



# Morphological, thermal and mechanical properties of polyamide and ethylene vinyl alcohol multilayer flexible packaging after high-pressure processing

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## ABSTRACT

The effect of high-pressure processing on the morphological surface, thermal and mechanical properties of three PA and EVOH based multilayer flexible packaging materials were evaluated. LDPE/PA/LDPE, LDPE/EVOH/LDPE and PET/LDPE/PA/EVOH/PA/LDPE packaging samples were processed under three different conditions (600 MPa/25 °C/10 min, 600 MPa/90 °C/10 min and 0.1 MPa/90 °C/10 min) and an unprocessed sample was used as control. The LDPE/PA/LDPE and LDPE/EVOH/LDPE samples showed good thermal stability and no surface defects after processing, because of the greater thickness of the external material, the film withstood the compression and decompression of the processing, consequently resulting in the protection of the other layers. The PET/LDPE/PA/EVOH/PA/LDPE sample showed a significant increase in the  $T_m$  of PET, LDPE and PA after processing at 600 MPa/90 °C/10 min. Regarding the mechanical properties, reductions in the tensile strength of the PET/LDPE/PA/EVOH/PA/LDPE sample were observed after processing at 600 MPa/90 °C/10 min. In addition, a significant reduction in heat seal strength was observed in the three materials after processing at 600 MPa, but these values are accepted in practice. Therefore, in terms of morphological, thermal and mechanical properties, LDPE/PA/LDPE and LDPE/EVOH/LDPE materials are recommended for the processing conditions of this study, whereas PET/LDPE/PA/EVOH/LDPE materials require further evaluation of other functional properties.

## 1. Introduction

The food and beverage industry has been demanding processing technologies capable of reducing additives while maintaining sensory and nutritional quality, which has stimulated the development of emerging technologies compared to conventional thermal processes such as pasteurization and sterilization (Huang et al., 2017). High-pressure processing (HPP) is a promising alternative for replacing conventional heat treatments, as it can be applied at room temperature, resulting in foods with virtually unchanged nutritional and sensory quality (Mensitieri et al., 2013; Sloan, 1999). One of the limitations to be overcome is the difficulty of inactivating bacterial spores and some

enzymes (Denoya et al., 2015). Therefore, an alternative to overcome the limitations of HPP is pressure-assisted thermal sterilization (PATS), which aims to process foods with low acidity and that are stable at room temperature. This technology involves the simultaneous application of high-pressure (500–700 MPa) and high-temperature (90–120 °C) for a shorter time compared to conventional sterilization treatments, thus reducing the thermal effects on the food processed (Ayvaz et al., 2012; Rastogi et al., 2008).

Another relevant aspect is to know the environmental impacts of some traditional and innovative food preservation technologies in order to provide environmental criteria when selecting food preservation methods as a way to develop more efficient and sustainable food

*Abbreviations:* DSC, Differential Scanning Calorimetry; EVOH, Ethylene Vinyl Alcohol; HPP, High-Pressure Processing; LDPE, Low-Density Polyethylene; MD, Machine Direction; PA, Polyamide; PATS, Pressure-Assisted Thermal Sterilization; PE, Polyethylene; PET, Polyethylene Terephthalate; PP, Polypropylene; RH, Relative Humidity; SEM, Scanning Electron Microscopy; TD, Transverse Direction.

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products throughout the world your life cycle (Pardo and Zufia, 2012). The Pardo and Zufia (2012) study performed the life cycle assessment comparing autoclave pasteurization with high-pressure processing. HPP showed reduced environmental impacts in terms of energy demand and CO<sub>2</sub> emissions compared to conventional pasteurization. In addition, lower water requirements were observed for HPP technology compared to equivalent thermal processes. Fig. 1 shows an example of how high pressure processing can introduce new variables in food processing and contribute to more sustainable processing by reducing the number of processing operations, avoiding overuse of water, gas emissions and energy.

HPP and PATS are in-pack food processing technologies. Therefore, the packaging used must meet a number of requirements. According to Marangoni Júnior et al., (2019) and Dobiáš, and Vápenka (2017) main packaging material requirements for HPP and PATS are: flexibility, dimensional stability, heat seal integrity, head space reduction, heat transfer, proper barrier characteristics and resistance to the total volume changes.

