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Original Scientific Paper

Fulfilling the requirements for exporting fish meat products (tuna pâté) to the United States market

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ABSTRACT

The objective of this work was to ensure all necessary prerequisites for exporting canned fish meat (tuna pâté) to the United States market are met. To accomplish this, thermocouple measurements were conducted during sterilization in the autoclave to validate the heat treatment process. Initially, a temperature distribution study was carried out during regular production, followed by a heat penetration study. In both cases, the success criteria were satisfied. The temperature distribution was monitored using nine thermocouple probes, and within one minute after the holding time (come up time), the temperature at all measured points in the autoclave medium exceeded 120°C. For the heat penetration study, eleven probes were used, eight placed in the thermal centre of the cans and three in the autoclave medium. All probes located at the thermal centre achieved F_0 values ranging from 6.88 to 8.56 minutes, confirming the safety and effectiveness of the sterilization process for tuna pâté intended for export to the United States.

1. Introduction

Despite the advancements in emerging conservation technologies which are often categorized as non-thermal processing methods (*Furukawa S., Hayakawa I.,* 2000), thermal sterilization remains the most widely used technique for food preservation (*Farid M., Abdul Gani M.A.,* 2004). Pressured vapor sterilization is economic and has a short processing time. It is non-toxic and safe for the environment (*Silindir M, et Ozer Z.A.*, 2009). In accordance with advice from the Food and Agriculture Organization of the United Nations (FAO), the extended shelf life of heat-treated meat and meat products is obtained by inhibiting or destroying micro-organisms through a heat-based process. The main steps of this preservation method involve placing the product into a container (such as a can, glass jar, or synthetic or laminated aluminium pouch) that is hermetically sealed after filling, ensuring it is impervious to external substances, and subjecting the sealed container to a controlled thermal treatment with specified temperature and duration (*FAO*, 2025).

To ensure food safety, producers are required to implement hazard analysis and critical control point (HACCP) plans. In support of this, the United States (US) Food Safety and Inspection Service (FSIS) issued a generic HACCP model as a useful

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tool (*FSIS*, 2025). The US Food and Drug Administration (FDA) has firm and strict rules and regulations which have to be met in order to import low acid canned foods (*FDA*, 2025). In order to assist food business entities, the Institute for Thermal Processing Specialists (IFTPS) issued a guideline for proper thermal processing studies (*IFTPS*, 2014). Finally, to elaborate and simplify the application to importation of canned foods to the US, the US Department of Health and Human Services issued instructions regarding the procedures to be followed in thermal processing studies in 2016 (*FDA*, 2016) and in 2017 (*FDA*, 2017).

Steam sterilization is widely employed to ensure the sterility of manufacturing equipment and products. Regulatory standards require the validation of sterilization processes, which depends on accurately estimating key microbial resistance parameters (*Faya P. et al*., 2017). It is widely acknowledged that the thermal process is a critical control point that must be closely monitored to ensure the microbiological safety of processed foods. The hazard analysis and critical control points (HACCP) system is the globally recognized approach for enhancing the safety of sterilized food products. Common challenges in implementing a HACCP system lie in identifying critical factors, determining their critical limits, and defining acceptable tolerances for those factors (*Akterian G.S. et al.*, 1999).

The industrial sterilization process relies on a specific temperature-time profile designed to ensure product shelf life and quality by inactivating target microorganisms or enzymes while minimizing the loss of nutritional components. The heating rate and the mechanism of heat transfer in canned foods—whether by conduction or convection—are influenced by various factors, including the temperature difference between the retort and the product, the surface-to-volume ratio, and the viscosity of the product (*Tattiyakul J.M., et al*, 2002).

The intensity of heat treatment significantly influences both the elimination of micro-organisms and the sensory qualities of the product. This thermal intensity can be described using physical parameters, most commonly the F-value, which quantifies the lethal effect of heat on micro-organisms. The thermal death time, measured in minutes at 121°C, is used as a reference standard for different micro-organisms (*FAO*, 2025). The minimum exposure time, denoted as F, for a decontamination process at a specific temperature is typically estimated using an empirical model that incorporates the decimal reduction time D and the temperature resistance coefficient z as parameters. These parameters are generally assumed to be independent of temperature. However, a microbiological analysis reveals that both D and z are actually temperature-dependent, suggesting that conventional models are only accurate within a limited temperature range (*Vuković*, 2012). The temperature dependence of F derived from this approach aligns closely with existing experimental data. Additionally, safety margins to ensure sterility can be easily incorporated into the process (*Doornmalen and Kopinga, 2009*). Calculating heat penetration parameters and microbial inactivation efficiency is essential for evaluating the uniformity of heat transfer in the canning industry (*Smout et al*., 2000).

