



Optimization of supercritical fluid extraction: Polydisperse packed beds and variable flow rates

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ABSTRACT

This theoretical study examines variable-in-time flow rates $v(t)$ and different ways of packing polydisperse ensemble of ground particles as controls in supercritical fluid extraction (SFE) to maximize extraction yield. The so-called packing function χ is introduced to describe the local particle-size distribution in the pack along the extraction vessel. The research is based on the modified shrinking-core (SC) model for the mass transfer inside particles and assumes the pseudo-steady solvent flow in the vessel. It is rigorously proven that for any variable flow rate $v(t)$ and overall particle-size distribution the corresponding locally-monodisperse stratified (LMS) packing χ_0 maximizes the current amount of extracted solute, while the appropriate filtration policy extends the domain of efficient particle-size distributions. Sufficient conditions that guarantee a certain extraction degree at a fixed time are deduced and formulated in terms of F -distribution. Being of obvious practical significance for finely ground substrates optimization is shown to be rather limited for relatively big particles (commonly used in laboratory experiments), and only longer extraction times, higher oil solubility and diffusion rates allow noticeable increase in the extraction yield in this case.

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1. Introduction

Industrial technologies which use supercritical fluids become more and more popular in production of new materials, natural products, pharmaceuticals and others. One of the most attractive applications of supercritical fluids is extraction of natural products from packed beds of ground plants or other granular material – the supercritical fluid extraction (SFE) [1]. This technique gradually substitutes for traditional extraction methods all over the world, being environmentally friendly and providing selective extraction. Supercritical CO₂ is conventionally used in SFE as a solvent because it is non-toxic, non-flammable and readily available. It also has low critical temperature (~31 °C) which is crucial for extraction of thermally labile compounds (such as essential oils).

As a consequence, significant attention is drawn to understanding and optimization of the SFE process. SFE practice is usually focused on experimental tuning of extraction conditions such as solvent flow rates, temperature, and pressure in the framework of “experimental design approach” or “response surface method” to achieve the highest mass of extract at a fixed extraction time [2].

However, these research lines require a large number of experiments and do not consider more sophisticated controls which account for temporal variation of solvent flow rates and spatial inhomogeneity of particle-size distribution in a polydisperse packed bed for given thermodynamic conditions. Practical significance of such controlling factors together with other technological parameters (grinding rate and extractor dimensions) could be theoretically evaluated on the basis of an appropriate SFE model [3].

Various mass-transfer models [4–7] have been presented and employed for SFE predictions and data interpretations [4–10]. Among them, the two, shrinking core (SC) [5] and broken-and-intact cell (BIC) [4], models cover a wide range of possible approaches and have shown [4,8,9,12,13] to match the overall extraction curves (OECs) with high accuracy. Based on the general description of oil diffusion in ground material, one could deduce that, in principle, the BIC model is valid in the limit of relatively small particles, when the inner part of a particle can be described in terms of spatially averaged characteristics. On the other hand, the SC model becomes more preferable in the case of relatively large particles with high oil content, when the diffusive front thickness in particles is much less than their typical size [5].

Herein we employ the modified SC modeling approach discussed [12–16] previously in detail and verified (validated) [12,13] on various available data sets.

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