In this context, the flexible material used should be able to withstand the rapid compression and decompression, and provide flexibility to compensate for the collapse of the head space and possible reduction of the food volume within the packaging (Caner et al., 2000; Caner et al., 2004; Galotto et al., 2009; Schauwecker et al., 2002). However, irreversible changes to the materials may occur during processing, such as visible deformation, possibly impacting the packaging's functionality and visual appearance (Richter et al., 2010). In addition, the choice of flexible packaging materials must ensure that processing will not affect the integrity of the heat seal and its mechanical properties (Marangoni Júnior et al., 2019, 2020), since failures in the sealing regions could allow pressurization fluid to enter, consequently contaminating or leaking the product (Koutchma et al., 2010).

The suitable flexible and/or semi-rigid packages for HPP comprise the polymer bottles (made of polyethylene terephthalate or high density polyethylene) or the packages formed from layered polymer films (e.g. sachets, thermoformed or semi-rigid trays sealed with a film). Flexible packaging materials include mostly multilayer films formed with a sealable layer (polyolefin), a barrier ply (polyamides, polyesters, metalized polymer films, aluminum foil, ethylene vinyl alcohol copolymers) and sometimes with a mechanically resistant outside layer (polyamides, polyesters) (Dobiáš and Vápenka, 2017).

Ethylene vinyl alcohol (EVOH) and polyamides (PA) are widely used in the packaging of in-pack processed foods. These materials work as an

excellent gas barrier and have high thermal resistance as well as good optical characteristics. However, the hydrophilic nature of EVOH and PA causes a significant decrease in their oxygen barrier properties when exposed to an environment with high relative humidity (López-Rubio et al., 2005; Robertson, 2013). For this reason, in most food packaging applications, EVOH and PA are used in multilayer structures between at least two layers of a hydrophobic material (inner and outer layer), such as polyethylene (PE) or polypropylene (PP), e. g. PE/PA/PE and PE/EVOH/PE.

Therefore, evaluating the influence of HPP and PATS on the properties of multilayer structures containing EVOH and PA is fundamental to guide their use towards these applications. The aim of this research was to evaluate the effect of different conditions of high-pressure processing and pressure-assisted thermal sterilization on the morphological, thermal and mechanical properties of EVOH and PA based multilayer flexible packaging materials.

## 2. Materials and methods

### 2.1. Materials

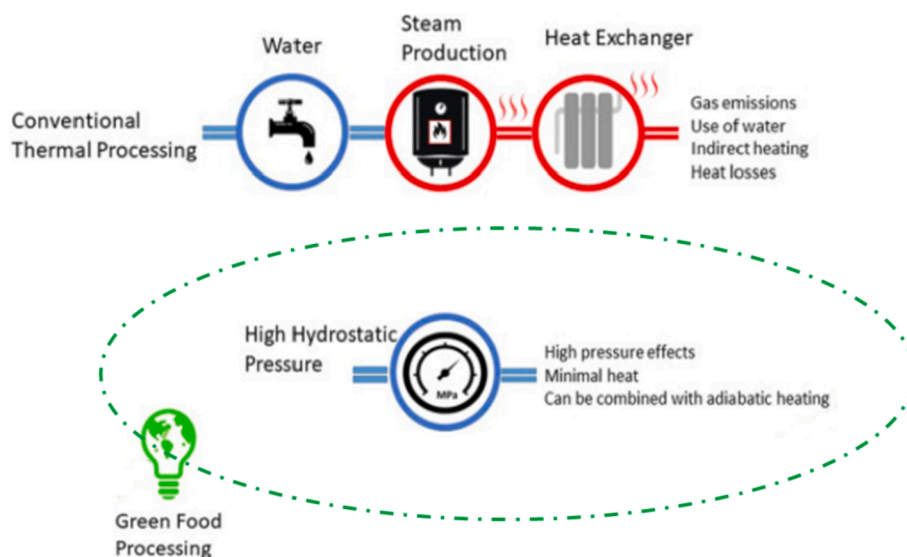
Three different multilayer flexible materials containing PA and/or EVOH were used to produce 80 × 150 mm samples that were sealed on three sides with an electric impulse sealing machine (Haramura – A380 Regente, São Paulo, Brazil). (Table 1). Then, the samples were filled with 70 mL of distilled water and vacuum sealed (Selovac – 200, São Paulo, Brazil), minimizing their head space.