To accurately measure the heat parameters during thermal processing, thermocouples are used. These are temperature indicator and measurement devices composed of two dissimilar metals which are joined together to form two junctions. When one junction is kept at an elevated temperature as compared to the other, a small thermoelectric voltage or electromotive force is generated which is proportional to the difference in temperature between the two junctions (*IFTPS*, 2014).

Aluminium is frequently used for smaller and easy-to-open cans as deep-drawn two-piece cans (the body and the bottom end are formed out of one piece and only the top end is seamed on after the filling operation). The advantages of aluminium cans compared to tin cans are their better deep-drawing capability, low weight, resistance to corrosion, good thermal conductivity and easy recyclability.

The objective of this work was to ensure all necessary prerequisites, for exporting canned fish meat (tuna pâté) to the US market are met. To accomplish this, thermocouple measurements were recorded during thermal processing of the tuna cans in the autoclave to validate the process. Specifically, a temperature distribution study was carried out during regular production, followed by a heat penetration study.

2. Materials and methods

All export requirements for the canned fish meat (tuna pâté 60 g, packed in aluminium foil with a thermally glued lid) to the US market, regarding sterilization procedure, are stated in IFTPS guidelines (*IFTPS*, 2014).

Thermal processing of tuna pâté 60 g packed in aluminium foil took place according to the following thermal formula:

$$To = 20' + \frac{20'}{120°C/2.5bar} + 15'$$

At the start of thermal processing, the first 20 minutes was the active heating phase in which temperature increased to the defined level of 120°C in the autoclave medium, then 20 minutes was the effective sterilization time at 120° C with a medium pressure of 2.5 bar in the autoclave medium. The terminating phase was 15 minutes of cooling time prior to opening the autoclave door.

To conduct a temperature distribution study (TDS), the autoclave medium temperature was 121°C, since an acceptable temperature distribution is a requirement for process establishment. A heat penetration study (HPS) then was performed under a defined thermal formula. The stages and duration of the sterilization process were the same for the TDS and the HPS.

Thermocouple measurements, required to meet the export requirements for canned fish meat for the US market, were used during the TDS in the autoclave medium and during the HPS in the thermal centre of the cans and in the autoclave medium. The TDS and HPS were carried out in accordance with the IFTPS Guidelines, which contain success criteria for the two studies (*IFTPS*, 2014). Measurements were made with an Ellab thermocouple device (E-Val Pro, serial number 411982, validated software - US FDA, 21 CFR part 11, GMP, ver. 4.6.1.0, Picture 2), hooked to thermocouples with compensating cables. The Ellab Valsuit software, Val Suite Pro ver, was used to prepare the technical report. 5.2.015.

Both the TDS and HPS were performed in a Steriflow autoclave (Roanne, France), which operated on a cascading water principle. It is a horizontal overpressure autoclave that accommodates four carts. Considering the waiting time of the canned filling before sterilization, every sterilization cycle was carried out with three carts filled with product to the top, while one cart, always positioned in the same spot in the autoclave medium, remained empty. In this way, the three-cart work practice ensured the safety of the canned product before sterilization by monitoring the residence time of the canned filling. The internal procedures of the food business entity, based on the conducted microbiological and sensory testing, defined a maximum waiting time of the filling, before sterilization, of up to 2 h. During that defined time, the filler machine in the production department can completely fill the three full carts. The position of the carts in the autoclave is shown in Figures 1 and 2. Altogether, there were 8697 cans in the autoclave during both the TDS and the HPS. Tuna pâté cans were placed in one layer on the trays, always placed and lined up in an identical manner (Figure 3). Altogether, 116 individual cans fitted on one layer on a tray, with 25 layers in the cart, making 2899 cans on one cart. The dimensions of the tuna pâté 60 g cans were L x W x H – 97 × 60 × 25.4 mm. Identical autoclave conditions were met for the separate HPS.

