Chapter 5
High Pressure Effects on Packaging Materials

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Abstract During high pressure processing, the prepackaged food material is pasteurized or sterilized by subjecting it to combined pressure-thermal treatment of different intensity. This chapter summarizes the impact of pressure-thermal treatment on moisture and gas barrier properties, seal and mechanical strength, as well as migration characteristics of the packaging polymer. Packaging also play important role in preserving quality of pressure treated foods during extended storage life. Some of the recommended future research needs in the development of novel packaging material for high pressure processing are summarized.

Keywords Package • Polymer • Pressure • Temperature • Barrier properties • Migration

5.1 Introduction

Packaging is a vital aspect for any food preservation method to be successful since it protects the food products from adverse environmental conditions (Ozen and Floros 2001). It helps to preserve the internal properties of foods, including flavor components, by acting as a barrier between internal and external environments.

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During the high pressure pasteurization, the food material is prepackaged in flexible container and then pressure treated at ambient or chilled conditions (see Chap. 3). Package also protects the food material from contamination of the external pressure transmitting fluid and other contaminants. Pressure treatment can potentially alter various moisture and gas barrier properties of the packaging polymer. Understanding how different polymer properties altered by the pressure treatment can help to select suitable packaging material that can withstand pressure treatment. During semicontinuous operations, food products are processed in bulk and are not packaged before the application of high pressure (see Chap. 4). Therefore, package is not exposed to high pressure, and food product is packaged aseptically after processing to ensure shelf-stability.

Pressure treatment can also alter heat compression and other thermo physical properties of packaging polymers (see Chap. 6). For instance, it has been shown that polypropylene (PP) and polyethylene (PE) undergo compression heating greater than water under both high pressure at low and mild temperatures of 10 and 50 °C and pressure treatment at high temperature (90 °C) conditions (Schauwecker et al. 2002; Knoerzer et al. 2010). In particular, the temperature increase with pressure was not linear, and the relative increase with respect to water depended on the pressure range selected as well as the initial temperature. Assuming minimum 5 % of the HP vessel is filled with packaging material, changes in thermophysical properties of packaging materials may affect temperature distribution within the pressure vessel.

When high pressure and high temperature combinations are used to achieve sterilization (see Chaps. 11, 14, 29), prepackaged food products need to be preheated to a target initial temperature before the high pressure is applied. This process is generally known as pressure-assisted thermal processing (PATP). Reasonably harsh preheating process encountered during PATP has the potential to affect the properties of the packaging materials used, including changes in their structure and alterations of their mechanical and barrier properties (Koutchma et al. 2010). Moreover, these changes in the package may result in product quality loss during extended storage. That is why the packaging aspect of high pressure processing is very essential for the successful application of the technology. Researchers have investigated the effect of high pressure processing on packaging materials and food-packaging interactions (Bull et al. 2010).

5.2 Packaging Requirements for High Pressure Processing

Pressure pasteurized products are packaged using flexible, polymeric based pouches, jars, trays and bottles. Rigid packaging materials made from metal and glass are not suitablable as they could be deformed by elevated pressure treatment.

The following are some key considerations when selecting packaging material for high pressure processing:

5.2.1 Flexibility

A packaging material needs to be flexible enough to withstand compression forces so that it can avoid irreversible deformations and maintain its physical integrity. This statement is supported by understanding of volume change during high pressure processing. Typically, high-barrier flexible pouches made of polymers or copolymers with at least one flexible side can be used for processing solid or liquid food products by batch high pressure systems. It can be possible to retain the quality and freshness of the product throughout the shelf life if only a compatible type of packaging material is selected (Sorrentino et al. 2007). When an inelastic packaging material is used, severe deformation is expected to occur during the process (Guillard et al. 2010). Metal cans collapse permanently and glass bottles are likely to break. Thus, packaging containers made from metals and glass material may not be suitable packaging material for high pressure processing (HPP). Additionally, packages which are made of paperboard are not suitable either, since they can degrade under pressure (Caner et al. 2004).

5.2.2 Sealing Strength

Sealing is an important point for flexible pouches, and seal strength should be maintained during processing in order to avoid product leaks or infiltration of pressurization medium into the containers (Koutchma et al. 2010).

5.2.3 Robustness

Barrier, mechanical, and mass transfer properties (sorption and migration) of the package must be resistant to changes occurring during the process. During the process, the volume of the package and the food material decrease due to the pressure applied. The decrease in volume is expected to be temporary, and once the pressure is released, the package needs to regain its original conditions. This is possible only if the thermomechanical stress generated during combined pressure-heat treatment is within the limit that the package can tolerate and return to its original condition (Caner et al. 2004). Containers should also maintain their aesthetic qualities and any other built-in features included in their design, in order to ensure consumer acceptance and overall product convenience (Koutchma et al. 2010; Lambert et al. 2000).

5.2.4 Vacuum Packaging

Vacuum packaging is very important for a uniform treatment because air trapped after sealing the package has higher compressibility than food products and this could potentially lead to a nonuniform treatment and package deformation (Lopez-Rubio et al. 2005). Additionally, excessive amount of gases can increase the come-up time of the processing and cause unnecessary physical stress on the pouch (Schauwecker et al. 2002). Vacuum packaging can also help avoiding oxygen-related reactions including lipid oxidation during both processing and storage. Another advantage of vacuum packaging is that it can improve the loading factor and more packaged product can be processed at one time in the limited volume of the pressure vessel since the amount of air in the package is minimized. Package size and shape are also critical in terms of maximizing the number of packages which can be fitted in the chamber. Therefore, proper package design can contribute to the economical processing.

5.2.5 Barrier Properties

Packaging materials need to have adequate barrier properties to maintain the quality of food products throughout shelf life. In this regard, water and oxygen permeabilities are two of the most important considerations (Galotto et al. 2008). Foods that are sensitive to change in moisture can spoil quickly and lose their characteristics by either absorbing or losing water. Similarly, foods that are sensitive to oxygen changes can become rancid (Yoo et al. 2009). Developing packaging materials with improved water, oxygen, and light impermeability is essential, and choosing the correct type of polymer (or polymer blend) to be used as packaging material is key. Efforts are also underway to utilize modified atmosphere packaging (MAP) during high pressure processing (see Chap. 3.7).