The LDPE/PA/LDPE film is obtained through the co-extrusion process, being the PA layer for oxygen barrier and vacuum maintenance in the packaging and the last LDPE layer for heat sealing. This material is used in packaging for meat products and cheese in general under vacuum. LDPE/EVOH/LDPE is obtained by the co-extrusion process, being the EVOH layer for oxygen barrier and the last LDPE layer for heat

**Table 1**  
Packaging materials used in the study.

Packaging material	Total thickness (µm)	Partial thickness (µm)
LDPE/PA/LDPE	100	40/18/42
LDPE/EVOH/LDPE	70	30/11/29
PET/LDPE/PA/EVOH/PA/LDPE	65	13/16/5/4/5/22

Values referring to the mean of 5 determinations in 5 specimens.



**Fig. 1.** Reduction in the number of processing operations and environmental footprint through the use of high pressure processing (adapted from Fasolin et al. (2019)).

sealing. In general this material is intended for packaging of meat products and cheese. PET/LDPE/PA/EVOH/PA/LDPE is also obtained by co-extrusion of the 6 layers, the PET layer being for gloss, printing and mechanical strength, the internal LDPE being an adhesive; PA and EVOH layers for oxygen barrier and the last LDPE layer for heat sealing. Material used as film for thermoformed tray lids for meat products and sliced cheeses, and can be used in modified atmosphere packaging systems.

## 2.2. Processing

The experiments were performed on a high-pressure pilot equipment (QFP 2L-700, Avure Technologies, OH, USA), operating at pressures up to 690 MPa and at temperatures up to 90 °C. The temperature of the equipment's chamber and the initial water temperature were adjusted for the different processing conditions, taking into account the rate of temperature increase under adiabatic conditions (3 °C/100 MPa). The samples were processed at 600 MPa/90 °C/10 min to evaluate the synergistic effect of pressure and high temperature, and at 600 MPa/25 °C/10 min to evaluate the effect of pressure at room temperature. In addition, samples processed at 0.1 MPa/90 °C/10 min in an ultra-thermostatic bath were evaluated to assess the effect of temperature (MA184, Marconi, Piracicaba, Brazil). After processing, the water contained within the packaging was discarded, unprocessed samples (control) were prepared for comparative purposes, duplicates of all experiments were performed, and the samples were conditioned at 23 ± 2 °C until the time of analysis.

## 2.3. Surface morphological properties

The samples underwent a visual evaluation after processing to identify the presence of defects on the materials' surface, as described by Sarantopoulos and Teixeira (2017). In addition, a scanning electron microscope (Zeiss – DSM 940A, Jena, Germany) was used to analyze the surface of the films before and after processing. The samples were prepared in 10 × 10 mm formats of each material. The SEM observations were performed with up to 5000× magnification using a secondary electron detector (relief contrast, image topography, SE) and a back-scattered electron detector (atomic weight contrast, identification of similarity by hue, BSE), the working distance was 25 mm and the high voltage was 5 keV (Goldstein et al., 1992). Before the analysis, the samples were coated with gold on a metallizer (Balzers – SCD 050, Balzers, Liechtenstein).

## 2.4. Thermal properties

Differential scanning calorimetry (DSC) was performed using a calorimeter (TA Instruments – DSC 250, New Castle, USA) according to the ASTM D 3418–15 (ASTM INTERNATIONAL, 2015a) methodology, at a heating rate of 20 °C/min in the range from –70 to 300 °C. The sample weight was approximately 10 mg, and the determinations were performed under dry nitrogen purging. Melting temperature ( $T_m$ ) and melting enthalpy ( $\Delta H$ ) were estimated based on the DSC thermograms. The results were the mean of 3 repetitions.

## 2.5. Mechanical properties

The samples were conditioned for 48 h at 23 ± 2 °C and 50 ± 5% RH in a laboratory with controlled temperature and humidity conditions to meet the mechanical properties testing conditions specified in ASTM standards. The determinations were made with 25 mm wide specimens, cut with high-precision equipment to avoid burrs (RDS-100-C, Chem-Instruments, OH, USA). All tests were performed at 23 ± 2 °C and 50 ± 5% RH, with five repetitions.

### 2.5.1. Tensile strength

The tensile properties were determined according to ASTM D882-18 (2018), using a universal testing machine (Instron, 5966-E2, Norwood, USA) operating with 1 kN load cell. The test speed was 500 mm min<sup>-1</sup>, and the distance between test devices in the machine direction was 50 mm, while in the transverse direction, it was 25 mm.

### 2.5.2. Heat sealing tensile strength

The heat seal strength was determined according to ASTM F88/F88M (2015b), using a universal testing machine (Instron, 5966-E2, Norwood, USA) operating with 1 kN load cell at a speed of 300 mm min<sup>-1</sup>. The distance between test devices was 25 mm.