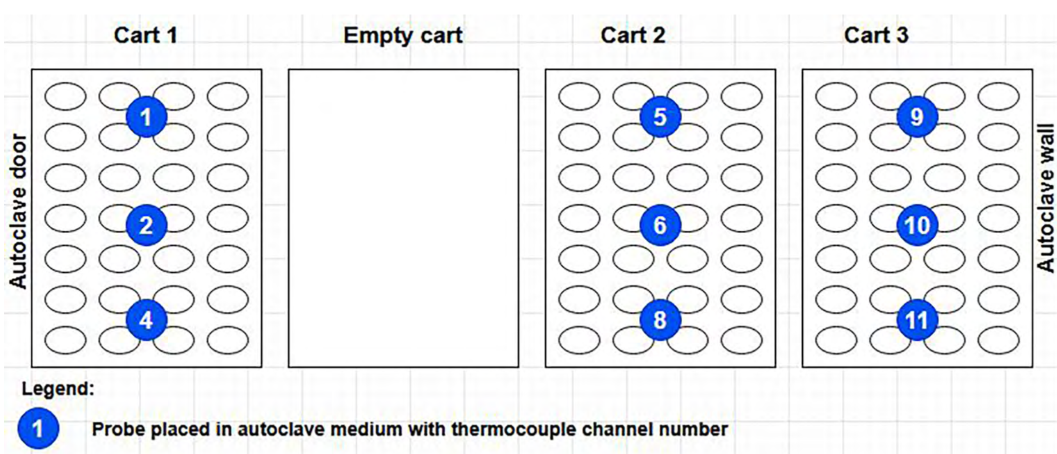

Figure 1. Plan of thermocouple probe placement in the autoclave medium (blue colour) for the temperature distribution study, seen from the side

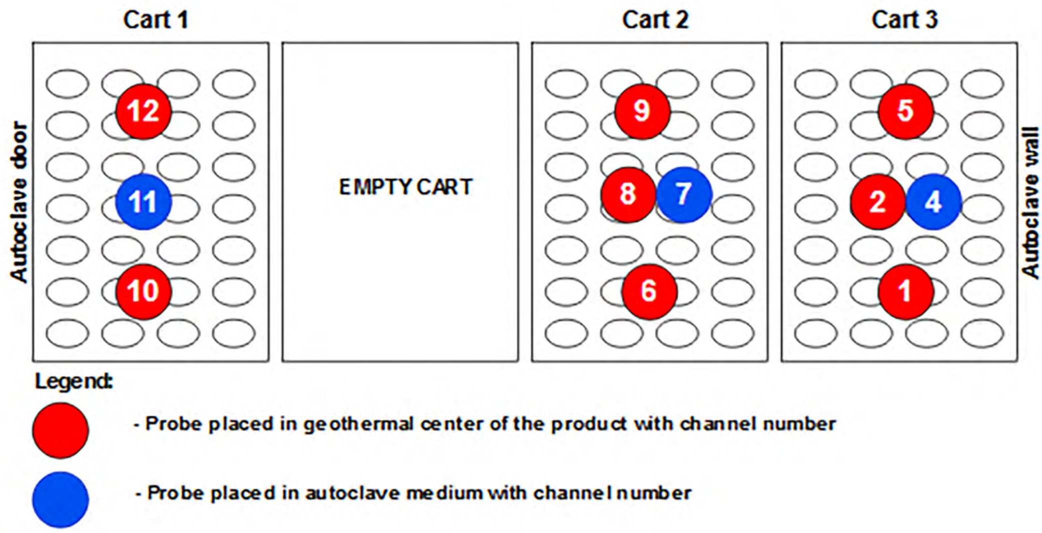

Figure 2. Schematic diagram of thermocouple probe placement in the thermal centre of the product (represented by red circles) and in the autoclave medium (blue circles), seen from the side

Figure 3. Tuna pâté cans of 60 g positioned in a cart and ready for thermal processing

For the TDS, nine thermocouple probes were used (three thermocouple probes in each of the three carts (Channels: 1, 2, 4, 5, 6, 8, 9, 10 and 11). Probes were placed outside the containers in autoclave medium (Figure 4), in accordance with IFTPS Guidelines (Chapter 4, *IFTPS*, 2014), and in the locations shown schematically in Figure 1. Temperatures were recorded at one minute intervals.

The HPS, conducted after the TDS, utilised eight thermocouple probes placed in the thermal centre of the products (Channels 1, 2, 5, 6, 7, 9, 10 and 12), in the locations depicted schematically in Figure 2. Each probe was inserted in the thermal centre of a can with an airtight seal, as depicted in the photograph, Figure 5. These eight probe locations in the autoclave were the same as in the TDS,

Figure 4. Thermocouple probes placed outside the tuna pâté cans in the carts

but in the HPS, three other probes were placed outside the cans (Channels 4, 7 and 11) to monitor the autoclave medium (Figure 2).