5.2.6 Heat Transfer

Heat transfer properties of packaging materials need to be well characterized and enable fast preheating (Koutchma et al. 2010) prior to PATP. The preheating step can cause undesirable quality changes in the food product and enhance the negative developments in the properties of the package materials during subsequent high pressure application and storage. Therefore, it should be done as quickly as possible. Koutchma et al. (2010) evaluated the impact of packaging materials on heat transfer during the preheating stage and compared four plastic-laminated and two aluminum-laminated pouches. The authors reported that foil-laminated materials reached the targeted preheating temperature faster, providing shorter preheating time than polymeric materials even though foil-laminated materials were thicker than the plastic-laminated materials tested. They attributed faster preheating of foil-laminated pouches to higher thermal conductivity of aluminum layer in the pouch.

5.2.7 Transparency

Consumers in recent years are more attracted towards transparent packages, which provides better product visibility (Lange and Wyser 2003). According to Lange and Wyser (2003), it is important to have good barrier polymers which can also provide transparency. Metal based containers such those made from aluminum lack transparency and thus consumer may not able to see package product content.

5.3 Commonly Used Packaging Materials in High Pressure Applications

Packaging materials used in the food industry can vary drastically from each other depending on specific properties of the polymers used, such as: functional groups in the structure, chain backbone, cross-linking, orientation, melting point, density, thickness of the polymer, and transparency (Valentas et al. 1997). Most of the flexible packaging materials used in the food industry show reasonably good compatibility with high pressure pasteurization when there is no severe heat involved (see Chap. 1, 3). Single and combinations of PET, PE, PP, and EVOH films are some of the commonly used packaging materials for high pressure processing applications in the food industry (Juliano et al. 2010). Additionally, co-extruded films with polymeric barrier layers, adhesive laminated films on a polymer base, or inorganic layer such as aluminum foil (a few micrometer thick) or more recently vacuum deposited coating (nanometer levels thick) are also used (Richter et al. 2010).

For pressure-assisted thermal treatment, where the packaging material is exposed to harsher pressure-thermal conditions, most of the packaging materials used by the food industry may not survive. Nylon, EVOH, PET, PP, aluminum oxide coating, and Al or metalized layer are some of the barrier materials which can survive the PATP conditions (Koutchma et al. 2010). High-barrier materials are needed in order to prevent the interaction of the internal atmosphere of the packaged food with the ambient environment so that the packaged food can retain the properties during the shelf life. A common technique used for creating a barrier layer is lamination or co-extrusion with a good barrier polymer. Polyvinylidene chloride (PVDC), ethylene-vinyl alcohol (EVOH), polyvinyl alcohol (PVAl), and polyamide (PA) are common high-barrier polymers. Good oxygen barrier polymers are generally effective when they are dry. Therefore, they are usually sandwiched between good water-vapor barrier films so that their oxygen barrier properties can be still retained (Lange and Wyser 2003). Another way of improving the barrier properties of a package is blending. By mixing a high-barrier material with a regular film, tortuosity, which is the length that a diffusing compound such as oxygen or water vapor needs to travel, can be extended, and therefore a better barrier property is provided (Lange and Wyser 2003).

5.4 Effect of High Pressure Processing on the Properties of Packaging Materials

In the last decade, numerous research efforts assessed the effect of pressure on the mechanical, barrier, structural, and sealing properties of commonly used mono or multilayer packaging films (Guillard et al. 2010). Typically these studies utilized milder treatment conditions (pressure levels up to 800 MPa; temperature up to 60 °C) (Koutchma et al. 2010). Very limited studies reported the impact of pressure-assisted thermal processing (where temperature levels are above 100 °C) on the properties of the packaging materials (Caner et al. 2000; Schauwecker et al. 2002; Bull et al. 2010; Juliano et al. 2010). Similarly, the changes in properties of pressure-assisted thermally processed packaging materials during extended storage and how these changes in packages influence product quality during storage have been poorly understood (Ayvaz et al. 2012).

In general, two different phenomena occur in the packaging materials exposed to high pressure processing: reversible and irreversible changes (Fig. 5.1). Reversible changes are commonly seen in the packaging materials and occur as a result of compression, particularly on the amorphous regions in the packaging material structure. This generally results in a decrease in permeability values of the polymer, and the packaging material remains intact, which even could be considered as advantageous. As of irreversible changes due to high pressure, visible damage and considerable increase in the permeability values can be observed. In other words, functionality and aesthetic appearance of the packaging material can be negatively affected. One of the possible explanations for the irreversible damage is that the gases are absorbed within the structure of the packaging material under pressure and quickly released upon decompression (Richter et al. 2010).

5.4.1 Gas Barrier Properties

Polymers have repeating functional groups in their structures, and these groups contribute to the formation of packed and highly ordered crystalline regions. As opposed to amorphous regions (void spaces) in the structure, atmospheric gases as well as organic compounds cannot penetrate through crystalline regions. Therefore, high crystallinity in a polymer can result in better barrier to oxygen, water vapor, carbon dioxide, and organic compounds. Additionally, increase in crystallinity in a polymer improves its strength and stiffness (Schauwecker et al. 2002). For instance, EVOH is known as a high oxygen barrier, and this property is

