Blends of PP/PE Co-Octene for Modified Atmosphere Packaging Applications

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The aim of this work was to obtain films of polypropylene (PP)/polyethylene co-octene (POE) blends and study the influence of their composition and mixing conditions on final morphology and ultimate properties (thermal, mechanical, oxygen and water vapour transmission rates).

Scanning electron microscopy showed segregation of POE domains in the PP matrix. Thermal analysis indicated that the addition of POE modified neither the melting temperature nor the crystallinity of PP in the pressed films. It was found that permeabilities of films were mainly related to POE content rather than morphological features. For the maximum POE concentration used (40%), the oxygen permeability increased up to ~100% from that of neat PP. A similar trend was found for water vapour permeability. Rigidity of blends dropped significantly with the addition of POE although their ductility slightly increased when compared to pure PP. No significant effect of blend preparation conditions on these properties was found. Mechanical and permeability characteristics of films make them very attractive to be used for MAP of fresh produce such as apple, blueberry and mushroom. Copyright © 2011 John Wiley & Sons, Ltd.

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KEY WORDS: polypropylene; polyethylene co-octene; packaging; modified atmosphere packaging; fresh produce

INTRODUCTION

It is known that fruits and vegetables carry out their aging and senescence processes by a respiration cycle, which consumes oxygen (O_2) and evolves carbon dioxide (CO_2). Such respiration cycle remains active even after harvest. The higher the rate of respiration, the faster the stages of aging and senescence proceed. So it is advisable to decrease the respiration process in order to maintain freshness and to extend shelf life of produce.^{1–3} Low temperature along with low O_2 and high CO_2 levels are effective in reducing respiratory activity and spoilage mechanisms of fresh produce.^{3,4} Modified atmosphere packaging (MAP) is a passive way to create an appropriate gas composition around the produce, which is typically packaged in plastic bags during shipping, storage and marketing.^{1,5}

Modified atmosphere packaging is a dynamic system: the gas composition inside the package depends on the interaction between the natural process of respiration of fresh produce and the gas flow through the package plastic film. Zagory and Kader¹ gave emphasis to the proper balance between O_2 consumption and CO_2 production of the fresh produce, and the diffusion rate of these gases across the film as a key factor to develop a suitable modified atmosphere. Besides, the relative humidity inside the package is influenced by the rate at which the product losses water and by the water vapour transmission rate of the film.^{2,3}

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Each foodstuff item has often unique packaging requirements, so the ability to customize the package to the product requirements has been the aim of produce package development efforts. Polymeric films of polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC) and polyethylene terephthalate (PET)^{2,5,6} are most often found in flexible package structures (nearly 90%) for fresh produce. They provide not only a range of permeability to gases and water vapour but an adequate mechanical performance for package integrity. Frequently, different materials are combined through coating, lamination and coextrusion processes to achieve packaging films exhibiting the best relation between good properties and low cost. Although for many applications, advanced technology for instance perforated, microperforated, microperous, metallocene catalysed films, etc.^{7–10} are used.

There are many reports of successful applications of polymeric films for MAP of fresh produce.¹¹⁻¹⁴ For example, Rocha et al.¹⁵ packed apples in bags of 100 μ m PP films for 6.5 months at 4°C and 85% relative humidity (RH) and found that they lost less weight, presented better colour and firmness than fruits stored in air. Geeson et al.¹⁶ used microperforated low-density PE films (30 μ m) for packing apples and found that it was able to provide a favourable atmosphere during a simulated 4-week marketing period under ambient conditions. Avella et al.¹⁷ stored minimally processed apples in PP films filled with calcium carbonate nanoparticles and found that they were able to preserve the apple slices for up to 10 days, limiting oxidation processes and microbiological growth. Many studies were made using PE and PVC films to extend tomato shelf life up to 21 days.^{18,19} An et al.²⁰ studied the quality of honey peach fruit stored at 2°C in low-density PE bags of different thickness. They found that peaches packed in 25 μ m films showed the best quality at the end of 20 days of storage. Liu *et al.*²¹ evaluated the effect of MAP on the quality of fresh-cut pineapples stored at 4°C for 7 days with different pre-treatments. They found that either MAP or ascorbic acid/sucrose pre-treatment could reduce respiration rate, ethylene production, textural and colour deteriorations. Also, they found that MAP was more effective than pre-treatments in restraining microbiological growth. Serrano et al.²² stored broccolis using macroperforated, microperforated and non-perforated PP films and observed that all changes related to loss of quality were significantly reduced and delayed in time.

