



## Transport properties of multilayer active PET films with different layers configuration



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### ABSTRACT

The integration of active scavengers inside polymeric films is still facing technological difficulties related to material storage and handling, because of the rapid exhaustion of the scavenger due to the reduced thickness involved. Therefore, the development of multilayer active structures and the optimization of active and inert layers configuration represent crucial points to achieve the best performance. In this article, the effect of different layouts on the transport properties of multilayer active PET films is investigated. Symmetrical, “IAI” films were produced by means of cast co-extrusion process, inserting the active layer (A), containing the oxygen scavenger, between two inert layers (I). Four different layouts were obtained, combining two thicknesses for the active layer and two for the inert layers. Oxygen absorption measurements pointed out the effectiveness of the inert layers in prolonging the scavenging activity of the active phase, controlling the oxygen diffusive flux through the core layer. The results highlighted the impact of the multilayer films configuration on the scavenging parameters (initial oxygen scavenging rate, exhaustion time, amount of oxygen absorbed per unit surface and scavenging capacity), the possibility to discriminate the individual contributions of active and inert layers and the good reproducibility of the films scavenging performance. Furthermore, results revealed the effect of the oxygen scavenger phase on PET morphology and functional properties, regarding the crystallization process, the oxygen permeability and tensile parameters.

### 1. Introduction

Nowadays, the increasing demand for safe food products with prolonged shelf life, as well as functional, low cost, environmental friendly packaging, represent the driving forces that have prompted the food packaging industry towards innovations in materials and technologies.

Among all these innovations, active packaging solutions are certainly the most challenging: this technology allows the packaging to interact directly with food or food surfaces by releasing or absorbing specific substances, to prolong the shelf life of sensitive foods and to guarantee their safety and freshness, through a continuous modification of the environment inside the packaging. Food and packaging are no more seen as two separate entities, and the old, traditional role of generic containment of the packaging is overcome.

One of the main causes of food deterioration is due to the presence of oxygen inside the package of sensitive products. Oxygen causes changes in flavors, color, fat rancidity, degradation of vitamins and nutrients, as well as encourages microbial growth and spoilage.

Traditional packaging methods for extending O<sub>2</sub>-sensitive food shelf life include modified atmosphere packaging (MAP), in combination

with passive barriers, such as multilayer structures containing EVOH or aluminum foil, or polymer blends or nanocomposites [1–17].

However, these methods cannot remove completely the oxygen into the package headspace, dissolved into the food, or permeating through the package walls during the storage [18,19].

Oxygen Scavenger (OS) systems have revolutionarily simplified the process of gas reduction inside the package, thanks to their capability to remove oxygen permeating through the packaging material and/or residual oxygen that may have been trapped inside the package prior to sealing [20], and also eliminated costly equipment and technologies [21].

Although iron-based oxygen scavengers still occupy a leading position among the commercialized systems [22–25], many concerns related to their potential health risks, the consumer acceptability, and their difficulty of use with liquid products prompted the research towards incorporation of oxygen-scavenging compounds into the packaging material [26–32]. In particular, the design and development of non-visible, integrated OS systems, easy to blend with the polymer matrix and to process through conventional technologies, is raising growing interest, and new patents are continuously registered [33–37].

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Currently, polymeric OS are widely used to improve barrier properties of Poly(ethylene terephthalate) containers. PET is relevant for packaging foods, such as beverages and oils, thanks to its capability to ensure good protection from environmental contamination, the good optical and mechanical properties, the recyclability advantages, and the ease of use with traditional processing technologies [38,39]. Despite its intrinsic low oxygen permeability with respect to other commercial polymers [40], efforts are still made to increase its barrier performance, especially in those cases where particularly compelling requirements are imposed by the nature of the packaged food [45]. The addition of a scavenging compound to the plastic matrix provides an active protection, which results in the same advantages of heavier and more expensive packaging materials [41].

The necessity to improve the barrier properties of the polymeric material becomes even more compelling in the case of flexible packaging, due to the reduced thicknesses involved. The replacement of rigid containers with films for packaging foodstuff is becoming increasingly popular thanks to several advantages, such as reduced weights and volumes, reduced raw materials employment, reduced waste and disposal issues, and therefore reduced costs for production and transport.

However, many literature studies were interested in the development of integrated OS systems for PET rigid packaging [41–46], but only few have dealt with the application of these scavengers to flexible packaging.

The integration of the OS inside polymeric films to achieve a significant active barrier is still facing some practical difficulties, particularly in case of self-activating oxygen scavengers. Major issues concern the necessity: (i) to adopt precautions during material storage and handling, to avoid premature depletion of the OS films as soon they are manufactured, prior to filling the package; and (ii) to prevent the excessively fast oxidation of the scavenger, delaying the exhaustion of the active phase until the end of the food shelf life is reached.

Among all possible strategies, joining the active and the passive barrier in a multilayer structure could represent an interesting solution to control the diffusive flux of the oxygen and to modulate the performance of the active system.

The realization of a multilayer structure, by inserting the active layer between two or more inert, barrier layers, would create a synergistic system in which the central layer traps and reacts with oxygen, thus providing an active protection, whereas the outer layers improve the passive protection, controlling the rate at which the OS exhausts.