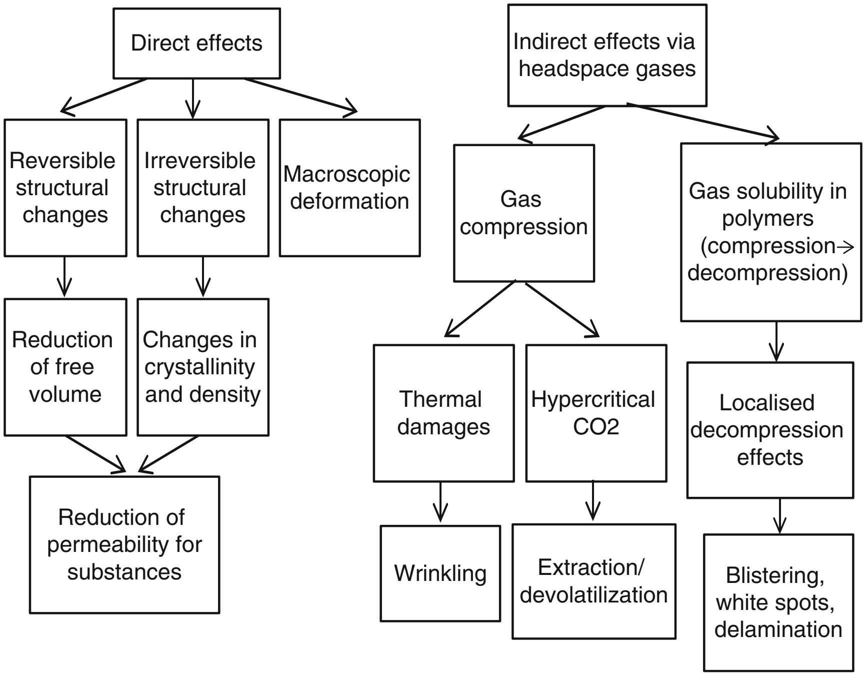

Fig. 5.1 Summary of the effects of high pressure on polymeric packaging materials (adapted from Richter et al. 2010)

a result of hydroxyl groups in its structure, which tend to make a high degree of hydrogen bonds and reduce the free volume between the chains in the polymer. This results in higher barrier to gas exchange. However, these hydroxyl groups also make the copolymers sensitive to water. That is why the barrier properties of EVOH are weakened in high relative humidity (Lopez-Rubio et al. 2005). To avoid this problem, EVOH is sandwiched between other high water-vapor barrier packaging materials in its applications. Additionally, there is a tendency for highly crystalline polymers to be more brittle which can be considered as a disadvantage (Kovarskii 1994). Some factors including the presence of nucleating materials, the rate of cooling, the molecular weight of the polymer, and the degree of agitation and alignment during the process can affect the crystallinity of a polymer or the size of an individual crystal (Jenkins and Harrington 1991).

5.4.2 Oxygen Permeability

The effect of combined pressure and heat treatment on the oxygen barrier properties of the packaging films differs greatly. Even though there are numerous research studies available about high pressure processing and oxygen transmission rates (OTRs) of packaging materials, there is very limited research focusing on pressure-assisted thermal processing.

Earlier researchers documented that in general, high pressure processing did not increase the oxygen permeability of many tested packaging materials. Surprisingly, high pressure processing even decreased the oxygen permeation rates of some of the packaging films significantly. Masuda et al. (1992) reported no significant changes in barrier properties of tested OPP/EVOH/PE and PVDC-coated OPP/CPP at 400 and 600 MPa and maximum process temperature (T_{max}) of 40 °C for 10 min. On the other hand, Mertens (1993) applied 400 MPa and 60 °C for 30 min on LLDPE/EVA/EVOH/EVA/LLDPE and observed 15 % decrease in OTR. Similarly, Kovarskii (1994) evaluated the effect of HPP on PET and reported 70 and 25 % decreases in OTR and WVTR, respectively. Fradin et al. (1998) reported a slight reduction in the oxygen permeabilities of low-density polyethylene (LDPE)/ethylene-vinyl acetate (EVA)/ethylene-vinyl alcohol/EVA/LDPE and polyethylene terephthalate (PET)/aluminum (Al)/polypropylene (PP) after high pressure thawing of a model food at 200 MPa and 15–45 min holding time. Le-Bail et al. (2006) tested LDPE, which is the packaging material commonly used for HPP applications. They processed at 200, 400, and 600 MPa and 10 °C for 10 min and found that barrier properties were not significantly affected and even slightly improved (1 and 2 % decreases in OTR and WVTR, respectively). This could be due to high pressure compression that increased the stiffness due to volume reduction, which caused restrictions in macromolecular mobility and increase in microscopic ordering. In other words, increase in pressure improves the ordering of molecules of a material. Similarly, Lopez-Rubio et al. (2005) reported 2 and 7 % improvements in OTRs of PP/EVOH26/PP and PP/EVOH48/PP pouches in the pressure range of 400–800 MPa and 40 °C applied for 5–10 min, which was attributed to the increase in crystallinity of the structure caused by high pressure compression.

Some studies have indicated that there can be losses in barrier properties of flexible packaging materials. It was reported that metalized films were damaged by high pressure treatment and their barrier properties were impaired. Caner et al. (2000) processed water-filled PET/SiO_x/LDPE, PET/Al_2O_3/LDPE, PET/PVDC/Nylon/PE, Met-PET/EVA/LLDPE, PP/Nylon/PP, and PET/EVA/PET at 600 and 800 MPa and 45 °C for 5, 10, and 20 min. They found no statistical difference in barrier properties except for the Met-PET film, in which they observed that the oxygen permeation and water-vapor transmission rates (WVTRs) were almost doubled (95 % increase) and tripled (150 % increase), respectively. Lambert et al. (2000) processed different packaging materials and reported significant increase in OTR of PA 20 μm/adhesive 10 μm/PE 70 μm, which is similar to the result of a research conducted by Moueffak et al. (1997), who also found a 60 % increase in OTR of PE/PA/PE film after HPP (550 MPa at 55 and 85 °C for 20 min). In these films, the thickness of the polyamides was very low compared to the thickness of polyethylene layer, which minimized the contribution of the good gas barrier property of the polyamides. Galotto et al. (2008) reported a significant increase in OTR of metalized pouches due to the damage in the metalized surface coating. It could be explained by lower compression rate of metals which can accelerate delamination of the packaging structures or discontinuities in the metal coating caused by the low mechanical resistance of the thin aluminum layer (Lopez-Rubio et al. 2005). Schauwecker et al. (2002) found that there were visible signs of delamination between the polypropylene (PP) and aluminum (Al) layers in the meals-ready-to-eat (MRE) pouches processed at ≥200 MPa and 90 °C for 10 min.