Another attractive way to obtain new materials with tailored properties for MAP applications is the blending of different polymer species. It usually results in immiscible two-phase systems with final properties that depend on the composition, the viscoelasticity of the individual components and the blending conditions.^{23–28}

Among polyolefins, PP is a thermoplastic with a number of desirable properties such as high melting temperature, low density and high chemical inertness. In addition, its low cost places PP in an advantageous position with respect to most other plastic materials. However, a relatively low permeability to gases, low flexibility and high rigidity mainly at low temperatures, limit the range of its applications.²⁹

Alternatively, the advancements in metallocene catalyst technology allowed for an efficient control of the molecular structure of polyolefins. For example, the incorporation of various degrees of long chain branches to polyethylene-produced materials with very low densities, elastomeric characteristics and high permeability to gases^{30,31} like metallocenic ethylene-octene copolymers (POE).

With the aim of improving PP properties mainly at low temperature, POE seems to be an attractive material. The objective of this work was to study PP/POE blends focusing in the effects of dispersed phase (POE) amount and morphology, as well as the mixing conditions on blend permeabilities and final mechanical and thermal properties. We were interested in the design and preparation of films of PP/POE blends exhibiting high levels of oxygen and water vapour permeabilities to match the respiratory quotient (CO_2 production/ O_2 consumption) and moisture loss of selected fresh produce at storage conditions. In this way, optimal atmosphere could be achieved inside the package and an extended shelf life of the produce would result.¹

MATERIALS AND METHODS

Blends were prepared in a counter-rotating batch mixer (Brabender GmbH & Co. KG, Duisburg, Germany) at different temperatures and screws rotational speed. The basic materials used in this study

		Blending conditions			
Sample code	wt% POE	205°C	215°C	50 rpm	80 rpm
8020 T ₁ V ₁	20	•		•	
$8020 T_1 V_2$		•			•
$8020 T_2 V_1$			•	•	
8020 T ₂ V ₂			•		•
7030 T ₁ V ₁	30	•		•	
7030 T_1V_2		•			•
7030 T_2V_1			•	•	
7030 T ₂ V ₂			•		•
6040 T ₁ V ₁	40	•		•	
6040 T ₁ V ₂		•			•
6040 T ₂ V ₁			•	•	
6040 T ₂ V ₂			٠		•

Table 1. Sample codes and blending conditions for blends with 20, 30 and 40 wt% of POE.

were a commercial grade PP (density 0.908 g/cm³, melt flow index (MFI) 3.4 g/10 min measured under 2.16 kg at 230°C) and a commercial grade POE (density 0.885 g/cm³, MFI 1.0 g/10 min measured under 2.16 kg at 190°C).

Calculated amounts of the polymer species were mixed at 205°C or 215°C and at 50 rpm or 80 rpm for 10 min. Table 1 summarizes the sample codes and the blending conditions used (i.e. 8020 T_1V_1 means a blend with 20 wt% POE mixed at 205°C and 50 rpm).

Film samples with a thickness of about 0.1 mm were obtained from blend pellets by applying \sim 140 kg/cm² in a hydraulic press at 205°C during 60 s. Then films were quenched into water at room temperature. Films at other pressing times (30 s, 180 s, 300 s and 600 s) were also prepared to be used in further morphological analysis.

