



## Stability of low-fat oil in water emulsions obtained by ultra turrax, rotor-stator and ultrasound homogenization methods



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### ABSTRACT

The stabilities of 20 formulations of low-fat oil in water emulsions were evaluated with Ultra Turrax, rotor-stator and ultrasound applied as homogenization methods. Emulsions were formulated with extra virgin olive oil (15–20% w/w), egg yolk (0–5% w/w), mustard (0–5% w/w) and vinegar (10–25% w/w). The stability was determined at 7 days of storage through the parameters:  $\zeta$  potential (mV), droplet size (nm), phase separation (cm), and viscosity ( $\mu$ ). The most stable emulsions were found with less phase separation distance in order ultrasound, rotor-stator and Ultra Turrax. Emulsions with smaller droplet sizes were obtained with ultrasound. The highest viscosity was found in emulsions obtained with the rotor-stator homogenizer. Regarding sensory preference, 43% acceptance was obtained with the rotor-stator method versus 21% for ultrasound. To cooks and chefs the use of rotor stator as homogenization technology could be an opportunity to prepare low fat emulsions with lower separation phases without effects on overall taste perception.

### Introduction

Various food products are made starting from different oil and water ratios that form immiscible phases, and in their stable form, they are found to form emulsions. Thus, an important quality characteristic in low-fat emulsions is the stability over time and acceptance in sensory perception. Therefore, the stabilizers and homogenization methods used constitute the most relevant aspects in the design of emulsions (Chemat et al., 2011; Juliano et al., 2011; Chung and McClements, 2014; Piorkowski and McClements, 2014). Additionally, the concentration of oil present into dispersed phase in water emulsions (O/W), is highlighted to salad dressing products (Dridi et al., 2016). In traditional culinary preparations, dressings are formulated in a 3:1 ratio of the water-vinegar dispersed phase to the fat continuous phase. Thus, it is important to evaluate the change in this ratio to obtain dressings with low fat contents. Among the most used ingredients to maintain stability in emulsions, egg yolk is found to be widely used in salad dressings, and its function has been described by Ma et al. (2013). It helps decrease interfacial tension, forming a layer that prevents the aggregation of drops in an emulsion. Additionally, Marcet et al. (2016); Orcajo et al. (2013) have defined the following two fractions in egg

yolk: plasma and granules. The plasma fraction contains 75–81% of the solids of the yolk and is largely composed of low density lipoproteins (LDL); the granular fraction largely contains high density lipoproteins (HDL). As an ingredient in emulsions, the use of egg yolk that has not undergone thermal treatment is relevant as has been explained by Marcet et al. (2016), who demonstrated that the thermal denaturation of egg yolk can alter its functionality as an emulsifier. An increase in the degree of denaturation of yolk proteins can increase the concentration of interfacial protein, which suggests that it could increase the steric repulsions between drops of the emulsion.

Cooks and chefs usually obtain emulsions by mechanical agitation, however, the use of other systems such as rotor-stator, ultrasound and Ultra Turrax could contribute to improve the stability in low fat emulsions. In this way, the use of ultrasound is justified due to agitation that occurs by cavitation, the mechanical homogenization with a rotor-stator (Maa and Hsu, 1996) has been used to obtain colloids, emulsions and dispersions. These are produced by colloidal or dispersion mills forming droplets or particles smaller than a micron, which can be suspensions constituted by separate particles, with feeds approximately equal to 100 mesh (50  $\mu$ m). For culinary applications these homogenization methods at less than 5 min of process, have not

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influence on the energy consumption to obtain emulsions.

In recent years, the use of ultrasound in the emulsification process has increased mainly because of its energetic efficiency, low cost of production, ease of handling and better control over components (Abbas et al., 2014). Power ultrasound applications have been considered emerging technologies, in addition to being considered a green technology that offers high potential for a variety of processes (Gallego-Juárez et al., 2010). Compared to other methods that influence heat or mass transfer phenomena (Cárcel et al., 2012), one of the main effects of the use of high power ultrasound has been stabilization in the processing of emulsions depending on the characteristics of the matrix. For example, in the case of two immiscible liquids, if a bubble collapses near the limit of the phase of the liquids, the resulting shock wave can provide very effective mixing of the layers requiring less surfactant, and thus, emulsions with smaller drop sizes within the size distribution are obtained (Mason et al., 2005).

Homogenization systems affect the stability of emulsions, and this can be represented by the  $\zeta$  potential, which is widely used to understand the behaviour of the interface of O/W emulsions (Acedo-Carrillo et al., 2006). Thus, a change in the  $\zeta$  potential according to composition conditions represents significant information on the condition of the interface, which determines the useful life of emulsion droplets in the continuous phase (Stachurski and Michalek, 1996). However, understanding the rheology in emulsions is important from a practical point of view to establish the best formulation, preparation, manipulation, storage and transportation conditions. In addition, knowledge of the rheological properties can provide useful information on the stability and microstructure of the emulsion (Pal, 2011). Taking measurements of the strain rate is useful for rheological control of products based on emulsions to which shear, compression or both are applied. For its part, viscosity is used to indicate thickness of the liquid (Chung and McClements, 2014). Thus, the importance of rheological properties in dressings is related to the quality, sensory attributes, useful life and microstructure. Different rheological tests have been performed to understand the internal structural organization and interaction of components within emulsions. An example of this is a flow test which provides information on the force of interactions between droplets within the emulsion (Ma et al., 2013).