Figure 5. Thermocouple probe placed in the thermal centre of a tuna pâté can

3. Results

The TDS was performed during the regular production process. The temperatures recorded in the autoclave medium by the thermocouple probes throughout the thermal processing are shown in Figure 6. The first condition for the successful implementation of the TDS was to reach a temperature of at least 120°C at all control positions (thermocouple locations) within 30 seconds of reaching the effective sterilization temperature. One minute after starting the hold time, at all control positions, the temperature was above 120°C. Temperatures in the autoclave at the end of the come up time and after one minute of hold time are presented in Table 1.

Table 1. Autoclave medium temperature recorded by the probes at the end of come up time and after one minute of hold time during the temperature distribution study

Thermocouple channel	Temperature (°C)
Channel 1	120.65
Channel 2	120.69
Channel 4	121.23
Channel 5	120.32
Channel 6	120.68
Channel 8	120.53
Channel 9	120.54
Channel 10	120.38
Channel 11	120.10

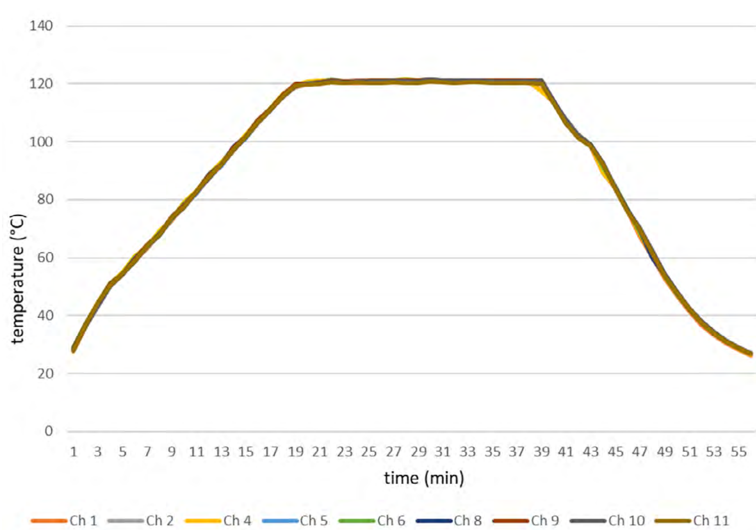

Figure 6. Temperature of the autoclave medium as recorded by the thermocouple probes at different locations in the autoclave during the thermal processing

The HPS was performed during a regular production process, and temperature results are shown in Figure 7. The temperature in the thermal centre of the product increased uniformly and consistently in every part of the autoclave medium.

At the end of the sterilization process (Table 2), acceptable F_0 values were recorded, which ranged from 6.88 (Channel 8) to 8.56 minutes (Channel 10). A graphical presentation of these F_0 values is shown in Figure 8.

Table 2. F_0 values at the end of the heat penetration study, measured in the thermal centre of the tuna pâté cans

Thermocouple channel	F_0 value (minutes)
Channel 1	7.97
Channel 2	8.18
Channel 5	7.58
Channel 6	7.78
Channel 8	6.88
Channel 9	8.05
Channel 10	8.56
Channel 12	7.29

Temperatures recorded during the HPS at the end of come up time by the probes that were placed in autoclave medium are presented in Table 3.

Table 3. Autoclave medium temperature at the end of come up time and one minute of hold time during the heat penetration study

Thermocouple channel	Temperature (°C)
Channel 4	120.78
Channel 7	120.70
Channel 11	120.28

