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Original Article

Properties of barrier shrink bags made with EVOH and polyamide for fresh beef meat preservation

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Abstract: The objective of this work was to compare the barrier and mechanical properties and shrinkability of coextruded films chlorine-free, with high barrier chlorine multilayer films traditionally used to preserve fresh beef. Four 9-layer barrier-shrink films containing PET, ethylene ionomers, polyamide PA66/6 modified with amorphous PA, 32 or 44 mol% EVOH and PE were produced in a commercial scale triple bubble co-extrusion line. Seal strength, puncture resistance, oxygen and water vapor permeability and film shrink were measured for the four films and compared to the EVA/PVDC/PE film properties. The results obtained under controlled laboratory conditions show that films made with one layer of EVOH 32 mol% of ethylene encapsulated between two layers of PA66/6 modified with amorphous PA have gas barrier properties and puncture resistance better than a typical EVA/PVDC/PE, seal strength and shrinkability comparable to this film and therefore have potential to preserve fresh beef.

Keywords: chlorine free, oxygen transmission rate, puncture resistance, shrink film, vaccum packaging.

1. Introduction

Trends in the food market clearly show an increasing demand for healthier and safer food and the need for packages with lower environmental impact[1]. In regards to health the food industry is striving to provide fresh products with reduced amounts or no preservatives that meet stringent safety requirements in the globalized market.

Beef is considered fresh if it is recently processed, vacuum-packed or packed in modified atmospheric gases, and has not undergone any treatment other than chilling to ensure preservation [2].

To avoid undesired changes in appearance, odor, texture, and flavor due to microbial activity or interaction with the environment, the packaging material used must be able to enclose the meat cuts and maintain the ideal atmospheric environment inside the package.

Therefore the packaging must provide a hermetic and reliable closure, must have the ability to retain vacuum and to minimize gas transfer through the film surface to maintain a low oxygen partial pressure to reduce oxidative reactions and aerobic bacteria growth[3].

Plastic films with structural strength and shrink ability are used for wrapping uneven cuts of fresh meat to achieve a skin-tight and compact pack. The skin-tight feature is also effective to prevent liquid purge from inside the muscle tissue. The advantages of plastic shrinkable films include ease handling, a contour fit and neat appearance[4].

Traditionally vacuum packaging bags are designed to optimize gas barrier, shrinking properties, toughness and sealing characteristics, among other features[5]. Those properties are highly dependent on the resins used, the manufacturing technology and the actual structure of the multilayer film[6].

According to Zhou et al.[2], vacuum packages for fresh beef are usually coextruded multilayer films composed of ethylene-vinyl acetate copolymer (EVA) and polyvinylidene chloride-methyl acrylate copolymer (PVDC) which have oxygen permeability lower than 15.5 mL (STP).m^{-2}.day^{-1} . In Brazil, the market for barrier shrink film for vaccum beef package is dominated by two global manufactures, that sells films with OTR lower than 25 mL (STP).m^{-2}.day^{-1}. Those films are treated with radiation along the conversion process so they become temperature sensitive and shrink when subjected to temperatures ranging between 80 °C and 90 °C.

Although shrinkable high barrier EVA/PVDC/PE (polyethylene) films are widely used and very effective to preserve fresh beef, they are considered not eco-friendly because they contain chlorine which produces dioxin during combustion and require controled atmospheric emissions in case they are submitted to energetic recycling after disposal. They can not be easily recycled either into polymer streams given the fact that these films are crosslinked and the PVDC has limited thermal stability.

In regards to sustainability, the impact of packaging to the environment can be minimized by following some criteria such as: (i) it has to be beneficial, safe and healthy for individuals and communities throughout its lifecycle; (ii) meet the designed performance and cost; (iii) maximize the use of renewable or recycled materials; (iv) manufactured using clean production technologies and best practices; (v) made from materials healthy in all probable end-of-life scenarios and; (vi) can be recovered effectively and used in biological and/or industrial cradle-to-cradle cycles[7].

To mitigate the environmental problems associated with PVDC films and to offer beef processors a more sustainable packaging alternative, multilayer coextruded films containing EVOH, PA and PE have been tested to package fresh meat products and are regarded as a valid alternative to traditional PVDC films used in wholesale distribution of chilled meat[8].

