



## Effects of film constituents on packaging-relevant properties of sodium caseinate-based emulsion films



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### ABSTRACT

The objective of this study was to examine the effects of the plasticizer type (sorbitol or glycerol) and lipid concentration (oleic acid or oleic acid-beeswax mixtures) on sodium caseinate (NaCas)-based films and to investigate how, and to which extent, these parameters are affecting the technofunctional properties of NaCas-based films. Additionally, we intended to demonstrate the antioxidant potential of quercetin as a component of the described formulations. Films prepared from the different composed emulsions were characterized in terms of selected packaging-relevant properties. The results showed that the type of plasticizer and the lipid concentration had significant effect on the technofunctional properties of NaCas-based films. Contact angle measurement and evaluation of the SFE revealed rather hydrophilic than hydrophobic surfaces. Determination of the WVTR showed a significant influence ( $p \leq 0.05$ ) of the type of plasticizer towards WVTR. WVTR was clearly decreased for sorbitol-plasticized films. On the contrary, the influence of the lipid concentration was also significant ( $p \leq 0.05$ ), but not as clear as the plasticizer effect. However, at 10% (w/w) beeswax (BW) addition, there was a sharp drop in WVTR visible for glycerol-plasticized films. Measurement of the OP did not yield as clear results as suggested in preliminary test series. Although, we did not conduct systematic studies regarding the effect of the antioxidant towards the OP we showed the antioxidant potential of the described compositions. Results for the mechanical performance showed a significant influence ( $p \leq 0.05$ ) of the type of plasticizer and lipid concentration. YM and TS values were clearly higher for sorbitol-plasticized films whereas E% values were lower for sorbitol-plasticized films. Regarding the effect of the lipid concentration on the mechanical performance, no apparently visible trend could be identified. Light transmittance measurement revealed that in terms of UV-absorptivity at wavelengths between 300 nm and 400 nm, the incorporation of quercetin led to promising barrier properties against UV-light.

### 1. Introduction

The selection of a suitable material composition for edible films and coatings is very important in respect to the specific requirements a food demands from its packaging and the intended field of application. Edible films derived from various plant and animal sources have been investigated because of their promising potential to enhance food quality and possible application to replace synthetic polymers for certain applications, e.g. caseins [1–4], and whey proteins [5–9]. An additional interest comes from the public awareness for the fact that petrochemical-derived raw materials are limited and their employment in any form can lead to waste-disposal problems, which has increased significantly in recent years [10].

Generally, the primary function of every packaging material is the

protection of the packaged good throughout its entire life cycle. Of particular importance in this context is the ability of packaging materials to control mass transfer throughout the film matrix, e.g. permeation of gases like water vapor or oxygen, as well as the migration of oils, fats and organic compounds from and to the good in order to prolong the shelf-life and maintain or improve organoleptic properties [11].

Materials capable of forming edible films and coatings can generally be divided into three categories: hydrocolloids, lipids, and composites. Hydrocolloids consist of proteins, e.g. caseins and whey proteins, and polysaccharides, e.g. starch and cellulose derivatives. Milk proteins, such as caseins and whey proteins, have been extensively studied due to their various functional properties, such as barrier- and mechanical properties, solubility- and swelling behavior, and emulsifying properties [2,6,12–20]. Caseins, with a share of approximately 80%, represent the

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major protein fraction in bovine skim milk. It mainly consists out of  $\alpha_{s1}$ -,  $\alpha_{s2}$ -casein,  $\beta$ -casein, and  $\kappa$ -casein [21,22]. All casein fractions contain high amounts (35–40%) of non-polar amino acids, e.g. Valin, Leucin, and Prolin. The disorganized (random coil) structure, high amount of polar amino acid residues and their uneven distribution is believed to be responsible for the good emulsifying properties and thermal stability of caseinates [22]. Caseins possess the ability to form extensive intermolecular hydrogen, electrostatic and hydrophobic bonds [18], which is the reason for the good film-forming properties out of aqueous solutions.

Lipids include fatty acids, e.g. oleic acid or stearic acid, and waxes, e.g. beeswax or carnauba wax. Edible composite films contain mixtures of hydrocolloids and lipids and can appear either in the form of dried emulsions or as bilayer films [23]. The incorporation of lipids and waxes into protein-based films may weaken the three-dimensional protein network and thus can result in a plasticizing effect [11]. As reported by Gontard, Marchesseau [24], the poor cohesive energy of lipids and waxes are responsible for a reduction of film strength and increase of film flexibility. Among lipids, waxes are the most effective substances due to their high hydrophobicity compared to fatty acids. Beeswax consists of wax esters, wax acids, and hydrocarbons and possesses over viscoelastic properties [25]. It is reported to decrease water vapor permeability of edible films due to its high hydrophobicity and solid state at room temperature and is used in food to prevent water loss [26]. It is approved for food use in most countries and in the European Union under the E number “E901” [27].

Plasticizers used for protein-based films commonly include polyols, mono-, di-, and oligosaccharides. Polyol plasticizers have been used extensively for hydrocolloid and especially protein-based films. They are water soluble, protein-compatible, polar, non-volatile, and high boiling-point substances [28]. Polyols, like glycerol or sorbitol, are highly hygroscopic, low molecular weight substances which possess the ability to interact with polar amino acid residues via hydrogen bonding and facilitate sorption of water molecules from the surrounding environment and thus enhance plasticization of protein-based films [29].