## 2.6. Statistical analysis

The results were statistically evaluated using the analysis of variance (ANOVA) and Tukey's test for comparison between mean values ( $p < 0.05$ ).

## 3. Results and discussion

### 3.1. Surface morphological properties

The samples underwent a visual evaluation before and after processing. No defects visible to the eye were detected after processing in ambient light. Regarding the scanning electron microscopy's results (Fig. 2), the LDPE/PA/LDPE and LDPE/EVOH/LDPE films did not show superficial microscopic defects after processing, differing from the results obtained in the study by Galotto et al. (2010), where the LDPE/EVOH/LDPE film showed surface defects after processing at 400 MPa/60 °C/30 min. In relation to the PET/LDPE/PA/EVOH/PA/LDPE film after processing at 600 MPa, microscopic defects were observed on the material's surface as shown in Figure C2 and C3, that is, the defects were probably caused by the stresses on the outer layer of PET, influenced by compression and decompression during high pressure processing.

### 3.2. Thermal properties

Table 2 shows the melting temperature ( $T_m$ ) results of each material and the total melting enthalpy ( $\Delta H$ ) of the films before and after processing. The LDPE/PA/LDPE and LDPE/EVOH/LDPE samples showed no significant difference ( $p < 0.05$ ) in thermal properties ( $T_m$  and total  $\Delta H$ ) after processing at 600 MPa compared to the controls. For the PET/LDPE/PA/EVOH/PA/LDPE sample, no significant increase ( $p < 0.05$ ) in the EVOH layer's  $T_m$  was observed after processing at 600 MPa. However, there was a significant ( $p < 0.05$ ) increase in the melting temperature of PET, LDPE and PA after processing at 600 MPa/90 °C/10 min compared to the control and to processing at 600 MPa/25 °C/10 min.

Similar results were found for EVOH's  $T_m$  in other studies, such as those by Dhawan et al. (2011), who evaluated PET/EVOH/PP and PET/PP/PA/EVOH/PA/PP films processed at 680 MPa/100 °C/5 min, the study by Ghamdi et al. (2019), with PA/EVOH/PP structures processed at 400 and 600 MPa/30, 60 and 90 °C/6 min, and the study by López-Rubio et al. (2005), with PE/EVOH/PE films processed at 400 and 800 MPa/40 and 75 °C/5 and 10 min, i.e., this study, using processing conditions and structures different from those already reported in the literature, has shown that high-pressure processing conditions (600 MPa) do not influence EVOH's melting temperature. This differs from the results obtained by Galotto et al. (2008), who observed a decrease in EVOH's  $T_m$  when in contact with an aqueous simulant and processed at 400 MPa/20 and 60 °C/30 min. The longer processing time may have caused these modifications.

The most severe changes in the PET/LDPE/PA/EVOH/PA/LDPE samples'  $T_m$  occurred in PA and may be justified by the complexity of the