Multilayer barrier shrinkable films made with polyethylene terephthalate (PET), ionomeric ethylene copolymers, ethylene-vinyl alcohol copolymer (EVOH), polyamide (PA) and polyethylene (PE) are chlorine free and do not require radiation crosslinking to become thermally shrinkable. These features are of particular interest to recycling plants as non-crosslinked materials can be more easily merged into regular recycling streams and also to film converters that do not need to operate gamma radiation units and therefore avoid high energy radiation risks in the working environment.

Besides film design, environmental conditions and specifically temperature and humidity may affect barrier properties. A possible drawback of films containing EVOH and PA is that these materials are hygroscopic and regarded to be sensitive to moisture in a way that when exposed to high moisture environment they may have limited ability to provide adequate oygen barrier [9].

According to McKeen[9] the permeability of EVOH and PA films is depending on the relative humidity. The oxygen permeability coefficient of a 15 μm thick film made of EVOH with 32 mol % of ethylene varies from 0.01 to 0.05 $mL.mm.m^{-2}.day^{-1}.atm^{-1}$ at 20 °C as the relative humidity (RH) varies from 0% to 85%, while for 20 μm thick films made of EVOH with 44 mol% of ethylene, the oxygen permeability coefficient varies from 0.04 to 0.08 $mL.mm.m^{-2}.day^{-1}.atm^{-1}$ at 20 ° C as RH increases from 65% to 85%. In the case of 25 μm thick films made of polyamide 66/6 copolymer the coefficient varies from 0.94 to 5.91 $mL.mm.m^{-2}.day^{-1}.atm^{-1}$ at 23 °C as the RH varies from 0% to 90%.

It has been reported that multilayer PET/PVDC/PE films with layer thicknesses of 12/4/50 μm respectively have oxygen transmission rate as low as 5 $mL.m^{-2}.day^{-1}.atm^{-1}$ (23 °C, 50% RH) and water vapor barrier of 2 $g.m^{-2}.day^{-1}$ (23 °C, 85% RH), whereas a PET/EVOH/PE film (12/5/50 μm) has oxygen and water vapor transmission rates of 1 and 2 to 4 in similar units and testing conditions [10].

Therefore, and to optimize the oxygen barrier of multilayer films containing EVOH and/or PA, the structure must contain other outer layers, typically polyolefins, that can minimize water vapor transmission and can protect the inner EVOH and PA layers so the film as a whole can perform as an effective gas barrier.

The ethylene content in the EVOH copolymer also has influence on the oxygen permeability of the polymer[9]. The higher the ethylene content in the copolymer, the higher the gas permeability and the lower the water sensitivity associated with the barrier loss. The permeability of EVAL F Series (EVOH 32 mol% of ethylene) is 0.4 $mL.20\mu m.m^{-2}.day^{-1}.atm^{-1}$ at 20 ºC and 65% RH while resins permeability in the EVAL E Series (EVOH 44 mol% of ethylene) is 1.5 in the same units and conditions[11].

Differently than polyamide 66/6 or EVOH resins, amorphous polyamides have excellent oxygen, carbon dioxide and water vapor barrier, even at extremely wet conditions such as 95% to 100% RH. The oxygen permeability coefficient of amorphous PA is reduced at higher humidity, and has values of 1.50 and 0.59 $mL.mm.m^{-2}.day^{-1}.atm^{-1}$ at RH of 0% and 95% respectively (both at 30ºC). Blending amorphous PA (aPA) into PA 6 or PA 66/6 polymers results in a product that behaves like an amorphous polymer with enhanced barrier properties at high humidity as well as toughness, strength and flexibility[12]. Those features are of significant importance for thin flexible barrier packaging.

Foreseeing an increased interest from fresh beef producers to adopt more environmentally friendly packaging and given the sensitivity of EVOH and PA to high humidity, four different nine layer shrink-barrier film structures were developed and manufactured in a commercial scale triple bubble film co-extrusion line.

The objective of this study was to compare the gas barrier and mechanical properties and shrink performance of films made with PET, ionomer, EVOH, PA and polyethylene with those of commercially available EVA/PVDC/PE films used to preserve the quality of fresh beef in the Brazilian market.