During PATP, the packaged products need to be preheated to a target initial temperature (typically 75-90 ºC) prior to pressure-thermal treatment. Therefore, the preheating step can also be an important factor affecting the OTRs of the packaging films. Koutchma et al. (2010) applied PATP treatment (688 MPa and 121 °C) on scrambled egg patties packaged in four plastic-laminated materials (Nylon/co-extruded ethylene-vinyl alcohol, Nylon/polypropylene, polyethylene terephthalate/aluminum oxide/casted PP, and PET/polyethylene) and two aluminum foil-laminated pouches (PET/aluminum and Nylon/Al/PP). They observed an increase in oxygen permeability of the packaging materials and claimed that it was due to thermal damage occurred during the preheating of the packaged egg patties since the OTR values increased following preheating. According to the researchers, preheating caused 369 times increase in OTR of PET/AlOx/CPP pouch. Even though the PE pouch lost 40 % of its oxygen barrier property after preheating, the subsequent PATP treatment at 688 MPa and 121 °C led to recovery of the oxygen barrier property up to 200 %. They also reported that the magnitude of the increase in oxygen permeability in PATP-treated pouches was significantly less than that observed in traditional thermal treatment (121 °C for 3 min in steam retort). There were significant increases in the OTR values of the packaging materials tested, following the high pressure application which did not cause any further increase in OTRs except for PP. The reason for the further increase of OTR of PP was the structural damage occurred in PP pouches. Oxygen permeability of the foil-laminated pouches experienced less loss in oxygen barrier property compared to their thermally treated counterparts. Bull et al. (2010) processed 11 commercially available packaging materials used for thermal sterilization at 600 MPa and 110 °C for 5–10 min and reported that barrier properties of vapor-deposited oxide and Nylon containing films were negatively affected whereas barrier properties of aluminum foil and PVDC-MA containing films survived the PATP treatment well. They observed that regardless of the temperature of the product inside the pouch or the length of holding time of the process, high-barrier laminates with both SiOx and AlOx coatings were severely damaged by the process and lost their barrier properties. This observation was in agreement with that reported by Galotto et al. (2008) and Galotto et al. (2010), who found a complete destruction of the SiOx vapor-deposited barrier layer in PPSiOx, processed at 400 MPa and 60 °C for 30 min. Galotto et al. (2010) evaluated the effect of high pressure treatment (400 MPa and 20 and 60 °C for 30 min) on OTRs of four different flexible packaging materials (PE/EVOH/PE, metalized polyester/polyethylene, PET/PE, and PPSiOx). They observed significant increases in OTRs of packaging films tested except PET/PE film, which experienced almost no change. They reported delamination in metalized polyester/polyethylene. Additionally, water sensitivity and swelling of EVOH in PE/EVOH/PE caused partial loss of their barrier properties, resulting in significant

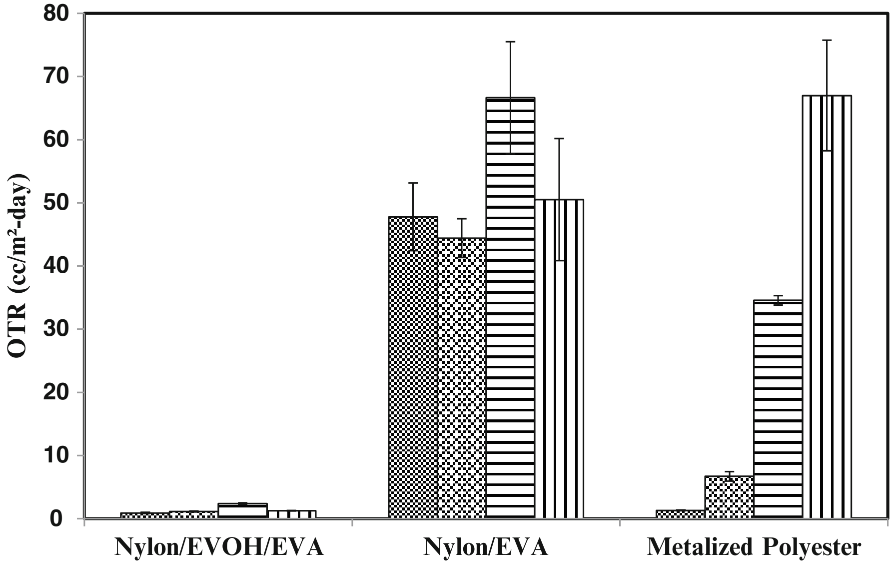

Fig. 5.2 Oxygen transmission rates of control and PATP-treated films stored for 4 weeks at 25 and 37 °C (▩), untreated control; (▦), preheated only; (▤), PATP and stored at 25 °C for 4 weeks; (▥), PATP and stored at 37 °C for 4 weeks (reprinted from Ayvaz et al. 2012, with permission from Elsevier Ltd.)

increase in OTR. Similarly, according to Bull et al. (2010), aluminum barrier layer showed great compatibility with the PATP processing and OTRs of the materials tested experienced the lowest oxygen barrier before and after PATP. This result was not consistent with findings reported by Ayvaz et al. (2012) who processed the Met-PET/PE pouch at 600 MPa and 110 °C for 10 min and observed dramatic increase in oxygen permeability values (Fig. 5.2). Ayvaz et al. (2012) processed baby carrots in three different pouches (Nylon/EVOH/EVA, Nylon/EVA, and Met-PET/PE) at 600 MPa and 110 °C for 10 min and stored the pouches in the dark at 25 and 37 °C for 4 weeks. As shown in Fig. 5.2, the authors observed significant increase in OTR values for Nylon/EVA and Met-PET/PE while high-barrier packaging material Nylon/EVOH/EVA had minimal impact. Moreover, even preheating the Met-PET/PE pouch without any pressure application caused significant increase in OTR of Met-PET/PE. They also reported that change in barrier properties of packaging materials (particularly change in oxygen transmission rates) accelerated the color change and β-carotene degradation of baby carrots processed during extended storage. In other words, packaging type had significant effect in color and β-carotene content of pressure-assisted thermally processed carrots.

5.4.3 Water-Vapor Permeability

Effect of HPP on WVTRs of the packaging materials shows similarity to the changes in OTRs. Several research studies showed that high pressure processing at ambient or chilled conditions did not cause any significant changes in WVTRs of the tested packaging materials. Additionally, high pressure processing even decreased the WVTRs of some of the packaging films remarkably. Masuda et al. (1992) reported no significant changes in water-vapor transmission rates (WVTRs) of the polyvinylidene chloride (PVDC)-coated oriented polypropylene (OPP) and polyethylene terephthalate (PET)/aluminum (Al)/cast polypropylene (CPP) by high pressure in the range of 400–600 MPa at process temperatures of up to 40 °C. Similarly, Halim et al. (2009) processed several co-extruded packaging materials at 800 MPa and 70 °C for 10 min. They reported no significant differences in WVTRs of any tested co-extruded films including Nylon 6/EVOH caused by processing. Mertens (1993) applied 400 MPa pressure at 60 °C on LLDPE/EVA/EVOH/EVA/LLDPE and PET/Al/PP for 30 min and observed 5 and 25 % reduction in WVTRs, respectively. Fradin et al. (1998) processed LDPE/EVA/EVOH/EVA/LDPE and PET/Al/PP laminates at 200 MPa for 15 and 45 min holding time and determined a slight decrease in water-vapor permeability.