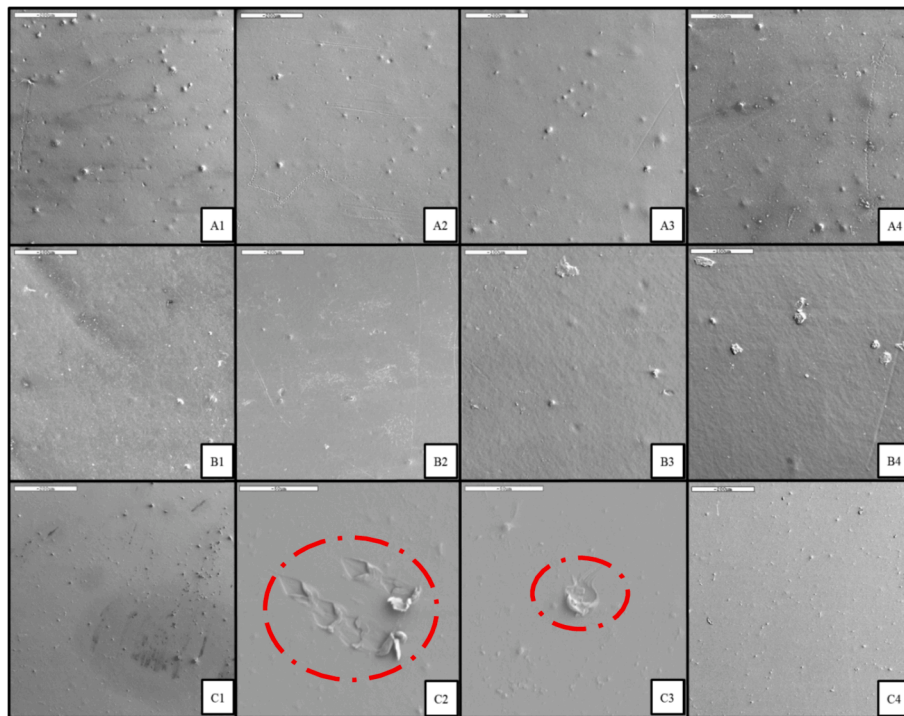


Fig. 2. Surface morphology (Scanning Electron Microscopy – SEM with SE detector) of the samples. A = LDPE/PA/LDPE, B = LDPE/EVOH/LDPE, C = PET/LDPE/PA/EVOH/PA/LDPE, 1 = Control, 2 = 600 MPa/25 °C/10 min, 3 = 600 MPa/90 °C/10 min and 4 = 0.1 MPa/90 °C/10 min, where A1, A2, A3, A4, C1 and C4, bar = 200  $\mu$ m, B1, B2, B3 and B4, bar = 100  $\mu$ m and C2 and C3, bar = 50  $\mu$ m.

structure and its exposure to a humid environment, in addition to the influence of high-pressure and high temperature. According to Parodi et al. (2017), PA has polar monomers, the amide and carbonyl groups that can form hydrogen bonds between chains, leading to high resistance. Therefore, if the polymer absorbs moisture when in contact with a humid environment, part of the hydrogen bonds are broken and new hydrogen bonds are formed with the water molecules, resulting in a plasticizing phenomenon. This results in the modification of the mobility of the chains, causing a decisive impact on the stress-strain response (Miri et al., 2019). Consequently, it affects the thermal properties of the material and possibly causing a considerable deterioration of its mechanical properties, which can be seen in the tensile strength results of the next section. In addition, PA's melting temperature may change with the increase in heating and pressure cycles. This change can be explained by alterations in the degree of crystallinity and redistribution in the size of the polymer's crystals after processing, as described by Galotto et al. (2009); Radlmaier et al. (2017). In addition, we emphasize that the different  $T_m$  values of the same polymer, but in a different sample, is attributed to the fact that each packaging material was supplied by different manufacturers, and by industry secrecy we do not have accurate information about the resin grade, additives and process conditions used in the manufacture of films.

### 3.3. Mechanical properties

The maximum tensile strength of the LDPE/PA/LDPE and LDPE/EVOH/LDPE films showed no significant difference ( $p < 0.05$ ) after processing at 600 MPa/25 °C and 90 °C/10 min in both directions of the material (MD and TD) when compared to the control (Table 3). Similar results were obtained by Galotto et al. (2008), who also detected no significant differences in tensile strength in both directions of the PE/EVOH/PE structure at 400 MPa/20 °C and 60 °C/30 min.

The PET/LDPE/PA/EVOH/LDPE film showed a significant reduction ( $p < 0.05$ ) in tensile strength after processing at 600 MPa/90 °C for 10 min compared to the control, from  $46.2 \pm 1.0$  MPa to  $41.2 \pm 2.3$  MPa in

the MD and from  $52.4 \pm 2.1$  MPa to  $47.2 \pm 0.9$  MPa in the TD. The same processing condition differed significantly ( $p < 0.05$ ) from the results of the films processed at 600 MPa/25 °C/10 min, but there was no difference ( $p < 0.05$ ) from the results of the films processed at 0.1 MPa/90 °C/10 min. The results obtained in this study show that the reduction in the PET/LDPE/PA/EVOH/LDPE film's tensile strength values in both directions of the material was mainly influenced by the combination of high processing pressure (600 MPa) and temperature (90 °C). However, these results have no negative impact in practice, as variations of up to 25% in this property are acceptable to industries (Galotto et al., 2008; Lambert et al., 2006).

Regarding the elongation in the maximum strength of the materials, we observed that for the LDPE/PA/LDPE film, there was a significant increase ( $p < 0.05$ ) of 8.6% in the results in the MD after processing at 600 MPa/90 °C/10 min compared to the control (Table 4). In the LDPE/EVOH/LDPE and PET/LDPE/PA/EVOH/LDPE films, the results showed no significant difference ( $p < 0.05$ ) in both directions of the material after processing at 600 MPa/25 °C and 90 °C/10 min compared to the control.