2. Materials and Methods

2.1 Packaging materials and structure

Four different high barrier shrinkable bi-oriented tubular films containing one PET outer layer; one ionomeric ethylene copolymer layer; one EVOH layer; one or two layers of PA 66/6 blended with amorphous PA; one or two layers of PE blends made by blending pellets of Ziegler-Natta and metallocene catalysts polyethlenes and two layers of polyethylene grafted with maleic anhydride resins used as co-extrusion adhesive. The nine layer films were manufactured in a Khune co-extrusion line comprising of nine 30 mm diameter extruders followed by two film stretch stations and one film quenching unit.

A five layer film, made with EVA (outerlayer), PVDC and PE (selant layer) reticulated with gamma-ray, commonly used in the Brazilian market was used as the "control" to which the four competing alternatives were compared. Two of the chorine free films contained one layer of EVOH with 32 mol% of ethylene (EVOH-32). In one case the EVOH layer was encapsulated between a layer of modified PA and a PE layer, while in the other case the EVOH layer was encapsulated in-between two modified PA layers. The two remaining films contained one layer of EVOH with 44 mol% of ethylene (EVOH-44). Like in the EVOH-32 case, the EVOH-44 layer was encapsulated between a layer modified PA layer and a PE layer in one case and in-between two modified PA layers in the other.

In all the chlorine free film structures just described, the modified PA layers contained PA 66/6 and aPA (DuPont™ Selar® PA) blends. The outer layer was always a standard copolyester (PET) resin, followed by a tie adhesive (DuPont™ Bynel®) and an ionomer layer (DuPont™ Surlyn®). The sealing layer was always a blend of polyethylene resins. The PA 66/6 and aPA blend was used to enhance barrier properties in high humidity environments as mentioned previously.

The films structure of the five samples compared are summarized as follows:

- **Control:** EVA/tie/PVDC/tie/LLDPE;
- **EVOH32-1:** PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol %/tie/LMDPE/LLDPE (LMDPE: linear medium density polyethylene/ LLDPE- linear low density polyethylene);
- **EVOH32-2:** PET/tie/Ionomer/tie/ PA6/66+aPA/EVOH 32 mol %/ PA6/66+aPA/tie/LLDPE;
- **EVOH44-1:** PET/tie/Ionomer/tie/ PA6/66+aPA/EVOH 44 mol %/tie/LMDPE/LLDPE;
- **EVOH44-2:** PET/tie/Ionomer/tie/ PA6/66+aPA/EVOH 44 mol %/ PA6/66+aPA/tie/LLDPE.

Seal strength, puncture resistance, oxygen and water vapor transmission rates and film shrink were measured for the four films and compared to those obtained for a typical EVA/PVDC/PE film.

2.2 Film thickness

The total thickness and the total barrier layer thickness were measured in 1 cm x 2 cm specimens that were randomly cut from each film structure and placed in a sample holder between two polyester slip-sheets. Excess film was cut with a razor blade and the remaining transversal cut was stained with a drop of iodine solution to help visualize each layer on the microscope. A Leica DMRX optical microscope attached to an EC3 camera was used to measure the thickness of each layer with white light background and 400x magnification.

2.3 Seal strength

The maximum bottom heat seal strength was determined according to ASTM F 88/88M[13] standard procedure with an Instron universal testing machine model 5500R. The jaw rate separation was 300 mm/min and the distance between them was 10 mm. Ten 25.4 mm wide specimens per film sample were tested. These specimens were preconditioned at 23 °C and 50% RH and the test was carried under these conditions.

2.4 Puncture resistance

Puncture resistance was measured using an Instron universal testing machine model 5500R, equipped with appropriate compression load cells using blunt and sharp probes with radii of 6.35 mm and 0.79 mm respectively. Circular specimens of 95.25 ± 0.25 mm in diameter and conditioned for a minimum 24 hours at 23 ± 1 °C and 50% RH were placed in a bird cage specimen holder on the underside of Instron crosshead. The probe speed was 51 mm/min. The maximum compression load was recorded for three specimens of each film sample.

2.5 Water vapour transmission rate (WVTR)

WVTR was determined by a gravimetric method according to ASTM E 96/E 96[14]. This standard procedure is based on the weight gain of anhydrous calcium chloride placed inside an aluminum capsule that is isolated from room atmosphere by the specimen. The effective permeation area for each specimen was 50 cm^2. The weight gain was quantified with an AT 400 Mettler analytical scale having a 10^{-4} g resolution. The test was made in a Vötsch – VC 0057 chamber at 38.0 ± 0.1 °C and 90.0 ± 0.5% RH. Five specimens of each film sample were tested.

