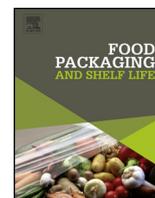




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A quantitative approach to assess the contribution of seals to the permeability of water vapour and oxygen in thermosealed packages

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ABSTRACT

Shelf-life of many foods is largely dependent on the barrier to moisture and oxygen of the respective package. Barrier is often assessed by measuring the transmission rate of films. However, in thermosealed packages leaks and weak seals can give rise to increased total mass transfer entering the system and reaching the food. While leaks are random defects and are associated to early failure, lower integrity of regular seals tends to affect whole batch causing a decrease in the product shelf-life. In the present work the contribution of the seals to total permeability of packages was assessed by measuring transmission rate of the film and of thermosealed packages of different sizes, therefore with different seals length. Packages made of PA/PE and PVDC coated PET/PE and their respective films were tested for moisture and oxygen transmission rate at different temperature and relative humidity. Results indicate that industrial regular produced seals can account for ca. 25% of the total mass transfer through the system. This decrease of the package barrier as compared to the material barrier will have a significant impact on the product shelf-life and it should be considered in the packaging design process.

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1. Introduction

The packaging system is, directly and indirectly, important for the physical, chemical and microbiological safety of foods. The packaging should protect and contain the product, which requires physical and mechanical strength and ability to maintain its integrity throughout the distribution chain and product shelf-life. For the majority of food products, the package is an essential part of the preservation process by assuring adequate barrier to gas, moisture, odours, and light, although the specific requirements may be different depending on the food processing conditions.

Package integrity is a key requirement for product safety and security, whatever the packaging material and corresponding closure system. Thermosealing can be made using different techniques, but the basic process involves the welding of two polymer layers when forced into intimate contact while they are in a semi-molten state. Different kind of seals, such as peelable or

non-peelable seals can be obtained by changing the conditions under which the material is sealed (Aithani, Lockhart, Auras, & Tanprasert, 2006). Seals defects may consist of leaks (pinholes and channel leaks) and weak seals. Defective seals can contain wrinkles, voids or minute amounts of food causing channelling and integrity loss. Weak seals are potentially formed by deficient sealing operation conditions or due to inadequate material properties (Mihindukulasuriya & Lim, 2011). Quality of thermoseals depends on pressure and profile of sealing jaw, temperature of the material to be sealed, dwell time and alignment of the surfaces to be sealed (Harper, Blakistone, Litchfield, & Morris, 1995; Farris, Cozzolino, Introzzi, & Piergiovanni, 1995). The jaw profile has a great influence on the seal performance. It can be flat or grooved and with different groove pitch, angle and orientation (Oliveira & Faria, 1996). Optimization of sealing temperature and dwell time can raise production rates (Aithani et al., 2006).

Seals failure has been considered a more frequent cause of product deterioration than the packaging material itself (Aithani et al., 2006). Methods for leak detection include simple visual inspection, assisted by dye penetration, pressure differential techniques, electrolytic conductance and biotest based on penetration of microorganisms. Seal quality is evaluated by mechanical resistance tests, such as bursting test and tensile test (peeling test).

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The strain energy, defined as the integration of the load-deformation curve obtained in the *T*-peel test (ASTM D1876), is often considered the best measure for quantifying the seal strength (Farris et al., 2009). These are destructive methods, unable to detect microleaks and unable to detect randomly occurring defects. More recently, imaging techniques, such as ultrasonic imaging and optical and scanning electron microscopy, have been used to characterise seal bond (Pascall, Richtsmeier, Riemer, & Farahbakhsh, 2002; Ayhan, 2004). These methods are very useful to visualize the surface of the packaging material and respective seal bond and diagnosis causes of failure. New on-line, non-destructive package evaluation systems have been developed including trace gas sensing, pressure or vacuum decay, acoustic micro imaging and high-voltage leak detection (Song, Gera, Jain, & Koontz, 2014).

