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Casein/wax blend extrusion for production of edible films as carriers of potassium sorbate—A comparative study of waxes and potassium sorbate effect

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ABSTRACT

Organic acids as potassium sorbate (KS) have a long history as being generally recognized as safe food preservatives. In this work, KS was incorporated in rennet casein sheets containing waxes (beeswax, candelilla and carnauba waxes) and its release and antimicrobial potential against *Escherichia coli* were analysed. KS was studied at 15 °C for its antimicrobial properties against *Escherichia coli* ATCC 10536. 15 °C is a non-common incubation temperature for E. coli but is often used for food storage of cheeses (in ripening cellars) or dry foods for example. Waxes are well studied blended to hydrocolloids to make coatings or films by solvent casting method but the novelty of this work is the use of a twin screw extruder to produce films. Morphological, barrier, tensile and antimicrobial properties were compared for casein/wax blends material produced by twin screw extrusion.

The presence of KS significantly inhibits *E. coli* and that even at low concentration and even after 20 days at 15 °C, which suggests effect of casein matrix as preservative carrier. Besides, a plasticizing action of KS was demonstrated at 10% addition on mechanical properties (decrease of TS and increase of %E) and on water barrier properties. Concerning water vapor permeability, only beeswax among waxes introduced in sheets was efficient to reduce WVP (by 20%). No significant impact on sheet color parameters was observed especially due to wax addition.

Finally, the work provided here might help researches on edible biopolymer as preservative carriers that can be produced by an easily industrial process (twin screw extrusion).

1. Introduction

Packaging wastes and pollution are meant to be reduced in the near future and new packaging solutions should be developed to enhance part of biodegradable and nontoxic packaging. Edible films or sheets are a response to this context concerning new, biodegradable and environmentally friendly packaging research. Additionally, healthier and high-quality foods with longer shelf life are requested nowadays by consumers. In recent years, researches have focusing on active packaging to act as carrier of active substances. Antimicrobials are extensively studied as active molecules incorporated in films to prevent microbial growth in food (Appendini & Hotchkiss, 2002; Cha & Chinnan, 2004; Chowdhury, 2013; Kowalczyk, Kordowska-Wiater, Solowiej, & Baraniak, 2015).

Biopolymers based on polysaccharides, lipids or proteins or blends of these categories are often used as matrix for active edible films. Casein is the main protein of milk and has been described as good film forming raw material, allowing production of flexible and transparent films with good oxygen-, aroma- and oil-barrier properties at low relative humidity (Khwaldia, Perez, Banon, Desobry, & Hardy, 2004; Krochta, 2002). Casein proteins were studied by several authors as active molecule carrier (Arrieta et al., 2014; Atarés, Bonilla, & Chiralt, 2010; Colak et al., 2016).

Protein films production can be achieved by two main casting solutions: wet and dry processes. Solution or wet casting is commonly used in the literature to produce edible films. This process allows the production of stand-alone films based on several raw materials (Arrieta et al., 2014; Basiak, Galus, & Lenart, 2015; Emiroğlu, Yemiş, Coşkun, &

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Candoğan, 2010). However, wet casting is not an easily industrial scale up process that could be used to produce commercial amount of films. Indeed, due to solvent use or energy- and time-consuming solvent evaporation step, this process doesn't seem to be a good replacer of synthetic polymer production. Dry process, like extrusion process, is at the opposite a large-scale production technic. Besides, this process is already used for synthetic polymer production and would just need to be adapted to new biopolymers blends. This continuous process, depending on the extruder size, can involve important feeding rates that imply rapid and large production with great commercial potential. Caseinate proteins have been successfully produced by extrusion process by Colak, Gouanve, Degraeve, Espuche, and Prochazka (2015) and Belyamani, Prochazka, and Assezat (2014).

Caseinates (sodium, calcium, magnesium or ammonium caseinates) corresponding to the neutralized form of acid casein and whey proteins have been widely studied for their ability to form films. However, insoluble milk proteins as acid or rennet casein on the contrary are not well known as film forming material (Chevalier, Assezat, Prochazka, & Oulahal, 2018). Proteins are recognized as good polymer matrix and imply excellent oxygen, aroma and oil barrier film production up to intermediate relative humidity conditions (Gennadios, 2002). However, hydrophilic nature of protein results in poor moisture barrier properties as WVP, swelling or solubility for example. Increasing interactions between protein chains by cross-linking and addition of lipids are the two principal techniques studied to decrease hydrophilic character of protein films. Caseinate cross-linking by enzymes, heating treatment, γ-irradiations, formaldehyde or calcium ions have been developed on solvent cast films (Audic & Chaufer, 2005; Avena-Bustillos & Krochta, 1993; Juvonen et al., 2011; Lacroix, Jobin, Mezgheni, Srour, & Boileau, 1998; Vachon et al., 2000). Protein/lipid composites could be interesting for commercial large-scale production by extrusion to reduce cost of protein based materials. Moreover, composite films are meant to take advantage of both components, in other words good oxygen barrier from protein and good moisture barrier from lipids for example.

