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Exploring the Effects of Ultrasound Processing on Food Packaging Laminated With Aluminum Foil

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ABSTRACT

This study investigates the effects of ultrasound (US) processing on laminated plastic packaging containing aluminum foil for food contact applications. Two multilayer structures, PET/Al/PE and PET/Al/PP, were exposed to US at 25 kHz and 8.75 W/L for up to 40 min at 25°C and 60°C, using a 3% acetic acid as a food simulant. The structural, mechanical, thermal, barrier, and migration properties of the packaging were evaluated. Results indicate that US exposure for up to 30 min did not alter chemical composition, thermal stability, seal strength, nor barrier properties. A slight rise in elongation at break in the machine direction of the PET/Al/PP film was noted when processed at 60°C with ultrasound, relative to both the untreated samples and those treated at 25°C. No significant overall, aluminum, or antimony migration was detected. These findings demonstrate that aluminum-laminated packaging can be safely used in US food processing applications, provided that exposure time does not exceed the time to prevent structural degradation.

1 | Introduction

Food packaging plays a crucial role in modern food manufacturing, ensuring that products remain safe, fresh, and appealing throughout their shelf life. Because of its vital role, as consumers increasingly seek convenient, ready-to-eat, and long-lasting food options, packaging must effectively protect against environmental factors such as moisture, oxygen, and light, among others, which can lead to product deterioration [1]. In addition to preserving food quality, packaging must meet hygiene and safety standards, provide essential product information, and facilitate handling and storage [2]. Given its vital function, food packaging must be carefully designed to integrate with food processing methods, minimizing contamination risks while maintaining nutritional and sensory properties [3].

High-barrier flexible packaging is extensively utilized in the food sector for its superior protective qualities, safeguarding items from physical, chemical, and biological contaminants, thereby extending shelf life and maintaining quality. To achieve this, manufacturers combine multiple materials in a composite structure, which enhances the barrier effect while reducing material thickness [2, 4]. Aluminum foil plays a crucial role in these composite systems, offering a lightweight yet strong

Summary

- Ultrasound processing on polymer packaging with aluminum foil was evaluated.
- Ultrasound exposure above 30 min led to aluminum foil perforation.
- No significant migration of aluminum or antimony was detected.
- Thermal, barrier, and seal strength properties remained stable.

material that can be easily shaped into various packaging formats. Furthermore, its stiffness and dead-fold characteristics allow for a variety of packaging applications, such as pouches, trays, lids, and laminated cartons to pharmaceutical blister packs [2]. Additionally, aluminum foil exhibits excellent resistance to corrosion and high temperatures (up to 150°C), making it an ideal choice for applications requiring high durability and long-term storage stability [5].

Additionally, a major benefit of aluminum foil in food packaging is its outstanding protection against oxygen, moisture, and light, the primary elements responsible for food spoilage. Even in its thinnest form, aluminum foil prevents flavor loss and aroma migration [6, 7]. To enhance its performance, aluminum foil is often combined with plastic layers or coatings that improve flexibility, heat-sealing properties, and resistance to mechanical stress. Studies have shown that aluminum foil–plastic laminates maintain their barrier properties more effectively than metallized films, making them a superior choice for high-barrier packaging solutions [2, 5].

The development of novel technologies for food processing aims to enhance safety, quality, and efficiency while reducing environmental impact and the need for preservatives. Among these, ultrasound (US) has gained attention because it can physically modify foods through cavitation [8]. Ultrasound applies high-frequency sound waves in different systems, and it can be used at low or high intensities. Low-intensity ultrasound ($<1\,W/cm^2$) is primarily employed for process monitoring and quality control, whereas high-intensity ultrasound ($>1\,W/cm^2$) is capable of actively altering material properties and enhancing manufacturing processes [9]. Its versatile nature and environmentally friendly profile—being a purely physical method—have positioned ultrasound as a promising green technology across various industries [10]. The specific effects of ultrasound on a product are influenced by multiple variables, including the system's physical and chemical properties, the nature of the product itself, and the specifications of the ultrasound equipment. In the food sector, ultrasound has been applied for numerous functions, such as accelerating mass transfer processes [11], improving nutrient extraction and bioavailability [12], altering enzymatic activities [13], and modifying the structure of polysaccharides [14, 15].

Ultrasound in packaged food processing provides multiple benefits, such as decreasing reliance on preservatives, minimizing thermal damage, and maintaining sensory qualities [16]. However, under these conditions, ultrasound may alter the packaging material's properties, affecting its permeability, mechanical strength, and potentially facilitating the transfer of compounds from the packaging into the food.

For example, ultrasonically treated pouches made from linear low-density polyethylene (LLDPE) and acrylic/poly(vinylidene chloride) polypropylene (PPAcPVDC) showed notable increases in overall migration levels, regardless of the food simulant employed [16]. Additionally, studies involving biaxially oriented polypropylene (BOPP) films coated with acrylic/poly(vinylidene chloride) (BOPPAcPVDC) and coextruded BOPP (BOPPcoex) have demonstrated that ultrasound exposure can significantly influence their gas barrier properties. In particular, high-power ultrasound treatments markedly affected oxygen permeation, altering the permeability, solubility, and diffusion rates of oxygen within these materials under certain testing conditions [8]. The same US-treated samples exhibited better thermal stability compared to untreated ones, and surface changes were observed in BOPPcoex films. These findings suggest that US treatment could be a viable option for enhancing the properties of packaged food materials [17]. Finally, a study focusing on the migration of monomers from multilayer packages composed of polyethylene (PE) and polyamide (PA) layers and filled with aqueous, acidic, and fatty food simulants showed that US processing influenced the migration of ε-caprolactam. The results met Brazilian and European Union legislation limits; however, the authors highlighted the importance of packaging design and evaluation in developing emerging food processing technologies [3].