According to our knowledge, there is little research regarding active multilayer systems development, or deeply studying the optimization of the layer thicknesses or the effects of the active phase on the transport and functional properties of the polymer [47–56].

Our previous studies [47,54] on active multilayer films pointed out the influence of film configuration on the oxygen absorption parameters. In the present work, a detailed analysis was conducted in order to discriminate the individual contributions of the active and the inert layers on the oxygen permeation and scavenging phenomena in terms of scavenging capacity, exhaustion time and oxygen absorption rate. This allows to obtain necessary information for the design of active multilayer structures with tunable OS performances.

These considerations inspired the aim of this work, which focuses on the production and characterization of PET active multilayer films, intended for food packaging applications.

Multilayer, “IAI” samples were produced by co-extrusion process by inserting the active layer (A), containing the oxygen scavenger, between two PET inert layers (I). Four multilayer films were obtained with different relative thicknesses of both the active and the inert layers. The scavenging performance of the films was assessed in terms of initial oxygen scavenging rate, exhaustion time, scavenging capacity and total volume of oxygen absorbed per unit surface. Moreover, the produced systems were characterized in order to ascertain the effect of the scavenger phase on the morphology and functional properties of the polymeric films.

## 2. Experimental

### 2.1. Materials

The selected polymeric matrix is the film grade PET resin Cleartur P60 (M&G Polimeri S.p.A., Patrica (FR), Italy), having intrinsic viscosity 0.58 dL/g. The active phase is a new generation of polymeric oxygen scavenger, named Amosorb DFC 4020E (AMS, supplied by Colormatrix Europe, Liverpool, UK). This is a copolyester-based polymer designed for rigid PET containers, characterized by an auto-activated scavenging mechanism. Both PET and AMS comply fully with FDA and EU food contact legislation.

For the preparation of the active systems, the PET was dried under vacuum at 130 °C for 16 h, before processing. The AMS, delivered dried in aluminum bags sealed under vacuum, was used as received. The percentage of the oxygen scavenger added to the active layer, equal to 10% w/wt, was already optimized by previous published studies [56].

The multilayer active films were produced by using a laboratory co-extrusion cast film line (Collin, Teach-line E20T), equipped with two single screw extruders (D = 20, L/D = 25) one feeding the active layer (A) and one feeding the inert layers (I), a flow convergence system (feed-block), a coat-hanger type head (slit die of 200 × 0.25 mm<sup>2</sup>) and a take-up/cooling system (chill rolls) thermally controlled by water circulation at 50 °C. The temperature profile for the two extruders was set at 280 °C from the hopper to the die. The chill roll speed was 7 m/min, thus allowing the films to be stretched to their final dimensions of about 170 mm wide and variable thicknesses.

The mass flow rates of both the extruders was varied in order to obtain a set of four multilayer films with a combination of two thicknesses for the active layer (a smaller one, equal to 13.5, and a larger one, equal to 23.5 μm) and two for the inert layers (a smaller one, equal to 6.75 and a larger one, equal to 11.75 μm). Films were named A<sub>I</sub>I<sub>S</sub>, A<sub>S</sub>I<sub>S</sub>, A<sub>I</sub>I<sub>L</sub> and A<sub>S</sub>I<sub>L</sub>, depending on the thickness of each layer (where S stands for Small and L stands for Large), as resumed in Table 1. Single layer films inert I (i.e. made of pure PET) and active A (i.e. made of PET + 10% w/wt AMS) were also produced, for comparison, using the same apparatus and processing conditions.

### 2.2. Physico-chemical characterization

#### 2.2.1. Thermal characterization of films

Thermal analyses on the produced films were performed using a Differential Scanning Calorimeter (DSC mod. 822, Mettler Toledo). The specimens were heated at a rate of 10 °C/min from 25 °C to 300 °C under a nitrogen gas flow (100 mL/min), in order to minimize thermooxidative degradation phenomena. The crystallinity degrees, X<sub>c</sub>, were calculated from first heating parameters, according to the following formula:

$$X_c = \frac{\Delta H_m - \Delta H_{cc}}{(1 - \phi * \omega) * \Delta H_{\infty}} \quad (1)$$

**Table 1**

Nominal thicknesses of the inert and active layers for I, A, A<sub>I</sub>I<sub>S</sub>, A<sub>S</sub>I<sub>S</sub>, A<sub>I</sub>I<sub>L</sub> and A<sub>S</sub>I<sub>L</sub> samples, calculated from the screw speed of extruders feeding layers I and A.

Sample	Total thickness [μm]	Inert layer 1 [μm]	Active layer [μm]	Inert layer 2 [μm]	Speed extruder A [rpm]	Speed extruder I [rpm]
I	35	35	–	–	–	40
A	25	–	25	–	25	–
A <sub>I</sub> I <sub>S</sub>	37	6.75	23.5	6.75	40	27
A <sub>S</sub> I <sub>S</sub>	27	6.75	13.5	6.75	27	27
A <sub>I</sub> I <sub>L</sub>	47	11.75	23.5	11.75	40	40
A <sub>S</sub> I <sub>L</sub>	37	11.75	13.5	11.75	27	40

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