2.6 Oxygen transmission rate (OTR)

OTR was determined by coulometry method according to ASTM F1927[15] using a MOCON OXTRAN equipment model 2/20, operating with pure oxygen as permeating at 23 °C and 75% RH. Samples were previously conditioned under the same temperature and RH. The effective permeating area for each specimen was 50 cm^2. Results for two specimens of each film sample were adjusted for 1 atm parcial pressure gradient of oxygen.

2.7 Film shrink

The free linear thermal shrinkage of the films was determined according to ASTM D 2732 [16] standard procedure. The initial test specimens dimension was 100 mm x 100 mm. Specimens were placed inside a hot water bath at 85.5 ± 0.5 °C for 5 seconds. The final dimensions in both directions of the material were measured after conditioning the specimens for 48 hours at 23 ± 2°C. Five specimens were tested for each film.

3. Results and Discussions

3.1 Film thickness

The total film thickness and the gas barrier layer thickness for all film structures are reported on Table 1 . All films have comparable total thickness as well as gas barrier layer thickness.

Table 1.
Total film thickness and gas barrier layer thickness of EVOH and PVDC (control) multilayer shrinkable films.

Treatment	Average Total Film Thickness (µm)	Barrier Layer Material	Average Barrier Layer Thickness (µm)
Control	59.2	PVDC	4.6
EVOH32-1	69.3	EVOH	4.6
EVOH32-2	67.9	EVOH	4.6
EVOH44-1	56.5	EVOH	3.5
EVOH44-2	62.1	EVOH	4.1

3.2 Seal strength

Fresh beef is typically packed under vacuum to remove as much oxygen as possible from the inside of the package. After it is sealed, a package must provide a hermetic closure to prevent oxygen ingress into the package allowing spoilage bacteria growth. Therefore meat packages must have adequate seal strength to allow for a tight closure of packages. Seal strength results for all film samples varied from 1.5 to 2.6 kN/m (Figure 1) and no significant difference can be assigned to any of the mean values obtained for the five film samples.

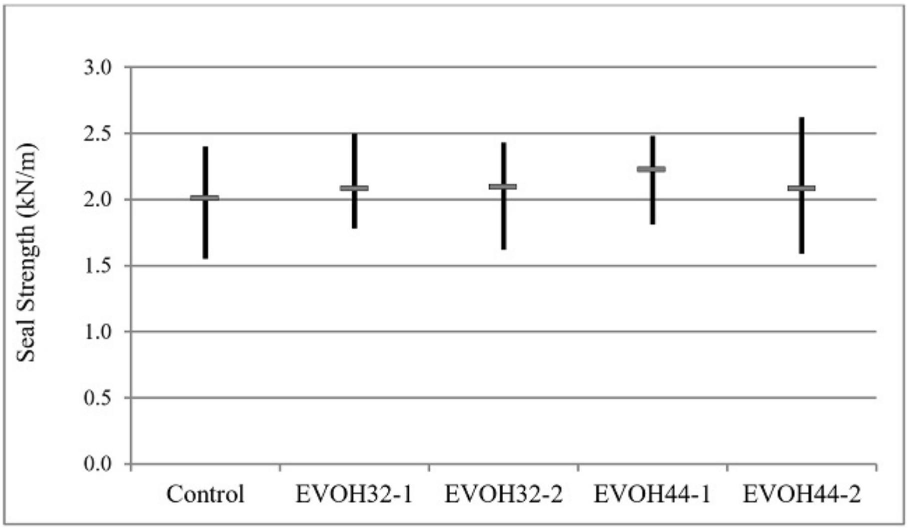

Figure 1.
Bottom seal strength at yield of EVOH and PVDC (control) multilayer shrinkable films. Control: EVA/tie/PVDC/tie/LLDPE. EVOH32-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/tie/LMDPE/LLDPE; EVOH32-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/PA6/66+aPA/tie/LLDPE; EVOH44-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/tie/LMDPE/LLDPE; EVOH44-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/PA6/66+aPA/tie/LLDPE.

