SUPERCRITICAL EXTRACTION OF SOLID MATRICES. MODEL FORMULATION AND EXPERIMENTS

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***Abstract*— Nowadays, supercritical extraction of solid matrices is used frequently to obtain valuable substances especially by food and pharmaceutical industries. Most often, solid particles are arranged in a fixed bed configuration and the supercritical fluid flows through the bed extracting the desired solute. In this study, a mathematical model is set to simulate this process. The influence on the extraction yield of processing variables such as temperature, pressure and flow velocity as well as some process parameters like particle size, void fraction and bed height are predicted by the model. This model was used to simulate the extraction of essential oil from clove buds with supercritical CO_2. Experiments were carried out at different pressures (90-120 bar) at a fixed temperature and CO_2 flow. A good agreement between experimental and theoretical results was obtained.**

***Keywords*— Supercritical Extraction, Mathematical Modeling, Solid Matrix.**

I. INTRODUCTION

Supercritical CO_2 extraction of vegetable oils and essential oils from plant materials is an alternative process to solvent extraction and steam distillation. Supercritical fluids have advantages over conventional solvents, such as: a)low viscosity and large diffusivity ensuing excellent mass transfer characteristics, b)variable dissolving power, b)very low surface tension allowing it to penetrate easily into the porous structure of a solid matrix to extract the solute and c)low latent heat of evaporation and high volatility favoring its removal from extracted products by simple expansion.

Process temperature and pressure affect significantly the dissolving power of supercritical fluids. At lower pressure (near the critical point) volatile components, such as essential oils are selectively extracted, while other components present in the vegetable matter such as waxes, resins and dyes have low solubility in these conditions. However, coextraction of waxes is common since they are located on the surface and they are extracted with a very limited mass transfer resistance (Reverchon, 1997).

Supercritical extraction has been applied to a large number of solid matrices to obtain valuable substances, mostly in food and pharmaceutical industries (King & Bott, 1993; Palmer & Ting, 1995). Most often, raw materials prepared to enhance solvent extraction are arranged in a fixed bed configuration and the supercritical fluid flows continuously through the bed extracting the desired solute. After the extractor, one or more separation stages are used to precipitate the solute from the supercritical solution.

The analysis of products and the assessment of their composition as a function of the extraction pressure and temperature have been reported for several vegetable matter (Stahl *et al*., 1988). However, some other process parameters like particle size, milling conditions, fluid velocity and extraction time are not conveniently incorporated into these studies. Such parameters influence the extraction process to an extent that they can not be neglected in the analysis

Reliable mathematical models for supercritical extraction process can be developed that include all these parameters. Mathematical models can be used to generalize the experimental results to new process conditions and extracted materials. Even more, they are useful to develop scale-up procedures from laboratory to pilot and to industrial scale. Different approaches have been proposed to obtain mathematical models that simulate supercritical fluid extraction from solid particles (Brunner, 1994; Reverchon, 1997; Goto *et al*., 1998). In this work, differential mass balances are used to elaborate a model that takes into account the following mechanisms: *i*)the dissolution of solid or liquid component into supercritical fluid filling the particle pores, *ii*)diffusion through porous cellular material and *iii*)external mass transfer around the solid particle to the main fluid stream. The resulting equations are solved using numerical techniques. Results obtained during the extraction of essential oil from clove buds with supercritical CO_2 are used to check the model.

II. METHODS

A. Model development

In order to obtain the mathematical model, the following simplifying assumptions were made: a) The extrac-