



Electrospraying of water in oil emulsions for thin film coating



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ABSTRACT

Electrospraying of water-in-oil emulsions was investigated to produce thin edible barriers. A reproducible model surface was used, namely cellulose membranes of which permeability is well-established. PGPR-based emulsions were stable during electrospraying and produced a fine stable jet spray; emulsions prepared with lecithin and span80 produced unstable jets and only sporadic sprays. The droplet size decreased to $50 \pm 10 \mu\text{m}$ by the addition of water droplets till 3% but remained constant at higher water volume fractions. On the other hand, the addition of protein and salt influenced the droplet size and radius values up to $200 \mu\text{m}$ were found. The addition of whey protein isolate (10% w/w) resulted in films with water vapour barrier properties that were at least 70% better than lipid only, therewith showing that addition of protein enhances the integrity of the films.

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1. Introduction

Various strategies and technologies have been developed to maintain the quality and to extend the shelf life of (ready-to-eat) food products. In this paper, focus is on edible coatings that can be used for these purposes, since they act as barriers to water and gases exchange. A range of ingredients such as proteins, polysaccharides and lipids can be used either as single ingredient or in combinations to produce edible coatings with varying mechanical and barrier properties (Chiumarelli and Hubinger, 2012).

Polysaccharides and protein based coatings are known to form good films having low oxygen permeability. These coatings mostly do not alter the taste and appearance of the product (Chiumarelli and Hubinger, 2012). Lipids are excellent moisture barriers due to their low affinity with water; examples of lipids used in coatings are waxes, lacquers, fatty acids, alcohols, acetylated glycerides, and cocoa based compounds (Debeaufort and Voilley, 2009; Khan et al., 2012c).

The films of pure components are mostly brittle which induces cracks in the films, resulting in poor food protection. This may be circumvented through combination of e.g. protein and lipid, which are known respectively as good barriers for oxygen and water vapour, ideally resulting in films with better moisture resistance, improved mechanical flexibility, and transparency (Hambleton et al., 2011; Monedero et al., 2009; Pereda et al., 2012). These blends are mostly applied through spraying of an emulsion in

which both components can be well distributed (de la Caba et al., 2012).

Various coating techniques have been developed for preparing thin films; spray coating (mechanical or ultrasonic) may be the most common method, because of low equipment cost, good control, and the availability of various 'sprayable' materials. During spraying, droplets are formed and their size co-determines the quality of the deposited film (Khan et al., 2012c; Oh et al., 2008).

Electrospraying is another spraying technique that has potential for coating operations. Applying a potential difference over a droplet emerging from a nozzle causes instabilities on the droplet surface which then spontaneously breaks up into smaller and relatively mono-disperse, charged droplets (Jaworek, 2007, 2008; Khan et al., 2012c). The charged droplets follow a trajectory to the nearest grounded surface as a result of electrostatic attraction, leading to a high transfer efficiency compared to conventional spraying methods (Luo et al., 2012; Maski and Durairaj, 2010; Oh et al., 2008). Upon impact with the target surface, release of the charge needs to take place to allow following droplets to deposit on the surface and subsequently form a complete film (Khan et al., 2012b,c).

Electrospraying of lipid-based coating materials has been well documented (Gorty and Barringer, 2011; Khan et al., 2012a,c; Luo et al., 2012; Marthina and Barringer, 2012). Mostly lecithin, an ionic surfactant, is used to increase the conductivity of the to-be-sprayed lipid material; lipids are otherwise non-conductive and not suitable for electrospraying. In this paper, an emulsion as a starting point was used, that ultimately make barriers with dual functionality. As information on electrospraying of emulsions is

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still lacking in literature, first the atomization behaviour of water-in-oil emulsions prepared with different surfactants was investigated, and later this was extended to electrospraying of protein containing emulsions. Various formulations and process conditions were used, and droplet deposition behaviour was reported in relation to film formation and barrier properties.

2. Materials and methods

2.1. Materials

Sunflower oil purchased from a local super market was used as continuous phase and MilliQ water was used as disperse phase. Span80 (Sigma–Aldrich, Germany), Alcolic-S lecithin (American lecithin company, USA) and polyglycerol polyricinoleate (PGPR) (Givaudan, Vernier, Switzerland) were used as surfactants. Whey protein Isolates (Bipro, provided by FrieslandCampina, The Netherlands) sucrose and sodium chloride (Merck KGaA, Darmstadt, Germany) were added to the water phase at different concentration and specified in respective sections.

2.2. Methods

2.2.1. Emulsion preparation

Water-in-oil emulsions were prepared using an Ultra-Turrax homogenizer (T18 basic, IKA-werke, Staufen, Germany). MilliQ water (0.5%, 1%, 3%, 5%, 7%, 10%, 20%, 30%, and 40% (w/w)) was gradually added to sunflower oil with 5% (w/w) surfactant. The conductivities of the oil phase and the resulting emulsion were measured using an electrometer (6514 system electrometer, Keithley, USA). A SevenMulti conductivity meter (Mettler Toledo international Inc.) was used to measure the conductivity of salt and sucrose solutions.