Melting behaviour was analysed in a Perkin Elmer Pyris I Calorimeter (Waltham, Massachusetts, USA). Film samples of about 7 mg were heated from 20°C to 190°C at 10°C/min. The temperature scale was calibrated at 10°C/min against indium (156.6°C) and the melting behaviour was recorded. Three specimens from each sample were tested.

The morphological aspects of films were studied by scanning electron microscopy (SEM) using an EVO 40 XVP microscope (Carl Zeiss NTS Ltd., Cambridge, UK). Film samples were fractured at cryogenic temperature, and then, their surfaces were etched with hot n-heptane and rinsed with fresh n-heptane to remove the dissolved polymer. Finally, they were gold coated. The incident beam was perpendicular to the surface and the voltage used was 7 kV.

The oxygen permeability (OP) of films was measured in an Ox-Tran 2/21 (Mocon, Minneapolis, USA) at dry condition and at 10°C, 23°C, 30°C and 40°C. Masked films with an open area of 5 cm² were exposed in one side to the carrier gas (nitrogen) and the other side to the test gas (oxygen). Both flowing gases were automatically controlled to the same selected temperature. At least three determinations were carried out for each film sample.

The water vapour permeability (WVP) was measured according to the desiccant method detailed in standard test methods for water vapor transmission of materials (ASTM E96). Films were sealed to the open mouths of acrylic test dishes containing dried calcium chloride and these assemblies were placed in a chamber with controlled atmosphere at 50% RH and 25°C. The cells were weighed periodically for at least 7 days and the weight against elapsed time was plotted. The slope of the linear portion of this curve is the rate of water vapour transmission (w/t) and WVP was calculated as

$$WVP = \frac{w \cdot e}{t \cdot A \cdot \Delta p} \tag{1}$$

where w is the weight gain (g), e is the film thickness (cm), t is the time of measurement (day), A is the permeation area (m²) and Δp is the vapour pressure difference between both sides of films (atm). All tests were carried out in at least three repetitions.

Tensile stress–strain behaviour of film samples was studied at room temperature in an Instron 3369 (Norwood, USA) operated at a cross-head speed of 5 mm/min for elastic modulus and 500 mm/min for yield strength and elongation at break determinations. A minimum of 10 samples were tested for each blend series. The average values as well as standard deviations were calculated.

Statistical difference (p < 0.05) analysis between mean values of properties obtained for different film compositions and blending conditions was performed by the unpaired Student's *t*-test with unequal variance.

RESULTS AND DISCUSSION

Thermal analysis

Figure 1 shows the superimposed thermograms of PP, POE and the blend films analysed. The observed melting peak of PP (~162°C) is characteristic of α -crystals melting.³² POE showed a bimodal thermogram with melting peaks at ~45°C and ~80°C. Thermograms of binary blends exhibited a broad melting endotherm in the range of melting of POE and a PP melting peak.

As it is shown in Table 2, the melting temperatures (T_m) of PP in all blends were similar and they did not show significant differences with the melting temperature value of pure PP. It suggests that PP crystal sizes and perfection were not greatly modified by the addition of POE. Also, no significant differences between the enthalpy of fusion of the PP melting peak (ΔH_{PP}) in pure PP and in the blends were found, meaning that the degree of crystallinity of PP was similar irrespective of the blend composition. Table 2 also shows the percentage of crystallinity calculated from the ratio of ΔH_{PP} to ΔH for a theoretical 100% crystalline PP (209 J/g).³³ From these results, it can be said that the addition of POE had little effect on the PP crystallization process.

The analysis of variance did not show statistical differences (p > 0.05) in the experimental data at each composition within the mixing conditions evaluated.



Figure 1. DSC thermograms of PP, POE and PP/POE blends. DSC heating rate was 10°C/min.