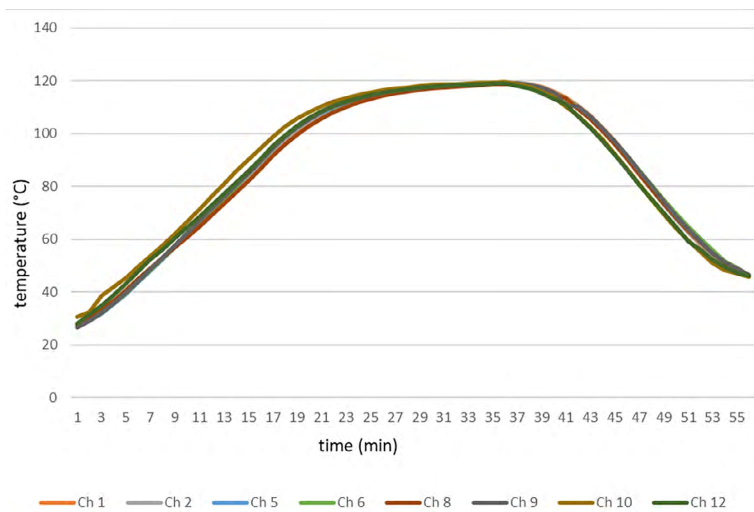

Figure 7. Temperature recorded by the different thermocouple channels in the thermal centres of tuna pâté cans during the thermal processing

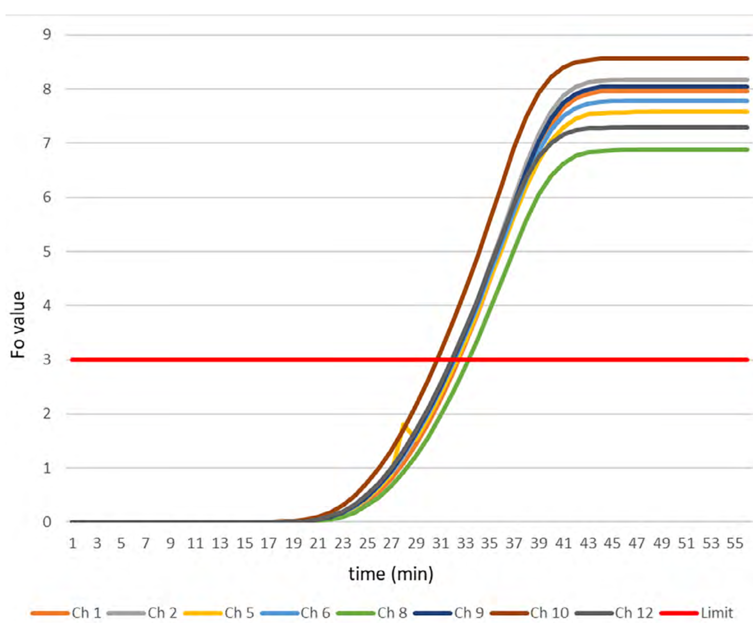

Figure 8. Achieved F_0 values during thermal processing of the tuna pâté cans

4. Discussion

Canning aims for so-called commercial sterility, meaning the food is free from microorganisms that can cause spoilage or illness under normal storage conditions (*Kinzhal M.*, 2018, *Kenneth Y.*, 2022). Validation of the sterilization process from the aspect of fulfilment all necessary conditions for export to the US market was realized through two successive studies using thermocouple probes: TDS and HPS. In both studies, the success criteria were met.

TDS is used to establish the ability of a retort process to uniformly mix and distribute the heat transfer medium, especially when the heat transfer into product may be limiting. The heating time of the autoclave medium was 20 minutes, and under the defined come up time, after 1 minute of sterilization hold time, thermocouple temperatures were above the minimum process temperature. The slowest heating point was measured by Channel 11 (120.1°C), but this was above the required temperature of 120°C, while all other thermocouple temperatures were higher at that moment. All thermocouple measurements were above the minimum process temperature after one minute of holding time. Both the uniformity and stability of temperatures in the autoclave medium were confirmed, without any fall below expected values. The position of the thermocouples was such that they were evenly distributed in all three product carts, at different levels. The success criteria for the TDS were met. Also, the three probes placed in the autoclave medium during the HPS (Table 3) revealed that the required temperatures were achieved for successful temperature distribution at the end of come up time. Product temperature increased uniformly and consistently regardless of thermocouple position, testifying to a uniform thermal process in all parts of the autoclave.

