



How composition and process parameters affect volatile active compounds in biopolymer films

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ABSTRACT

Active edible films based on chitosan and containing an active aroma compound have been investigated. The influence of the composition of the film forming solution (solvent, plasticizers, nanoparticles, emulsifiers and aroma traps) and process parameters (temperature and viscosity) on drying kinetics, water content, colour, aroma compound retention and partition coefficient was studied. Solvent evaporation rate was increased by addition of ethanol. Water evaporation was delayed by glycerol. The aroma retention during film drying was directly related to the water content. The drying temperature had opposite influences according to the composition of the solvent and additives. Increasing temperature from 20 to 100 °C could increase 3 times the retention of aroma compounds. The viscosity of film forming solutions was strongly increased by biopolymer content and glycerol but reduced by carvacrol and ethanol. To improve carvacrol retention, the most efficient additive was gum arabic, followed by glycerol whereas the effect of nanoclays or emulsifiers remained weak.

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1. Introduction

Dynamic changes in market trends and current consumer demands significantly increased the production of biopolymer active packaging materials. Active packaging is a type of packaging where product, packaging and surrounding environment interact in order to extend shelf life, improve organoleptic properties and/or food safety (Suppalku, Miltz, Sonneveld, & Bigger, 2003). The addition of an active substance in the material can improve packaging functionality (Appendini & Hotchkiss, 2002). There are several ways of its incorporation in the packaging system. One of the ideal ways of biopolymer packaging applications could be the incorporation of an active/antimicrobial compound directly within polymer matrix.

The effectiveness of antimicrobial films is determined by the release rate of the antimicrobial compounds, either directly to food or via air. Process conditions influence the final film structure and performances. Drying is generally accompanied by the loss of volatiles. Their retention during oven drying is affected by the nature of the matrix, the volatile concentration and the experimental conditions (e.g. drying rate, sample thickness,

humidity) (Flink & Labuza, 1972). Carvacrol is a monoterpene phenolic constituent of essential oils with antimicrobial properties. Its retention and release depend on the polymer, the film preparation method, the interactions between polymeric and antimicrobial materials and environmental conditions (Cagri, Ustunol, & Ryser, 2004; Cha, Cooksey, Chinnan, & Park, 2003; Rico-Pena & Torres, 1991). Up to date, there are few studies dealing with the detection of the amount of the aroma compound in the film after processing and drying. Numerous authors have reported the initial amount of antimicrobial substances and not the real concentration in the dry films. Quantifying compounds mobility is a crucial element in understanding the mechanisms of release in the headspace or in a contact with food. Therefore, the retention of the active/antimicrobial compound is one of the most important features of active biopolymer film processing. Generally, when loss during drying is greater, the release in the vapour phase is faster. Sánchez-González, Cháfer, González-Martínez, Chiralt, and Desobry (2011) showed that limonene loss during drying ranged from 39 to 99% when added from 0.5 to 3% (w/w) in chitosan films.

Macromolecules such as chitosan show abilities to retain different antimicrobial substances. Chitosan (CS) offers real potential for applications in the food industry due to its particular physico-chemical properties, short time biodegradability, biocompatibility, antimicrobial and antifungal activities, non-toxicity, toughness, durability, moderate values of water and oxygen permeability (Agulló, Rodríguez, Ramos, & Albertengo, 2003; Bégin & Van

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Calsteren, 1999; Coma et al., 2002; Jeon, Kamil, & Shahidi, 2002; Muzzarelli et al., 1990; Ouattara, Simard, Piette, Bégin, & Holley, 2000; Shahidi, Arackchi, & Jeon, 1999; Wang, Du, Luo, Lin, & Kennedy, 2007). Drying methods, conditions and matrix formulation (e.g. polymer concentration, suitable solvent system, addition of plasticizers, dispersants, emulsifiers) have to be set up to engineer chitosan films likely to monitor the retention and thus the release of the active compound (Chillo et al., 2008; Fernández Cervera et al., 2004; Park, Marsh, & Rhim, 2002; Suyatma, Tightzert, & Copinet, 2005). The choice of the solvent in the preparation of a film forming solution is of a particular importance because it affects the solubility and dispersion of hydrophobic volatile molecules as well as the physico-chemical properties of the final products.

Polysaccharides are believed to induce the retention of aroma compounds caused either by molecular interaction with specific molecules or by an increase in viscosity of the matrix (Guichard, 2002). Changes in polymer concentration or mixing chitosan with other polymers may meet the above mentioned requirements when polymers are in solution. Arabic gum (AG) is a biocompatible and biodegradable polymer with more interaction sites and negative charges for interaction with polycationic polymers such as chitosan (Wareing, 1999). Because of its unique properties (e.g. emulsification, acid stability, low viscosity at high concentrations) it is used in many food applications such as beverages, emulsions, flavour encapsulation, as an agent that protects from oxidation and volatilization. The recovery and the oxidation stability of orange oil encapsulated in AG has been reported (Qi & Xu, 1999). Up to date, not a lot of work has been done to understand the aroma compound retention capacity of the chitosan blends with AG, particularly in dry matrices.

