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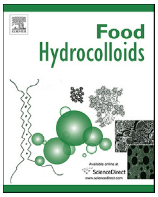

Mechanical and water barrier properties of isolated soy protein composite edible films as affected by carvacrol and cinnamaldehyde micro and nanoemulsions

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ABSTRACT

Edible films may be used in food packaging, for which they must deliver good barrier and mechanical properties. Films based on proteins have good gas barrier and mechanical properties, but poor water barrier properties. Films made from lipids have good water barrier properties, but poor mechanical properties. Protein and lipids were then combined to form composite films, and the particle size of the lipid phase was reduced to evaluate its effect on the mechanical and barrier properties. Micro and nanodroplets of Acetem and Tween 60 were added into an isolated soy protein (ISP) solution. Oil-in-water droplets were formed by direct emulsification at 1300 rpm for 30 min (microdroplets) or 5 h (nanodroplets), dispersed into the ISP solution, and cast into films. Emulsified films showed reduced strength and increased elongation, indicating a plasticizing effect of emulsions. The water barrier properties were either unchanged or slightly improved by the addition of hydrophobic compounds. Reducing droplet size improved the barrier properties as surface area of the lipid increased. Carvacrol and cinnamaldehyde were also added, and they either improved or unaffected the mechanical and barrier properties of ISP films. Addition of emulsions and reduction of droplet size generally decreased total pore volume in the films as well as water vapor permeation, indicating that the microvoid model suitably explains the water barrier properties of ISP films. This work further clarifies possible differences in barrier properties by examining water diffusivity and adsorption/desorption kinetics.

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1. Introduction

One of the intended uses of edible films is in food packaging. Films having poor mechanical properties may not withstand handling, whereas poor barrier properties may lead to food physical, chemical, and microbiological spoilage. Protein- and polysaccharide-based films generally have good mechanical properties and are good barriers to gases, but not to water vapor. Contrastingly, lipid-based films have good water barrier properties but form brittle films (Kester & Fennema, 1986). The combination of proteins and lipids is thus a way of developing composite edible films matching the requirements for use as food packaging (Bilbao-Sáinz, Avena-Bustillos, Wood, Williams, & McHugh, 2010).

Casting a lipid over a protein film is a way of preparing a bi-layer composite film, but this is a multi-step process that could lead to layer delamination. Dispersing a lipid into a film-forming solution and casting it produces single-step composite films (Kamper & Fennema, 1984). Although steps are reduced, the film-forming solution usually requires an emulsifier to avoid phase separation during drying (Shellhammer & Krochta, 1997). An emulsion can be kinetically stabilized by reducing the particle size of the dispersed phase (Yi, Li, Zhong, & Yokoyama, 2014) and by adding amphiphilic

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Abbreviations used: AC, Acetem; BET, Brunauer, Emmet and Teller; CARV, carvacrol; CINN, cinnamaldehyde; DVS, dynamic vapor sorption; EB, elongation at break; EM, elastic modulus; HLB, hydrophilic-lipophilic balance; ISP, isolated soy protein; ISPS, isolated soy protein solution; PdI, polydispersity index; RH, relative humidity; TPV, total pore volume; TS, tensile strength; TW, Tween 60; WVP, water vapor permeability.

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molecules having polar and nonpolar regions, so called surfactants (McClements, 2004; Rao & McClements, 2012). This way, it is preferable to elaborate composite films through the dispersion of lipids into film-forming solutions followed by addition of an emulsifier and homogenization, a process of reducing the droplet size of oil-in-water emulsions (Otoni et al., 2014a).

Edible films that contain micro or nanoemulsions into the film matrix may be used as active packaging for delivering lipophilic active compounds to the packaged food. This is of interest because active films may carry a wide range of food additives, including antimicrobials (Pranoto, Salokhe, & Rakshit, 2005), since the lipid phase may act as a solvent (McClements, 2004). Antimicrobial films have received special attention due to their capacity to avoid illness outbreaks caused by increased consumption of fresh-cut produce (Rojas-Graü et al., 2007). Carvacrol and cinnamaldehyde (Fig. 1) are the major components of oregano and cinnamon essential oils, respectively, both of them delivering antimicrobial effect against foodborne pathogens (Du et al., 2009a, 2009b; Friedman, Buick, & Elliott, 2004a; Friedman, Henika, Levin, & Mandrell, 2004b; Otoni et al., 2014a; Otoni, Pontes, Medeiros, & Soares, 2014b; Passarinho et al., 2014; Ravishankar, Zhu, Olsen, McHugh, & Friedman, 2009; Rojas-Graü et al., 2007).

