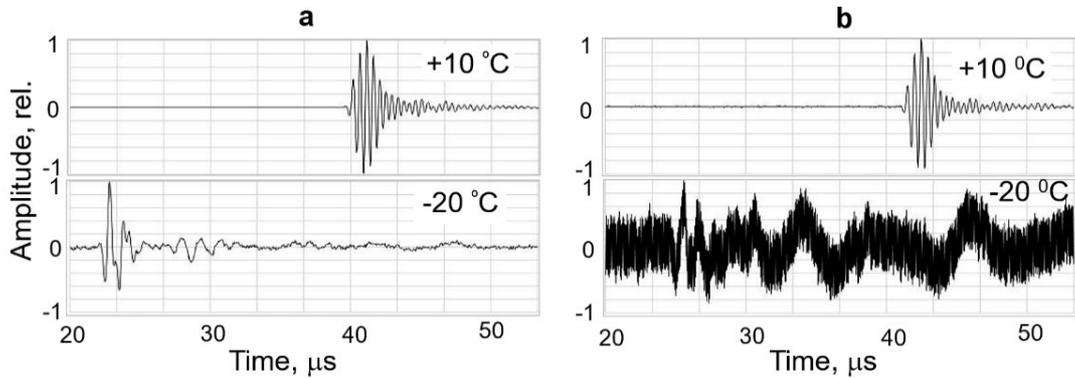

Figure 3. Ultrasonic signals normalized by beak amplitude in lean (a) and fatty (b) pork meat specimens before freezing at +10 °C (upper graphs) and in frozen condition at -20 °C (lower graphs).

Changes of ultrasonic signals during the processes of cooling and freezing of the meat samples are visually presented by time-temperature diagrams in Fig. 4. Five areas with characteristic features of ultrasound propagation can be distinguished in the correspondence to the stages of cooling and freezing (denoted as Stages I–V). Stage I, cooling of samples room temperature to temperature 0 °C is marked by strong and pronounced ultrasonic signals and intact waveform with no distortion during this period.
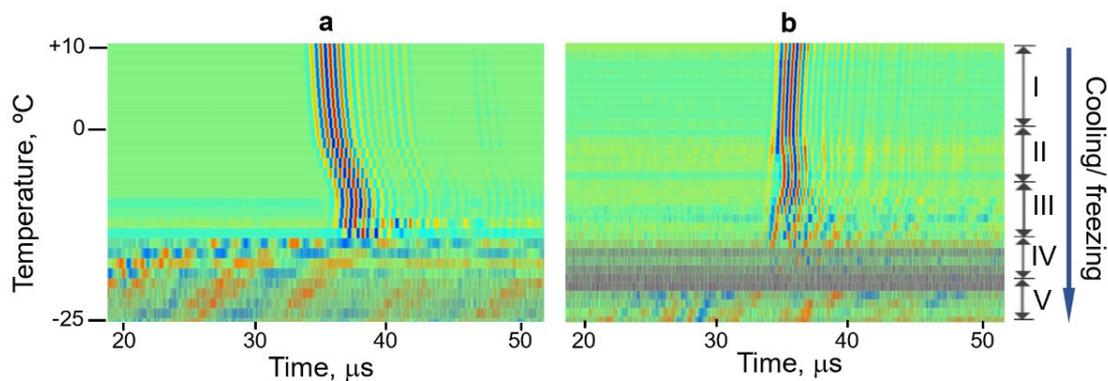

Figure 4. Time-temperature diagrams of ultrasonic signals in lean (a) and fatty (b) pork meat samples during continuous cooling from +10 °C to -25 °C. Amplitudes of signals are normalized by peak amplitude and presented in color-coded scale. I–V are stages of cooling and freezing (explanation in text).

Slight shifts of the signals by time with the temperature decrease occurred due to the known temperature effects on ultrasound velocity in water and fat. In lean meat containing up to 70% of water, decrease of ultrasound velocity is the same as in water, while there are competing trends in fatty tissue, where stiffening of the fat component strives to increase the velocity. Stage II below 0 °C is a period, where the liquid phase is prevalent inside the tissues, but the appearance of ice nuclei lead to deflection of signals from the regular trends observed before. Stage III is the active crystallization process, where the liquid phase and ice crystals are in about equal proportion forming some kind of sludge. This stage is featured by a dramatic drop of the signals' amplitude down to the noise level due to increased scattering of ultrasound on ice crystals caused by a large difference in acoustic impedances of water and ice. The signals are dispersed and parameters of ultrasound propagation are not measurable. Stage IV is the stage of further freezing to temperatures below -15 °C, when the ice component becomes to prevail. Ultrasonic signals again become notable, but shifted left by time due to the increase in velocity to values like in ice. Stage V is complete freezing to the monolithic state of the meat samples, where the steady trend of ultrasound velocity to increase with temperature corresponds to the same in ice. Ultrasonic signals are well detectable and the waveforms shapes are stable again.