Emulsifiers are surface active molecules that interact simultaneously with polar and hydrophobic sites on the casting matrix, thus being able to improve the functional film properties (di Gioia & Guilbert, 1999). Both ionic and non-ionic surfactants, with or without plasticizer addition have been studied in different film productions (Andreuccetti et al., 2011; Bravin, Peressini, & Sensidoni, 2004; Morillon, Debeaufort, Blond, Capelle, & Voilley, 2002; Rodríguez, Osés, Ziani, & Maté, 2006; Villalobos, Hernández-Muñoz, & Chiralt, 2006). In an emulsion, droplets of aroma compound partition themselves among polymer matrix, according to their polarity and surface activity.

Montmorillonite (MMT) is a compound that can potentially be used to control the release of antimicrobial agents from film materials as displayed by Tunc and Duman (2010). Moreover, functional film properties were improved by the reinforcement of the polymer matrix with layered silicates (Park, Liang, Mohanty, Misra, & Drzal, 2004; Sothornwit, Rhim, & Hong, 2009; Tunc et al., 2007).

One way of investigating the retention (or release) of volatile compounds by polysaccharides is to measure the gas/matrix partition coefficient (K). This parameter describes the distribution of volatile compounds between the gas phase and the support matrix, after equilibrium is reached (Boland, Buhr, Giannouli, & van Ruth, 2004). Thus, a better understanding of this property is needed to adjust the volatile concentration in the packaging headspace. Several parameters should be taken into account. These include the type and the concentration of components along with the nature and the physicochemical properties of the aromatic compound (e.g. volatility and hydrophobicity) (Seuvre, Philippe, Rochard, & Voilley, 2006). According to these authors, polysaccharides influence the volatility of the molecules of the aroma compounds and their partitioning between different phases mainly by two mechanisms. The first one is diffusion decrease, predicted by the Stokes–Einstein equation where diffusion is inversely proportional to viscosity (Baines & Morris, 1987; Wilke & Chang, 1955). The second mechanism involves specific molecular interactions of the aroma compounds with the macromolecule. It is often due to

adsorption, entrapment in microregions, complexation, encapsulation and hydrogen bonding (Godshall, 1997; Kinsella, 1989).

In this study, the aim was to investigate influence of drying parameters (temperature and relative humidity) and film formulation (polymer, casting solvent, and additives) on carvacrol retention and partition coefficient. The drying kinetics and some physical film properties were studied in order to develop chitosan films as vehicles for controlling aroma compound retention and release for further packaging applications.

2. Materials and methods

2.1. Materials and reagents

Commercial grade chitosan (CS) (France Chitine, Marseille, France, powder 652, having a molecular mass of 165 kDa, low viscosity, food grade, degree of deacetylation of 85%) and arabic gum (AG) (Spraygum, CNI, France) were used to constitute the film matrix. Anhydrous glycerol (Fluka, 98% purity, Fluka Chemical, Germany), Tween 20 (Sigma–Aldrich), lecithin (Sigma–Aldrich), nanoclays (montmorillonite, synthesis and characterization is detailed elsewhere, Reinholdt, Miehé-Brendlé, Delmotte, Le Dred, & Tuilier, 2005), polyethylene glycol (PEG750, Aldrich Chemicals) and glycerol monostearate (Sigma–Aldrich) were used in order to improve aroma compound retention and mechanical properties. Acetic acid (glacial 100%, Merck, Darmstadt, Germany) and pure ethanol (absolute, Sigma–Aldrich) were used as solvents in the preparation of the film forming solutions (FFS). All the chemicals were used without further purification and freshly prepared solutions were used in all experiments. Carvacrol with a purity of about 97% (Fluka) was used as a model antimicrobial volatile compound. *n*-Hexane (Chromasolv, purity of >97%, Sigma–Aldrich) was used as an extraction solvent.

2.2. Film formation

2.2.1. Chitosan films (CS)

A chitosan solution was prepared by dissolving the chitosan powder in the 1% (v/v) aqueous acetic acid, to obtain 1, 2 or 3% (w/v) solutions. To achieve the complete dispersion of chitosan, the solution was stirred for 2 h at room temperature. To prepare aqueous hydroalcoholic acid media, ethanol was mixed in ratio ethanol:aqueous acetic acid 30:70. Different amounts of glycerol (up to 50%, w/polymer dry matter (p.d.m.)) were added to the chitosan solution under stirring. The aroma compound (from 0.01 to 3%, w/v) was dispersed in the film forming solution (FFS) and the mixture was homogenized at 24,000 rpm for 10 min with an Ultra Turrax (T25 IKA). FFS was then poured into a glass Petri dish. In order to obtain films, solvents were removed by drying in a ventilated climatic chamber (KBF 240 Binder, ODIL, France) at fixed temperatures ranging from 20 °C to 100 °C and RH ranging from 10 to 80%. After drying, the films were peeled off the surface and stored in a ventilated climatic chamber (KBF 240 Binder, ODIL, France) before measurements. The aroma compound-free films were prepared using the same procedure, instead of the step where the aroma compound was added. PEG (up to 30%, w/p.d.m.), lecithine (from 0.5 to 30%, w/p.d.m.), Tween 20 (from 2.5 to 30%, w/p.d.m.) and glycerol monostearate (up to 5%, w/p.d.m.) were added in some film formulations after solubilisation of chitosan powder.

2.2.2. Chitosan/arabic gum blends (CS/AG)

To achieve CS/AG composite films, AG was added in the FFS of CS (50%, w/w) and mixed under magnetical stirring for 2 h. The next steps were the same as for the CS film.

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