



Material properties

Physical and mechanical testing of essential oil-embedded cellulose ester films



Maria do Socorro Rocha Bastos ^{a,*}, Larissa da Silva Laurentino ^b, Kirley Marques Canuto ^a, Luana Guabiraba Mendes ^c, Camila Mota Martins ^a, Sarah Maria Frota Silva ^b, Roselayne Ferro Furtado ^a, Sanghoon Kim ^d, Atanu Biswas ^d, H.N. Cheng ^e

^a Embrapa Agroindústria Tropical, Dra. Sara Mesquita, 2270, 60511-110, Planalto Pici, Fortaleza, Ceará, Brazil

^b Universidade Federal do Ceará, Campus do Pici, s/n, Fortaleza, Ceará, Brazil

^c Universidade Estadual do Ceará, Av. Paranjana, 1700, Campus do Itaperi, Fortaleza, Ceará, Brazil

^d National Center for Agricultural Utilization Research, USDA Agricultural Research Service, 1815 N. University St., Peoria, IL, 61604, USA

^e Southern Regional Research Center, USDA Agricultural Research Service, 1100 Robert E. Lee Blvd., New Orleans, LA, 70124, USA

ARTICLE INFO

Article history:

Received 23 September 2015

Accepted 7 November 2015

Available online 19 November 2015

Keywords:

Food packaging

Essential oil

Cellulose acetate

Cellulose acetate propionate

Cellulose acetate butyrate

ABSTRACT

Polymer films made from cellulose esters are useful for embedding plant essential oils, either for food packaging or air freshener applications. Studies and testing were done on the physical and mechanical properties of cellulose ester-based films incorporating essential oils (EO) from lemongrass (*Cymbopogon citratus*), rosemary pepper (*Lippia sidoides*) and basil (*Ocimum gratissimum*) at concentrations of 10 and 20% (v/w). Results obtained showed that, in all films, the addition of the essential oil caused a decrease in the water vapor permeability due to the hydrophobic nature of the oil. The use of 20% of EO caused lower transparency of the films, although the change was not observed visually. Mechanical testing was done on cellulose acetate, cellulose acetate propionate and cellulose acetate butyrate. It was found that incorporation of lemongrass, basil and rosemary pepper EO significantly affected the Young's modulus, tensile strength and elongation at break of the cellulose ester films. The results suggested that the essential oils interacted with the polymers like plasticizers. The results were confirmed with thermal and microscopic studies.

© 2015 Elsevier Ltd. All rights reserved.

1. Introduction

There is current interest in using agri-based polymers and their derivatives in food packaging. Thus, many polysaccharides, such as starch, cellulose derivatives and food gums are employed to produce edible coatings, films and food wrappings. For these purposes, cellulose esters can be utilized to great advantage. Cellulose esters are derived from cellulose, are commercially available and have good physical properties [1]. The most popular product is cellulose acetate, which is biodegradable, amorphous, non-toxic, odorless, stable in mineral oils, permeable to water vapor, and soluble in acetone depending on the degree of substitution [2]. This compound can form transparent and flexible films which can be used in food packaging, photographic films and other applications.

Essential oils are aroma-containing compounds obtained from

plants. In addition to their fragrance, many essential oils are utilized in medicine and are known for their antimicrobial properties. There are at least two applications where cellulose esters can be used together with essential oils. First, an antimicrobial essential oil can be incorporated in a cellulose ester film for food packaging and reduction in microbial contamination [3,4]. Some essential oils like rosemary, clove, cinnamon, oregano, nutmeg and basil, have stood out because of their strong antimicrobial efficacy. Secondly, the embedding of a fragrant essential oil into a cellulose ester permits its use as a slow-release air freshener material.

For food packaging application, an antimicrobial compound can be incorporated into a packaging film, and common modes of practice include dispersion of the antimicrobial compound in the packaging, coating it on the packaging surface, and immobilizing it in the film by ion or covalent linkages [5,6]. However, for successful incorporation of the essential oils to the polymer, it is necessary to evaluate the effects of these compounds and their interactions with cellulose ester. Thus, polymer testing is necessary to study the

* Corresponding author.

E-mail address: socorro.bastos@embrapa.br (M.S.R. Bastos).

mechanical and thermal properties of the packaging [7]. It is also useful to evaluate the film's water vapor barrier properties because the foods can be susceptible to deterioration due to moisture gain [8]. It is known that the incorporation of additives in cellulose acetate films can significantly change the mechanical characteristics of the film [9–11], such as sorbic acid [12] and essential oils [13].

The objective of this study was to evaluate the influence of the incorporation of three essential oils (EO) in cellulose esters with respect to water vapor permeability (WVP), opacity, mechanical and thermal properties, and microscopic observations.

2. Materials and methods

2.1. Raw materials

An initial sample of cellulose acetate (CA) was obtained from Rhodia (São Paulo, Brazil). The degree of esterification was 2.5. Later, samples of CA (39.8 weight % acetyl, $M_n \sim 30,000$), cellulose acetate propionate (CAP, 2.5 weight % acetyl, 45 weight % propionyl, $M_n \sim 25,000$), and cellulose acetate butyrate (CAB, 12–15 weight % acetyl, 36–40 weight % butyryl, $M_n \sim 30,000$) were acquired from Sigma Aldrich (Milwaukee, Wisconsin, USA). Acetone was obtained from Vetec[®] (Rio de Janeiro, Brazil) and from Fisher Scientific (Pittsburgh, Pennsylvania, USA).

