

## Microstructural changes of some multilayer polymer films applied in patp food treatment

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### Abstract

Pressure Assisted Thermal Processing (PATP) provides an alternative to classical processing that can provide safe products without loss of sensory quality or nutrients. The objective of the present work was to study the behaviour of two commercially available multilayer polymeric films exposed to PATP treatment in order to assess their usefulness as food packaging materials. The microstructural changes during the treatment were monitored by Scanning Electron Microscopy (SEM). Two commercially multilayer polymeric films: I (combination of biaxially oriented polyamide with coextruded barrier film of the structure PE/EVOH/PE) and II (coextruded high barrier multilayer film with the structure PA/EVOH/PA/PE) were PATP treated (600 MPa for 10 min. at 70°C). The surface topography was evaluated after the PATP and compared to the control samples. The results showed that the combined pressure-heat treatment can compromise the integrity of tested multilayer films

**Keywords:** Pressure-assisted thermal processing, scanning electron microscopy, food packaging films

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### 1. Introduction

The thermal food treatment is a conventionally technique for ensuring the microbiological safety of food products [1-2]. This treatment leads to unwanted changes in the foods' sensory attributes or to low nutritional value of the food products [1-4]. In response to consumer demand, novel food preservation techniques such as: High Hydrostatic Pressure, Pulsed Electric Fields, High Voltage Arc Discharge, Cold Plasma and Pressure-Assisted Thermal Processing (PATP) have received considerable attention during the last few decades [1-6]. Among these alternative technologies, PATP is the most investigated one due to its advantages, including: greater retention of colour, flavour and nutrients [7]. On the other hand, simultaneous application of high pressure and temperature treatments can produce undesirable effects on multilayer polymeric films [5]. Moreover, the choice of the polymeric films to be used in PATP should guarantee that the high pressure associated with the heat treatment do neither affect package

integrity nor its functional properties [1, 10]. Very limited information is available on impact of combined pressure-heat treatment on different polymeric films during PATP treatment [6, 10]. In fact, PATP treatment has been proven to produce in some cases undesirable effects on food packaging films [10] such as: delamination phenomenon and unacceptable modifications of the integrity of the packaging structure [5]. In this case, the selection of the packaging structures for PATP processing has an extreme importance to food manufacturers in terms of correct processing, shelf-life enhancement, economy and marketing [5]. In this research it was investigated the influence of PATP treatment (600 MPa, 70 °C/10 min) on the behaviour of two commercially packaging films. The microstructure was qualitatively studied, aiming to clarify the impact of PATP processing on the structure of the packaging films.

## 2. Materials and methods

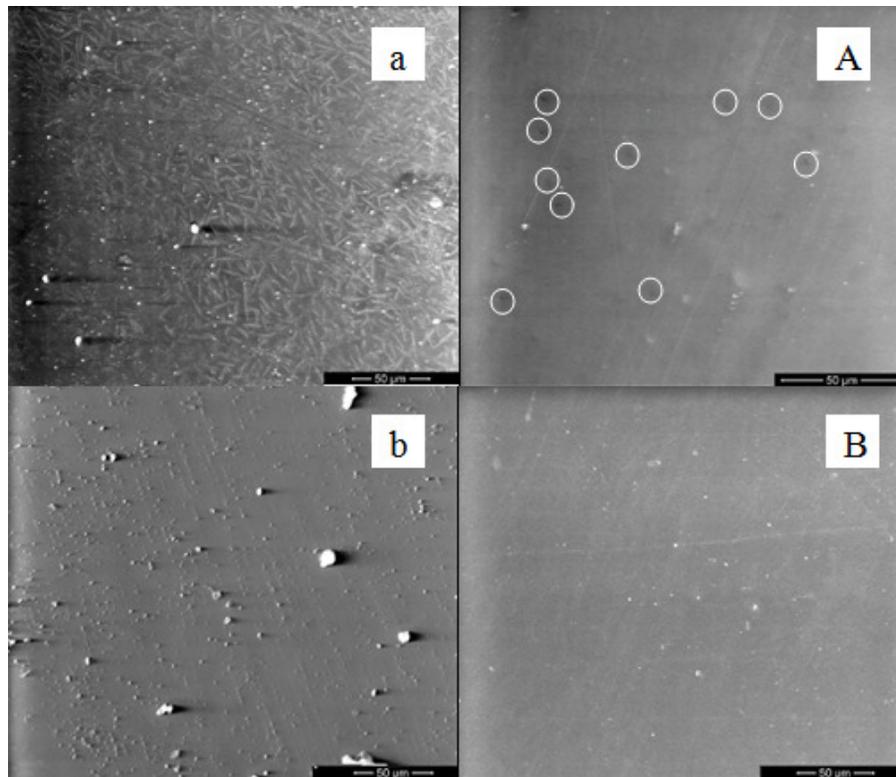
Two commercially available multilayer polymeric films were analyzed for their performance under PATP conditions: I (combination of biaxially oriented polyamide with coextruded barrier film of the structure PE/EVOH/PE) and II (coextruded high barrier multilayer film with the structure PA/EVOH/PA/PE). To perform the PATP experiment, the polymeric films were treated at PATP in a pilot unit built by Resato (Holland). The PATP treatment at constant pressure of 600 MPa was carried out at 70°C for 10 min. Six hundred MPa pressure is considered by many authors as threshold value and also is considered to be economical and microbiologically safe at the pasteurization level [6-8]. and combined with high temperatures, such as the ones selected for the current experiment, it lead to a sterilization effect. PATP treatment was carried out in duplicate for each type of multilayer film. Immediately after PATP processing the films' surface and their microstructure were investigated using a Quanta 200 microscope (FEI Company, Holland) with an operating voltage of 20 KV [9].