Generally, especially at low relative humidity, proteins possess over good barrier properties against oxygen, carbon dioxide, aroma components, and lipids due to their hydrophilic nature [30,31]. However, the application of protein-based packaging materials is limited as these materials exhibit poor moisture barrier and mechanical properties [26,32]. By combining the properties of protein and lipid films, in which the dispersed hydrophobic phase provides suitable moisture barrier properties, it is possible to overcome the poor moisture barrier properties, but keep the good oxygen barrier properties [33,34].

Shellhammer and Krochta [8] reported that the moisture permeability properties in protein-lipid emulsion-type films are dependent on the lipid type and amount. They found that for viscoelastic lipids such as beeswax and a high melting anhydrous milk fat a critical concentration existed at which the water vapor permeability of whey protein-based emulsion-type films suddenly decreased compared to less viscoelastic and much harder waxes, such as carnauba and candelilla wax. The type of composite film, being either bilayer or emulsion-type film, is reported to have significant influence on film strength. Emulsion-type films have been shown to possess better mechanical properties compared to bilayer films [1]. In order to overcome film brittleness and impart flexibility to the protein matrix, the incorporation of plasticizers into hydrocolloid networks is essential [35]. Plasticizers function by interrupting protein–protein interactions and increasing the sorption of water molecules into the film matrix [36] thus increasing the polymer free-volume [29] and lowering the glass transition temperature [37]. However, an additional effect of improving the mechanical performance is the simultaneous increase of the permeation coefficient due to the increased free-volume [38].

Flavonoids are particularly antioxidant and universally present in plants, hence also in human food. Quercetin, 2-(3,4-dihydroxyphenyl)-3,5,7-trihydroxy-4H-chromen-4-one, is one of the most abundant

dietary flavonoids [39] and an effective antioxidant [40]. In general flavonoids are instable in contact with air, but entrapped in films composed of caseins and caseinates they can be protected against rapid oxidation [41] during the film preparation process.

To our knowledge, no information regarding antioxidant activity of quercetin in NaCas-based films is available from the literature. This study aims on the development of a bio-based antioxidant packaging film or coating material. In order to ensure suitable properties for packaging applications, the effects of the plasticizer type and lipid concentration on the technofunctional properties, including water vapor transmission rate (WVTR), surface free energy (SFE), contact angle, Young's modulus (YM), tensile strength (TS), elongation at break (E%), and light transmittance were assessed. Quercetin was assessed regarding its promising antioxidant properties entrapped within the biopolymer network as a part of the film composition.

## 2. Materials and methods

### 2.1. Materials

Sodium caseinate (NaCas) was supplied by Sigma-Aldrich GmbH (Steinheim, Germany). Distilled water was provided by the Fraunhofer Institute for Process Engineering and Packaging IVV (Freising, Germany), and the Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB (Stuttgart, Germany). Film-forming dispersions contained oleic acid (technical grade 90%) and beeswax (reagent grade  $\geq 95\%$ ; BW) to increase film hydrophobicity, glycerol (reagent grade  $\geq 99\%$ ; Gly) or sorbitol (reagent grade  $\geq 98\%$ ; Sor) as plasticizing substances, as well as quercetin (reagent grade  $\geq 95\%$ ; Q) for antioxidant activity. All utilized substances were purchased from Sigma-Aldrich GmbH (Steinheim, Germany).

### 2.2. Preparation of sodium caseinate-based emulsion films

Sodium caseinate (NaCas) was mixed into distilled water (10% w/w) at room temperature and  $\sim 1000$  rpm and stirred continuously for approximately two hours using a magnetic stirrer (Ikamag RCT, IKA-Labortechnik, Staufen, Germany). After the protein is completely suspended, either glycerol or sorbitol was added to the mixture and stirred for another 3 h at 1200 rpm. The relation NaCas:plasticizer was 0.66 parts plasticizer per part protein. Before the lipid materials were added, the magnetic stirrer was removed. Oleic acid was added in a share of 0.35 parts oleic acid per part protein. The amount of beeswax added to the mixture varied according to the film composition (0%, 6.6%, 10%, 15%, 20% (w/w) of dry matter). In preparation of the homogenization process, the mixture was heated to 85 °C in a water bath. Consequently, due to the heat treatment, phase separation occurs. After the water bath reached the desired temperature, the mixture was homogenized using an IKA Ultra-Turrax Model T25 (IKA-Labortechnik, Staufen, Germany). The first homogenization step was performed at 20,200 rpm for 1 min. Subsequent to the first homogenization step the dispersion was allowed to sit for 5 min in order to let incorporated air escape. The second homogenization step was performed at 20,200 rpm for 2 min. The mixture remains in the tempered water bath at 85 °C throughout the entire homogenization process. Following homogenization, the mixture was cooled down to room temperature using a magnetic stirrer (Ikamag RCT, IKA-Labortechnik, Staufen, Germany). Once the mixture reached room temperature, the quercetin was added, accounting for 1% (w/w) in respect to the total mass of the dispersion. The dispersion was continuously stirred for 1 h, until the quercetin was completely dispersed. Following dispersion preparation, the mixture was allowed to sit for at least 1 h in order to let incorporated air escape. Films were prepared by pouring 15 mL of the dispersion into a series of Petri dishes (12 cm x 12 cm, PS, Greiner Bio-One, Germany) on a leveled surface. The mixture was spread with the aid of a spatula in order to ensure even distribution. The dispersions were dried at 23 °C and 40% r.h. for 3 days.

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