Table 2. Me yield stre	elting tempers $\operatorname{ngth}(\sigma_y)$ and	ttures (T _m), enth elongation at b	alpy of fusion of PP (2 reak (El _b) of neat PP, F	MPp), crystallinity, oxygen peri OE and PP/POE blends prepar	meability (OP), water vapour ted at different blending condi	permeability (W itions. Standard	VP), elastic mo deviations are i	odulus (E), ncluded.
Sample	T _m (°C)	ΔH _{PP} (J/g)	Crystallinity (%)	OP (cm ³ .mm/m ² .day.atm)	WVP (g.cm/m ² .day.atm)	E (MPa)	σ _y (MPa)	El_{b} (%)
PP	162	85	41	114 ± 3	0.70 ± 0.04	527 ± 12	23.4 ± 0.5	571 ± 49
POE	45 - 80	I	I	948 ± 32	2.98 ± 0.30	N/A	N/A	N/A
$8020 T_1 V_1$	162	86	41	173 ± 6	1.14 ± 0.09	416 ± 11	15.2 ± 0.4	670 ± 52
$8020 T_1 V_2$	162	86	41	172 ± 5	1.22 ± 0.05	420 ± 12	14.9 ± 0.2	663 ± 60
$8020 T_2 V_1$	162	87	42	175 ± 7	1.23 ± 0.04	409 ± 9	14.8 ± 0.3	667 ± 41
$8020 T_2 V_2$	162	86	41	169 ± 3	1.15 ± 0.09	411 ± 9	15.2 ± 0.4	673 ± 48
$7030 T_1 V_1$	162	87	42	182 ± 9	1.31 ± 0.07	380 ± 12	13.6 ± 0.4	692 ± 61
$7030 T_1V_2$	162	88	42	181 ± 8	1.32 ± 0.09	368 ± 10	13.1 ± 0.3	684 ± 55
$7030 T_2 V_1$	161	88	42	185 ± 4	1.34 ± 0.08	364 ± 11	13.5 ± 0.4	683 ± 32
$7030 T_2 V_2$	161	87	42	184 ± 3	1.31 ± 0.06	375 ± 10	13.5 ± 0.3	669 ± 60
$6040 T_1 V_1$	161	88	42	235 ± 3	1.58 ± 0.10	285 ± 9	10.2 ± 0.3	712 ± 25
$6040 T_1 V_2$	162	88	42	233 ± 4	1.57 ± 0.08	280 ± 11	10.1 ± 0.4	718 ± 63
$6040 T_2 V_1$	162	85	41	232 ± 8	1.66 ± 0.16	297 ± 12	10.1 ± 0.3	710 ± 34
$6040 T_2 V_2$	161	85	41	233 ± 10	1.67 ± 0.12	288 ± 7	10.7 ± 0.5	716 ± 35

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Morphology

Figure 2–4 show SEM micrographs of PP/POE blends of different compositions and prepared at different mixing and moulding conditions. The dark holes correspond to amorphous POE particles, which were dissolved out by the selective etching process.

Blends with 20 wt% of POE (Figure 2a–d) presented a quite homogeneous elastomeric phase distribution in the PP matrix through the whole transverse area of the film. The POE particles were mostly spherical; only a few of them were elongated and their sizes were in the range of $0.1-1 \mu m$. Analogous morphologies were found by changing temperature or mixing velocity.

Figure 3a–b show micrographs of 7030 blends. Similar morphologies as for 8020 blends were observed, with a somewhat larger content of elongated particles. This effect could be assigned to elastomer coalescence due to the higher POE concentration.²³

When POE content was increased to 40 wt%, like in the 6040 blends (Figure 4a–d), two different morphologies appeared: platelets near the surface (named '1' in Figure 4) and spherical domains in the core of the film (named '2' in Figure 4). It could be associated to a squeezing effect near the surface during pressing for a time not long enough to allow the relaxation of stresses of the elastomer particles.



Figure 2. Comparison of SEM micrographs of batch-mixed PP/POE blends with 20 wt% of POE and pressed for 60 s: (a) 8020 T₁V₁; (b) 8020 T₁V₂; (c) 8020 T₂V₁; (d) 8020 T₂V₂. The temperature variation is indicated as a row while the mixing velocity variation is shown as a column.