3.3 Puncture resistance

Puncture resistance measures the ability of films to resist pinholes caused by rough handling or sharp objects, such as meat bones. Similarly to seal failure, pinholes must be avoided in order to maintain vacuum inside the package.

The results in Figure 2 show that film samples containing EVOH have similar or better puncture resistance than the control EVA/PVDC/PE film. They also show that samples containing two modified PA layers (EVOH 32-2 and EVOH44-2) have superior puncture resistance in comparison to the control film for either the blunt or sharp probes. In fact, the best result obtained for the control sample is smaller than the lowest value obtained for the films containing two modified PA layers (EVOH32-2 and EVOH44-2).

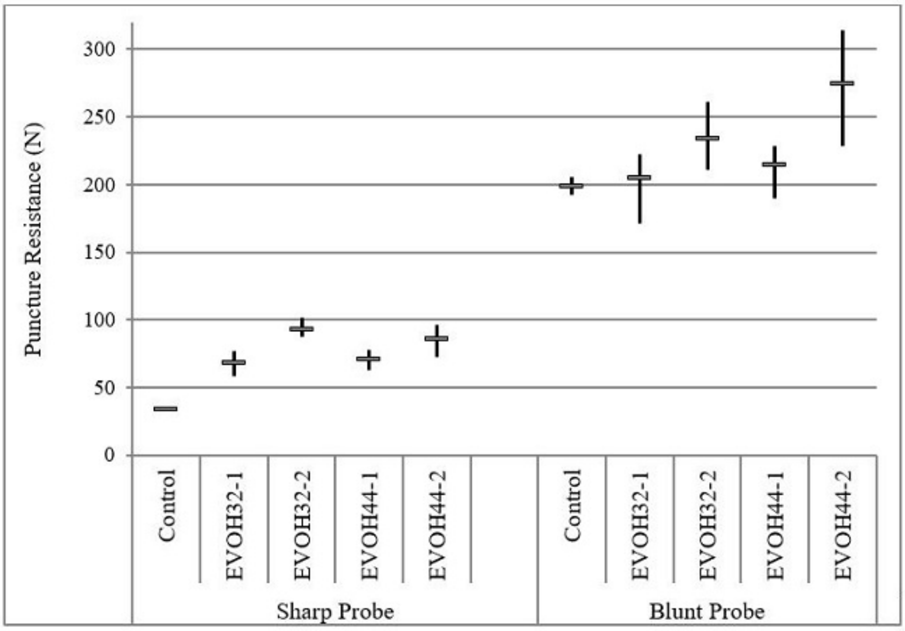

Figure 2.

Puncture resistance using sharp and blunt probes of EVOH and PVDC (control) multilayer shrinkable films. Control: EVA/tie/PVDC/tie/LLDPE; EVOH32-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/tie/LMDPE/LLDPE; EVOH32-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/PA6/66+aPA/tie/LLDPE; EVOH44-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/tie/LMDPE/LLDPE; EVOH44-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/PA6/66+aPA/tie/LLDPE.

3.4 Water vapour transmission rate (WVTR)

Although the WVTR is not a primary concern in fresh beef packages, water vapour barrier must be enough to protect the inner EVOH layer against moisture absorption to prevent oxygen permeation into the package.

The results on Figure 3 show that all samples containing EVOH layers in their structures have significantly higher WVTR values than the control. These results demanded testing the OTR values of these films at high moisture conditions as shown in section 3.5.

