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Spatial and temporal temperature distributions in fixed beds undergoing supercritical fluid extraction

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ABSTRACT

Most of the studies dealing with extraction of compounds commonly consider homogeneous beds with constant temperature, although variations might exist. However, understanding that temperature can influence the fluid dynamics and solubility of compounds, this work proposed to evaluate temperature distribution in fixed beds during supercritical CO_2 extraction of compounds from turmeric and clove. Two beds of 1 L with different height to diameter ratios were tested. Kinetic curves were determined, extract composition was measured and temperature distribution was mapped. Heating the beds to the desired temperature was readily reached (< 5 min) when the beds were pressurized with pre-heated CO_2. During pressurization, a significant increase in temperature was observed. Gradients of temperature (until 20 °C) were formed during the extraction mainly in the axial direction. Furthermore, slight variations of ar-turmerone, eugenol, beta-caryophyllene, eugenyl acetate, and alpha-humulene are displayed as a function of spatial and temporal temperature variations in the beds.
Industrial relevance: Experimental data of temperature distribution in fixed beds undergoing supercritical fluid extraction in industrial scale can contribute to improving the productivity (yields) and the quality (composition) of extracts containing bioactive compounds. In food-related areas, bioactive compounds are searched for applications as antioxidant, anti-inflammatory, and antimicrobial agents.

1. Introduction

In the last few years, the interest of using CO_2 as a solvent on its supercritical condition for obtaining several products in industrial scale has strongly emerged. Such interest is mostly based on the outcomes taken on the scientific database since the ending of 90s and beginning of this century. Several advantages of supercritical fluid extraction (SFE) process using CO_2 have been listed (Abaide et al., 2017; Machado, Pereira, Nunes, Padilha, & Umsza-Guez, 2013), as the most important ones the high quality of products and the high efficiency of this environmentally friendly technology (Chemat et al., 2017; Confortin et al., 2017). The density of supercritical fluids can be closer to the density of liquids, thus conferring high solvation power to the former, that is, high capacity of dissolving groups of compounds to reach large extraction yields (Rosa et al., 2009). Supercritical fluids also exhibit high diffusion coefficient and low viscosity (Smith, Inomata, & Peters, 2013). These characteristics favor the solvent penetration in the solid particles and provide processes with high mass transfer rates.

In this sense, the success of establishing the supercritical technology in industrial scale depends on the reproduction of results in laboratory scale. During the scale-up some drawbacks can raise, as variations in bed porosity, deficiency in solvent dispersion, axial dispersion and gradients of temperature, among others. These factors can affect the equilibrium and transport phenomena in the processes (Pronyk & Mazza, 2009). On this subject, the scale should be taken into account, as well as the geometry of beds regarding the height (H_B) to internal diameter (D_B) ratios. In short vessels (short H_B/D_B ratios) axial dispersion might be significant and radial effects can cause heterogeneous distributions of temperature and mass (Brunner, 1994; del Valle et al., 2004). Consequently, different H_B/D_B ratios can influence the gradients of temperature, mainly at higher temperatures. Therefore, variations in the saturation conditions of the system and viscosity of solvent might occur.

The temperature is a fundamental parameter for developing SFE of target compounds. This parameter influences the phase equilibrium between solvent and solutes, as the density of the supercritical fluid and vapor pressure of solute. Furthermore, temperature influences the transport properties, as the viscosity of solvent and diffusivity of solutes