Caner et al. (2000) evaluated the effect of HPP (up to 800 MPa and maximum 50 °C) on eight high-barrier laminated films and observed up to 150 % increase in water transmission for PETmet/EVA/LLDPE. Similarly, Galotto et al. (2010) evaluated the effect of high pressure treatment (400 MPa and 20 and 60 °C for 30 min) on WVTRs of four different flexible packaging materials (PE/EVOH/PE, metalized polyester/polyethylene, PET/PE, and PPSiOx). They observed significant increases in WVTRs of packaging films tested except PET/PE film, which experienced almost no change. They reported delamination in metalized polyester/polyethylene and water sensitivity, and swelling of EVOH in PE/EVOH/PE caused partial loss of their barrier properties, resulting in significant increases in WVTRs. Additionally, PPSiOx that was seriously damaged by a drastic increase in WVTR was observed. Bull et al. (2010) studied the effects of PATP (600 MPa and 110 °C for 5–10 min) on 11 commercially available packaging materials and reported that the foil laminates gave the lowest WVTRs of all the films examined. Additionally, although the laminates containing AlOx and SiOx had low WVTRs before processing, they were damaged by the PATP treatment, and the packaging films almost completely lost their barrier properties. Ayvaz et al. (2012) evaluated the effect of PATP (600 MPa and 110 °C for 10 min) and storage conditions (4 weeks at 25 and 37 °C) on Nylon/EVOH/EVA, Nylon/EVA, and Met-PET/PE and reported a significant increase in WVTR of the Met-PET/PE pouch by preheating (15 min in boiling water). Similarly, Caner et al. (2000) also reported that WVTR of Met-PET was significantly affected by pressure application. According to Ayvaz et al. (2012), Nylon/EVOH/EVA and Nylon/EVA, were not affected significantly in terms of WVTRs. Additionally, PATP treatment followed by 4-week storage at either 25 or 37 °C did not further increase the WVTRs of any pouches significantly.

5.4.4 Mechanical Strength

Mertens et al. (1993) reported no changes in mechanical properties, including tensile strength and elongation of LLDPE/EVA/EVOH/EVA/LLDPE and PET/Al foil/PP films by high pressure application at 400 MPa and 60 °C for 30 min. Similarly, according to the research conducted by Caner et al. (2003), no major impact of pressure alone was found in tensile strength, elongation, and modulus of elasticity for the tested packaging films (PET/SiO_x/LDPE, PET/Al_2O_3/LDPE, PET/PVDC/Nylon/HDPE/PP, PE/Nylon/EVOH/PE, PE/Nylon/PE, metallized PET/EVA/LLDPE, PP/Nylon/PP, and PET/PVDC/EVA). Lopez-Rubio et al. (2005) observed no damage on the structure of EVOH processed at 400–800 MPa and process temperatures of 40 and 75 °C for 5–10 min and reported some improvements in the crystallinity of EVOH. Similarly, Le-Bail et al. (2006) also conducted a research on the effect of HPP (200, 400, and 600 MPa and 10 °C for 10 min) on the mechanical properties of PA-PE, PET/BOA/PE, PET/PVDC/PE, PA/SY, LDPE, and EVA/PE and reported no significant effects as a result of HPP.

However, there can be some increase in the tensile strength of the packages after high pressure processing irrespective of the pressure level, the initial rigidity, and the thickness of the package. This shows that the package becomes more rigid and less flexible (Lambert et al. 2000). For instance, Lambert et al. (2000) reported that processing at 200, 350, and 500 MPa and 20 °C for 30 min resulted in an increase of tensile strength and rigidity for five out of six polymers tested (PA/EVOH/PE, PA/LDPE, PA/LDPE, PET/PVDC/LDPE, PE/PE, PA/PP/LDPE). Only PA/PP/PE film experienced a decrease in strength at 200 and 500 MPa.

5.4.5 Migration and Sorption

In general, three different mass transfer processes need to be emphasized.

- First, the *permeation* of the gases present in the environment such as oxygen, water vapor, and so forth. These gases can permeate from the environment into the packaged food.
- Second, the *migration* or movement of the low molecular weight substances including solvents, plasticizers, antioxidants, and monomers from the packaging material into the food. The migrated substances also have the potential to create some by-products through degradation reactions. This migration needs to be controlled since the substances may be toxic for humans and the environment. There are global and specific migrations and they can be measured. In order to measure the global migration, weight differences between the evaporated extracts of the processed and unprocessed samples are determined, whereas further instrumental analysis is required for specific migration measurement (Schauwecker et al. 2002).

- The third and the last mass transfer which can occur is the scalping of low molecular weight substances such as aroma compounds from food. Scalping of aroma compounds can alter the flavor and compromise the quality of food (Guillard et al. 2010; Mauricio-Iglesias et al. 2011).