For biopolymers, a swelling induced release is the main mechanism of active compounds release by diffusion of water molecules through polymeric matrix (Mastromatteo, Mastromatteo, Conte, Nobile, & Alessandro, 2010). Therefore, hydrophobic substances incorporation in the biopolymeric matrix is meant to delayed hydration and swelling and subsequently active agents diffusion (Ouattara, Simard, Piette, Bégin, & Holley, 2000; Ozdemir & Floros, 2003).

Concerning protein/lipid blending, few authors presented a work on caseinate and lipid or wax addition where films were exclusively prepared by wet casting (Aliheidari, Fazaeli, Ahmadi, Ghasemlou, & Emam-Djomeh, 2013; Avena-Bustillos & Krochta, 1993; Chick & Hernandez, 2002; Fabra, Talens, & Chiralt, 2008; Jiménez, Fabra, Talens, & Chiralt, 2013). To the best of our knowledge, incorporation of wax by an extrusion process was only reported by Janjarasskul et al. and Rauch with whey protein isolates as matrix of the composite (Janjarasskul, Rauch, McCarthy, & Krochta, 2014; Rauch, 2008). They proved that wax powder could be incorporated in the dry blend powder up to 8.1% and that extruder operating conditions were affecting films properties.

Casein used in this study are not common in literature indeed, rennet casein is less studied than caseinate because of its insolubility in water without any other treatment and the poor dispersibility observed when solvent casting solution are prepared. Aim of this work, first of all, is to assess the potential of extrusion process on composite material made of rennet casein and different edible waxes known as food additives (beeswax, candelilla wax and carnauba wax). Waxes were not added at more than 5% (w/w) to avoid breakthrough inside the material. Then, characteristics of sheets produced have been evaluated to determine the extent of modifications occurring by wax addition and particularly concerning water sensibility of sheets. Three different waxes were chosen in this work to have a comparative study of the effect of wax origin and physicochemical characteristics on the formulation and process. Organic acids are well known to be efficient food preservatives and their salts are preferentially used in food industry (Flores, Costa, Yamashita, Gerschenson, & Grossmann, 2010; Neetoo, Ye, & Chen, 2008; Stanojevic & Comic, 2009). Active edible sheet based on casein/wax have been studied regarding potential carrier of potassium sorbate as antimicrobial agent and antimicrobial properties have been assessed.

2. Material and methods

2.1. Materials/chemicals

Rennet casein native powder was purchased from Eurial Ingredients (Nantes, France). Different waxes, beeswax, candelilla wax and carnauba wax with the following melting temperature 63 °C, 68 °C, 84 °C and were purchase from Aroma-Zone (Cabrières d'Avignon, France). Glycerol and potassium sorbate were obtained from Sigma Aldrich (St Louis, MO, USA).

2.2. Casein sheet preparation

Casein sheets were prepared through a dry process with a co-rotating twin screw extruder as previously described by Colak et al. with modifications (Colak et al., 2015). A blend of rennet casein powder, wax powder and potassium sorbate (depending on the formulation, listed in Table 1) was introduced into the first zone of the extruder. Glycerol (13.2% w/w of protein powders) and an aqueous solution of acetic acid at 5° were added into the second zone to facilitate processability through amphiphilic property of acetic acid. Temperature from hopper to tie (5 cm width and 1 mm thickness) ranged from 10 °C to 75 °C. Sheets thickness is about 500 μm +/− 50 μm depending on formulation (determined with a digital micrometer). Residence time in twin screw extruder with a flow powder rate of 2 kg/h and screw speed at 170 rpm was determined between 2 and 6 min. Prepared sheets were stored in plastic bags in cold room (4 °C) until further experiments. All sheets specimens were conditioned 48 h in a climate room at 50%

Table 1
Formulations of extruded sheets studied in this work and their abbreviations.

Abbreviations		Formulations (ingredients concentration expressed as percentage w/w of casein)			
KS 0	Without wax	Rennet casein	13.2% glycerol		
	BW1				Beeswax (BW) 1%
	CW1				Candelilla wax (CW) 1%
	CaW1				Carnauba wax (CaW)1%
KS 2	Without wax	Rennet casein	13.2% glycerol	Potassium sorbate (KS) 2%	
	BW1				Beeswax 1%
	BW5				Beeswax 5%
	CW1				Candelilla wax 1%
	CW5*				Candelilla wax 5%
	CaW1				Carnauba wax 1%
	CaW5				Carnauba wax 5%
KS 10	Without wax	Rennet casein	13.2% glycerol	Potassium sorbate 10%	
	BW1				Beeswax 1%
	CW1				Candelilla wax 1%
	CaW1				Carnauba wax 1%

CW5*/KS2: High concentration (5%) of candelilla wax did not produce a coherent and homogenous matrix through extrusion technic.