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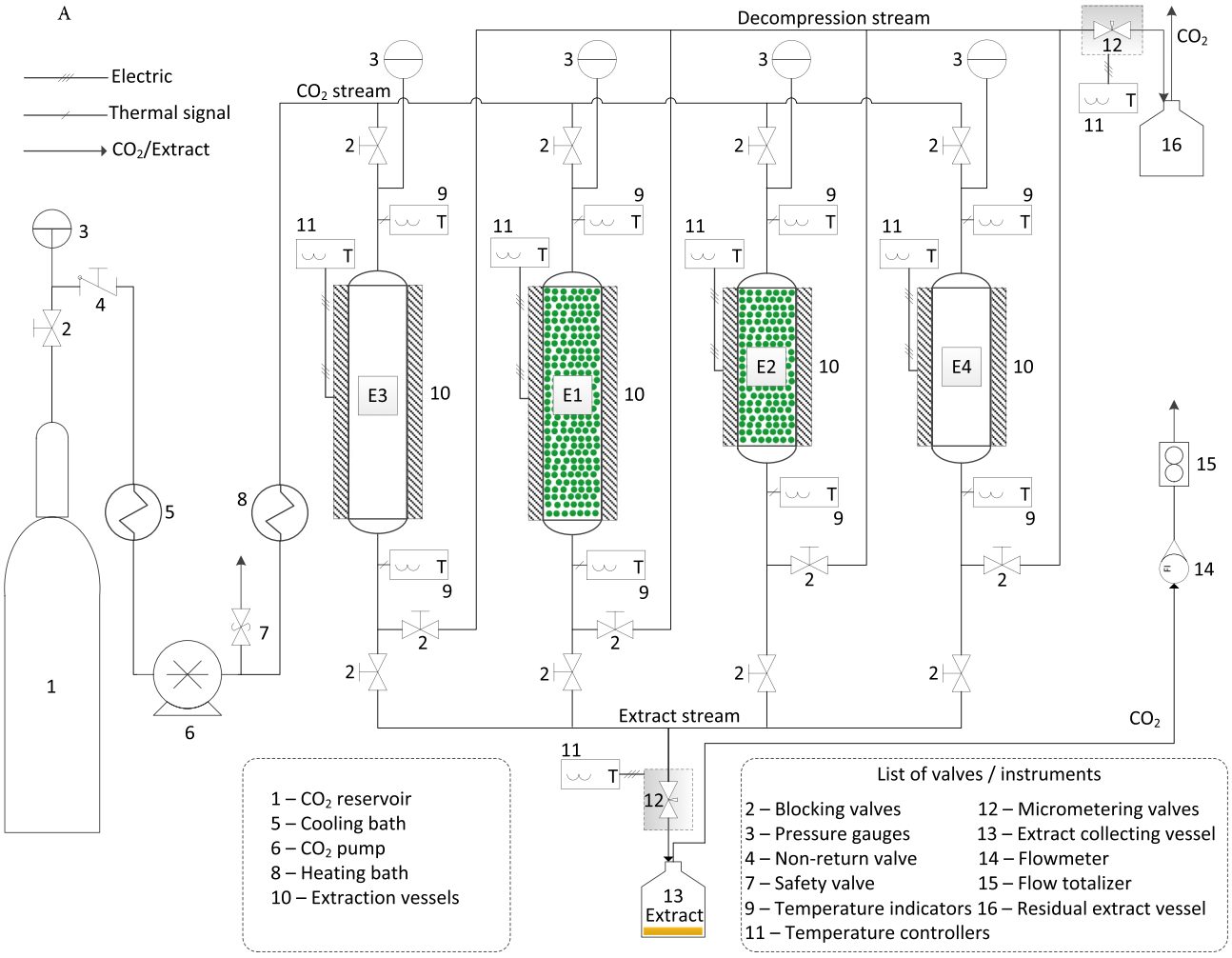

Fig. 1. (A) Schematic drawing of SFE-4 × 1L equipment and (B) axial and radial positions of the thermocouples (T1 and T2) used for the temperature study; h: axial vector; H: height of the vessels; r: radius vector; R: internal radius of the vessel; D: internal diameter.

in the fluid phase (Brunner, 1994). Comprising most of the studies reporting the use of SFE, either about experimental optimization of parameters or about kinetic modeling, temperature is generally treated as a homogeneous constant during the whole process (Barry, Dinan, & Kelly, 2017; Carvalho, Osorio-Tobón, Rostagno, Petenate, & Meireles, 2015; Sallet et al., 2017; Santana et al., 2017; Valente et al., 2018). However, this approach is not as real as the theory suggests. Controlling temperature during SFE process is a hard and systematic work, because temperature depends on the pre-heating of solvent, the volume of bed, the geometry of the vessel, the mode/efficiency that the bed is heated, and the pressurization rates.

Therefore, gradients of temperature in the bed can exist, which are more remarkable when the scale increases. According to some reports in the scientific literature, gradients of temperature can affect the fluid dynamics of solvent + solute mixture and its phase equilibrium (Brunner, 1994; del Valle et al., 2004; Zabot & Meireles, 2016). This occurrence might influence global yields of extractions and composition of extracts. Learning about temperature distribution through extraction beds during SFE process can contribute to validate further studies performed in different beds and scales. Up to now, the systematic determination of temperature distribution in this field has been rarely reported. A lack of relevant studies demands that this subject requires further investigation. In an effort to generate more experimental information, the objective of this work was to fulfill a descriptive and exploratory study on temperature distribution in two different beds during the SFE-CO_2 of bioactive compounds from turmeric rhizomes and clove buds. The relationships among gradients of temperature, extraction yields and kinetic composition of extracts were also assessed and provided.

2. Materials and methods

2.1. Raw materials

Turmeric rhizomes were acquired from "*Oficina de Ervas Farmácia de Manipulação*" (lot 065DM, Ribeirão Preto, Brazil). Clove buds were acquired from a local Market (Campinas, Brazil). Both raw materials were stored at −18 °C in a domestic freezer (Metalfrio, model DA420, São Paulo, Brazil). Before the assays, the raw materials were comminuted in a knife mill (Marconi, model MA340, Piracicaba, Brazil) and the samples were packed in air impermeable bags and stored again at −18 °C until performing the extractions and evaluations of temperature inside the beds. The particle size distribution was determined using a vibratory system (Bertel, model 1868, Caieiras, Brazil) with sieves of mesh sizes 8–80 (WS Tyler series, Wheeling, USA). The mean particle diameter (d_p) was determined according to the ASAE standards (ASAE, 1998).

The moisture (U) content in the raw materials was determined using the xylene distillation method (Jacobs, 1973). The true density of the particles (ρ_r) was measured by pycnometry (Quantachrome