Mertens (1993) used olive oil as food stimulant and measured the global migration for PET/Al/PP and LLDPE/EVA/EVOH/EVA/LLDPE at atmospheric and high pressure (400 MPa). The results showed a decrease in migration of olive oil under 400 MPa for PET laminate, while a considerable increase was observed under high pressure for LLDPE laminate. Pastorelli (1997) processed PET/Al/PP and PE/EVOH/PE at 400 MPa and 25 °C for 30 min and observed no significant overall migration or by-products of substances from the packages. Similarly, Lambert et al. (2000) tested different packaging materials including PA/EVOH/PE, PA/LDPE, PET/PVDC/LDPE, PE/PE, and PA/PP/LDPE after processing at 200–500 MPa and room temperature for 30 min. Authors reported insignificant amount of overall migration. Galotto et al. (2010) evaluated the effect of high pressure treatment (400 MPa and 20 and 60 °C for 30 min) on total migration of four different flexible packaging materials (PE/EVOH/PE, metallized polyester/polyethylene, PET/PE, and PPSiOx) into distilled water and olive oil. They found that total migration from high pressure-treated packages was lower than that of controls due to more compressed structure and higher degree of crystallinity caused by high pressure treatment. On the other hand, total migration from high pressure-treated packaging materials into olive oil was higher than that of controls. The authors mentioned that oil could be absorbed by packaging material and acted as a plasticizer causing changes in the structures of packaging materials such as swelling, pinholes, and delamination. Additionally, total migration values were lower at higher processing temperature (60 °C as opposed 20 °C) due to recrystallization and therefore higher crystallinity at high pressure and high temperature. In terms of specific migration, Caner and Harte (2005) reported no considerable changes caused by Irganox 1076 migration from polypropylene flexible structures by HPP at 800 MPa and 60 °C for 10 min.

Pressure-transmitting fluids such as glycol, castor oil, and silicone oil are used in HPP systems for the purpose of transmitting the pressure to the packaged product. Therefore, the fluid gets in contact with the package. Schauwecker et al. (2002) evaluated the migration of 1,2-propanediol (PG) into EVOH and MRE pouches treated by HPP (400, 600, and 827 MPa at 30, 50, and 70 °C for 10 min) and observed no detectable migration. It could be due to the high pressure induced reduction of the size of the micro-channels within the pouches.

Very limited studies investigated the impact of pressure treatment on aroma barrier properties of packaging material. Kuebel et al. (1996) found that HPP had no significant effect on the sorption of p-cymene and acetophenone into tested packaging materials (LDPE/HDPE, HDPE, and PET/Al/LDPE). In situ measurement of p-cymene, which had a very low polarity during HPP (50 MPa and 25 min) in LDPE/HDPE/LDPE and PET/Al/LDPE, was conducted by Götz and Weisser (2002). Similar to some other works in the area, they reported a slight decrease in the permeation rate of the p-cymene versus increasing pressure levels during processing due to the structural change in the packaging materials used.

Caner et al. (2004) treated model food containing ᴅ-limonene with HPP (800 MPa and 60 °C for 10 min) in PP and PE/Nylon/EVOH/PE films and observed no significant change in the sorption of ᴅ-limonene by the process. On the other hand, the Met-PET/EVA/LLDPE experienced less ᴅ-limonene sorption after the treatment.

It appears that during compression by high pressure, the polymer matrix loses its capacity to absorb compounds from food or the surrounding media due to a decreased free volume. As the pressure is released, the polymer quickly recovers its original dimensions, and thus sorption and diffusion processes proceed as expected at normal atmospheric pressure. Therefore, permeation and sorption values are statistically the same as for the non-compressed samples.

Rivas-Canedo et al. (2009) evaluated the effect of high pressure processing (400 MPa and 12 °C for 10 min) and packaging on volatile profile of minced beef and chicken breast. In terms of packaging, they compared wrapping the food with aluminum foil and subsequent vacuum packaging in multilayer package (LDPE/EVA layers/VDC (vinylidene chloride)) to only vacuum packaging in multilayer package with no initial aluminum wrap. They reported that upon high pressure processing, volatile profile of minced beef and chicken breast changed significantly and migration was the main phenomenon. Migration increased with increasing fat content since packaging material was more lipophilic, and therefore minced beef which had higher fat than chicken experienced more migration. According to the authors, wrapping the food with aluminum foil helped maintaining flavor profile better. Mauricio-Iglesias et al. (2011) studied the effect of high pressure pasteurization (800 MPa and 40 °C) and pressure-assisted thermal processing (800 MPa and 115 °C) on scalping of four aroma compounds (2-hexanone, ethyl butanoate, ethyl hexanoate, ᴅ-limonene) by LDPE and PLA (polylactate). They reported that tested aroma compounds were quite stable after pasteurization treatment whereas significant losses of aroma compounds were observed after sterilization treatment. They concluded that both LDPE and PLA were suitable for high pressure pasteurization but not for sterilization conditions. Especially for PLA, temperature becomes very critical if the temperature of the processing exceeds the glass transition temperature of PLA.

5.4.6 Packaging Integrity

Any damage or alteration in the packaging materials can potentially cause the loss of hermeticity, and therefore the quality, safety, and shelf life of the product can be adversely affected. According to Fradin et al. (1998), the presence of air and the volume of headspace could be a reason for delamination in HPP-treated packages. The authors conducted an experiment in which they processed vacuum-packaged pouches filled with water at 200 MPa and 25 °C for 45 min. They observed no delamination for any pouches without headspace. However, delamination was found in the regions close to the air bubbles in the material (on the top end of the pouches, close to seams) in the presence of less than 10 % headspace. Headspace in the packaged product should be controlled in order to avoid any potential delamination, seal damage, flexing, cracking, and deformation (Schauwecker et al. 2002).

Götz and Weisser (2002) applied 500 MPa pressure for 5 min on PA/PE film at room temperature and observed delamination in the package. They explained it as a result of elasticity difference between the layers similar to the findings reported by Caner et al. (2003) who utilized scanning electron microscopy and ultrasonic imaging and also attributed the delamination in the pressure-treated (600 and 800 MPa, 45 °C for 5–20 min) Met-PET/EVA/LLDPE to the differences in elasticity and compressibility of the metallized and polymeric layers. Schauwecker et al. (2002) tested PE/Nylon/Al/PP and Nylon/EVOH/PE laminates filled with water after processing at 200 and 690 MPa and approximately 100 °C for 10 min. They observed that delamination occurred between PP and aluminum layer at processes above 90 °C while no delamination was observed when temperature levels decreased to 85 °C or processing was conducted at atmospheric pressure. Galotto et al. (2008) processed four different pouches at 400 MPa and 60 °C for 30 min and observed delamination and wrinkling except in PE containing pouches (PE/EVOH/PE and Met-PET/PE). On the other hand, Bull et al. (2010) processed eleven commercially available packaging materials used for thermal sterilization at 600 MPa and 110 °C for 5–10 min and also reported delamination and deformation. The authors explained the effect as a result of solubilization of the gases into the packaging film during compression and quick release of the gases upon decompression. They further explained that the concentration of the air in the headspace and in the gap of the laminate from manufacturing becomes denser due to the high pressure applied and dissolves into the polymer layers more easily. Then, once the depressurization step is reached, the dissolved gases are released quickly forcing the layers to separate. This resulted in delamination. Moreover, authors proposed that degree of solubility of the gases located in the material may be a factor affecting the magnitude of delamination. Similar to this, Fairclough and Conti (2009) also mentioned that upon depressurization of solubilized gases in the PP films processed at 695 MPa and 86 °C for 10 min, opaque areas and bubbles are formed.