The temperature dependences of ultrasound velocity presented in Fig. 5 quantitatively depicts the same five stages of cooling and freezing. Although ultrasound velocity differs in lean and fatty meat and has different slopes of the temperature dependence in the unfrozen state, its values are quite close comparing to those in the frozen state. Ultrasound velocity in frozen lean meat is close to that in ice, but ultrasound velocity in fatty meat is much lower due to fat content. A close effect was noted in Miles's early work without providing quantitative data (Miles & Cutting, 1974). Stage III is characterized by a fall of the signal amplitude to the level of noise that makes it impossible to measure ultrasound velocity. This temperature interval of the signal absence is more prolonged in fatty meat. The findings confirm the proposition that ultrasound velocity indirectly can indicate the propagation of ice front inside a meat specimen during freezing (Sigfusson et al., 2004).

Figure 5. Changes of ultrasound velocity C in lean (1) and fatty (2) pork meat specimens during continuous cooling from +10 °C to -25 °C. I–V – stages of cooling and freezing.

The signal intensity was found as another quantitative indication of freezing stages (Fig 6). The intensity reflecting changes in ultrasound attenuation is affected by the ratio between the tissue components - liquid, ice crystals and fat at all stages of freezing. A sharp drop in signal intensity was observed at III and IV stages when the mix of ice
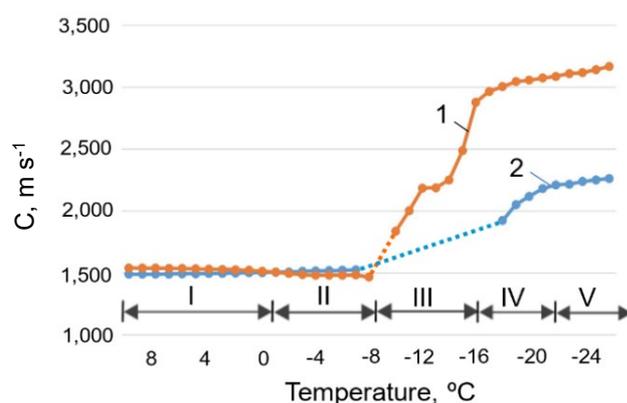
crystals and liquid phases inside the tissues were in an approximately equal proportion. The signals are stronger in lean tissue than in fatty one both in unfrozen and frozen states due to a higher ultrasound attenuation in fat.

In general, our findings confirm statements of other authors (Jha et al., 202; Charrier et al., 2015) that different food products had different ultrasonic responses to freezing manifesting in ultrasonic waveforms, spectral composition, power and temporal parameters of ultrasonic signals. The peculiarities of the dimensional heat transfer and crystallization processes in meat, in contrast to pure water and aqueous tissues, appear in the presence of 'mushy' regions in
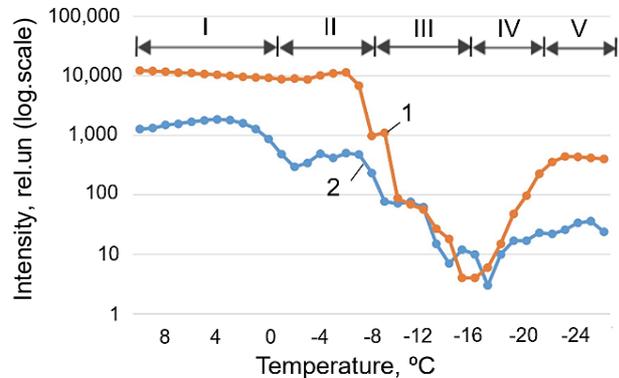

Figure 6. Changes in the intensity of ultrasonic signals in lean (1) and fatty (2) pork meat specimens during continuous cooling from +10 °C to -25 °C. I–V – stages of cooling and freezing.

meat,where crystallization and/or solidification may take place in separated areas (David et al., 2021).

CONCLUSIONS

The overview of SF processes emphasizes the necessity of analyzing temporal and temperature parameters, particularly for conducting localized studies and SF in small volumes. An experimental SF system for materials and products has been developed, enabling SF in small volumes and localized research. Solutions have been proposed for stabilizing the SF product temperature within a specified range, with temperature fluctuations not exceeding 2–3 °C.

The integration of ultrasonic monitoring holds promise for elucidating freezing processes in meat products with varying fat contents. In lean and fatty meat samples, discernible alterations in ultrasound propagation characteristics, including changes in ultrasound velocity, intensity of ultrasonic signals and waveform shapes were revealed. By leveraging the distinct ultrasonic properties of lean muscle and fat tissues, alongside the dynamic changes in tissue properties during freezing, this study unveils valuable insights into the freezing dynamics of meat. The observed trends in ultrasound propagation parameters, such as velocity and attenuation, provide a foundation for conducting nuanced assessments of SF processes and optimizing freezing regimes. Future studies will be aimed at investigation of influence of freezing regimes on different types of meat with different tissue composition moving from random observations to statistically based ones.

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