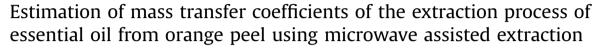
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# ABSTRACT

Microwave assisted extraction (MAE) is an emerging technique of extraction that improve yields and reduce process time and energy. The aim of this work was to evaluate the effect of different MAE process parameters in the extraction yield of orange peel essential oil (EO), and the description and simulation of this process with mathematical models based on mass transfer fundamentals. For the assessment of process parameters effects on EO yield, different extractions were carried out following a two-level factorial design varying orange peel particle shape (spheres or plaques) and moisture content (dry or not), as well as microwave potency (360 or 540 W). Results demonstrated that particle size, moisture content, and its interaction significantly affected (p < 0.05) the yield obtained and had an influence on the extraction mechanism. Model parameter values associated to mass transfer coefficients ( $k_1$  and  $k_2$ ) indicated that diffusion was the process that defined extraction velocity.

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# 1. Introduction

*Citrus* are the most cultivated fruits in the world, being oranges 60% of the world fruit production. The orange peel, depending on the orange variety, may represent 45% of the total bulk. Orange peel includes the epidermis covering the exocarp that consists of irregular parenchymatous cells, which encloses numerous glands or oil sacs (Farhat et al., 2011; Velázquez-Nuñez et al., 2013). The oil in these sacs represents the citrus essential oil (EO) that is obtained as by-product of citrus processing (Bousbia et al., 2009). Besides its use as a flavoring agent, citrus EO has gained relevance in the food industry due to its antimicrobial effect against both bacteria and fungi (Rezzoug and Louka, 2009; Velázquez-Nuñez et al., 2013; Lago et al., 2014). Typical processes for obtaining essential oils from citrus include cold pressing, solvent extraction, and different distillation techniques (Bica et al., 2011; Lago et al., 2014). However, these procedures involve several disadvantages, such as the use of volatile and hazardous solvents, low yields, long extraction times, and high energy consumption (Zu et al., 2012).

The extraction method is one of the prime factors that determine the quality of EOs (Tongnuanchan and Benjakul, 2014); thus

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the use of new extraction techniques for natural substances, which typically use less solvent and energy, such as supercritical fluid extraction, ultrasound extraction, microwave assisted extraction, and sub-critical water extraction are being evaluated (Bousbia et al., 2009; Périno-Issartier et al., 2010). Among these emergent technologies, microwave assisted extraction (MAE) has showed many advantages such as convenience, less processing times, and high efficiency (Zhai et al., 2009; Flamini et al., 2007).

MAE uses microwave radiation as the source of heating for the solvent—sample mixture. Due to the particular effects of microwaves on matter (namely dipole rotation and ionic conductance), heating with microwaves is instantaneous and occurs in the interior of the sample, leading to rapid extractions (Camel, 2001). The main advantage of MAE resides in its heating mechanism (Périno-Issartier et al., 2010); acceleration of the extraction can be partly explained by the specific effect of microwaves on plant material (Camel, 2001).

Mathematical modeling of extraction processing must be considered as a fundamental step during operation of an efficient industrial process (Xavier et al., 2011; Reyes-Jurado et al., 2014). Mathematical models are used to simulate different process scenarios without the need to run a large number of experimental trials and identify the best conditions for the process (Cassel et al., 2009; Rezzoug and Louka, 2009). Different mathematical approaches have been reported that explain the process involved



during traditional methods of extraction. Some authors have described the extraction variables effects on essential oil yield through statistical analysis of experimental data (Ghasemi et al., 2011; Parikh and Desai, 2011; Zhang et al., 2012), being response surface methodology (RSM) successfully applied for optimizing extraction conditions. However, this type of analysis represents parameter values for the optimization process within the experimental values, thereby; it just can be used in the experimental range tested and just represents the evaluated variables effect on analyzed response (Almeida et al., 2008). For this reason, models that try to explain the physical mechanism of the extraction process based on mass transfer fundamentals have been developed (Sovová and Aleksovski, 2006; Cassel et al., 2009; Moreno et al., 2010; Xavier et al., 2011) with the aim of determining physical parameters that make possible extrapolations.