Another way to evaluate the stability and acceptance of a product is through sensory analysis, which has led to humans evaluating products that can be consumed in terms of how good or bad they are perceived (Meilgaard et al., 2006). Regarding emulsions, the organization of the composition and structure determines the desirable sensory attributes, as they are influenced by the continuous and dispersed phases as well as characteristics of the interfacial region (Chung and McClements, 2014). In recent years, low-fat salad dressings have received more attention from the food industry due to the higher salad consumption as a healthy food option (de Melo et al., 2015). On the other hand, nowadays is a need to integrate the scientific research with the development in the culinary field, for innovation in new culinary techniques (Ruiz et al., 2013). That is why the present study is an opportunity for chefs, cooks and researchers to incorporate these technologies as an alternative to bring close scientific knowledge and gastronomy in order to generate new applications (Valverde et al., 2016). Thus, the objective of this research was to evaluate the influence of the composition of low-fat O/W emulsions obtained by Ultra Turrax, rotor-stator and ultrasound, which is represented on the stability following these parameters: phase separation time,  $\zeta$  potential, pH, droplet size, viscosity and sensory acceptance.

## Materials and methods

### Materials

To obtain O/W emulsions, the following were used: distilled water, Borges® extra virgin olive oil (Tarragona-Spain), Colman's® English mustard, Colman's® white vinegar (pH = 2.63), and egg yolk extracted

manually followed by drying the excess white on absorbent paper (Chatsisvili et al., 2012; Magnusson and Nilsson, 2011). The eggs used were AA organic from La Granja® and were acquired from a local supermarket in Bogotá, Colombia.

### Preparation of O/W emulsions

Prior sensory analysis was conducted to determine the preference thresholds of vinegar flavour in a non-trained panel of 20 people between 20 and 40 years old. From the preference threshold results, the percent composition was defined between 10% and 25% (w/w) for vinegar in the formulation. The dispersed phase was defined between 15% (w/w) (Kaci et al., 2014) and 20% (w/w) (Santipanichwong and Suphantharika, 2009) oil. For yolk and mustard, incorporation values in the formulation were established from 0% to 5% (w/w) to evaluate their individual effects on the stability (Ma et al., 2013). An Ultra Turrax® homogenizer (T-25 basic, Janke and Kunkel IKA, Germany) with a S25KV-25F model stem was used at a speed of 9500 rpm during 5 min, the sample volume was 200 ml. For the emulsions prepared by ultrasound, an ultrasonic processor (Qsónica®, Q700 sonicator, 700 W RMS, United States) was used at 20 kHz for 5 min (Paradiso et al., 2015). The ultrasound probe was placed inside a noise reduction box whose interior walls were lined with water resistant acoustic foam. A 25.4 mm tip made with a titanium alloy and having a cylindrical diameter and geometry was used. The probe was placed 10 mm below the surface of a pre-emulsion with a 50 ml volume, and amplitude of 42  $\mu$ m with continuous sonication was used. For the emulsions obtained by rotor-stator, a rotor-stator apparatus (IKA®, Magic Lab, United States) was used at 26,000 rpm for 5 min, recirculating the emulsion, and a 6 F generator was also used, the sample volume was 200 ml.

### Determination of the $\zeta$ potential

The  $\zeta$  potentials of emulsions were evaluated as a stability parameter. The evaluation of the  $\zeta$  potential was performed in a Malvern Zetasizer Nano ZS apparatus (United Kingdom). A 1:100 (w/w) dilution was made (Shukat and Relkin, 2011) in ultrapure type I deionized water with a resistance of 18.2 M $\Omega$ -cm (PURELAB option-Q). Measurements were taken in triplicate for each sample.

### pH

Measurement of the pH value was performed on 50 ml of emulsion placed in Falcon tubes, using an UltraBASIC pH/mV meter (United States), Method AOAC 981.12 (AOAC,1997).

### Droplet size

The droplet size of the emulsion was measured in a Malvern Zetasizer Nano ZS apparatus (United Kingdom) (Shukat and Relkin, 2011). A 1:100 (w/w) dilution was made in ultrapure type I deionized water with a resistance of 18.2 M $\Omega$ -cm (PURELAB option-Q). Measurements were taken in triplicate for each sample.

### Phase separation

For each formulation, phase separation was evaluated every day for 7 days. Emulsions were deposited in plastic Falcon tubes with a 50 ml volume, and a visible mark was made upon observing phase separation (Acedo-Carrillo et al., 2006; Darine et al., 2011; Nesterenko et al., 2014). Emulsions were stored at room temperature in the presence of daylight. At the end of the evaluation time, images were captured with a SONY 14.2 Mpx camera (DSLR-A330/A380). Images were analysed through the program ImageJ® version 1.48. The results that are shown correspond to emulsions that did not show separation in the evaluated technologies (Shanmugam and Ashokkumar, 2014).

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