Heat seal strength is a critical feature of flexible packaging in high pressure food processing. The results for the LDPE/PA/LDPE sample showed no significant difference ( $p < 0.05$ ) after processing at 600 MPa/25 °C and 90 °C for 10 min in the MD compared to the control (Table 5). However, it was significantly lower ( $p < 0.05$ ) in the TD after processing at 600 MPa/25 °C/10 min and 600 MPa/90 °C/10 min compared to the control and to the material processed at 0.1 MPa/90 °C/10 min, the heat seal strength of which decreased from  $1826 \pm 53.4$  N/m to  $1704 \pm 34.3$  N/m and  $1678 \pm 31.8$  N/m, respectively. It may be concluded that these results were mainly influenced by the compression and decompression efforts caused by the high processing pressure (600 MPa). These results are similar to those obtained by Lambert et al. (2000), who observed significant changes in the PA/LDPE film's heat seal strength after processing at 500 MPa/25 °C/30 min.

For the LDPE/EVOH/LDPE film, significant reductions ( $p < 0.05$ ) in heat seal strength were observed in both directions of the material after

**Table 2**  
DSC results of each material before and after processing.

LDPE/PA/LDPE					
Material	Thermal property	Processing condition			
		Control	600 MPa 25 °C/10 min	600 MPa 90 °C/10 min	0.1 MPa 90 °C/10 min
LDPE	T <sub>m</sub> (°C)	121.1 ± 0.1 <sup>a</sup>	122.7 ± 0.7 <sup>a</sup>	121.5 ± 2.8 <sup>a</sup>	120.9 ± 0.4 <sup>a</sup>
PA	T <sub>m</sub> (°C)	184.6 ± 4.2 <sup>a</sup>	186.3 ± 4.7 <sup>a</sup>	179.1 ± 3.2 <sup>a</sup>	177.6 ± 4.0 <sup>a</sup>
	ΔH <sup>a</sup> (J/g)	72.1 ± 1.6 <sup>a</sup>	68.7 ± 1.9 <sup>a</sup>	62.6 ± 6.4 <sup>a</sup>	69.9 ± 3.2 <sup>a</sup>
LDPE/EVOH/LDPE					
Material	Thermal property	Processing condition			
		Control	600 MPa 25 °C/10 min	600 MPa 90 °C/10 min	0.1 MPa 90 °C/10 min
LDPE	T <sub>m</sub> (°C)	124.2 ± 0.1 <sup>a</sup>	124.8 ± 0.4 <sup>a</sup>	124.9 ± 0.7 <sup>a</sup>	124.2 ± 0.2 <sup>a</sup>
EVOH	T <sub>m</sub> (°C)	177.1 ± 0.2 <sup>a</sup>	181.5 ± 5.4 <sup>a</sup>	170.2 ± 3.6 <sup>a</sup>	172.0 ± 6.6 <sup>a</sup>
	ΔH <sup>a</sup> (J/g)	71.7 ± 0.9 <sup>ab</sup>	67.7 ± 1.3 <sup>b</sup>	68.0 ± 2.4 <sup>b</sup>	76.5 ± 3.2 <sup>a</sup>
PET/LDPE/PA/EVOH/PA/LDPE					
Material	Thermal property	Processing condition			
		Control	600 MPa 25 °C/10 min	600 MPa 90 °C/10 min	0.1 MPa 90 °C/10 min
PET	T <sub>m</sub> (°C)	255.7 ± 0.2 <sup>b</sup>	253.8 ± 1.0 <sup>b</sup>	259.1 ± 0.7 <sup>a</sup>	258.1 ± 0.6 <sup>a</sup>
LDPE	T <sub>m</sub> (°C)	120.5 ± 0.1 <sup>b</sup>	120.6 ± 1.9 <sup>b</sup>	123.9 ± 1.4 <sup>a</sup>	122.4 ± 0.4 <sup>ab</sup>
PA	T <sub>m</sub> (°C)	179.8 ± 2.9 <sup>b</sup>	174.4 ± 2.0 <sup>b</sup>	222.5 ± 2.5 <sup>a</sup>	221.7 ± 5.5 <sup>a</sup>
EVOH	T <sub>m</sub> (°C)	153.3 ± 7.6 <sup>bc</sup>	140.6 ± 1.5 <sup>c</sup>	161.9 ± 1.7 <sup>ab</sup>	167.1 ± 2.3 <sup>a</sup>
	ΔH <sup>a</sup> (J/g)	50.9 ± 2.3 <sup>b</sup>	49.0 ± 2.4 <sup>b</sup>	50.9 ± 2.4 <sup>ab</sup>	59.3 ± 2.3 <sup>a</sup>

Values referring to the mean of three repetitions ± standard deviation.  
a,b,c means followed by the same letter in the line do not differ at the 95% confidence level (p < 0.05).