This study differs from the previously published work by Marangoni Júnior et al., which investigated the effects of US processing on metallized flexible materials, analyzing their crystalline structure, chemical–physical composition, and migration of metals such as aluminum and antimony in PET_{met}/PE and PET_{met}/PP films subjected to similar processing conditions (25 kHz, 8.75 W/L, varying temperatures) [18]. While the previous study focused on assessing metallized packaging under higher ultrasonic exposure times, the present work expands this approach by specifically examining laminated packaging containing aluminum foil, evaluating the effects of US at shorter exposure times. Thus, this study provides a more detailed understanding of the impact of US on the integrity of packaging containing laminated aluminum foil in different polymers, aspects that are crucial for its safe application in the food industry.

Despite the advancements in US technology and its applications, packaging criteria for products subjected to US treatments remain poorly defined. Given the growing adoption of emerging technologies in the food industry, it is crucial to understand the impact of US on multilayer flexible packaging performance and its safety in food contact applications. This study investigates the effects of US on flexible multilayer packaging containing aluminum foil, focusing on its mechanical and barrier properties, and considering implications for food safety and preservation through migration. Understanding these effects is crucial for optimizing the application of US technology in food packaging

and ensuring the development of more sustainable and effective packaging solutions.

2 | Material and Methods

2.1 | Materials

For this research, two flexible packaging laminated with aluminum foil were selected: PET/Al/PE (14/10/55 μm) and PET/Al/PP (14/9/56 μm) (PET = polyethylene terephthalate, Al = aluminum foil, PE = polyethylene, and PP = polypropylene). These materials are commonly utilized for food products that demand high barriers to oxygen, water vapor, and light, such as tomato products (sauce, ketchup), salad dressings, sauces, and fruit juices, since the aluminum foil in the multilayer polymer-based structure imparts these protective properties.

2.2 | Sample Preparation and Ultrasonic Processing

This study employed an acidic food simulant: a 3% acetic acid solution (≥ 98.0%, Merck, Germany) in deionized water, with a pH of ≤ 4.5, following RDC No. 51 from Brazil's Anvisa [19] and EU Regulation No. 10/2011 [20]. The acidic simulant was selected to mimic low-pH foods like sauces, juices, and other processed items that frequently utilize aluminum foil packaging. Acidic foods are known to have a higher potential to promote the migration of metals like aluminum and antimony from packaging into the food, compared to low-acidity or fatty simulants, which tend to have lower interactions with metal layers. Therefore, this simulant allows us to evaluate the worst-case scenario regarding migration and interactions between the aluminum foil components and the food matrix. Packages (120 mm × 150 mm) were created by sealing two films with a 3 mm wide heat sealer (Haramura—A380, SP, Brazil). Each package was then filled with 100 mL of the food simulant and securely sealed.

The packaged samples were treated in an ultrasonic bath (Q 13/25 A CR, Ultronique, Indaiatuba, Brazil) operating at 25 kHz with a volumetric power of 8.75 W/L, determined via calorimetric measurement. The processing was performed at two different temperatures: 25°C and 60°C. These temperatures were selected because the ultrasound treatment is intended as a pre-treatment step prior to other processes, like thermal treatments, which typically occur around these temperature ranges. The ultrasound treatment was first performed for up to 40 min, as detailed in item 3.1, to observe the onset of surface defects. As defects appeared at this time, the processing time was subsequently reduced to 30 min for the remaining analyses, ensuring no surface damage occurred. Water was used as the medium to propagate the ultrasonic waves evenly throughout the bath. For comparison, samples were also processed at 60°C for 30 min without ultrasound to isolate and evaluate the effect of temperature alone. Untreated samples served as controls. All treatments were performed in triplicate to ensure reproducibility. Following treatment, the samples were analyzed for their morphology, structural characteristics, properties, and migration.

2.3 | Evaluation of Packaging Films

2.3.1 | Optical Microscopy

To detect defects in the outer surface (PET) and the cross-section of laminated films resulting from ultrasonic processing, we conducted a detailed analysis using an M165C stereomicroscope (Leica, Germany) with a magnification of up to 120× and LAS EZ software. This equipment provided a clear view of the surfaces, allowing for the identification of imperfections that may affect the performance of the films. For the cross-section images, the films were prepared using a microtome (Leica—RM2245, Buffalo Grove, USA), set to cut at 40 μm thickness. The cuts were made with precision in the areas where the packaging exhibited defects.

2.3.2 | Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectra were obtained for both the inner layers (PE and PP) and the outer PET layer of the laminated packaging samples. The analysis used a Spectrum 100 spectrometer (PerkinElmer, Waltham, MA, USA) with ATR accessories containing a zinc selenide crystal. Spectral data were collected between 4000 and 650 cm^{-1} at a resolution of 4 cm^{-1}, using Spectrum software version 10.4.00. To ensure data reliability and account for sample variability, three spectra were collected from different locations on each layer, minimizing measurement inconsistencies [21, 22].

2.3.3 | Differential Scanning Calorimetry (DSC)

The thermal characteristics of the laminated packaging were evaluated using DSC at a heating rate of 10°C/min, from 40°C to 300°C, with a DSC 250 calorimeter (TA Instruments, New Castle, USA). About 5 mg of each sample was tested under a nitrogen atmosphere, in accordance with ASTM-D3418-21 standards [23]. The resulting thermograms enabled the identification of the melting temperature (T_m), which was determined from three replicate analyses of each sample.

2.3.4 | Mechanical Properties

Tensile strength (TS), elongation at break (EB), seal strength, and bond strength were tested under standardized conditions: 23° ± 2°C and 50% ± 5% RH, after conditioning samples for 48 h in the same environment. Laminated samples, 25.4 mm wide, were cut with a precision cutter (JDC Twing Albert). All assessments used an Instron 5966-E2 universal testing machine (Norwood, USA), with five replicates per sample. Tensile strength and elongation at break were measured with a 1 kN load cell at 50 mm/min and 50 mm clamp distance [24]. Seal strength was determined using a 100 N load cell at 300 mm/min and a 25 mm clamp distance [25]. Tests were performed in both the machine direction (MD) and transverse direction (TD) to evaluate orientation effects. Bond strength was evaluated by applying a 100 N load to delaminate approximately 75 mm of material at 280 mm/min, calculating the average

over a 50 mm segment, excluding initial and final peaks for accuracy [26]. This thorough analysis offers valuable insights into the mechanical performance of the laminated materials, especially relevant for their behavior during ultrasound processing.

