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## Effect of antifungal hydroxypropyl methylcellulose (HPMC)–lipid edible composite coatings on postharvest decay development and quality attributes of cold-stored 'Valencia' oranges

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### ABSTRACT

Edible composite coatings based on hydroxypropyl methylcellulose (HPMC), hydrophobic components (beeswax and shellac), and food preservatives with antifungal properties were evaluated on 'Valencia' oranges during long-term cold storage. Selected food preservatives included potassium sorbate (PS), sodium benzoate (SB), sodium propionate (SP), and their mixtures. Intact oranges or oranges artificially inoculated with *Penicillium digitatum* or *Penicillium italicum* were coated and stored up to 60 d at 5 °C followed by 7 d of shelf-life at 20 °C. Some antifungal HPMC-lipid coatings significantly reduced incidence and severity of both green (GM) and blue (BM) molds on inoculated and cold-stored oranges, and PS+SP-based coating was the most effective. In general, the coatings controlled GM better than BM. After 30 and 60 d at 5 °C plus 7 d at 20 °C, fruit weight loss, rind firmness, internal gas concentrations, ethanol and acetaldehyde contents of the juice, sensory flavor, off-flavor, and fruit appearance were not adversely affected by application of the antifungal coatings, which showed promise as potential substitutes for citrus commercial waxes. However, further studies should follow to improve some coating physical characteristics in order to provide better water loss control and higher gloss on coated oranges.

#### 1. Introduction

Fruit and vegetables have been traditionally kept under cold storage to maintain their postharvest quality. In many cases, however, benefits from refrigeration are not important enough to preserve produce quality. In the citrus industry, in addition to cold storage, fruit coating is a normal practice to replace the natural waxes that are generally removed during washing. Wax coatings are made of natural or synthetic waxes and fatty acids (beeswax, carnauba, polyethylene, oleic acid), oils, shellac, emulsifier, plasticizers, antifoam agents, and surfactants (Baldwin, 1994; Hagenmaier and Baker, 1994; Hagenmaier, 1998). Coatings form a semi-permeable barrier to water vapor and gas exchange, leading to weight loss reduction, respiration rate modification, and senescence delay of coated produce (Hagenmaier and Baker, 1994; Nisperos-Carriedo, 1994; Olivas et al., 2008). Synthetic chemical fungicides are often incorporated into conventional citrus waxes to control postharvest molds. However, effectiveness of decay control is often lower when the fungicides are dissolved into the coatings than when fungicides are applied alone. It has been reported that a fungicide concentration increase of two- to threefold may be needed to obtain the same level of decay control than with the use of pre-coating fungicidal aqueous solutions (Grant and Burns, 1994). Prolonged and extensive use of postharvest chemical fungicides, either alone or into conventional coatings, may deposit harmful residues on fruit and contaminate the environment.

Edible films and coatings are alternative, non-polluting methods that have been developed to extend produce shelf-life (Banker, 1966; Greener and Fennema, 1994; Park, 1999; Rhim and Shellhammer, 2005). Ingredients used in edible films and coatings are proteins, polysaccharides, and lipids, which include natural waxes and resins. In addition, emulsifiers and plasticizers are added to improve coating performance (Nisperos-Carriedo, 1994; Baldwin et al., 1997; Pérez-Gago and Krochta, 2001; Yoshida and Antunes, 2004). Edible films and coatings can also act as carriers of food additives, including colorants, flavoring agents, antioxidants, or antimicrobial compounds (Cuppet, 1994; Franssen and Krochta, 2000; Ozdemir and Floros, 2003; Cha and Chinnan, 2004; Valencia-Chamorro et al., 2008). There is much in the literature on the effect of edible films and coatings containing a broad variety of components on postharvest quality of coated fruit and vegetables (Hagenmaier and Baker, 1995; Mannheim and Soffer, 1996;

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Pérez-Gago et al., 2002; Porat et al., 2005; Chien et al., 2007; Navarro-Tarazaga et al., 2007).

In the last decade, several studies have focused on the development of coatings based on proteins and polysaccharides with natural food preservatives to control microbial growth on fruit. For example, starch-based coatings with potassium sorbate (PS) reduced microbial growth on strawberries (García et al., 1998), and chitosan-based coatings containing PS showed an effective antifungal action against *Rhizopus* sp. and *Cladosporium* sp. on inoculated fresh strawberries (Park et al., 2005). Coatings based on carboxymethyl cellulose-shellac containing parabens reduced coliform bacteria on coated citrus fruit (McGuire and Hagenmaier, 2001). In recent work, hydroxypropyl methylcellulose (HPMC)-lipid edible composite films containing sodium salts of parabens, PS, sodium benzoate (SB), and their mixtures exhibited clear in vitro inhibitory activity against the main postharvest diseases of citrus fruit, green (GM) and blue (BM) molds, caused by Penicillium digitatum and Penicillium italicum, respectively (Valencia-Chamorro et al., 2008). In subsequent work, in vivo selected coatings reduced the incidence and severity of GM and BM on 'Clemenules' clementine mandarins, 'Ortanique' hybrid mandarins, and 'Valencia' oranges stored at 20 °C (Valencia-Chamorro et al., 2009). However, no information is available on the performance of these edible coatings on cold-stored citrus fruit.