<sup>a</sup> Total melting enthalpy (ΔH = ΔH<sub>LDPE</sub> + ΔH<sub>PA</sub>, ΔH = ΔH<sub>LDPE</sub> + ΔH<sub>EVOH</sub> e ΔH = ΔH<sub>PET</sub> + ΔH<sub>LDPE</sub> + ΔH<sub>PA</sub> + ΔH<sub>EVOH</sub>).

**Table 3**  
Tensile strength (MPa) of different packaging materials processed by high pressure.

Packaging material	Direction of the material	Processing condition			
		Control	600 MPa 25 °C/ 10 min	600 MPa 90 °C/ 10 min	0.1 MPa 90 °C/ 10 min
LDPE/PA/LDPE	MD	28.0 ± 1.7 <sup>a</sup>	27.3 ± 2.4 <sup>a</sup>	30.0 ± 0.8 <sup>a</sup>	29.5 ± 1.0 <sup>a</sup>
	TD	28.1 ± 2.3 <sup>a</sup>	26.3 ± 2.1 <sup>a</sup>	27.9 ± 0.6 <sup>a</sup>	26.5 ± 2.3 <sup>a</sup>
LDPE/EVOH/LDPE	MD	23.3 ± 1.9 <sup>a</sup>	21.2 ± 0.7 <sup>ab</sup>	21.9 ± 0.6 <sup>ab</sup>	21.6 ± 0.4 <sup>b</sup>
	TD	21.5 ± 0.6 <sup>a</sup>	21.4 ± 0.6 <sup>a</sup>	21.2 ± 0.5 <sup>a</sup>	20.8 ± 1.8 <sup>a</sup>
PET/LDPE/PA/EVOH/PA/LDPE	MD	46.2 ± 1.0 <sup>a</sup>	44.3 ± 2.4 <sup>a</sup>	41.2 ± 2.3 <sup>b</sup>	44.4 ± 1.4 <sup>ab</sup>
	TD	52.4 ± 2.1 <sup>a</sup>	50.9 ± 2.5 <sup>a</sup>	47.2 ± 0.9 <sup>b</sup>	50.4 ± 2.0 <sup>ab</sup>

MD: machine direction and TD: transverse direction.  
Values referring to the mean of five repetitions ± standard deviation.  
a,b,c means followed by the same letter in the line do not differ at the 95% confidence level (p < 0.05).

**Table 4**  
Elongation at maximum strength (%) of different packaging materials processed by high pressure.

Packaging material	Direction of the material	Processing condition			
		Control	600 MPa 25 °C/ 10 min	600 MPa 90 °C/ 10 min	0.1 MPa 90 °C/ 10 min
LDPE/PA/LDPE	MD	604.6 ± 28.0 <sup>b</sup>	643.0 ± 16.4 <sup>ab</sup>	661.6 ± 20.8 <sup>a</sup>	638.5 ± 35.3 <sup>ab</sup>
	TD	892.3 ± 47.8 <sup>a</sup>	886.6 ± 53.1 <sup>a</sup>	937.6 ± 12.6 <sup>a</sup>	887.6 ± 44.6 <sup>a</sup>
LDPE/EVOH/LDPE	MD	527.3 ± 45.7 <sup>b</sup>	556.6 ± 15.9 <sup>ab</sup>	558.3 ± 10.2 <sup>ab</sup>	579.8 ± 23.5 <sup>a</sup>
	TD	715.9 ± 29.2 <sup>b</sup>	776.2 ± 31.6 <sup>ab</sup>	755.2 ± 25.1 <sup>ab</sup>	824.9 ± 77.1 <sup>a</sup>
PET/LDPE/PA/EVOH/PA/LDPE	MD	108.5 ± 10.2 <sup>a</sup>	100.8 ± 9.2 <sup>a</sup>	99.1 ± 9.3 <sup>a</sup>	103.8 ± 8.4 <sup>a</sup>
	TD	120.3 ± 17.7 <sup>a</sup>	122.3 ± 14.2 <sup>a</sup>	110.3 ± 15.3 <sup>a</sup>	115.3 ± 4.0 <sup>a</sup>

MD: machine direction and TD: transverse direction.  
Values referring to the mean of five repetitions ± standard deviation.  
a,b,c means followed by the same letter in the line do not differ at the 95% confidence level (p < 0.05).