2.3.5 | Barrier Properties

The water vapor transmission rate (WVTR) was determined using a PERMATRAN analyzer with an infrared sensor (model 3/34 G, MOCON, Minneapolis, USA), following ASTM-F1249 guidelines [27]. Tests were conducted at 38°C and 90% RH to simulate ideal conditions for assessing the moisture permeability of the packaging samples. The oxygen transmission rate (OTR) was measured using an OXTRAN analyzer (model 2/20H, MOCON, Minneapolis, USA) in accordance with ASTM-D3985 [28]. During this test, the sample's outer surface was exposed to pure oxygen (100% O_2). Data adjustments accounted for a pressure gradient of 1 atm, with measurements performed at 23°C under dry conditions. Both measurements considered an effective permeation area of 50 cm^2 per sample, and all tests were conducted in duplicate. The information obtained on WVTR and OTR is essential for understanding how aluminum-laminated plastic packaging protects food from external factors, contributing to the preservation of food products.

2.3.6 | Overall and Specific Migration of Metals

Overall migration from the laminated packaging was evaluated immediately following US treatment and again after 10 days of storage at 40°C, simulating the most critical contact conditions for food safety. The testing procedures adhered strictly to the regulations specified in RDC No. 51/10 [19] and EU Regulation No. 10/2011 [20], following standardized official methods such as EN-1186-1 [29] (2002) and EN-1186-2 [30].

The overall migration was analyzed gravimetrically using an analytical balance with a precision of 0.00001 g (MSA225P-1 CE-DA, Sartorius). The acidic food simulant was evaporated on a heating plate (TE038, Tecnal) to enable subsequent chemical analysis. All tests were conducted in triplicate to ensure reliability and accuracy of the results.

Furthermore, the release of aluminum (Al) and antimony (Sb) from the packaging into the simulant was measured both immediately after treatment and after 10 days of storage at 60°C. These measurements followed the contact conditions specified by EU Regulation No. 10/2011 [20]. The analysis was conducted using inductively coupled plasma optical emission spectrometry (ICP-OES) (Optima 2000DV, PerkinElmer). The ICP system operated at a plasma power of 1500 W, with a detection height set at 15 mm, an axial torch configuration, and gas flow rates of 17 L/min for argon, 0.2 L/min for auxiliary flow, and 0.55 mL/min for the nebulizer. The emission lines used for quantification of Sb and Al were 206.836 and 396.153 nm, respectively, with all analyses monitored via Syngistix software (PerkinElmer). Each measurement was repeated three times to ensure the robustness of the results.

2.3.7 | Statistical Analysis

The data were analyzed using statistical methods to ensure their accuracy and consistency. An ANOVA test was performed to identify any statistically significant differences among the different data sets. If differences were detected, Tukey's post hoc test was conducted to determine which specific groups differed from each other. A p-value of less than 0.05 was considered indicative of significant differences.

3 | Results and Discussion

3.1 | Preliminary Treatments and Definition of Process Conditions

Initially, the samples were subjected to US processing for periods of up to 40 min that resulted in defects on the surface of the packaging. According to microscopic observations, a processing time equal to 40 min (at the conditions here employed) led to visible damage on the films' surface, as illustrated in Figure 1. The images of the films treated by US for 40 min revealed that the application of US at temperatures of 25°C and 60°C caused perforation of the aluminum layer in the multilayer films. It is noteworthy that these perforations occurred exclusively in the aluminum foil, without affecting the underlying polymer materials, as demonstrated in the cross-sectional images of the films. This result is further supported by the fact that there was no leakage of the food simulant from inside the packaging to the outside. Even so, this failure restricts the application of the processed packaging, limiting the experiments conducted to pre-failure conditions.

The primary hypothesis regarding the perforation of the aluminum layer in the multilayer structure suggests that this phenomenon may be more closely related to the effects of prolonged cavitation during US processing. Although the aluminum layer is positioned between different polymer layers, it is likely that during extended US treatment, alterations in the mechanical properties of the aluminum layer occur due to the intensified forces generated by cavitation.

As a result of these findings, the processing time was set at 30 min, a duration that did not lead to the formation of such defects attributed to bubble collapse during cavitation. This approach, therefore, ensures the viability of using laminated films with an aluminum layer in applications involving US processing.

3.2 | FT-IR Spectroscopy Analysis

FT-IR spectroscopy was employed to examine the functional groups present in both the outer layer and the inner layers of the films. The goal was to assess how interactions with the food simulant, US treatment, and the properties of the packaging affected these chemical groups. The FT-IR spectra of the outer PET layer from the various samples are shown in **Figure 2a,b**. The analysis identified key absorption bands at 3430 cm^{-1}, corresponding to hydroxyl (OH) groups, and between 3053 and 2908 cm^{-1}, indicative of symmetric CH stretching. A prominent peak at 1715 cm^{-1} was assigned to C=O stretching, while the