Sovová and Aleksovski (2006) developed a model for the hydrodistillation process; in their model two different kinds of particles were considered: spherical particles or slabs, with different distribution of the oil in each one. In their assumptions, the only mass transfer process that had to be taken into account was the oil diffusion from the particle core to the region of broken cells, ignoring the initial rate of distillation from intact cells. Similar to Sovová and Aleksovski, Xavier et al. (2011) proposed a model based on mass transfer fundamentals, their model considers a fluidized bed and is based on the concept of broken and intact cells, but it considers the presence of two extraction periods (each one associated with the velocity of EO extraction from cells) for the description of the extraction curve based on mass balance of the solute (EO).

Despite that MAE has proven to be a more effective extraction process than traditional methods, to our knowledge, available approaches to describe this process are primarily based in statistical analysis of process variables (Wang et al., 2007; Fang et al., 2010; Farhat et al., 2011; Song et al., 2011), while the study in terms of mass transfer is limited. Since MAE is based in a distillation process with a different source of heating, it should be expected that MAE can be properly modeled by describing the mass transfer mechanisms during distillation, taking into account the microwave effect on the diffusion coefficients. The aim of this work was to evaluate the effect of different MAE process parameters in the extraction yield of orange peel EO, to describe the process through statistical analysis based on a factorial design, and as well as description of this process with a mathematical model based on mass transfer fundamentals.

# 2. Materials and methods

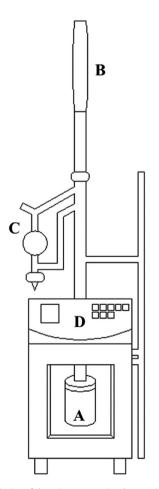
# 2.1. Plant material

The essential oil was extracted from orange (*C. sinensis* var. Valencia) peel without bagasse obtained from a local juice producer of San Andres Cholula, Puebla (Mexico) during April–August 2014. The peel was used at two moisture levels, high (50% of moisture) and low (10% of moisture), and using two different shapes of particles, plaques (1 cm) and spheres (40  $\mu$ m). To adjust orange peel moisture content, the peel was dried in a tray dehydrator (Excalibur, USA) for different times at 35 °C according to the final target moisture content, the final moisture was determined in a forced air oven at 105 °C for 24 h according to the official method reported by A.O.A.C (2000). Peels were ground in a domestic mixer to obtain the orange peel spheres and their particle size was determined with a particle analyzer (Bluewave, Microtrac, USA), while the peel plaques were obtained manually.

# 2.2. Microwave assisted extraction

MAE was carried out on a NEOS microwave extraction system (800 W, 60 Hz) (Milestone, Italy), which is designed to utilize different beakers for the extraction of different sample/solvent volumes. The MAE system is equipped with a TFT multicolor liquid crystal screen, a power sensor (power range 0-1000 W), an infrared temperature sensor, a temperature controller, and a magnetic stirrer in the base of the reactor. Fig. 1 displays a schematic description of the hydro-distillation system. During extraction, a sample of 250 g of orange peel and 700 mL of distilled water were placed into the beaker (A), which was introduced and fixed to the MAE system. The sample was then subjected to the extraction process for 50 min at two different irradiation powers (360 or 540 W). A condenser (B) was used to collect the extracted essential oil in a graduated trap (C). Irradiation power and extraction time were controlled from the equipment front panel (D). During the process, system temperature rises until the sample starts to boil and the extraction starts; since each sample was processed at different conditions of moisture content, particle size and microwave power, the time required to achieve the boiling temperature (90–94 °C) was also different depending on the characteristics of the sample, such delay time was registered as t<sub>CUT</sub>.

Extracted essential oil was dried with anhydrous sodium sulfate and stored at 4 °C in dark vials until used. The amount of essential oil was determined during extraction and gravimetrically after collection; the extraction yield was expressed as the percent ratio



**Fig. 1.** Schematic description of the microwave assisted extraction system. Beaker (A), condenser (B) graduated trap (C), and equipment front panel (D).

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