There are several studies about new destructive and non-destructive techniques to identify leaks and characterize them, particularly in relation to microbiological integrity, as discussed above. Studies on the effect of leaks on the gas barrier properties of packages have also been performed (Chung, Papadakis, & Yam, 2003; Lange, Büsing, Hertlein, & Hediger, 2000). Chung (2003) presented a theoretical analysis of the effect of leaks on water vapour and oxygen transport through a LDPE package and through a higher barrier package. The effect of temperature on the significance of leaks to the total mass transport through the package was also analysed. A simple mathematical model was proposed combining gas transfer through leaks with gas transfer through material wall, both based on Fick's law of diffusion. Leaks were approximated to cylindrical pores. The effective permeability was considered to depend on the leak diameter and length, diffusion coefficient of the gas in air, permeability of the material, material thickness and a correction factor to account for no equilibrium between the air around the leak end and the surrounding air. Lange et al. (2000) studied the water vapour transport through large pores (0.5–1 mm) in flexible packaging of dehydrated soup. Two models, both based on Fick's law of diffusion were compared: the Becker (1979) and the Heiss (1954) models. The Heiss model also considered an additional factor to account for the fact that the air next to the pore ends is not in equilibrium with the surrounding air and was found to represent better the experimental data as compared to the Becker model. Mass transfer through macro perforations have also been particularly focused in studies targeting modified atmosphere packaging for fresh produce (Fonseca, Oliveira, Lino, Brecht, & Chau, 2000; Techavises & Hikida, 2007).

However, there is still little information on the contribution of seals to the permeability of the overall package, which is of critical importance to the design of modified atmosphere packaging systems. It is pointless to develop high-end capacity to design packages that ensure optimum atmospheres for maximum shelf life from the properties of the packaging material only, if the seal can modify the permeability significantly and is not accounted for. There have been insufficient investigations in relation to the integrity of regular seals and the prediction of how they modulate the total gas and moisture transfer to the whole system. Alves, Garcia, and Bordin (1999) verified that the better the barrier of the material, the higher the relative contribution of the seal; seals were found to permit water vapour transfer in packages up to 3 times the moisture transfer through the film itself. A more recent study has focused on food wraps but not on sealed packages (Steven & Hotchkiss, 2002).

While leaks are frequently associated to early failure and tend to be random defects, the lower integrity of regular seals tends to affect the whole batch, decreasing the ability of the packaging system to maintain the protective conditions required for shelf life. The objective of this study was to determine the influence of the

seals on the water vapour and oxygen transfer rates through small packages used for dried products. A mathematical model to describe the transfer through the whole packaging system, both the film and the seal, was developed.

2. Material and methods

Two multilayer materials were analysed for their barrier to water vapour and oxygen in different conditions of temperature and relative humidity. Packages made with these materials in industrial form-fill-seal machines were also analysed and the contribution of the seals to the permeance of the whole system was estimated.

2.1. Packaging samples

Films: coextruded unprinted PA/PE (15 μm /50 μm) was used for the moisture transfer experiments and a printed PVDC coated PET/PE (12 μm /55 μm) was used for the oxygen transfer experiments. These structures were characterized by microscopy and the layers identified by differential scanning calorimetry. The PA was found to be a PA6: a semicrystalline polyamide made of caprolactam.

Packages: formed empty packages, were provided by local industries Ernesto Morgado and Fromageries Bel Portugal. The packages samples were collected during regular production i.e., during normal operations of forming, filling and sealing in industrial operation conditions. The packages were, however, sealed without the product. The sealing operations were performed according to the material supplier instructions. Integrity of the seals of the packages was checked with the dye penetration method.

For the moisture transfer experiments, two different package sizes were used: small package (SP)—6 \times 9 cm with an effective area available for mass transfer of 0.0108 m²; large package (LP)—11.5 \times 14 cm with an effective area of 0.0322 m². The SP is used for dried spices and the LP for dehydrated rice and vegetables. The oxygen transfer experiments were conducted in one single size package —13 \times 18 cm with an effective area available for mass transfer of 0.04682 m², typically used for modified atmosphere packaging of sliced cheese.

2.2. Water vapour transmission rate (WVTR)

WVTR was measured with a Permatran W-200 (Mocon) according to the ASTM F-1249 Test Method for Water Vapor Transmission Rate Through Plastic Film and Sheeting Using a Modulated Infrared Sensor. The equipment measuring range is 0.01–60 g/m²/day. The WVTR of the flat film, SP and LP were determined at different test conditions. The film WVTR was determined at 15 °C, 23 °C and 38 °C and at a relative humidity gradient across the film of 100%. The WVTR of SP and LP was determined at 23 °C \pm 2 °C and with a relative humidity gradient across the package wall of ca. 50%, 75% and 90%. The measurements at 23 °C \pm 2 °C and 50% \pm 5 relative humidity were obtained at laboratory controlled conditions. For the measurements at 75% and 90% relative humidity, high barrier flexible containers, made of polyethylene and aluminum laminate, were used in order to submit the external face of the packages to the required relative humidity. The containers were hermetically closed, with the testing package inside. The relative humidity inside the containers was created with saturated salt solutions of sodium chloride (measured experimentally as 75% at 23 °C) and distilled water (possibly due to losses from the chamber, the relative humidity inside the containers with distilled water was 90%, not 100%).

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