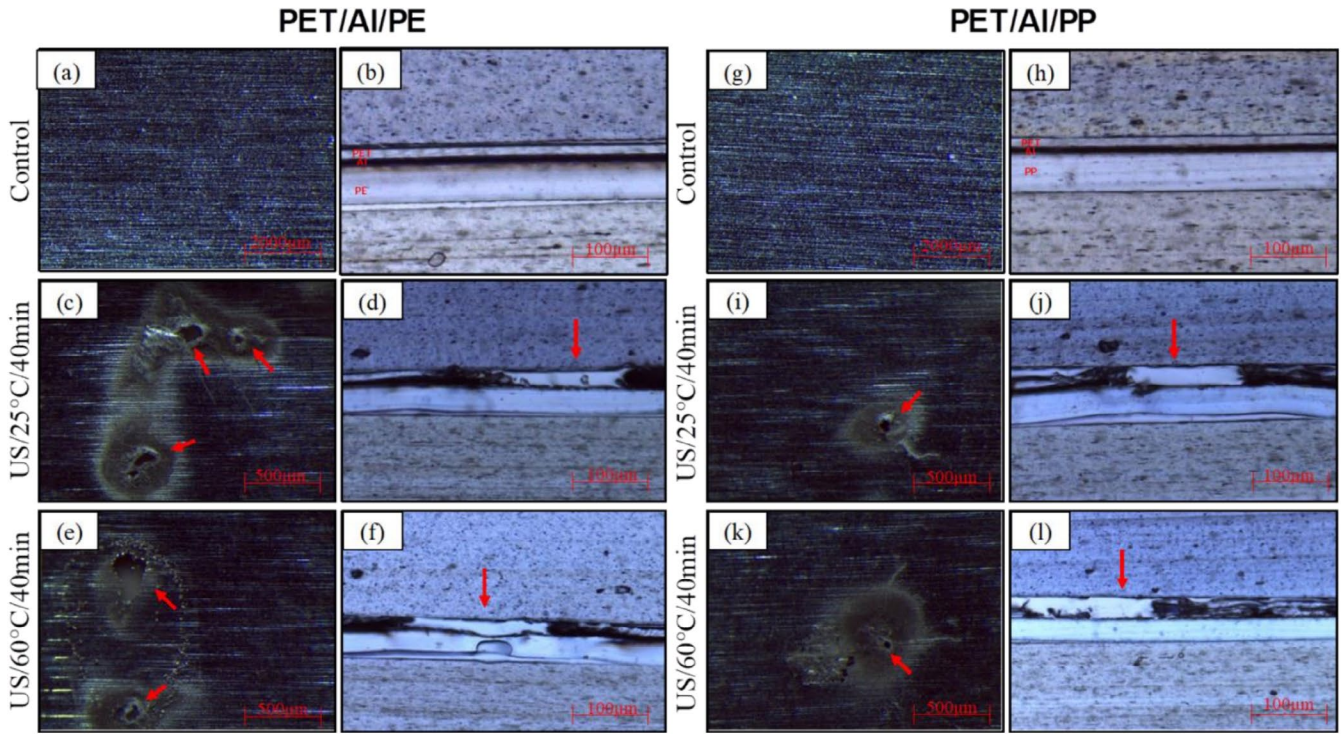

FIGURE 1 | Surface of the outer layer of the analyzed samples viewed under a stereomicroscope at 4× magnification (a, c, e, g, i, and k) and the cross-section observed at 100× magnification (b, d, f, h, j, and l). The red arrows highlight the defects observed in the packaging material.

bands at 1580 cm^{-1} and 1505 cm^{-1} are related to aromatic C=C stretching vibrations. The spectral region from 1470 to 1330 cm^{-1} reflects CO group stretching, associated with bending and oscillatory motion of segments like ethylene glycol. The presence of terephthalate groups is evidenced by the band at 1245 cm^{-1}, and bands at 1097 and 1045 cm^{-1} correspond to methylene ester bonds and C—O vibrations. Additionally, signals between 1017 and 720 cm^{-1} are linked to aromatic ring vibrations, as reported by Pereira et al. [31].

The FT-IR spectrum of the inner PE layer is shown in Figure 2c. The peaks at 2915 and 2850 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of the C—H bonds in CH_2 groups, respectively. Additionally, bands in the 1470–1460 cm^{-1} range are associated with CH_2 deformation modes, while those at 1370 and 720 cm^{-1} indicate bending vibrations of CH_3 groups and stretching of C—C bonds in CH_2, respectively, as documented by Turriziani et al. [32].

The FT-IR spectrum of the inner PP layer is presented in Figure 2d. The peaks at 2950 and 2840 cm^{-1} are associated with the stretching vibrations of CH_3 and CH_2 groups, respectively. The bands observed at 1455 and 1375 cm^{-1} correspond to CH_3 deformation and bending vibrations of CH_2, while the peak at 1360 cm^{-1} indicates the symmetric deformation of CH_3. Additionally, weaker bands in the 1300–700 cm^{-1} range are linked to the asymmetric stretching of C—C bonds and vibrations of CH_3 and CH_3 groups, as discussed by Alaburdaitė and Krylova [33] and Smith [34].

Different ultrasound processing parameters and their effects on the food simulant showed no substantial impact on the surface spectra of the multilayer packaging polymers. This suggests that the chemical makeup of the materials—mainly PET, PE, and PP—was preserved after ultrasound treatment, irrespective of the temperature used. Consistent findings were also noted in samples made of PE and PA films [35], BOPPAc/PVDC, and BOPPcoex [17] that also underwent ultrasonication treatment. This consistency in chemical properties suggests that the applied treatments, even under variable conditions, did not interfere with the structural or functional integrity of the polymers, highlighting the robustness of these materials in the face of US processing.

3.3 | Thermal Properties

The melting curves of the two films, both before and after exposure to different conditions of US processing, are presented in Figure 2e,f and in Table 1. The analyses conducted through DSC for the PET/Al/PE film clearly revealed two distinct melting peaks, each corresponding to a specific component of the material: the first peak, associated with PE, and the second peak, referring to PET. The melting temperatures (T_m) recorded for these peaks were found to range between 124.1°C and 125.0°C for PE, while for PET, the T_m varied between 230.3°C and 237.8°C. In the case of the PET/Al/PP film, two distinct melting peaks were also detected, reflecting the presence of different polymer components: the first peak was attributed to PP, while the second peak corresponded once again to PET. The T_m for these materials ranged from 156.7°C to 157.5°C for PP and from 234.4°C to 236.1°C for PET. These obtained values are in solid agreement with those previously reported in the specialized literature for the polymers PE, PP, and PET [32, 36, 37], reinforcing the validity and accuracy of the measurements conducted.

