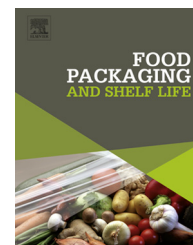


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Evaluation of polyamide composite casings with silver–zinc crystals for sausages packaging

Jairo H. Patiño^a, Luis E. Henríquez^a, Diego Restrepo^b, María P. Mendoza^c,
María I. Lantero^d, Mario A. García^{d,*}

^a Food Science and Technology Institute (INTAL), Carrera 50 G No. 12 Sur-91, Itagüí, Colombia

^b Universidad Nacional de Colombia Sede Medellín, Colombia

^c Alico S.A., Calle 10 Sur 50FF 63, Medellín, Colombia

^d Pharmacy and Food Institute, University of Havana, St. 222 No. 2317, ZC 13600 Havana, Cuba

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ABSTRACT

This study assessed the manufacture of polyamide (PA-6) composite casings based on silver–zinc crystals with potential application in sausages packaging. The antimicrobial activity of silver–zinc crystals was estimated by the minimum inhibitory concentration method for microbial strains related with the spoilage of meat products. PA-6 films were prepared as a multilayer film by bubble film sheet co-extrusion (130 °C–14,000 kPa), identified according to the layers distribution as control and active casings by adding 3% (w/w) of silver–zinc crystals to obtain a film with good barrier and mechanical properties. DSC, DMTA and SEM were also evaluated for determining the effects of microparticles in the polymeric matrix. Silver–zinc crystals inhibited the growth of various microbial strains, being *Salmonella typhimurium* ATCC 14028 which presented greatest resistance. Silver–zinc crystals were well impregnated in PA-6 films. Inclusion of silver–zinc crystals did not modify the mechanical properties and decreased the oxygen transmission rate of the films.

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1. Introduction

Antimicrobial active packaging constitutes a promising form of food packaging, especially for meat products. Antimicrobial substances incorporated into packaging materials can control microbial contamination by reducing the growth rate and expansion of the lag phase of the target microorganism, or by inactivation of microorganisms by contact. Inclusion of GRAS, non-GRAS and natural antimicrobials, such as metal nanoparticles (silver, gold and zinc), metal oxide nanomaterials and carbon nanotubes, into plastic matrix intend their slowly migration on the product surface and inhibition of the growth of microorganisms, increasing the shelf life and safety of the product (Silvestre, Duraccio, & Cimmino, 2011).

Silver nanoparticles are effective against a wide range of bacteria, yeasts and molds (Zapata et al., 2011), by altering their metabolism (Boschetto, Lerin, Cansian, Castellà, & Di Luccio, 2012). Silver ions cause DNA damage due to the inactivation of the membrane proteins and by generating chemical species reactive to oxygen, form complexes with sulphur, nitrogen and oxygen, damaging cell division mechanism (Awuah, Williams, Kenward, & Kenward, 2007; Damm, Munstedt, & Rosch, 2008).

In addition, the incorporation of micro- and nanoscale particles into polymeric matrix is related with the improvement of barrier (oxygen, carbon dioxide and ultraviolet rays) and mechanical properties, dimensional stability and heat resistance of packages (Silvestre et al., 2011).

Although some studies have reported the application of silver compounds in polymeric films such as polypropylene

* Corresponding author. Tel.: +53 7 2716389; fax: +53 7 2603894.

E-mail address: marioifal@gmail.com (M.A. García).

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(Pehlivan, Balköse, Ülkü, & Tihminioglu, 2005), polyurethane (Triebel et al., 2011) and polylactide (Martínez-Abad, Ocio, Lagarón, & Sánchez, 2013), and silver nanoparticles already found in some commercial uses, the information about impregnation of polymeric materials with antimicrobial agents is still limited. In this context, this study assessed the mechanical, barrier and thermal properties of polyamide (PA-6) composite casings based on silver–zinc crystals with potential application in sausages packaging.

