



## Development of low-density polyethylene films with lemon aroma

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### ABSTRACT

The purpose of this study was to develop active films that impart flavour and to test them in the packaging of biscuits. The films were mechanically analysed, with respect to colour and water vapour permeability (WVP) to evaluate the changes in the films resulting from the active agents and conditioning time. We used low-density polyethylene with incorporated lemon essential oil (EO) and/or lemon aroma to create the films that were used in biscuits and evaluated over a 30 day period. The results showed that the films showed a lower elongation due to the incorporation of active agents, and they showed a reduction of tensile strength over time. In addition, the combined use of EO and aroma did not affect the WVP value. As for colour, flavouring films had a more yellow colour and were opaque. Sensory biscuits packed with flavouring films showed an average acceptance of approximately 8.0 based on aroma and taste. These films represent an innovation for the packaging industry, and, based on our results, we recommend the combined use of EO and aroma to develop films for flavouring.

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### 1. Introduction

Citrus essential oils (EOs) contain 85–99 g/100 g volatile components and 1–15 g/100 g non-volatile components. The volatile constituents are a mixture of monoterpene hydrocarbons (limonene), sesquiterpene hydrocarbons and their oxygenated derivatives, which include aldehydes (citral), ketones, acids, alcohols (linalool) and esters (Sawamura et al., 2004; Vaio et al., 2010). The major chemical component of citrus oils is limonene, ranging from 45 to 76 g/100 g or lemon. Citral and linalool are thought to be the most potent aromatic compounds in citrus fruits, but they do not exceed 3 g/100 g in lemon oil. Fatty acids make up a negligible percentage (about 0.2 mL/100 mL) of citrus oils, and the major fatty acid in lemon oil is linoleic acid (Fisher & Phillips, 2008; Svoboda & Greenaway, 2003).

*Abbreviations:* WVP, Water vapour permeability; EO, Essential oil; LDPE, Low-density polyethylene; PP, Polypropylene; TS, Tensile strength; E, Elongation at break; RH, Relative humidity; PCA, Principal Component Analysis.

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It is widely recognised that most of the essential oils have antimicrobial properties (Emiroğlu, Yemiş, Coşkun, & Candoğan, 2010; Fisher & Phillips, 2008; Suppakul, Sonneveld, & Bigger, 2011; Tsigarida, Skandamis, & Nychas, 2000). Individual components of EO, which are either extracted from plant material such as flowers, buds, seeds, leaves, twigs, bark, herbs, wood, fruits and roots (Bajpai, Baek, & Kang, 2011), or synthetically manufactured, are also used as food flavourings.

The ability of citrus oils to delay spoilage and add organoleptic qualities in food products may be interesting from a commercial point of view (Bajpai et al., 2011; Tunç & Duman, 2011). However, there are few studies evaluating EO compounds used to modify the sensory properties of foods (Gutiérrez, Batlle, Andújar, Sánchez, & Nerín, 2011; Kostaki, Giatrakou, Savvaidis, & Kontominas, 2009).

Food processing, heat treatment, concentration, evaporation, boiling, baking and the food matrix effect (Taylor, 2002) can result in a loss of flavour quality. To prevent this loss, active packaging materials can be used. Through of the incorporation of active agents in the polymer matrix, food can be aromatised by an interaction between the package and product. In addition to improving the sensory characteristics of foods, flavouring active packaging can be used to develop new products. From a processing line, you can obtain products of different flavours with the use of flavouring packaging in the conditioning stage. This is useful in a food industry

that relies mostly on incremental innovation for new product launches; there is an increasing awareness in the industry that innovations are needed to remain competitive.

The transformation of cereal products from dough to biscuit, for example, is a very complicated process involving numerous mechanisms and many properties that must be controlled, such as colour, shape, aroma and crispness (Perrot et al., 2000). Biscuits are an important class of bakery products that are produced in a large variety of flavours. Every day, new types of biscuits, often with innovative flavours, are launched on the market. The degree of protection required by biscuits is determined to a great extent by their composition and the manufacturing process. However, in general, the shelf life of biscuits depends fundamentally on the barrier properties of the packaging materials used to preserve and protect the product from the ingress of atmospheric moisture and other agents that negatively affect flavour (Alves, Garcia, & Bordin, 1999).

Our objective was to develop low-density polyethylene flavouring films imbued with essential oils and/or lemon aroma. We sought to apply these films to the packaging of biscuits to evaluate the mechanical properties, water vapour permeability and colour of the films and the sensory properties of the biscuits packaged in the active films.