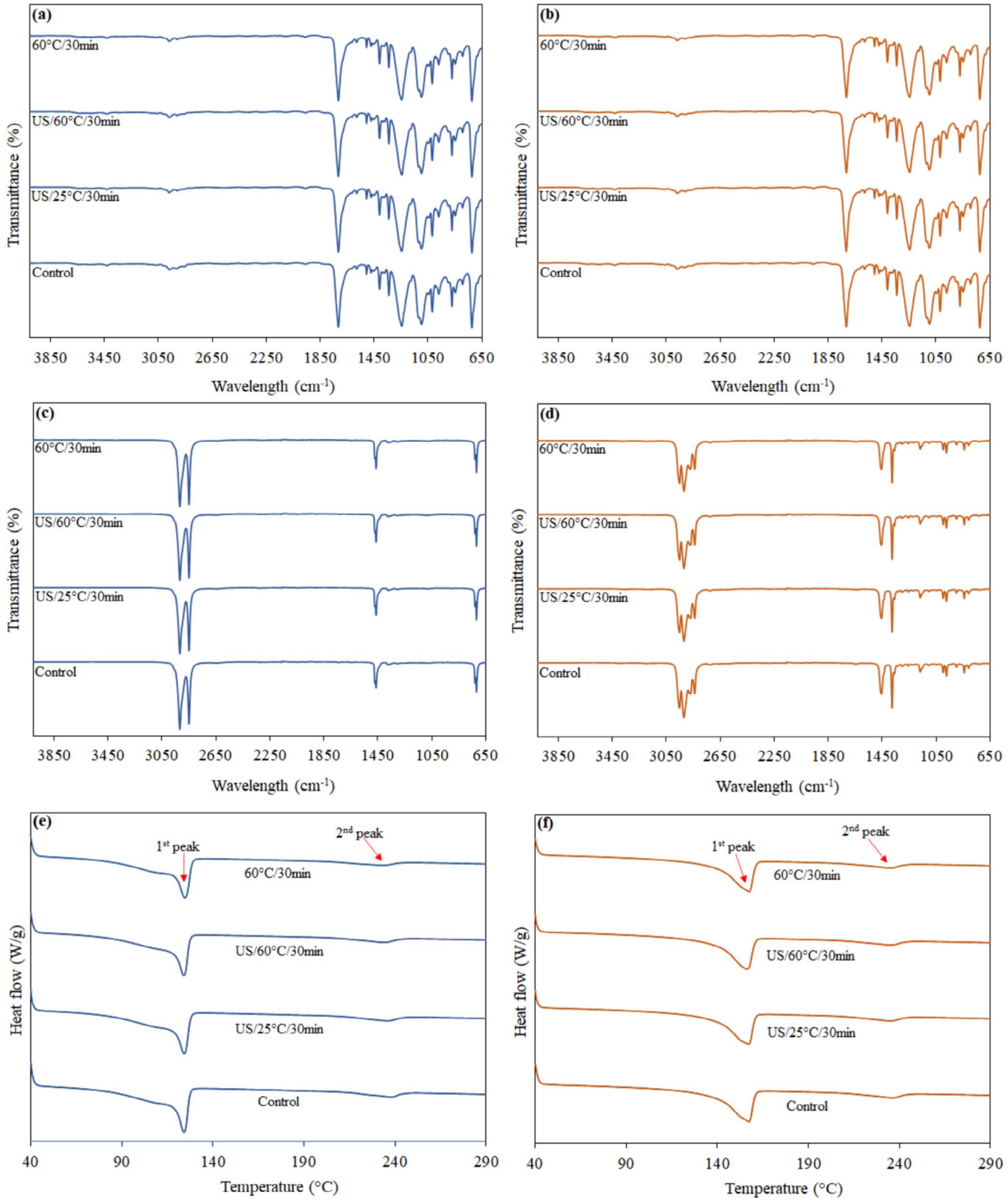

FIGURE 2 | FT-IR spectra of the outer layer (a and b) and inner layer (c and d), along with differential scanning calorimetry (e and f), of various packaging materials processed by ultrasound ($n = 3$). The samples include PET/Al/PE (a, c, and e) and PET/Al/PP (b, d, and f).

Exposure to various ultrasound processing conditions did not produce notable differences in the melting temperatures of the films when compared to unprocessed control samples. This implies that the combined effects of the food simulant, packaging materials, and ultrasound treatment did not significantly influence the films' thermal behavior. The maintained melting points indicate that the molecular structures of the polymers remained stable and unaffected by the process. These findings are in agreement with prior studies on PE and PA films, which also showed consistent thermal profiles after contact with fatty and

TABLE 1 | DSC results for various materials processed by US ($n = 3$).

Packaging material	Thermal property—T_m (C°)	Processing condition			
		Control	US/25°C/30 min	US/60°C/30 min	60°C/30 min
PET/Al/PE	PE	124.2 ± 0.1[a]	124.3 ± 0.6[a]	124.1 ± 0.1[a]	125.0 ± 1.0[a]
	PET	237.8 ± 0.7[a]	235.7 ± 0.8[a]	233.7 ± 0.7[a]	230.3 ± 6.1[a]
PET/Al/PP	PP	157.4 ± 0.3[a]	157.3 ± 0.1[a]	156.7 ± 0.9[a]	157.5 ± 0.3[a]
	PET	236.1 ± 0.5[a]	234.6 ± 0.8[a]	235.1 ± 1.0[a]	234.4 ± 0.7[a]

Note: T_m is the melting temperature. Values sharing the same letter (a, b, c) within a row are not significantly different from each other at the 95% confidence level ($p < 0.05$).

aqueous simulants under similar ultrasonic treatment parameters [3]. In that study, the films were subjected to ultrasound at 25 kHz frequency, with a volumetric power of 9.74 W/L, maintained at 25°C, for periods between 30 and 60 min. The similarities in the results emphasize the robustness of the material under the processing conditions, highlighting that there was no impairment of the thermal properties, regardless of the type of simulant used or the duration of exposure to the treatment.