The objective of this work was to determine the effect of selected HPMC–lipid edible composite coatings containing food additives with antifungal properties on the development of penicillium molds and the physico-chemical and sensory quality of 'Valencia' oranges during long-term cold storage.

#### 2. Materials and methods

#### 2.1. Materials

HPMC (Methocel E15) was purchased from Dow Chemical Co. (Midland, MI, USA). Shellac and beeswax (BW) (grade 1) were supplied by Fomesa Fruitech, S.L. (Beniparrell, València, Spain). Stearic acid and glycerol were from Panreac Química, S.A. (Barcelona, Spain). Silicone antifoam (FG-1510) and ammonia (25%) were from Dow Corning<sup>®</sup> (Belgium) and Scharlau (Sentmenat, Barcelona, Spain), respectively. Food preservatives were purchased from Sigma (Sigma–Aldrich Chemie, Steinheim, Germany) and included the salts of organic acids, PS, SB, and sodium propionate (SP). These chemicals are all classified as food additives or generally recognized as safe (GRAS) compounds by EU and US regulations.

#### 2.2. Emulsions preparation

HPMC-lipid edible composite emulsions were prepared combining the hydrophilic phase (HPMC) and the hydrophobic phase (BW and shellac) suspended in water. Glycerol and stearic acid were used as plasticizer and emulsifier, respectively. Ratios of HPMC-glycerol (2:1) (dry basis, db) and lipid components (BW/shellac)-stearic acid (5:1) (db) were kept constant throughout the study. BW and shellac content was 50% (db) at a ratio BW: shellac of 1:1. Emulsions were prepared as described previously by Valencia-Chamorro et al. (2008). Briefly, an aqueous solution of HPMC (5%, w/w) was prepared. Food preservative (w/w), BW, glycerol, stearic acid, water, and two drops of antifoam were added to the HPMC solution and heated at 90 °C to melt the lipids. Shellac was previously dispersed in water at 40  $^{\circ}$ C and ammonia (15%, w/w, shellac/ammonia) was added to dissolve the resin. Shellac solution was heated separately at 90 °C and added to the HPMC dispersion. Samples were homogenized with a high-shear probe mixer (Ultra-Turrax model T25, IKA-Werke, Steufen, Germany) for 4 min at 22,000 rpm. Emulsions were cooled to less than 25 °C and further agitated for 25 min. Emulsions were kept 2-3 d at 5 °C before use. Table 1 shows the total solids content of the formulations and the concentration of each food preservative (%, wet basis, wb). The formulations were selected from HPMC-lipid edible composite coatings that were effective inhibiting the development of GM and BM on citrus fruit stored at 20 °C (Valencia-Chamorro et al., 2009). These formulations were stable and no phase separation was observed.

#### 2.3. Effect of coatings on disease development

#### 2.3.1. Fungal inoculum

*P. digitatum* isolate NAV-7 and *P. italicum* isolate MAV-1, obtained from decayed citrus fruit from packinghouses in the València area (Spain), were isolated, identified, and maintained in the IVIA culture collection of postharvest pathogens. These strains were selected for their aggressiveness on the most commercially important mandarin and orange cultivars. Prior to each experiment, the isolates were grown on potato dextrose agar (Sigma) in petri dishes at  $25 \pm 1$  °C for 7–10 d. A high-density conidial suspension was prepared in Tween 80 (0.05%, w/v; Panreac Química, S.A.) in sterile water, passed through two layers of cheesecloth, measured with a haemacytometer and diluted with sterile water to achieve the desired inoculum density.

#### 2.3.2. Fruit inoculation and coating application

Oranges (*Citrus sinensis* [L.] Osbeck) cv. 'Valencia' from commercial orchards in València were selected by hand and used in the experiments before any postharvest treatment was applied. The fruit were stored up to 1 week at 5 °C and 90% relative humidity (RH) before use. Before each experiment, the fruit were randomized, washed with fresh water, and allowed to air-dry at room temperature. Fruit were artificially inoculated (inoculum density of  $10^8$  conidia L<sup>-1</sup>) by immersing a stainless steel rod with a probe tip 1 mm wide and 2 mm in length into the spore suspension and wounding each fruit once on the equator. Each fruit was inocu-

#### Table 1

Characteristics of hydroxypropyl methylcellulose (HPMC)-lipid edible composite coatings containing antifungal food preservatives and mixtures.

Food preservatives <sup>a</sup>	Food preservative concentration (%, wb) <sup>b</sup>	Solid concentration (%, wb)	Viscosity (cp) <sup>c</sup>	pH
Organic acid salts				
PS	2.0	6	19.20	7.30
SB	2.5	8	18.90	7.30
Mixtures				
PS + SP	1.5 + 0.5	6	12.20	7.20
SB + PS	2.0+0.5	8	18.70	7.20
SB + SP	2.0+0.5	8	16.10	7.10

<sup>a</sup> PS: potassium sorbate; SB: sodium benzoate; SP: sodium propionate.

<sup>b</sup> wb: wet basis.

<sup>c</sup> cp: centipoises.

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