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## Physicochemical properties of chitosan-essential oils film-forming dispersions. Effect of homogenization treatments

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### Abstract

Chitosan films enriched with basil (B) or thyme (T) essential oils (EO) were prepared by means of two homogenization treatments, without (H1) and with (H2) microfluidization. H2 treatment led to a significant decrease in the average particle size and the type of EO significantly affected  $d_{43}$  values ( $p < 0.05$ ).  $\zeta$ -potential significantly increased with the use of H2 treatment. Treatment H2 changed the rheological behaviour of the FFD. In all cases, the viscosity of the FFD was reduced by this treatment. The type of EO had a different effect on rheological behaviour depending on the homogenization treatment. CH:T were more viscous at H2 and CH:A showed higher apparent viscosity values than CH:T at H1.

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*Keywords:* chitosan, essential oils, microfluidization, basil, thyme

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### 1. Introduction

Droplet size is a determining factor for emulsion stability, and affects other important properties of the emulsion, including its viscosity. Rotor-stator homogenizers are often used in the food industry, and are able to reach particle sizes in the range of 1  $\mu\text{m}$ . This can be further reduced by applying an increased pressure in the system. Microfluidization (MF) can provide emulsions with narrower particle size distributions than those obtained at low pressure. This is attained in the interaction chamber of the microfluidizer, where the emulsion is submitted to high shear stress. There are few studies dealing with the effect of microfluidization on the properties of the film-forming dispersions (FFD) used to obtain edible films and coatings [1]. The combination of hydrophilic constituents such as chitosan and some lipids could produce films with optimized characteristics [2]. Many lipids have been incorporated to composite films, mainly aiming to reduce the water vapour permeability of these hydrophilic materials.

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Essential oils (EO) are interesting lipids to be incorporated into the films since these plant extracts exhibit additional characteristics, such as antimicrobial and antioxidant effects [3, 4]. This would be in line with current research into the reduction of the use of chemical additives in the food industry. The aim of this work is to study the influence of homogenization treatments on particle size distribution,  $\zeta$ -potential and rheological properties of chitosan-essential oils FFD as compared to pure chitosan dispersions.

## 2. Materials and Methods

### 2.1 Materials

High molecular weight chitosan (CH) (Batch 12913CJ, Sigma-Aldrich Quimica, Madrid, Spain) was used to prepare the film-forming dispersions (FFD) (0.8 Pa s viscosity, at 1% w/w in 1% w/w acetic acid). Basil (B) and thyme (T) essential oils were provided by Herbes del Moli (Alicante, Spain) and  $\text{Mg}(\text{NO}_3)_2$  by Panreac Quimica, S.A. (Castellar del Vallés, Barcelona, Spain).

### 2.2. Preparation of the film-forming dispersions

High molecular weight CH (1 wt%), dispersed in an acetic acid solution (1 % v/w), and basil (B) or thyme (T) essential oils (1 wt%) were mixed by two homogenization treatments, namely H1 and H2. FFD submitted to H1 were mixed by means of a rotor-stator homogenizer at 21500 rpm for 4 minutes. H2 treatment consisted of H1 plus high-pressure homogenization at 165 MPa in a single pass by means of a Microfluidizer® M110-P processor.

### 2.3 Characterization of the film-forming dispersions

#### 2.3.1 Particle size distribution

The particle size analysis of the emulsions was carried out using a laser diffractometer (Mastersizer 2000, Malvern Instruments, Worcestershire, UK) with ultrasound application. The samples were diluted in a sodium acetate buffer solution (175 mM) under the appropriate solvent conditions (pH = 4.8) at 2,000 rpm until an obscuration rate of 10% was obtained. Mie theory was applied considering a refractive index of 1.47 and 1.50 for T and B essential oils, respectively, and 0 absorption in both cases. Three replications per formulation were made. The volume-surface mean diameter ( $d_{3,2}$ ) as well as the droplet size distribution were determined [5].

#### 2.3.2 pH and $\zeta$ -potential

FFD were analyzed at 25°C in terms of pH.  $\zeta$ -potential was measured using a Zetasizer nano-Z (Malvern Instruments, Worcestershire, UK). The electrophoretic mobility of the droplets was transformed into  $\zeta$ -potential values using the Smoluchowsky mathematical model. The samples were diluted to a droplet concentration of 0.02% using a sodium acetate buffer solution (175 mM) at pH 4.8.

#### 2.3.3 Rheological behaviour

The rheological behaviour of FFD was analysed at 25°C using a rotational rheometer (HAAKE Rheostress 1, Thermo Electric Corporation, Karlsruhe, Germany) with a sensor system of coaxial cylinders, type Z34DIN Ti. Samples were left to rest for 5 minutes before the measurements were taken. The shear stress ( $\sigma$ ) was measured as a function of shear rate ( $\dot{\gamma}$ ) between 0 and 300  $\text{s}^{-1}$ , taking 3

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