Moreover, these findings align with the results obtained through FT-IR spectroscopy, which supports the conclusion that the level of energy applied during the US processing was not sufficient to induce significant molecular modifications in the evaluated polymers. This consistency across different analytical methods reinforces the understanding that the tested materials maintain their integrity, ensuring their appropriate functionality in the intended applications.

3.4 | Tensile Strength and Elongation at Break

Figure 3a,b illustrates the impact of US on the TS of the films. At the same time, Figure 3c,d displays the EB for these films, assessed in two orientations: MD and TD. The data obtained demonstrated that the TS of the PET/Al/PE films, both in the MD and in the TD, did not show significant changes in response to the various processing conditions applied. The measured values were statistically equivalent to those recorded for the control film, indicating the stability of the mechanical properties of this material. For the PET/Al/PP film, similar behaviors were observed in the TD. Under the conditions of US/60°C/30 min, a slight increase in TS was observed in the MD compared to both the untreated control and the sample processed at US/25°C/30 min. However, this increase appears to be minimal and may lack practical significance. Notably, such an improvement was not seen in samples exposed to 60°C/30 min without ultrasound. Concerning EB, the trend mirrored the TS results, with a significant enhancement detected in the PET/Al/PP film processed at 60°C for 30 min under US conditions relative to the control group.

The statistical analysis indicates differences in both TS and EB, even though the observed changes may not be significant from a practical mechanical perspective. A possible positive interaction can be attributed to the layer of PP, which is the only polymer distinguishing this film from the other multilayer film studied. The increased energy provided by the treatment (through high temperature and acoustic cavitation) can facilitate molecular movement and create more space between polymer units [8, 38], possibly changing the molecular network and local crystallinity. This condition may favor the movement of polymer chains, leading to greater elongation and improved mechanical characteristics, although these improvements should be the topic of new research.

Summarizing, the applied conditions of ultrasound processing can be considered not to affect both TS and EB of both evaluated materials, which is a relevant result from the application perspective.

3.5 | Seal Strength

The seal strength measurements in both the MD and TD for the films are depicted in Figure 3e,f. The results indicated that US treatment, across the different temperature conditions tested, did not produce significant alterations in the seal strength of either film. This phenomenon can be attributed to the ideal sealing conditions employed in the films, in which acoustic cavitation was not sufficiently strong to break the intermolecular bonds between the sealing layers—specifically, the PE–PE bonds in the PET/Al/PE film and the PP–PP bonds in the PET/Al/PP film. It is important to highlight that, as a result of this positive outcome, the packaging retained its closure integrity after being subjected to different US treatment conditions. This suggests a good resistance of the sealing layers to the processing, which is crucial for the functionality of the packaging.

On the other hand, in previous studies, it was observed that the films of PE and PA exhibited a reduction in seal strength after US processing [35]. These results indicate a more intense interaction between the applied processing conditions, the packaging, and the food simulants used. It is worth noting that, in that case, the processing time for US was greater than that used in the present study, which may have contributed to the differences observed in sealing properties. This divergence highlights the complexity of the interactions between processing variables, materials, and food simulants, emphasizing the need for optimization of these parameters to ensure the effectiveness of the packaging.

3.6 | Bond Strength

The bond strength measurements for the films are shown in Figure 4a,b. These tests assessed the adhesion between the PET/Al layer and the PE or PP layers, demonstrating that the bond