## 2. Materials and methods

### 2.1. Materials definer

Low-density polyethylene (LDPE, Braskem, Brazil), high-density polyethylene with a high absorption capacity (Accurel XP200, Braskem, Brazil), lemon essential oil (EO) and lemon heat resistant aroma (Duas Rodas Industrial Ltda., Brazil) were used to prepare the flavouring film. These films have the ability to aromatize food by diffusion of the active compounds added to the polymer matrix.

### 2.2. Experimental design

We used a complete factorial design with the following factors: level of EO/aroma (film 1: without EO and without aroma; film 2: with 10 mL of EO and 5 mL of aroma/100 g of polymer; film 3: with 5 mL of EO and 5 mL of aroma/100 g of polymer; film 4: with 10 mL of aroma/100 g of polymer) (Table 1) and observation times (0, 10, 20, 30 days). The experiment was conducted using a completely randomised design, and all samples were prepared and analysed in triplicate.

### 2.3. Preparation of the film

For the development of films with LDPE lemon flavouring, the resin Accurel XP200 was imbued with EO and/or lemon aroma. Subsequently, the blend (LDPE + Accurel XP200) was extruded using a monorosca extruder HaakePoly Drive (Thermo, Germany) with an extruded tube and five temperature stages (temperatures of 120, 130, 140, 150, and 160 °C, respectively).

**Table 1**  
Flavouring films developed.

Films	Lemon	
	Essential oil (mL/100 g of polymer)	Aroma (mL/100 g of polymer)
1	0	0
2	10	5
3	5	5
4	0	10

### 2.4. Antimicrobial activity of EO

The antimicrobial activity of EO was evaluated by measurement of the inhibition zone sizes against *Staphylococcus aureus* (ATCC 6538), *Listeria innocua* (ATCC 33090), *Escherichia coli* (ATCC 11229), *Salmonella choleraesuis* (ATCC 6539), *Pseudomonas aeruginosa* (ATCC 15442) (Fundação Oswaldo Cruz, Rio de Janeiro, RJ, Brazil) according to the Solid Diffusion Assays described by López, Sanchez, Batlle, and Nern (2005).

Strains of microorganisms were cultured over two nights to obtain nearly  $10^8$  viable cells mL<sup>-1</sup>. The cultures were diluted in 0.1 g of peptone water/100 mL of solution to  $10^6$  cells mL<sup>-1</sup> and inoculated in duplicate Petri dishes containing Mueller Hinton culture medium (Acumedia, Michigan). Filter paper (1 cm in diameter), previously sterilised by treatment with a UV lamp for 2 min in each side, was dampened with the essential oil of lemon and placed in the centre of each Petri dish. The dishes were incubated at  $36 \pm 2$  °C for 48 h, and the diameters of the inhibition zones formed around the films were measured.

### 2.5. Experiment: application of films to biscuits

The flavouring films (primary packaging) were sterilised in a chamber with a UV lamp (Prodicil, 110 V, 254 nm) for 15 min and they were used to package biscuits (15 units). The biscuits wrapped in flavouring film were packed in polypropylene (PP) plastic bags (secondary packaging) that were sealed in sealing machine (Selovac® 200B, São Paulo, SP – Brazil) and stored at a controlled temperature of  $20 \pm 2$  °C.

### 2.6. Analysis of films

#### 2.6.1. Thickness

The thickness of the films was measured using a manual micrometre (Mituyoyo SulAmericana Ltda., Brazil, precision 0.002 mm), and the average of five measurements for each film was used to calculate the tensile properties. For water vapour transmission (WVT) calculations, the average of three thickness measurements of each sample was used (Kechichian, Ditchfield, Veiga-Santos, & Tadini, 2010).

#### 2.6.2. Mechanical properties

The mechanical properties of the films were determined by the tensile test using a Universal Testing Machine (Instron, model 3367, USA) with the following parameters: a load cell of 1 kN and a speed of 50 mm min<sup>-1</sup>. For each film, five samples with dimensions of 50 mm × 150 mm were analysed. The tensile strength (TS, MPa) and elongation at break (*E*, %) values were measured. TS was calculated by dividing the maximum load by the cross-sectional area of the film, and *E* was calculated by dividing the extension at the moment of rupture of the specimen by the initial length of the specimen and multiplying the result by 100 (ASTM, 2008).

Mechanical analysis were performed at 0, 10, 20 and 30 days of storage.

#### 2.6.3. Water vapour permeability

The water vapour permeability (WVP) of the films was determined according to ASTM Standard Method 96-00 (ASTM, 2000), method E96, with some modifications. The test film was sealed in a permeation cell containing anhydrous calcium chloride. The permeation cell was then placed in a controlled temperature–humidity chamber maintained at 75% relative humidity (RH) and 25 °C to maintain a 75% RH gradient across the film. Because the RH inside of the cell was always lower than the outside, water vapour

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