Lopez-Rubio et al. (2005) processed EVOH-based packaging materials at 800 MPa and 75.8 °C as well as in the retort at 121 °C. They found that as opposed to the severe damage occurred in thermally treated packaging materials, high pressure processed packages were minimally damaged. Similarly, regarding the overall package integrity, Koutchma et al. (2010) reported that the pouches (four plastic-laminated materials and two aluminum foil-laminated pouches) processed at 688 MPa and 121 °C showed no leakage or severe damage except some blisters and ruptures formed on the outer walls of the metal layers. Good understanding of the delamination and reasons behind it such as headspace volume can be useful to avoid this problem and develop better packaging materials suitable for high pressure applications (Bull et al. 2010).

In several research studies, further analyses were conducted to better evaluate the damage on the surface or in the structure of packaging material. Caner et al. (2003), Galotto et al. (2008), and Ayvaz et al. (2012) utilized scanning electron microscopy (SEM) to locate damage on the surface of high pressure processed

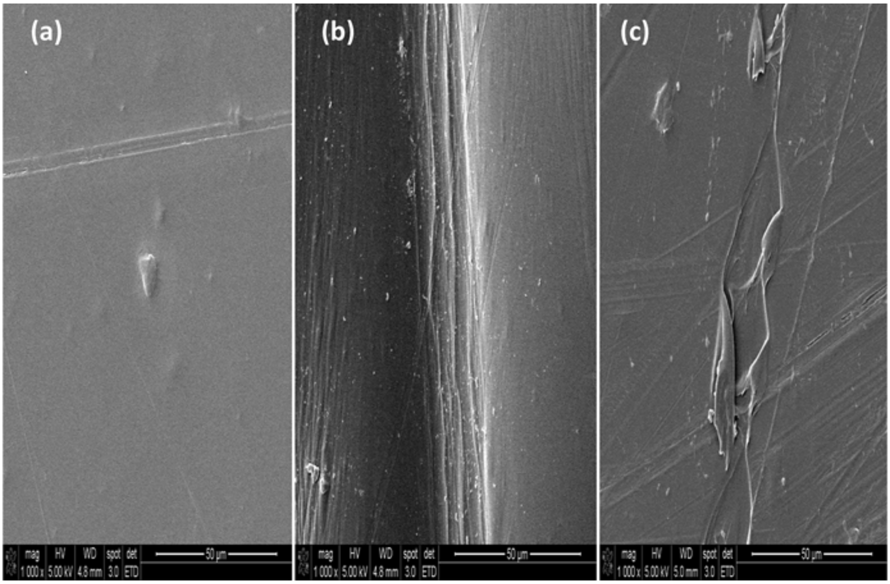

Fig. 5.3 Scanning electron micrographs of Met-PET/PE pouch illustrating the damage caused by PATP treatment: (**a**) control; (**b** and **c**) PATP-treated (magnification × 1000) (reprinted from Ayvaz et al. 2012, with permission from Elsevier Ltd.)

films. They observed the delamination in tested metalized polymers. Figure 5.3 shows how the pressure-assisted thermal processing (600 MPa, 110 °C for 10 min) affected the Met-PET/PE packaging material in research conducted by Ayvaz et al. (2012).

In the same study, pinholes created in pressure-assisted thermally processed Nylon/EVA package and effect of storage for 12 weeks at 25 °C can be seen in Fig. 5.4.

In some other research studies, differential scanning calorimeter (DSC) was utilized to determine the effect of high pressure and pressure-assisted thermal processing on thermal transition characteristics of different packaging materials (Schauwecker et al. 2002; Lopez-Rubio et al. 2005; Galotto et al. 2008; Ayvaz et al. 2012). T_m (melting temperature) and ΔH (heat of fusion) of polymers were calculated from DSC thermograms to support the change in structure of polymers after high pressure applications compared to control samples.

5.4.7 Seal Strength

For high pressure applications of flexible packages, sealing is an important step. Package needs to be sealed properly prior to high pressure applications so that it can survive the process maintaining the packaging integrity with no leakage (Lambert et al. 2000).

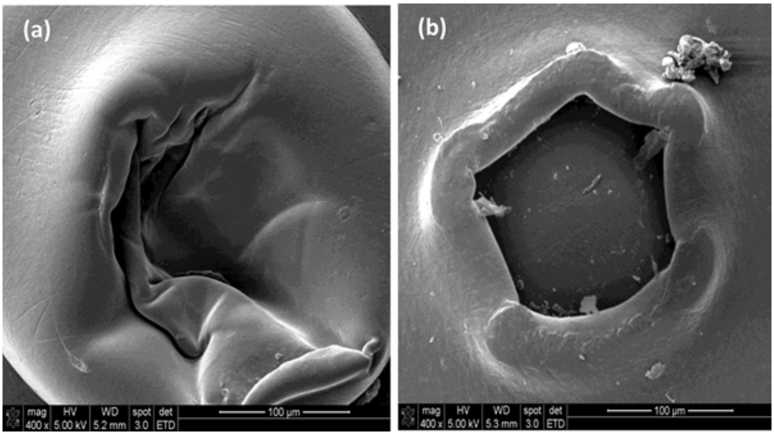

Fig. 5.4 Scanning electron micrographs of Nylon/EVA pouch illustrating the pinholes: (**a**) PATP-treated only with no storage; (**b**) PATP-treated and stored at 25 °C for 12 weeks (magnification ×400) (reprinted from Ayvaz et al. 2012, with permission from Elsevier Ltd.)