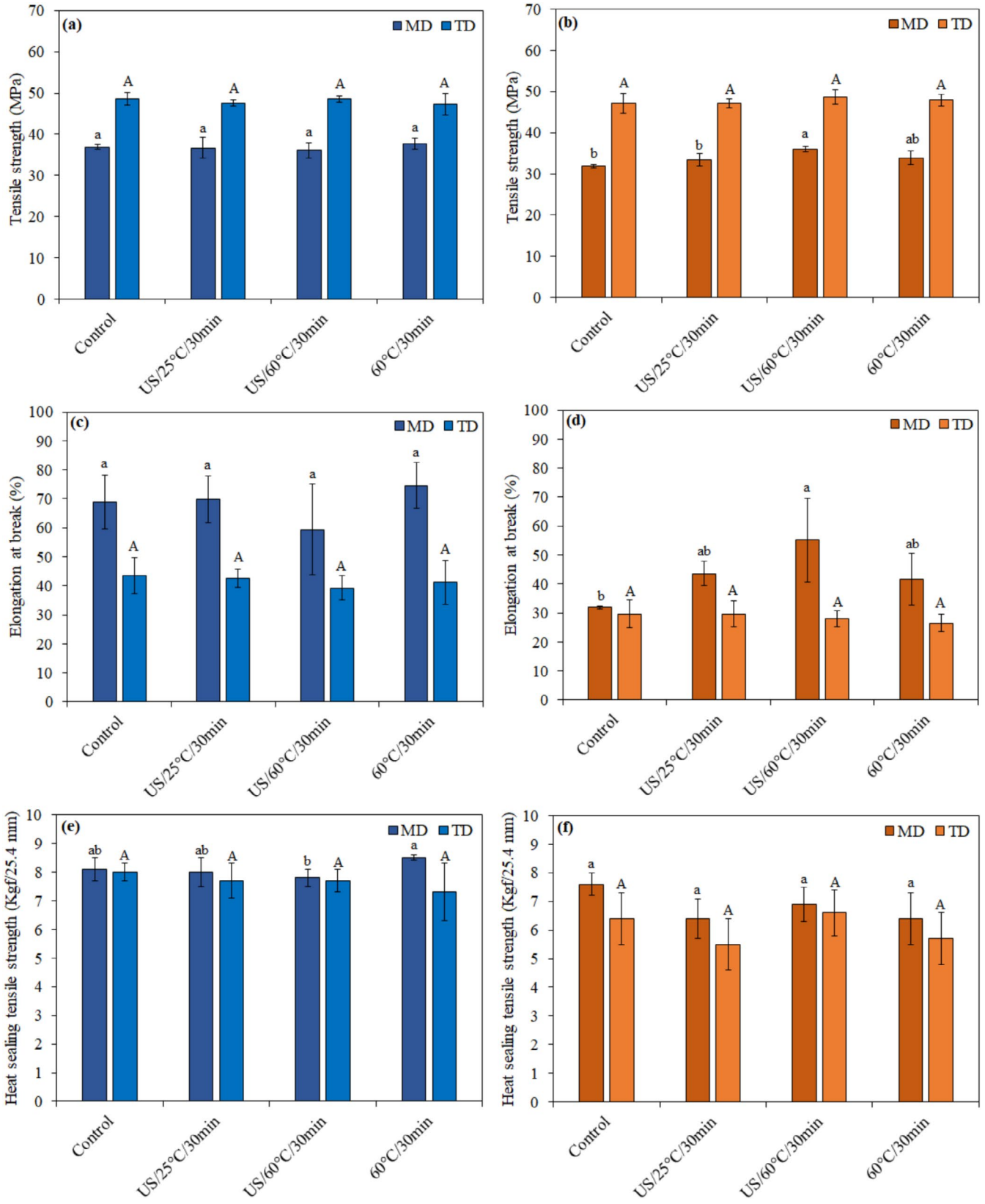

FIGURE 3 | Tensile strength (a and b), elongation at break (c and d), and seal strength (e and f) for various packaging materials processed by ultrasound ($n = 5$). Samples consist of PET/Al/PE (a, c, and e) and PET/Al/PP (b, d, and f), with measurements obtained in both the MD and TD. If lowercase (MD) and uppercase (TD) letters are the same, it indicates no significant difference between those measurements at the 95% confidence level ($p < 0.05$).

strengths were within acceptable ranges. This suggests that the lamination process produced a durable bond, as reflected by the high strength values observed. For the PET/Al/PE films, the various processing conditions—including ultrasound treatment at different temperatures—did not cause significant changes in bond strength, indicating that the adhesive properties remained stable. Conversely, in the case of the PET/Al/PP films, a notable decrease in bond strength between the aluminum layer and the PP film was detected when the samples underwent ultrasound combined with elevated temperature, relative to the control. It is important to highlight that this reduction was still within the range of measurements observed under other processing conditions.

The decrease in bond strength is likely due to the effects of US treatment on the adhesive layer employed during lamination. It is likely that the adhesive lost part of its bonding capability after exposure to the energy applied during the treatment. This phenomenon indicates that the combination of high temperature and acoustic cavitation led to a deterioration of the adhesive's integrity. Nevertheless, it is essential to emphasize that, despite the reduction in bond strength, no defects were detected at the interfaces between the Al and PP. This suggests that the packaging continues to maintain its functionality, as the recorded strength values remain high and suitable for their intended purposes. This observation highlights the robustness of the packaging system, demonstrating its effectiveness even when subjected to rigorous processing conditions.

3.7 | WVTR and OTR

The data for WVTR and OTR of the PET/Al/PE and PET/Al/PP films, measured prior to and following various processing conditions, are summarized in Table 2. In both cases, the films exhibited WVTR and OTR values below the detection thresholds of the instrumentation (less than 0.01 g water m^{-2} day^{-1} for WVTR and less than 0.05 mL (STP)·m^{-2} day^{-1} for OTR), both before and after US treatment. These results corroborate observations made in previous studies, which reported similar performances for laminated films containing aluminum foil [39, 40].

The superiority of the barrier to oxygen and water vapor in Al-laminated films is attributed to the exceptional barrier properties provided by the Al foil [41]. It is important to highlight that multilayer films laminated with Al foil are often subjected to testing against other processing technologies, demonstrating that their barrier properties remain intact even when exposed to high-pressure processing and ohmic heating technologies [36, 40]. This resistance to changes in barrier properties