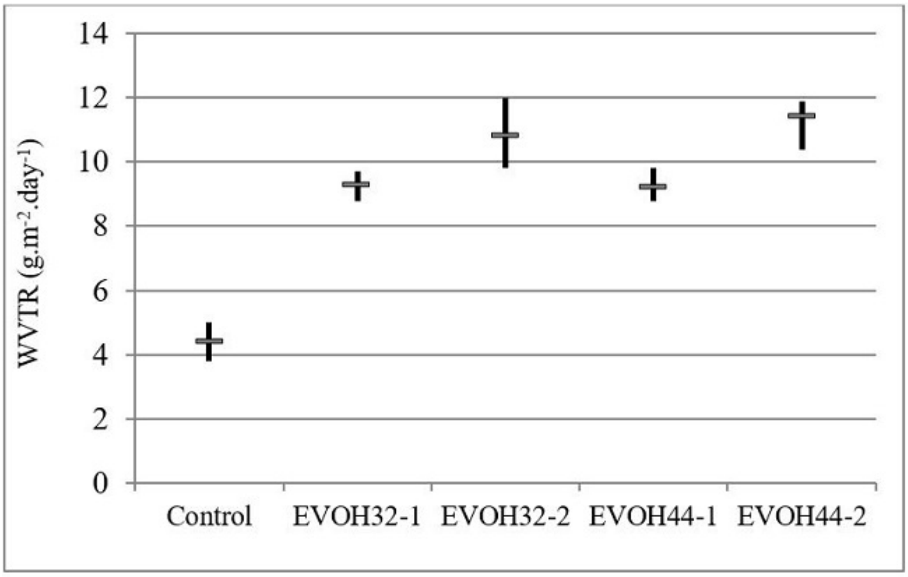

Figure 3.
Water vapour transmission rate of EVOH and PVDC (control) multilayer shrinkable films. Control: EVA/tie/PVDC/tie/LLDPE; EVOH32-1: PET/tie/Ionomer/tie/PA 6/66+aPA/EVOH 32 mol%/tie/LMDPE/LLDPE; EVOH32-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/PA6/66+aPA/tie/LLDPE; EVOH44-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/tie/LMDPE/LLDPE; EVOH44-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/PA6/66+aPA/tie/LLDPE.

3.5 Oxygen transmission rate (OTR)

The OTR results at high humidity conditions (75% RH) compared in Figure 4 show that films containing EVOH 44% mol of ethylene (EVOH44-1 and EVOH44-2) have higher permeation rates compared to films containing EVOH 32% mol (EVOH32-1, EVOH32-2) and to the control samples. Additionally the EVOH32-2 film, containing two PA blend layers shows the best oxygen barrier results of the films tested suggesting that the amorphous PA in the PA layer protects the EVOH layer against moisture gain. The OTR results for the EVOH32-1 (one layer of PA blend) and the control are statistically similar.

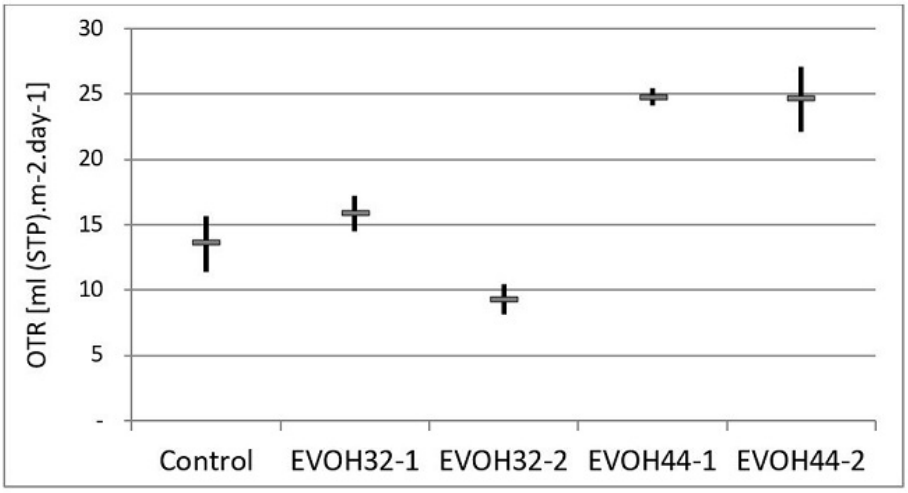

Figure 4.

Oxygen transmission rate at 23 ºC and 75% RH of EVOH and PVDC (control) multilayer shrinkable films. Control: EVA/tie/PVDC/tie/LLDPE; EVOH32-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/tie/LMDPE/LLDPE; EVOH32-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/PA6/66+aPA/tie/LLDPE; EVOH44-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/tie/LMDPE/LLDPE; EVOH44-2: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/PA6/66+aPA/tie/LLDPE.

3.6 Film shrink

The shrink results (Figure 5) indicate that all films made with EVOH have balanced shrink ratios in the machine (MD) and transversal directions (TD). This indicates that for round or cubic shape beef cuts those films may provide a more homogeneous wrapping, retaining liquid inside the muscle tissue tough rendering better product retail display.