2. Materials and methods

2.1. Antimicrobial activity of silver–zinc crystals

The antimicrobial activity of a mixture of silver–zinc crystals fine powder with a mean particle size of 1.8 μm (IRGAGUARD[®] B7000, Ciba Specialty Chemicals Corporation, Tarrytown, NY, USA) was performed by determining the minimal inhibitory concentration (MIC) using the following microbial strains: *Pseudomonas aeruginosa* ATCC 27853, *Penicillium chrysogenum* ATCC 8507, *Lactobacillus acidophilus* ATCC 4356, *Listeria monocytogenes* ATCC 19112 and *Salmonella typhimurium* ATCC 14028, selected for their relationship with spoilage of meat products. The MIC was determined by the indirect method based on the estimation of microbial growth by turbidity in broth culture (Boschetto et al., 2012). IRGAGUARD[®] B7000 (silver, as elemental, 0.37%; zinc, as elemental, 17.90% and other ingredients, 81.73%) is an antimicrobial powder for the manufacture of polymeric, plastic, and latex products only. It suppresses the growth of bacteria, algae, fungus, mold and mildew, which cause unpleasant odors, discoloration, staining, deterioration or corrosion of those manufactured products. Finished products containing IRGAGUARD[®] B7000 may not make public health claims relating to antimicrobial activity. IRGAGUARD[®] B7000 may be incorporated into materials that may be used in the finished product at 0.1–3.0% (w/w) of the powder (Ciba Specialty Chemicals Corporation, Tarrytown, NY, USA).

2.2. Polymer films preparation

Four types of biaxially oriented polyamide 6 (PA-6) casings with 45 μm of thickness were manufactured by ALICO S.A. (Medellín, Colombia) as a multilayer film by bubble film sheet co-extrusion (130 °C–14,000 kPa), identified according to the layers distribution as control (PA/adhesive/PE-pigment/adhesive/PA, with thickness distribution of 50/6.5/23/6.5/14% from outside to inside layers) and active casings by adding 3% (w/w) of a mixture of silver–zinc crystals fine powder (IRGAGUARD[®] B7000, Ciba Specialty Chemicals Corporation, Tarrytown, NY, USA), in the internal layer of the thermoplastic material. The control casings corresponded with commercial material (Aliflex, ALICO S.A.) for packaging chicken and beef sausages. The plasticizer (Ultramid[®], BASF Corporation) and active agent were mixed at the same time with the polymeric matrix for guaranteeing a homogeneous dispersion. The exact products used cannot be specified for proprietary reasons therefore these materials will be referred to using chicken and beef control and active casings, be used consistently throughout.

2.3. Film evaluation

2.3.1. Mechanical properties

Longitudinal and transversal tensile tests were performed on a Instron-5582 machine at 23 °C according to ASTM D638, ASTM D882-97 and ASTM F904-98, to samples with dimensions of 25 mm $\times$ 100 mm and putted in clamps separated 50 mm at a crosshead speed of 500 mm/min. Maximal tensile strength, yield strength, yield elongation, tensile strength, elongation at break and elastic modulus were evaluated.

2.3.2. Barrier properties

2.3.2.1. Oxygen transmission rates (OTR). OTR (cm^3 (STP) $\text{m}^{-2} \text{d}^{-1}$) through polyamide films, with and without microparticles, were obtained using an OX-Tran 2/20 (Mocon Inc., Minneapolis, MN, USA) at 23 °C; 0% RH and 1 atm (ASTM F1927). The detection limit of the instrument was 0.05 $\text{cm}^3 \text{m}^{-2} \text{d}^{-1}$. The system was operated according to ASTM D 3985.

2.3.2.2. Water vapor transmission rate (WVTR). WVTR ($\text{g m}^{-2} \text{d}^{-1}$) through films was determined gravimetrically using the pouch method according to ASTM E96-95. Pouches were placed in an environment of controlled humidity and temperature (90% RH and 35 °C). All tests were conducted in triplicate. The slope of the curve representing the weight increase of the pouches versus time at the steady state of transfer was obtained by linear regression.

2.4. Water absorption (WA)

WA (mg cm^{-2}) was calculated gravimetrically between the mass of the samples films (25 cm^2) before and after the immersion in 100 mL of distilled water at 23 $\pm$ 1 °C during 48 h, previously films drying at 50 $\pm$ 3 °C during 24 h (ASTM D 570).

2.5. Thermal properties

2.5.1. Differential scanning calorimetry (DSC)

Samples of each microcomposite films were placed in aluminum pans, hermetically sealed, in the equipment sample chamber (DSC-2920, TA Instruments), under nitrogen atmosphere, with a flow of 50 mL min^{-1} and warming rate of 5 °C min^{-1} until 300 °C, sustained by 5 min. The crystals transition temperature (T_g) was obtained at the inflection point between the base lines through the change in the heat capacity of the sample.

2.5.2. Dynamic mechanical thermal analysis (DMTA)

The samples used for DMTA were cut from narrow sections of tensile bars with dimensions of 17.0 mm $\times$ 6.0 mm. The samples were dried at 40 °C for about 2 days and then stored in desiccators until they were used. The thermomechanical experiments were performed on a dynamic mechanical analyser (DMA-Q800, TA Instruments) at a heating rate of 3 °C/min and at a frequency of 1 Hz.

2.6. Scanning electronic microscopy (SEM)

The microcomposites were analyzed using a scanning electron microscope (Jeol JSM 5910 LV, Tokyo, Japan) at

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