The HPS was performed to establish a consistent thermal process that would deliver commercially sterile products and to assist in evaluating process deviations (*IFTPS*, 2014). The same thermocouples were used during sterilization of the next production batch, in the same autoclave under the same production conditions. F_0 values were measured in thermal centres of the cans, in order to ensure safety and desired and expected product characteristics. Based on these microbiological considerations and including a sufficient safety margin, sterilized canned products should be produced with F_0 values of 4.0-5.5 minutes, while used autoclave medium temperatures can vary between 117 and 130 °C, which depends on the heat sensitivity of the individual products (*Desrosier et Singh*, 2025; *FAO*, 2025). In accordance with the FAO principles of meat caning and sterilization, a shelf life of up to four years at storage temperatures of 25°C or below can be achieved. The thermal death time for spores of *Clostridium botulinum* at 121°C is 2.45 minutes or in other words, an F_0-value of 2.45 is needed to inactivate all spores in the product at 121°C (*FAO*, 2025). It has been shown that F_0 values of 4 in heat-preserved products will guarantee commercial sterility (*FAO*, 2025). Measurements in our study yielded F_0 values from 6.88 (Channel 8) to 8.56 minutes (Channel 10), which is in accordance with the specified parameters for assessing HPS success, as shown in Figure 3. Comparing the F_0 values we obtained with those of other relevant pâté type products (F_0 ranged from 12.07 to 15.93 minutes and from 9.71 to 13.17 minutes), we observed a more optimal procedure that will still ensure product safety and likely will not reduce the food's biological and nutritional value (*Raseta et al.*, 2019). One condition for the successful implementation of the HPS is the achievement of a F_0 value of at least 3 in the thermal centre of the product, in accordance with the conditions that must be achieved by a can of meat intended for continental storage and transport conditions.

Overly intensive thermal processing should be avoided, since flavour changes in thermally treated meat products are caused by Maillard reactions, thermal degradation of lipids, and Maillard-lipid interactions (*Xie Q. et al., 2022*). The number and position of cans in the carts at each level was identical. The construction and shape of the cans (kidney-shaped) aluminium foil with a thermally glued lid favoured the intensity of heat transfer and the generation of suitable F_0 values in the thermal centre of the can. In similar work in Iran, it was found that cone-shaped containers exhibited the fastest heat transfer during sterilization. Container geometry significantly influences the shape and location of the slowest heating zone, as well as the final temperature, and F_0 value (*Ranjbar*, 2019).

The only thing that is constant in the entire work process is the operation of the sump water pump. During the entire process, the water circulates at the same intensity. During the one cycle of sterilization process, energy consumption was monitored and consumption of dry saturated steam at a pressure of 6 bar was 0.4 tons, while 4.6 m^3 of cold water was used.

The results obtained in both the TDS and HPS confirmed the required temperature distribution in all regions of the autoclave medium, which will ensure the safety of the tuna pâté during storage. Through these successful studies, the food business entity has increased the control level over the sterilization process as a mandatory critical control point in the production of tuna pâté intended for export to the US. Also the studies provide necessary data for further optimization of the sterilization process, from the aspect of increasing the nutritional and biological value of the product.

5. Conclusion

The success criteria for the both studies were met. First, the temperature distribution in the autoclave medium was uniform, and at the end of the come up period, all the thermocouples showed the temperature exceeded 120°C (TDS). After that, the heat penetration into the thermal centre of the product (HPS) was measured, and F_0 values were in the range of 6.88-8.56 minutes. These F_0 values are suitable to ensure the safety of the product, and all the necessary conditions were met for the export to the US market of the tuna pâté in 60 g cans, packed with a thermally glued lid.

Our findings indicate that for commercial sterilization processes, commonly used models significantly underestimate the necessary minimum exposure times at temperatures below 120°C. This has important implications for process optimization.

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References

Akterian G. S., Fernandez S. P., Hendrickx E. M., Tobbak P. P., Periago M. P. & Martinez A. (1999). Risk analysis of the thermal sterilization process.: Analysis of factors affecting the thermal resistance of microorganisms. *International Journal of Food Microbiology*, 47(1–2), 51–57. https://doi.org/10.1016/S0168-1605(99)00005-7

Desrosier, N. W. & Singh, R. P. (2025). *Food preservation.* Encyclopedia Britannica, Accessed 17 June 2025. https://www.britannica.com/topic/food-preservation

Doornmalen van M. C. P. J. & Kopinga K. (2009). Temperature dependence of F-, D- and z-values used in steam sterilization processes. *Journal of Applied Microbiology*, 107(3), 1054–1060. https://doi.org/10.1111/j.1365-2672.2009.04290.x

Farid M., Abdul Gani M. A., (2004). A new computational technique for the estimation of sterilization time in canned food. *Chemical Engineering and Processing: Process Intensification* 43(4). 523–531.