**Table 5**  
Heat sealing tensile strength (N/m) of the different packaging materials processed by high pressure.

Packaging material	Direction of the material	Processing condition			
		Control	600 MPa 25 °C/ 10 min	600 MPa 90 °C/ 10 min	0.1 MPa 90 °C/ 10 min
LDPE/PA/LDPE	MD	1833 ± 62.5 <sup>ab</sup>	1759 ± 64.9 <sup>b</sup>	1741 ± 59.4 <sup>b</sup>	1883 ± 36.1 <sup>a</sup>
	TD	1826 ± 53.4 <sup>a</sup>	1704 ± 34.3 <sup>b</sup>	1678 ± 31.8 <sup>b</sup>	1896 ± 67.0 <sup>a</sup>
LDPE/EVOH/LDPE	MD	1274 ± 89.6 <sup>a</sup>	1102 ± 44.3 <sup>b</sup>	1113 ± 71.2 <sup>b</sup>	1130 ± 61.3 <sup>b</sup>
	TD	1315 ± 34.7 <sup>a</sup>	1053 ± 10.7 <sup>c</sup>	1082 ± 8.7 <sup>c</sup>	1159 ± 15.4 <sup>b</sup>
PET/LDPE/PA/EVOH/PA/LDPE	MD	2973 ± 55.6 <sup>a</sup>	2693 ± 48.1 <sup>b</sup>	2713 ± 160.4 <sup>b</sup>	2805 ± 71.2 <sup>ab</sup>
	TD	3028 ± 206.9 <sup>a</sup>	3163 ± 228.5 <sup>a</sup>	3167 ± 105.4 <sup>a</sup>	3205 ± 157.8 <sup>a</sup>

MD: machine direction and TD: transverse direction.  
Values referring to the mean of five repetitions ± standard deviation.  
a,b,c means followed by the same letter in the line do not differ at the 95% confidence level (p < 0.05).

being subjected to all different processing conditions. The PET/LDPE/PA/EVOH/PA/LDPE film showed a significant reduction (p < 0.05) in heat seal strength in the MD after processing at 600 MPa/90 °C/10 min when compared to the control film. This result can be attributed to the synergistic effect of high temperature and high-pressure of processing.

However, it should be noted that although in some cases a reduction in heat seal strength was observed after processing, the samples were visually evaluated before and after processing and no leakage through the sealing area was detected. In addition, the heat seal strength's results may vary up to 25% in practice (Lambert et al., 2000).

#### 4. Conclusions

The LDPE/PA/LDPE and LDPE/EVOH/LDPE materials showed good thermal stability after being subjected to all different processing conditions, and no surface defects were detected. The PET/LDPE/PA/EVOH/PA/LDPE material showed changes in the melting temperatures of PET, LDPE and PA, influenced by the high temperature and high-

pressure of processing at 600 MPa/90 °C/10 min. It is important to evaluate in future studies whether these modifications will impact the component migration and barrier properties of the materials' polymers. Some changes in the mechanical properties of the three materials evaluated were observed after processing, but these values are accepted in practice by industries as they do not affect the packaging's performance. Therefore, in terms of morphological, thermal and mechanical properties, LDPE/PA/LDPE and LDPE/EVOH/LDPE films are recommended for the HPP and PATS conditions used in this study, and the PET/LDPE/PA/EVOH/LDPE film requires further evaluation of other functional properties of the material to learn if, in practice, the changes found in this study may have any influence on food processing, handling, transport, distribution and stability. In addition, these multilayer systems feature stand out for the cohesion property that ensures the transparency of packaging.

#### Declaration of competing interest

The authors declare that they have no conflict of interest.

#### CRediT authorship contribution statement

**Luís Marangoni Júnior:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing - original draft, Writing - review & editing. **Léa Mariza de Oliveira:** Conceptualization, Investigation, Methodology, Writing - original draft. **Paula Fernanda Janetti Bócoli:** Conceptualization, Methodology, Formal analysis, Investigation. **Marcelo Cristianini:** Conceptualization, Investigation, Methodology, Writing - original draft. **Marisa Padula:** Conceptualization, Investigation, Methodology, Supervision, Writing - original draft. **Carlos Alberto Rodrigues Anjos:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Writing - original draft.

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