To obtain detailed and accurate SEM images, the samples had to be left washed. The films were observed at a 500x magnification.

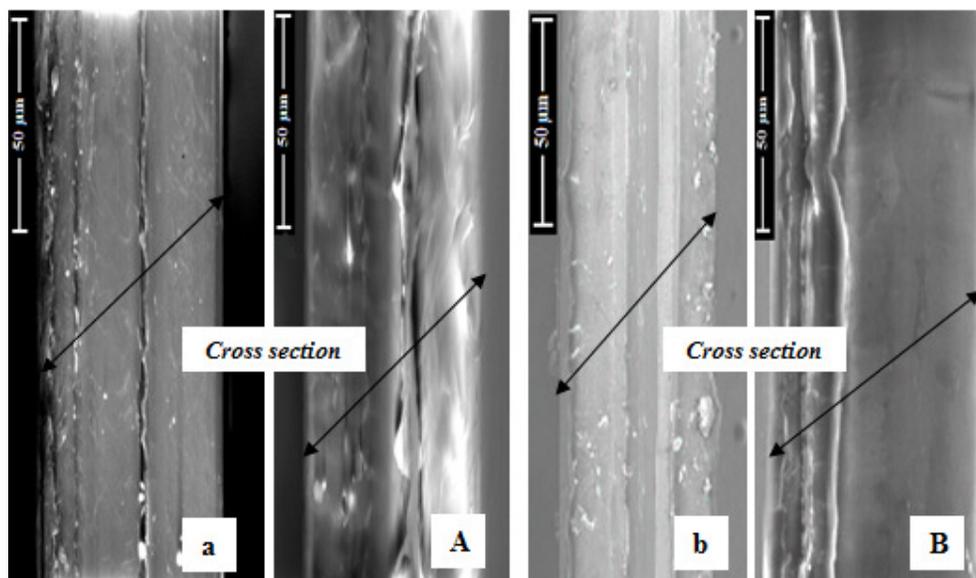
## 3. Results and Discussion

Scanning electron microscopy was used to observe the surface's topography and the structure of the multilayer films. A microstructural study of the tested multilayer films gives relevant information, allowing for a better characterization of the films. Figure 1 illustrates the surface of the multilayer films before (lower case) and after PATP treatment (upper case).

The control films show homogeneous surfaces, typical for a polymer [10]. In addition, in figures 1a and 1b it can be seen that the both untreated films have some irregularities present on the surface. Both the processed films structure I (Figure 1A) and II (Figure 1B) are heterogeneous and the fibers are not visible. The I processed film has an uneven surface with dark spots - indicated by white circles in Figure 1A. The II processed film presents a heterogeneous surface with white spots (Figure 1B). The micrographs of sections of multilayer films before and after PATP processing are presented in Figure 2.



**Figure 1.** The surface of multilayer films observed by SEM: (a) control I; (A) processed I; (b) control II; (B) processed II.



**Figure 2.** The section of multilayer films observed by SEM: (a) control I; (A) processed I; (b) control II; (B) processed II.

The microstructure was qualitatively studied, in order to clarify the impact of PATP processing on multilayer structure. We have noticed several changes in microstructural characteristics of processed II film compared to I processed film. The sections of untreated films (indicated by arrows) display a multilayer structure with a compact region at the film-air interface. The untreated films' cross sections show well defined layers with individual parallel fiber (Figures 2a and 2b). From the micrographs of processed films' sections is obvious that PATP treatment induces qualitative structural changes (Figures 2A and 2B). The I PATP processed film presents irregular and unclear layers and changes in its thickness (Figure 2A). A higher interlayer distance between the layers (delamination phenomenon) can be observed on the II processed film (Figure 2B). The II film layer is not clearly defined and thickness changes are demonstrated (Figure 2B) be the microstructural analysis.

#### 4. Conclusions

The objective of this work was to study the behaviour of two commercially available polymer films exposed to the PATP treatment, in order to evaluate their utility as packaging materials for foodstuffs. SEM analysis was used to observe the surface's topography and the structure of the

multilayer films. The control films show homogeneous surfaces, typical for a polymer. Both the processed films structure I and II are heterogeneous and the fibres are not visible. We have noticed several changes in the microstructural characteristics of processed II film compared to I processed film. The untreated films' cross sections show well defined layers with individual parallel fibres. The I PATP processed film presents irregular and unclear layers and changes in its thickness. A higher interlayer distance between the layers (delamination phenomenon) can be observed on the II processed film. The II film layer is unclear and thickness changes are clearly indicated by the microstructural analysis. The results showed that the combined pressure-heat treatment can compromise the integrity of tested multilayer films. Studied films don't represents options for packaging foods treated by PATP sterilization conditions, because the structural changes in the packaging materials, even the small deleaminated areas noticed after the treatment, could severe compormise the integrity and the stability of treated foods

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### Compliance with Ethics Requirements

Authors declare that they respect the journal's ethics requirements. Authors declare that they have no conflict of interest and all procedures involving human and/or animal subjects (if exists) respect the specific regulations and standards.

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