Emulsions with droplets ranging in diameter up to 500 nm are named nanoemulsions (Solans, Izquierdo, Nolla, Azemar, & Garcia-Celma, 2005), and they differ from traditional emulsions mainly due to their improved stability (Huang, Yu, & Ru, 2010). Nanoparticles are used in the food industry chiefly as food ingredients, additives, active agents in food packaging, and devices or materials for nanofiltration, water treatment, and nanosensors for food safety and traceability (Chaudhry et al., 2008). Nanoparticles can be created by either a 'bottom up' approach, where nanoparticles are created on a molecular level, or by a 'top down' approach, where bulk material is broken down into nanoparticles (Chaudhry et al., 2008). Micro or nanoemulsions mostly use the 'top down' approach to encapsulate ingredients, flavors, or healthful, bioactive, and antimicrobial compounds by emulsification. A delivery system is used to maintain the bioactivity of healthful ingredients (Chen, Remondetto, & Subirade, 2007; Luykx, Peters, VanRuth, & Bouwmeester, 2008; Weiss et al., 2008). The reduction on droplet size enhances the accessibility of the active agents due to increased surface area (Huang et al., 2010; Otoni et al., 2014a, 2014b; Tadros, Izquierdo, Esquena, & Solans, 2004; Yang, Marshall-Breton, Leser, Sher, & McClements, 2012). Otoni et al. (2014a) reported improved antimicrobial activity of cinnamaldehyde-containing edible films as droplet size was reduced. The authors attributed this finding to the facilitated migration of smaller droplets from films to microbial cells. Similar outcome was observed by Otoni et al. (2014b), who reported improved antifungal effect of clove bud and oregano essential oils when incorporated as nanodroplets in methylcellulose-based films. In this work, the shelf life of sliced bread stored within nanoemulsion-containing bags was longer than those stored alongside films incorporated with emulsions having larger droplets.

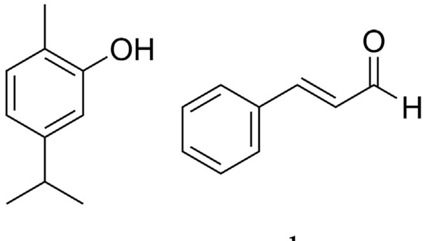

Fig. 1. Carvacrol and cinnamaldehyde molecules. Chemical structures of carvacrol (a) and cinnamaldehyde (b) molecules.

The addition of lipids, might however affects the films' physical properties (e.g., water barrier and mechanical properties), depending upon the physical state, type, amount, and hydrophobicity of the oil fraction (Kamper & Fennema, 1984; McHugh & Krochta, 1994a; Quezada-Gallo, Debeaufort, & Voilley, 2000). Water permeation through films involve the adsorption and dissolution of water, the mass transfer of water from an area of high moisture to another of low moisture, and the evaporation of water to the environment (Miller & Krochta, 1997). Shape and hysteresis of adsorption/desorption water vapor isotherms provide information on specific surface area, porosity and diffusivity of films and powders. Surface area is the means through which a solid material interacts with its surroundings. Nanoscale preparation of solid materials can increase its surface area (Lowell, Shields, Thomas, & Thommes, 2004). Water vapor permeability (WVP) is commonly used to measure the water barrier properties of films, which can be described by the microvoid or micropathway models. The microvoid model proposes that film drying creates shrinkage and voids or pores between water soluble polymers and microparticles, allowing mass transfer of moisture. The total pore volume (TPV) indicates the porosity of a film by a relationship between its bulk and true densities (Moura et al., 2011). The micropathway model proposes that water permeation occurs through the polymer matrix itself if it is compatible with water.

Emulsions made with or without carvacrol and cinnamaldehyde and having either micro or nanodroplets were mixed into an isolated soy protein (ISP) solution. This study aimed at producing composite edible films of micro and nanosize droplets by direct emulsification and at evaluating the effect of particle size on water barrier and mechanical properties. Additionally, this work clarifies possible differences in barrier and mechanical properties by examining diffusivity and porosity.

2. Material and methods

2.1. Materials

Isolated soy protein (Supro 500E) was kindly provided by The Solae Co. (St. Louis, MO). The films were plasticized by vegetable glycerin from Starwest Botanicals (Rancho Cordova, CA). The lipid phase of the emulsions was GRINDSTED® Acetem 90-50 K (an acetic acid ester of monoglycerides made from partially hydrogenated soybean oil), kindly provided by Danisco USA Inc. (New Century, KS), while the aqueous phase was purified double distilled water (Barnstead/Thermolyne, Dubuque, IA). Polyoxyethylene sorbitan monostearate (Tween 60) was purchased from Fisher Scientific Co. (Pittsburg, PA) and used as the surfactant. 98+% pure carvacrol and 93+% pure cinnamaldehyde were purchased from SAFC Supply Solution (St. Louis, MO).

2.2. Emulsification

Direct oil-in-water emulsions were prepared by adding Acetem, Tween 60 (with a hydrophilic-lipophilic balance of 14.9), and ultrapure water, and mixing on a Servodyne (model 50000-30) mixer (Cole-Parmer Instruments, Chicago, IL) at 1300 rpm using a 1.5 in diameter propeller. The mixing was done at 25 °C for 30 min to obtain microemulsions and 5 h to obtain nanoemulsions. Further emulsions were prepared likewise but with carvacrol or cinnamaldehyde in addition to other ingredients.

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