Lambert et al. (2000) processed six different types of plastic package. The packages were multilayer (PA/PE) with different thickness, permeability, and stress at yield point and at breakage. They processed at 200–500 MPa and 20 °C for 30 min and found that seal strength of the pouches was not significantly altered by the process except the heat-seal strength of one of the six pouches decreased more than 25 % compared to its initial strength before high pressure processing. Similarly, Dobias et al. (2004) applied 600 MPa pressure at 20 °C on some plastic-laminated films including PA/PE and PE/EVOH/PE and reported that seal strength of the laminates was not affected by high pressure application.

Koutchma et al. (2010) showed that pressure-assisted thermal processing (688 MPa and 121 °C for 3 min) did not affect the seal strength of Nylon/co-extruded ethylene-vinyl alcohol, Nylon/polypropylene, polyethylene terephthalate/aluminum oxide/casted PP, and PET/polyethylene pouches, but increased the seal strength of the two aluminum foil-laminated pouches (PET/aluminum and Nylon/Al/PP). Generally, PATP-treated pouches experienced increased seal strengths compared to traditional thermal treatments. The authors could not find any relationship between the vacuum levels in the pouches and the seal strengths and concluded that pressure-assister thermal processing does not alter the seal strength of plastic-laminated pouches significantly.

Patazca et al. (2013) studied the effect of packaging for inactivation of *Clostridium botulinum* spores by using PATP (750 MPa at 105 °C). Inactivation of spores in plastic pouches, cryovials, and transfer pipettes was compared in laboratory-scale and pilot-scale pressure systems. Approximately 6.6-log reduction of the spores packaged transfer pipettes was obtained after processing for up to 10 min at 118 °C and 700 MPa in both pressure test systems. Reduction of spores packaged in plastic pouches was the lowest (~4.8 log) for both pressure test systems when processed at the same conditions. It was concluded that the use of high-barrier plastic pouches for packaging of spores and subsequent high pressure processing can result in inaccurate spore inactivation data compared with other packaging systems studied. Since the type of flexible packaging systems might affect inactivation results, additional studies are needed to determine the cause of the packaging effect that was observed in this study.

5.5 Future Research Needs in Pressure Treatment of Packages

Food industry is exploring the concept of nanotechnology applications in food packaging, and it is expected that nanocomposite packages will become more commonly used in the food industry in the near future. Nanotechnology provides the ability to food processors to engineer the structure of the materials at a molecular scale. It may be possible to design packaging material with desired functionalities.

Nanoparticles are defined as particles which are less than 1 nm in at least one dimension; however, other side dimensions can be larger, which can provide high aspect ratios. When used in packaging materials, they provide highly desired exceptional properties (Brody et al. 2008) such as improved barrier, mechanical, and thermal performances as well as extended shelf life of food products (Sorrentino et al. 2007). For instance, montmorillonite clay increases the gas barrier properties of a polymer by simply extending the length of the tortuous path that gases need to pass through (Halim et al. 2009).

According to some modeling approaches, significantly higher barrier properties (up to 50 times higher or even more) can be accomplished by utilizing the high aspect ratio particles. Generally, the difficulty with the nanocomposite packaging is how well the filler is dispersed in the matrix (Lange and Wyser 2003). Another important point involving the usage of nanoparticles in food applications is that nanomaterials need to keep their beneficial properties after different processing conditions, including high pressure levels, elevated temperatures, and so forth (Halim et al. 2009). As for high pressure processing, very limited studies are available on the impact of combined pressure-thermal treatment on nanocomposite packaging materials.

Antimicrobial packaging is another promising tool for controlling the growth of food-borne pathogens or spoilage bacteria in ready-to-eat (RTE) post-processed products. Antimicrobials are impregnated into food packaging to protect the package or to extend the shelf life of the contained food. Known antimicrobials include organic acids and their salts, sulfites, nitrites, antibiotics, alcohols, enzymes, and natural components such as bacteriocins, especially nisin. Nisin is currently the only bacteriocin allowed to be used as a food additive. Enterocins A and B and sakacin K are bacteriocins whose antilisterial activity has been shown in a meat homogenate and have been applied experimentally as ingredients in several meat products. All uses of antimicrobial substances in or on food-packaging materials are considered food additive uses regardless of their intention. Jofré et al. (2008) studied the effectiveness of the application of interleavers containing enterocins A and B, sakacin K, nisin A, potassium lactate, and nisin plus lactate alone or in combination with a 400 MPa in sliced cooked ham spiked with *Salmonella* spp. It was reported that antimicrobial packaging, HPP, and refrigerated storage appear as an effective combination of hurdles to obtain value-added, ready-to-eat products for 3 months of storage at 6 °C. On the other hand, Marcos et al. (2013) evaluated the combined effect of antimicrobial packaging (polyvinyl alcohol with nisin added) and high pressure processing (at 600 MPa and 12 °C for 5 min) as a post-process treatment on *L. monocytogenes*-inoculated fermented sausages with no added sodium salt. They found that HPP did not contribute any protection against *L. monocytogenes* compared to antimicrobial packaging alone. The authors attributed this to possible protective effect by low water activity and lactate content of the fermented sausages.

5.5.1 Safety of Packaging Materials

Due to the novelty of HPP, food manufacturers may request that their packaging materials providers obtain a letter of no objection regarding the acceptability of their materials for HPP treatment from regulatory agencies such as US FDA or Health Canada. Information on the regulation and pre-market assessment of packaging materials can be found on US FDA and Health Canada's Packaging Materials webpage. In addition, a complete list of all packaging materials accepted by the Canadian Food Inspection Agency (CFIA) including those acceptable for HPP treatment is available on Health Canada and CFIA's website (http://www.hc-sc.gc.ca/fn-an/legislation/guide-ld/polymers_tc-polymere_tm-eng.php).

5.6 Conclusions

Pressure treatment has the potential to alter the gas and moisture barrier properties of packaging material. This in turn influence the quality of the packaged product during the extended storage. High pressure pasteurization treatment at ambient or chilled conditions generally do not have adverse effect on properties of the packaging materials commonly used in food industry. However, if the adhesion between the layers of multilayer structure of the packaging film is affected by processing, gaps may appear within the structure resulting in loss of integrity and therefore the safety and quality problems of the packaged food.

More research is necessary to identify suitable polymer packaging material with high barrier properties that can withstand severe treatment conditions during PATP.

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