Figure 3. Comparison of SEM micrographs of batch-mixed PP/POE blends with 30 wt% of POE and pressed for 60 s: (a) 7030 T_1V_1 ; (b) 7030 T_1V_2 .



Figure 4. Comparison of SEM micrographs of batch-mixed PP/POE blends with 40 wt% of POE and pressed for 60 s: (a) 6040 T₁V₁; (b) 6040 T₁V₂; (c) 6040 T₂V₁; (d) 6040 T₂V₂. The temperature variation is indicated as a row while the mixing velocity variation is shown as a column.



Figure 5. Comparison of SEM micrographs of batch-mixed PP/POE blends with 40 wt% of POE, mixed at 205°C and 80 rpm at different pressing times: (a) 30 s; (b) 180 s; (c) 300 s; (d) 600 s.

As can be seen in Figures 2–4 for each blend composition, the morphological features appeared to be independent of the preparation conditions used.

In order to study the pressing time effect on blend morphology, some $6040 \text{ T}_1\text{V}_2$ films were prepared using different pressing times. Micrographs of transverse area of films obtained at pressing times of 30 s, 180 s, 300 s and 600 s are shown in Figure 5a–d, respectively. While POE domains in the core of the film (named '2' in Figure 5) increased their sizes, platelets near the surface (named '1' in Figure 5) became smaller and more spherical. Particles sizes in part '2' were in the range of 0.1–1 μ m when pressing time was 30 s. Meanwhile, when pressing time was increased up to 600 s, the particles sizes were about $0.1-3.5 \ \mu\text{m}$. The observed morphological changes could be attributed to elastomer coalescence in the core of the film and to stress relaxation on the surface side.

Oxygen and water vapour permeabilities

Oxygen permeability coefficients at 23°C are summarized in Table 2 for pure components and blends. The statistical analysis performed showed that for a given amount of POE, the OP was almost the same irrespective of blending conditions (temperature and screws rotational speed). However, OP appeared to be more dependent on POE content. For example, when POE content was increased from 20 wt% to 30 wt%, blends OP increased from 50% to 60% with respect to neat PP. For 6040 blends, the OP was enhanced in about 105% from the value corresponding to neat PP. This behaviour could be directly attributed to the more permeable characteristics of the dispersed phase (OP_{POE}/OP_{PP} \approx 8.3).

As the intended use of these films is in the area of fresh produce packaging, it is very useful to know how the OP is affected by temperature fluctuations, which may occur during storage and shipping. The atmosphere generated inside the package depends on the respiration rate of produce and on the permeability of films, both of which are affected by temperature.³⁴ The dependence of OP with temperature is shown in Figure 6 for each blend composition and neat PP. Generally speaking, as the temperature was increased from 10°C to 40°C, the OP values of the films increased significantly following a relationship very close to an exponential fit.

A good fit of ln(OP) versus (1/T) was found for each composition tested evidencing that an Arrhenius-type equation was followed:



$$OP = OP_0 \cdot \exp\left(-\frac{E_a}{RT}\right) \tag{2}$$

Figure 6. Variation of oxygen permeability coefficients with temperature of neat PP and blends with 20, 30 and 40 wt% of POE. The error bars represent \pm standard deviation.

where E_a is the activation energy (KJ/mol), R is the universal gas constant, T is the absolute temperature (K) and OP_0 is a pre-exponential factor (cm³.mm/m².day.atm). No significant differences (p > 0.05) in E_a for OP were found for blends with increasing amounts of POE, meaning that the oxygen permeation rate through all films tested had almost the same sensitivity to temperature changes ($E_a \sim 40 \text{ kJ/mol}$). For packaging of fresh produce, the film must show a temperature sensitivity value similar to the activation energy of the product respiration rate (E_a^{ro2}). It assures that an optimal gas concentration inside the package will be generated and maintained. The magnitude of changes in package atmosphere is strongly related to the difference in both activation energies. Therefore, this parameter must be taken into account to make a good choice of a packaging material for a given fresh produce and storage conditions. For example, Song *et al.*³⁵ found that E_a^{ro2} for blueberry was in the range of 43–48 kJ/mol, Andrich *et al.*³⁶ reported values of E_a^{ro2} –44 kJ/mol for Golden Delicious apple, and Varoquaux *et al.*³⁷ found that E_a^{ro2} for mushroom was ~43 kJ/mol.

