Review

Design and scale-up of pressurized fluid extractors for food and bioproducts

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ABSTRACT

This article provides an in-depth review of the literature on the design, scale-up, and effects of scale on the extraction of food and bioproducts in pressurized fluid extractors. The design of pressurized fluid extraction systems such as supercritical CO_2, pressurized solvent, and pressurized low polarity water (subcritical water) are similar. Knowledge of phase equilibria, mass transfer rate, and solubility data are important first steps for the scale-up of extraction processes and equipment. The literature for the design, scale-up, and effects of scale on the extraction of bioproducts in pressurized fluid extractors is examined with particular attention to the mass transfer principal and important parameters for extraction as they relate to the design and scale-up of fixed bed pressurized fluid extractors. Often when two scales of an extractor are examined, the scale-up has not been done uniformly, leaving the effects of the scale-up on extraction in doubt. There has been some success in design of a continuous pressurized fluid extractor by utilizing a battery of vessels in series to operate on a quasi-continuous basis, and with the use of screw conveyors to produce a gas-tight plug of material, which allows the extraction to operate at the necessary elevated pressures and temperatures.

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Nomenclature

a_p	specific surface area of a solid substrate particle (m^{-1})	k_f	mass transfer coefficient in the fluid phase (m/s)
C_f	concentration of solute in the solvent (kg/m^3)	L	bed depth (m)
C_p	concentration of dissolve solute in the solvent contained in the particle pores (kg/m^3)	r	radial position within the particle (m)
		R	particle radius (m)
C_s	concentration of the solute in the particle (kg/m^3)	t	extraction time (s)
C_{po}	initial concentration of dissolved solute within particle pores (kg/m^3)	z	axial position along the bed (m)
		u	interstitial velocity (m/s)
C_{ps}	concentration of solute on external surface of the particle (kg/m^3)	ε	porosity of the bed (–)
		ε_p	porosity of the solid particle (–)
C_{so}	initial concentration of solute in the particle or adsorbed onto the particle surface (kg/m^3)	τ	residence time (s)
		u	U/ε
d_E	diameter of the extraction vessel (m)	U	F/A_E
D_e	effective diffusion coefficient of the solid particle (m^2/s)	A_E	cross-sectional area of the extraction vessel (m^2)
D_L	axial dispersion coefficient (m^2/s)	U	superficial velocity (m/s)
d_p	particle diameter (m)	F	volumetric flow rate of the solvent (m^3/s)

1. Introduction

Given enough time and money almost any process or equipment can be made to work no matter how poor the design (Ritcey, 2004). The design of industrial-scale equipment is usually preceded by laboratory (bench) and pilot-scale systems. Sometimes the pilot-scale system is skipped, and work goes straight from the laboratory to industrial production. It is thought that time and money from the pilot-scale work can be applied to making the industrial plant work. This is most useful when there is sufficient data and the process is similar to one already in existence (Ritcey, 2004). However, with quality data and determination of scale-up factors, the design of pilot or industrial sized equipment is much more efficient. There is a large body of work in the literature dealing with the leaching (dissolution and removal of soluble components from a solid matrix)/extraction of bioproducts from solid organic materials with liquid solvents. Most of the work has been done in the laboratory with bench-scale equipment. There are general texts on the design of pressurized vessels or extraction systems (Bertucco and Vetter, 2001; King and Bott, 1993; Tzia and Liadakis, 2003), but there is little discussion on the aspects of scaling up a process or equipment. Such texts generally do not include any discussions on the performance of the equipment or on the appropriate scaling of the extraction process. In this paper the design, scale-up, and effects of scale on the extraction of bioproducts in pressurized fluid extractors will be detailed by examining results available from the literature and patents. The mass transfer principles and important parameters for extraction will be discussed as they relate to the design and scale-up of fixed bed pressurized fluid extractors.

2. Pressurized fluid extraction

2.1. Types of pressurized fluid extraction systems

Solid–liquid extraction is a separation process involving the transfer of solutes from a solid matrix to a solvent. Solvents are chosen based on solubility characteristics of the desired solute. Ideally to achieve as pure a substance as possible, the solute should have high solubility in the solvent while other components in the solid matrix should not. Economics and safety are always a consideration and indeed, safer and less harmful solvents that are easy to remove, or recover, are gaining in popularity. There has been much interest in the field of pressurized fluids, as seen with the growing work with supercritical fluid extraction (SFE) with CO_2 (Díaz-Reinoso et al., 2006; Herrero et al., 2006; Reverchon and De Marco,

2006; Shi et al., 2007) and the use of high pressure solvents including pressurized low polarity water (PLPW) (Kim and Mazza, 2009; Cacace and Mazza, 2007, 2006; Carabias-Martínez et al., 2005; Mazza and Cacace, 2005; Kaufmann and Christen, 2002; Smith, 2002). The versatility of pressurized solvents is excellent due to the physicochemical properties of the solvent, including density, diffusivity, viscosity, and dielectric constant, which can be controlled by varying the pressure and temperature of the extraction system. In this way the solvating power and selectivity of the solvent can be effectively controlled.

Three methods of pressurized fluid extraction have gained popularity in the research community as a means of extracting bioproducts. They are: supercritical fluid extraction (SFE); pressurized solvent extraction (PSE, also known as pressurized liquid extraction (PLE), subcritical solvent extraction (SSE), or accelerated solvent extraction (ASE, Dionex trade name); and pressurized low polarity water (PLPW) extraction (also known as superheated water, subcritical water, pressurized hot water). All of these methods make use of vastly different pressurized solvents, each with a unique set of properties that influence the extraction of the desired compounds. Supercritical fluid extraction utilizes a solvent, usually CO_2, although other substances such as water or ethylene can be used, near its thermodynamic critical point, where it possesses the density of a fluid with the viscosity and diffusivity of a gas. By controlling the pressure of the supercritical fluid, the solubility within the supercritical fluid can be changed. This permits a high degree of selectivity, which allows for the fractionation of the extract by changing the pressure in the system's separators. Pressurized solvent extraction utilizes solvents such as hexane, methanol, or ethanol at temperatures above their boiling point, under high pressures, in order to increase the efficiency of the extraction process with respect to extraction time, solvent consumption, and extraction yields. This is accomplished through improved solubility and mass transfer effects, in addition to a disruption of surface equilibria, such as reduced viscosity and increased solvent penetration into the sample matrix (Richter et al., 1996). PLPW extraction is a specific case of PSE, where water is utilized as the solvent. Water is a polar solvent, but if heated from 25 °C to 200 °C under pressure to maintain a liquid state, its dielectric constant decreases from 79 to 35, reaching values similar to solvents such as ethanol (24) or methanol (33) (Cacace and Mazza, 2006).

2.2. Basic design of pressurized fluid extractors

The solvents used for all three pressurized fluid systems are vastly different, yet the basic design of the extraction equipment