2.2.2. Electrospraying and film formation

A single-nozzle electrospraying system (Terronics Development Cooperation, USA) was used to prepare the emulsion films. The emulsions were supplied at a flow rate of 15 ml/h via a syringe pump (Harvard 11 plus, Harvard Inc., USA). An electric potential was applied to the nozzle by a high voltage source (Heinzinger electronic GmbH, Germany). Moreover, the spraying conditions (emulsion flow rate and applied voltage) were adjusted to obtain a stable jet spray. Table 1 presents a summary of the applied conditions for electrospraying as a function of the water and salt content.

The emulsion films were prepared on two model surfaces; glass slides and cellulose membranes (dialysis membrane from Akzo Nobel N.V., the Netherlands). The surfaces were examined with a microscope (Axiovert 200 MAT, Carl Zeiss B.V., Sliedrecht, The Netherlands) attached to a camera (MotionPro HS4, Redlake MASD Inc., San Diego, CA, USA), and the microscopic images were analysed for droplet size using image analysis software (Image Pro plus 4.5). For the average value of droplet size, the average of five pictures con-

taining at least twenty droplets was determined. The permeability of the coated cellulose membrane was further tested as described in the next section.

2.2.3. Film properties

The emulsion films were analysed for water vapour permeability with a gravimetric method using a modified ASTM (1983) procedure (Khan et al., 2012a). The coated membrane was placed on top of a permeation cell containing MilliQ water (relative humidity inside the cell is 100%). The cell was tightly sealed and placed in a humidity chamber maintained at 20 °C and 60% RH. The cell was weighed daily, the weight loss as function of time was registered, and analysed by linear regression. Three replicates were measured for each film and the flux reduction was calculated as follows;

$$\text{Flux reduction (\%)} = \left(1 - \frac{M_c}{M_b}\right) \times 100 \quad (1)$$

where M_c and M_b are the rates of moisture loss as a function of time (g/h) through the coated and uncoated (bare) membranes, respectively. Moreover, barrier efficiency of the film was calculated as function of deposited amount (M) on the membrane surface.

$$\text{Barrier efficiency (\%/g)} = (\text{Flux reduction}/M) \quad (2)$$

2.2.4. Scaling of droplet size in cone-jet mode

The mechanism of electrospraying in the cone-jet mode has been studied by various authors (Fernandez de La Mora and Loscertales, 1994; Ganán-Calvo, 2004; Gañán-Calvo and Montanero, 2009; Hartman et al., 2000) who related the fluid properties and the operating conditions to the droplet size in air. The effective electric stress ΔP (due to the applied voltage) is defined as:

$$\Delta P = k_p \left(\frac{\sigma^2 \kappa^2 \rho}{\epsilon_0^2}\right)^{1/3} \quad (3)$$

where σ is the surface tension (N m⁻¹), κ is the electrical conductivity of the liquid (S/m), ρ is the liquid density (kg m⁻³), ϵ_0 is the permittivity of vacuum, and k_p is a constant in the order of unity. Based on analysis of the stability limits of the cone-jet mode, a scaling law for the cone-jet radius R_d was derived given certain process limitations that are summarised in $We \leq 20$.

$$R_d = \left(\frac{Q^2 \rho}{2\pi^2 \Delta P}\right)^{1/4} \quad \text{if } We = \frac{\rho Q^2}{\sigma \pi^2 R_d^3} \leq 20 \quad (4)$$

where Q is the flow rate and We is the dimensionless Weber number. The droplet size on the target surface depends on the contact angle (θ) of the droplet on the surface (Khan et al., 2012c), and can be calculated as follows;

$$a = R_d \left(\frac{4 \sin^3 \theta}{2 - 3 \cos \theta + \cos^3 \theta}\right)^{1/3} \quad (5)$$

where $2a$ is the diameter of the falling droplet on target surface. This equation was used to compare the experimental results of the study.

3. Results and discussion

3.1. Electrical conductivity

Electrical conductivity is an important parameter for electrospraying, which should normally be between 1×10^{-5} S/m and 1×10^{-10} S/m for successful atomization (Hayati et al., 1987). Low conductivity limits atomization due to insufficient electrical

Table 1

Minimum electrical potentials applied during electrospraying of water in oil emulsions to obtain a stable jet spray in the cone-mode at a flow rate of 15 ml/h.

Water content		NaCl content in 10% water emulsion	
Amount (%w/w)	Applied voltage (kV)	Amount (%w/w)	Applied voltage (kV)
0	12 ± 1.5	0.01	14 ± 0.5
0.5	12 ± 1.5	0.1	10 ± 1
1–20	14 ± 0.5	0.3	11 ± 0.5
30	17 ± 1	0.5	9 ± 1
40	18 ± 0.5	0.7	9 ± 0.5

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