Essentials oils were obtained from leaves of *Cymbopogon citratus*, *Lippia sidoides* and *Ocimum gratissimum*, which were cultivated in the Embrapa Tropical Agroindustry experimental field (Fortaleza, Brazil). The oils were extracted by hydrodistillation for 3 h using a Clevenger-type glass apparatus. The chemical composition of the essential oils studied was analyzed by gas chromatography coupled with mass spectrometry (GC – MS) and a flame ionization detector (GC – FID). GC–MS analysis was performed on a Varian 450-GC/240-MS instrument equipped with a non-polar VF-5MS fused silica capillary column (30 m \times 0.25 mm i.d., 0.25 μ m film thickness), using helium as carrier gas and a flow rate of 1.5 mL/min, with a split ratio of 1:30. The injector temperature and detector temperature were set at 250 °C. The oven temperature was programmed to increase from 70 to 180 °C at 4 °C/min, and afterwards to 250 °C at 10 °C/min. Mass spectra was recorded in a range of mass-to-charge ratio (m/z) between 30 and 450. GC-FID analysis was carried out on a Shimadzu GC-2010 Plus chromatograph under the same chromatographic conditions employed for the GC–MS analysis, except for the carrier gas (hydrogen). The retention indices were determined by the injection of a mixture of C7–C30 homologous n-alkanes (Sigma–Aldrich, St. Louis, Missouri, USA). The identification of the volatile compounds was performed through comparison of the mass spectra recorded with those provided by the spectrometer database (NIST – 147,198 compounds), as well as matching the retention indices and mass spectra with literature data [14]. The relative content of oil constituents was determined by the peak area normalization method and expressed as percentages.

2.2. Preparation of cellulose ester films

In the production of the film, a cellulose ester polymer (CA, CAP or CAB) were used with acetone. The polymer was dissolved in acetone in the ratio of (1:2) to give the filmogenic solution. In the next step, the lemongrass (*C. citratus*), rosemary pepper (*L. sidoides*) and basil (*O. gratissimum*) EO were incorporated at EO/polymer concentrations of 10 or 20% (v/w), moments before the production of the films, following complete homogenization. These concentrations were chosen according to previous studies of antimicrobial activity. Furthermore, it was found that the viscosity of the polymer/acetone solution with >20% EO would limit the spreading of

the film. Control samples (films without the addition of EO) were also evaluated.

The films were produced by the *casting* method according to Santiago-Silva et al. [15], with some modifications. The CA filmogenic solution was spread on glass plates (previously sanitized with 70% alcohol) for the formation of the film. After the complete evaporation of the acetone solvent at room temperature (24 ± 1 °C), the films were removed from the glass plates and conditioned in a desiccator. For CAP and CAB, the films could not be released easily from a glass surface and were cast on a Teflon surface. The films were stored in desiccators containing a solution saturated with magnesium nitrate and kept at 25 °C temperature and $55 \pm 2\%$ relative humidity (RH) for at least 40 h.

2.3. Mechanical properties of the films

The tensile strength (MPa), Young's modulus (MPa) and elongation at break (%) of each film were determined in an Instron DL – 3000 universal testing machine (EMIC, Brazil, and Norwood, Massachusetts, USA) using the ASTM method D882-00 [16]. Previously, the films were cut into rectangular shapes, 100 mm wide by 160 mm long, and stored in desiccators at $50 \pm 5\%$ RH and 24 °C temperature for 24 h according to the ABNT 67401 method. The initial gauge length was set at 125 mm, and the jaws were separated at a speed of 12.5 mm/min, using a 1 kN load cell. Each analysis was performed on five samples and the average was taken.

The thickness of CA, CAB and CAP films was determined by using a MiniTest 3100 device from ElektroPhysik (Cologne, Germany). The film was placed on a measuring plate, and a magnetic field was imposed on it to determine the thickness. Calibration was done using a number of probes supplied by the manufacturer. Five measurements were made for each sample, and the mean value was used in tensile strength, opacity and water vapor permeability studies.

2.4. Water vapor permeability (WVP)

The WVP was determined gravimetrically at 25 °C, based on the ASTM E96-00 method [17]. The films were cut (discs with a diameter of 600 mm), placed at the top of a permeation cell containing distilled water and then the cells were placed in a desiccator containing silica at 24 °C and 47% RH. The cells were weighed eight times over a 24-h period in intervals of at least 1 h. The calculations were carried out according to the ASTM method [17].

2.5. Opacity

The apparent opacity of the films was determined according to the method proposed by Gontard et al. [18], using a Cary 50 spectrophotometer (Varian, Australia). The films were cut into rectangles measuring 400 mm long and 100 mm wide and adhered to the spectrophotometer's quartz cuvette. The absorption of visible light between 400 and 800 nm was measured for each sample. The spectrum was calibrated with the cuvette without a sample as 100% transmittance. The opacity of the film was defined as the area under the curve divided by the film thickness. In practice, the area was determined by integration the curve using the trapezoidal method. The opacity was expressed as absorbance units/nanometers ($A \cdot \text{nm}^{-1}$). Measurements were made in triplicate for each sample.

2.6. Thermal analysis

Samples for differential scanning calorimetry (DSC) were run on a Q2000 MDSC (TA Instruments, New Castle, Delaware, USA). A small piece of film (~2 mg) was cut and weighed into a Tzero

Download English Version:

<https://daneshyari.com/en/article/5206011>

Download Persian Version:

<https://daneshyari.com/article/5206011>

[Daneshyari.com](https://daneshyari.com)