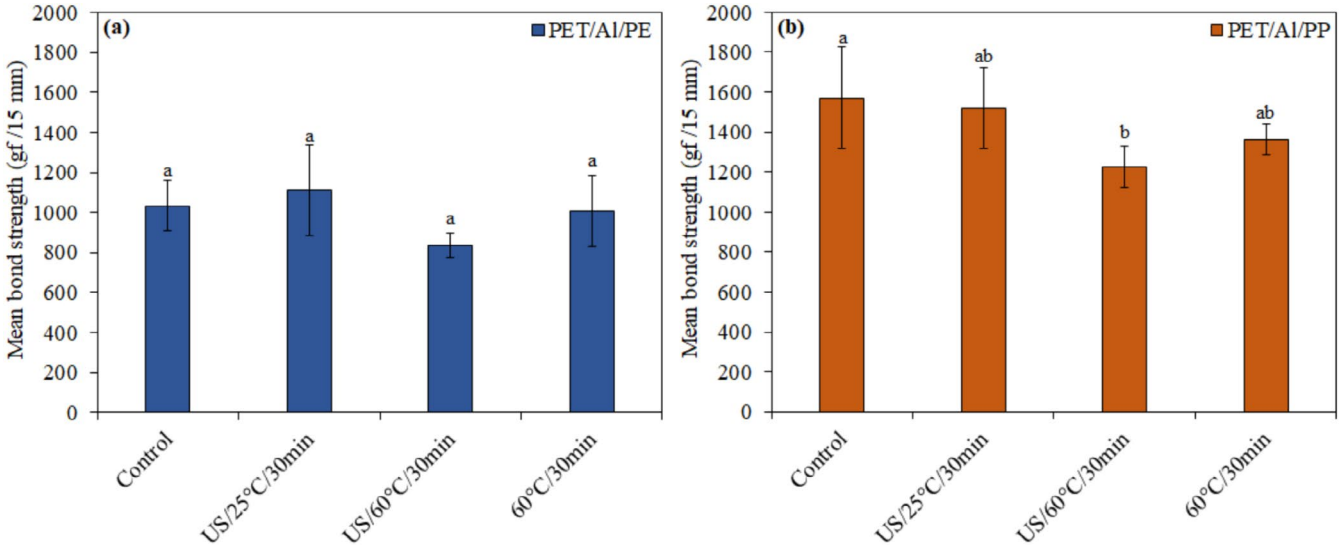

FIGURE 4 | Bond strength values for different packaging materials subjected to ultrasound treatment ($n = 5$) are shown here. The samples include PET/Al/PE (a) and PET/Al/PP (b). Identical lowercase letters indicate no significant difference between groups at a 95% confidence level ($p < 0.05$).

TABLE 2 | WVTR and OTR for various multilayer films processed by US ($n = 2$).

	WVTR (g water m^{-2} day^{-1})[b]		OTR (mL (STP) m^{-2} day^{-1})[c]	
Processing condition	**PET/Al/PE**	**PET/Al/PP**	**PET/Al/PE**	**PET/Al/PP**
Control	< 0.01[a]	< 0.01[a]	< 0.05[a]	< 0.05[a]
US/25°C/30 min	< 0.01[a]	< 0.01[a]	< 0.05[a]	< 0.05[a]
US/60°C/30 min	< 0.01[a]	< 0.01[a]	< 0.05[a]	< 0.05[a]
60°C/30 min	< 0.01[a]	< 0.01[a]	< 0.05[a]	< 0.05[a]

[a]Results that fall below the equipment's quantification limit.
[b]38°C/90% RH.
[c]23°C.

underscores the effectiveness and reliability of aluminum foil laminated films in applications that require protection against gas and moisture permeation, ensuring the preservation of the integrity of the packaged products.

3.8 | Overall, Al and Sb Migration

The laminated packaging containing aluminum foil was subjected to an analysis to assess its safety for food contact, both before and after US processing. For this evaluation, the overall, Al and Sb migrations were considered, which have specific migration limits: 10 mg/dm^2 for overall migration, 1 mg/kg for Al, and 0.04 mg/kg for Sb, as set out by the Resolutions of Anvisa No. 589/21 [42] and No. 326/2019 [43], as well as by European Regulation No. 10/2011 [20]. Al was analyzed due to its presence in the multilayer structure of the laminated packaging, while Sb is used as a catalyst during the PET polymerization process [44–46]. The measurements taken before and after processing indicated that both overall migration, as well as the migration of Al and Sb, were below the detection limits of the employed methods. The reported values were: total migration $\leq$2.33 mg/dm^2, Al $\leq$0.2 µg/L, and Sb $\leq$0.02 µg/L. These data indicate that all applied processing conditions, followed by appropriate conditioning, did not influence the migration of the analyzed materials, thus ensuring that the packaging is safe for food contact when packed products are processed with US under the tested conditions.

The findings comply with the regulatory limits established by both Brazilian law and European standards. Previous studies investigated films composed of PE and PA, which underwent ultrasound treatment at 25 kHz with 9.74 W/L, maintained at 25°C/30–60 min. Tests involving these materials in contact with different food simulants—such as aqueous, acidic, and fatty—revealed overall migration amounts that remained below the detection thresholds of the employed analytical techniques. However, it was observed that US processing influenced the migration of ε-caprolactam [3]. Thus, the importance of assessing the safety of materials intended for food contact under different processing conditions is emphasized, ensuring the protection of public health and compliance with current regulations.

4 | Conclusions

This work processed two flexible laminated packaging materials PET/Al/PE (14/10/55 µm) and PET/Al/PP (14/9/56 µm) with an acidic food simulant (3% acetic acid solution) by ultrasound (25 kHz, 8.75 W/L) at 25°C and 60°C for up to 40 min. The study demonstrated that exposure of the materials to US conditions for periods exceeding 30 min, especially when combined with high temperatures, leads to the perforation of the aluminum foil present in multilayer packaging. Consequently, these materials are not recommended for use under such conditions. Conversely, processing by US for 30 min, regardless of the temperature, did not alter the chemical structure of the polymers, nor did it affect the thermal behavior, seal strength, barrier properties, or migration potential of the analyzed packaging. Além disso, embora o tratamento com US combinado com temperaturas mais altas tenha resultado em nenhuma alteração ou em um ligeiro aumento no alongamento na ruptura do filme de PET/Al/PP, uma diminuição na resistência de união também foi observada neste filme. Essa redução na resistência de união não foi detectada no filme de PET/Al/PE nas mesmas condições. Despite these modifications in mechanical properties, they do not compromise the applicability of these films. Therefore, Al foil-laminated packaging can be used in US processing with high temperatures, provided that the exposure time does not exceed 30 min in the evaluated conditions (ultrasonic power and temperature). This finding reaffirms the versatility and effectiveness of the packaging under different processing conditions while ensuring the preservation of their essential properties.

Author Contributions

Matheus Morandi de Almeida: data curation, formal analysis, writing – original draft, investigation. **Nanci Castanha:** investigation, writing – original draft, data curation, formal analysis. **Paula Fernanda Janetti Bócoli:** data curation, investigation, writing – review and editing. **Paulo Henrique Massaharu Kiyataka:** investigation, methodology, writing – review and editing, data curation. **Beatriz Maria Curtio Soares:** conceptualization, writing – review and editing, validation. **Pedro Esteves Duarte Augusto:** conceptualization, writing – review and editing, methodology, formal analysis. **Fiorella Balardin Hellmeister Dantas:** conceptualization, resources, writing – review and editing, methodology. **Luís Marangoni Júnior:** conceptualization, investigation, funding acquisition, writing – review and editing, visualization, validation, project administration, resources, supervision.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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