Faya P., Stamey D. J. & Seaman W. J. (2017). *A Bayesian Approach to Determination of F, D, and Z Values Used in Steam Sterilization Validation. PDA Journal of Pharmaceutical Science and Technology*, 71(2), 88–98. https://doi.org/10.5731/pdajpst.2016.006981

FAO, (2025). Meat preservation by thermal treatment (accessed 10.06.2025), https://www.fao.org/4/x6932e/x6932e03.htm

FDA, (2025). *Low Acid Canned Foods/Acidified Foods (LACF/AF)* https://www.fda.gov/industry/importing-fda-regulated-products/importing-human-foods#LACF/AF

FDA, (2017). *Instructions for Electronic Submission of Form FDA 2541 (Food Canning Establishment Registration) for an Acidified/Low-Acid Food Canning Establishment*; Accessed 18 June 2025, https://www.fda.gov/media/83914/download

FDA, (2016). *Instructions for Electronic Submission of Form FDA 2541d (Food Process Filing for Low-Acid Retorted Method*); Accessed 18 June 2025, https://www.fda.gov/media/89916/download

FSIS, (2025). *A Generic HACCP Model for a Thermally Processed, Commercially Sterile Product*, Accessed 18 June 2025, https://www.fsis.usda.gov/sites/default/files/media_file/2021-08/FSIS-GD-2021-0010_0.pdf

Furukawa S. & Hayakawa I. (2000). Investigation of desirable hydrostatic pressure required to sterilize *Bacillus stearothermophilus* IFO 12550 spores and its sterilization properties in glucose, sodium chloride and ethanol solutions. *Food Research International*, 33(10), 901–905.

IFTPS, (2014). Guidelines for Conducting Thermal Processing Studies https://www.scribd.com/document/228948730/IFTPS-Retort-Processing-Guidelines-02-13-14-3-pdf#:~:text=This%20document%20provides%20guidelines%20for%20conducting%20 thermal,best%20practices%20for%20performing%20 thermal%20processing%20studies.

Kinhal, V., (2018). *Maintaining Canned Food Quality*, accessed 17 June 2025, https://www.desjardin.fr/en/blog/maintaining-canned-food-quality#:~:text=Maintaining%20 Canned%20Food%20Quality%20*%20Food%20 safety,canned%20has%20no%20microbiological%20 contamination%20or%20toxins.&text=*%20Sterilization%20is%20the%20process%20which%20 creates,can%20no%20longer%20grow%20and%20remain%20active.

Kenneth, YIP (2022). *Know More about Canned Foods*, Food Safety Focus Vol. 186, Article 2, Accessed 17 June 2025, https://www.cfs.gov.hk/english/multimedia/multimedia_ pub/multimedia_pub_fsf_186_02.html#:~:text=This%20 usually%20entails%20heating%20the,conveniently%20 with%20no%20refrigeration%20required.

Raseta, M., Branković-Lazić, I., Mrdović, B., Baltić, B., Zsolt, B., & Djordjević, V. (2019). Optimization of liver pâté sterilization from the aspect of preserving nutritional value and ensuring food safety. *Meat Technology,* 60(2), 97–105. https://doi.org/10.18485/meattech.2019.60.2.4

Ranjbar, A. (2019). Numerical Calculation F-value and Lethality of Non-Newtonian Food Fluid during Sterilization based on Can Geometry. *Iranian Food Science and Technology, Research Journal*, 14(6), 113–125.

Silindir, M., & Ozer, Y. A. (2009). Sterilization Methods and the Comparison of E-Beam Sterilization with Gamma Radiation Sterilization. *FABAD Journal of Pharmaceutical Sciences*, 34, 43–53.

Smout, C., Loey, V.A., Hendrickx, M., & Beirlant, J. (2000). Statistical Variability of Heat Penetration Parameters in Relation to Process Design. *Journal of Food Science,* 65(4), 685–693.

Tattiyakul, J., Rao A. M., & Datta K. A. (2002). Heat Transfer to Three Canned Fluids of Different Thermo-Rheological Behaviour Under Intermittent Agitation. *Food and Bioproducts Processing,* 80(1), 20–27.

Xie, Q., Xu, B., Xu. Y., Yao. Z., Zhu, B., Li, X., & Sun, Y. (2022). Effects of different thermal treatment temperatures on volatile flavour compounds of water-boiled duck after packaging. *LWT*, 154, 112625, https://doi.org/10.1016/j.lwt.2021.112625

Vuković I., (2012). Fundamentals of Meat Technology, 2012, Fourth Edition, Veterinary Chamber of Serbia, Belgrade, page 81.

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