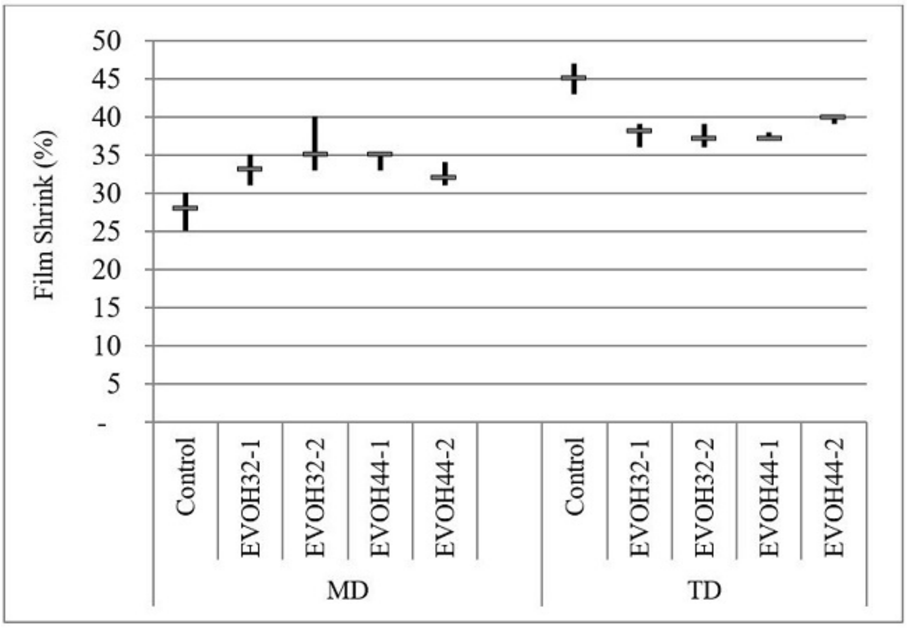

Figure 5.
Machine (MD) and Transverse Directions (TD) shrink of EVOH and PVDC (control) multilayer shrinkable films. Control: EVA/tie/PVDC/tie/LLDPE; EVOH32-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 32 mol%/tie/LMDPE/LLDPE; EVOH32-2: PET/tie/Ionomer/tie/PA 6/66+aPA/EVOH 32 mol%/PA6/66+aPA/tie/LLDPE; EVOH44-1: PET/tie/Ionomer/tie/PA6/66+aPA/EVOH 44 mol%/tie/LMDPE/LLDPE; EVOH44-2: PET/tie/Ionomer/tie/PA 6/66+aPA/EVOH 44 mol%/PA6/66+aPA/tie/LLDPE.

On the other hand, the control sample shows higher shrinking in the TD than in the MD. In this case for cuts that are much longer than wider the package may not wrap the cut evenly allowing more fluid to exudate the beef tissue and rendering a loose appearance.

4. Conclusions

We concluded that film samples containing EVOH 32% mol of ethylene are more effective to prevent oxygen permeation even under high moisture conditions than films made with EVOH 44% mol. Furthermore, the EVOH32-2, film structure with an EVOH layer encapsulated between two layers of PA 66/6+aPA blend, shows the lowest OTR values, supporting the positive roll played by the aPA in the blend to improve the gas and moisture barrier under high moisture.

Samples containing two layers of PA 66/6+aPA blends (EVOH32-2 and EVOH44-2) offered the best puncture resistance, clearly showing the contribution of the PA blend in improving film toughness.

The combination of triple bubble blow film technology to produce these films and the selected resins resulted in more even TD and MD shrink ability of all the films containing EVOH without affecting significantly the seal strength results as compared to the control sample.

In summary, the results obtained under controlled conditions indicate that the nine layer films containing EVOH 32% mol of ethylene, ionomer and PA 66/6+aPA blend in its composition, and especially the structure with EVOH 32% mol encapsulated between two layers of PA blend, have comparable or even slightly better performance features comparied to the control, in addition to be chlorine free and not requiring the radiation crosslinking used in the production of films made with EVA, PVDC and PE.

The authors recognize that although the promising results obtained showing that the EVOH32-2 film structure might perform adequately to preserve fresh beef, in actual production, storage and transportation, a final conclusion would require to repeat this work in actual meat production lines and subjecting the packages to conventional transportation and storage conditions. Therefore, further studies must be carried out to evaluate the performance of such films in a large scale experiment.

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