The in-package relative humidity required to minimize moisture loss from fresh produce and to prevent condensation, is strongly related to the WVP of the packaging film. Thus, it is important to know the rate at which water vapour is lost through the package to the surrounding atmosphere due to vapour pressure difference across the film.

Table 2 shows WVP coefficients of each assayed blend. Statistical analysis demonstrated that the different preparation conditions did not affect significantly WVP of blends with a given amount of POE (like the trend found for OP). These results were assigned to the similar morphologies obtained at equal-blend composition.

Considering WVP average values for each composition, a great increase from that of neat PP was observed when more POE was added to the blends: almost 70%, 90% and 130% of increment for blends containing 20 wt%, 30 wt% and 40 wt% POE, respectively. This fact was mainly attributed to the greater content of the more permeable phase (WVP_{POE}/WVP_{PP} \approx 4.3) rather than preparation conditions.

Tensile properties

Typical stress–strain curves for each blend composition and neat PP are shown in Figure 7 from which elastic modulus (E), yield strength (σ_y) and elongation at break (El_b) were calculated, and they are shown in Table 2.



Figure 7. Stress-strain curves for neat PP and PP/POE blends.



Figure 8. Variation of elastic modulus with wt% of POE. The error bars represent \pm standard deviation.

As POE was added to PP the value of E dropped, meaning that blends lost rigidity. The maximum decrease in E was shown by 6040 blends, being half the value for neat PP. No significant differences (p > 0.05) in E values were obtained when different mixing conditions were used.

All blends showed σ_y values significantly lower than that of neat PP and they decreased with the addition of POE. E and σ_y were highly dependent on blend composition and followed an almost linear relationship when they were plotted against POE content (Figures 8 and 9). It suggested that the linear law of mixtures was obeyed for all blends prepared regarding E and σ_y .

Elongation at break of blends was higher than that of neat PP. Ductility was not dependent of blending conditions and increased slightly with POE content. The experimental results for El_b were in the range of 650–750%. Blends also exhibited a strain hardening effect, which could be attributed to the plasticization effect of POE.

CONCLUSIONS

- The morphology of the PP/POE blends studied by SEM consisted of a PP matrix with dispersed POE domains. In the composition range studied, the mixing conditions did not affect significantly the shape or sizes of the domains. The blend composition had an important effect on the shape of POE domains, which changed from mainly spherical for 8020 blends, to a coexistence of platelets and spherical particles in 6040 blends.
- Differential scanning calorimeter (DSC) results indicated that PP crystallization process was not significantly modified by the addition of POE. The melting temperatures of the blends were similar to those of pure PP, irrespective of the blend composition.
- As POE was added to PP, blends lost rigidity and increased ductility exhibiting a strain hardening
 effect. Elastic modulus and yield strength varied according to the rule of mixtures, meaning that
 they were basically dependent on the blend composition. The values found for E and Elb of blends
 are in the range of commonly used flexible packaging materials.



Figure 9. Variation of yield strength with wt% of POE. The error bars represent \pm standard deviation.

- The addition of POE to PP was an effective way to prepare PP-based blends with enhanced oxygen and water vapour permeability. Both OP and WVP of blends increased with the addition of POE. No significant differences in the activation energies for OP were found for all blends prepared meaning that they had almost the same sensitivity to temperature changes.
- PP/POE films prepared seem to be suitable for MAP packaging of fresh produce such as apples, blueberries and mushrooms.
- Future work will be focused in the storage of apples under MAP conditions. Relevant parameters such as apple mass, respiratory quotient, proper permeability, area and thickness of films among others would be considered in order to achieve a successful MAP system.

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