CHAPTER 1 INTRODUCTION

1.1 Introduction

SFE technology has had an impressive growth until now. The unique properties of SFE were observed more than a century ago, but only in the last three decades has SFE evolved as a novel separation technique. It has the potential to be an environmentally friendly green processing technique, and in some cases, replacing the traditional organic solvent based extraction techniques with more benign solvents like supercritical carbon dioxide ($SCCO_2$), ethanol and water. In many countries, stricter regulations regarding the use of organic solvents such as hexane to address safety, health and environmental issues are forcing the industry to search for alternative processes (Pasquali and Bettini, 2008). As mentioned above, SFE is usually used as an alternative method for new or improved applications in the production of a variety of products including natural foods and flavours, pharmaceuticals, nutraceuticals, polymers, chemicals, and equipment cleaning (Herrero et al., 2010).

SFE is an extraction process using a supercritical fluid as a solvent. Principle of SFE is that the feed materials are arranged in a fixed bed. The supercritical fluid flows continuously through the bed and contacts with the feed materials then the volatile substances will partition into the supercritical phase. After the dissolution of soluble material in supercritical fluid (SCF), the dissolved substance is removed from the feed material. After extraction, the extracted component is completely separated from the SCF by means of a temperature and/or pressure change in one or more separation stages. The SCF may be then recompressed to the extraction conditions and recycled (Martinez, 2008).

When a fluid is taken above a particular temperature and pressure (critical point of the respective fluid), it exists in a condition called the SCF state. The physiochemical properties of a fluid in the supercritical state are in between those of a typical gas and liquid. For example, the density of a SCF can be changed by varying the pressure on the fluid. As a result, a SCF can have a density that ranges between those exhibited by gases to liquid-like values when the fluid is compressed at high pressures (Hauthal, 2001). CO_2 is the most widely used fluid in SFE. SFE with CO_2 delivers the most naturalsmelling and -tasting extracts because there are no volatiles removed in a residual solvent removal post-processing step. Additionally, because CO_2 processing requires low temperatures, there is less deterioration of heat-sensitive components in the extract. Furthermore, since there is no oxygen in the process, the potential for oxidation of the extract is significantly minimized (Brunner, 2005).

From an engineering point of view, the SFE units require both the knowledge of the relevant process parameters such as phase equilibrium (or solubility) and mass transfer kinetics, and also the optimum operating conditions. These parameters may be obtained by using an accurate mathematical description of the extraction process and experimental data. Optimization of the operating conditions is a critical step in development of successful SFE process concerning the effects of various parameters on the extraction yield. Generally, extraction pressure, temperature, particle size, solvent flow rate, and cosolvent type and concentration are considered as the important operative parameters. Besides the limitations concerned with mass transfer, the maximum theoretical separation that can be produced in a continuous extraction column is governed by thermodynamic phase equilibria (Ajchariyapagorn et al., 2008).

SFE has many advantages due to the strong dependence on a solute's solubility has on the operating temperature, pressure, and cosolvent type and concentration. This strong dependence means that the operating conditions for the extraction and separation process influence SFE recovery rates, selectivity and ultimately overall extraction costs. Therefore, the ability to predict the solubility of solute in supercritical fluids is important in understanding supercritical fluid extraction (SFE). In SFE, a solute must dissolve in the supercritical solvent before it can be extracted. Thermodynamic methods using a phase equilibrium approach have been used in the past to predict solubility in supercritical fluids (Gordillo et al., 2005). When the solubility can be well predicted, the modelling of supercritical fluid extraction could be developed more precisely due to the main mechanism can be described (Ajchariyapagorn et al., 2009).

1.2 Research Objectives

The objectives of this research work are to develop a methodology to mathematically characterize the extraction yields obtained from the supercritical fluid extraction of biomaterials in packed bed systems. Shrinking core model is selected as the best mathematical model, which characterises the extraction process, after taking into consideration of mass transfer mechanisms such as diffusion and solubility. A methodology of predicting solubility of biomolecules in supercritical fluids is also developed in this work.

1.3 Outline of the Research

The organization of the chapters of the thesis is as follows:

Chapter 1 Introduction: Present brief introduction of supercritical fluid extraction (SFE) process. The research problem in relation to relevant literature and conceptual framework of this research in supercritical fluid extraction model are also elaborated.

Chapter 2 Background and Literature Review: Presents the background issues related to the supercritical fluid extraction (SFE) process and the development for the modelling and simulation of SFE from previous studies. This chapter also summarises the major (dependent and independent) variables in this research.

Chapter 3 Model Developement: Investigates the concepts and the important theories which are used to develop a methodology of solubility estimation and yield prediction in the SFE systems. This chapter also includes extraction parameters controlling the mass transfer process.

Chapter 4 Modelling and Simulation: Presents numerical results of applying our developed models to the experimental data obtained from the literatures. The deviation on the results due to the assumptions and identified variables related to the model are also discussed. This chapter also includes a discussion of the extraction mechanism and the parameters which control the mass transfer process. Sensitivity analysis is performed to investigate the parametric effects on the model. Possible extractor configuration is also investigated in order to obtain the highest yield.

Chapter 5 Conclusions and Recommendations: Provides a summary of the overall findings of the research and suggestions for future work opportunities.

1.4 Conceptual Framework

The conceptual framework (as shown in Figure 1.1) describes the scope of our research. Many entities and relationships of developed supercritical fluid extraction model are contained and many of which overlap in their semantics and behaviour. The conceptual framework of supercritical fluid extraction model is divided into 4 main topics: solubility model validation, transport property estimation, SFE model validation, and extractor configurations. The variables and process parameters related to the model are identifies.

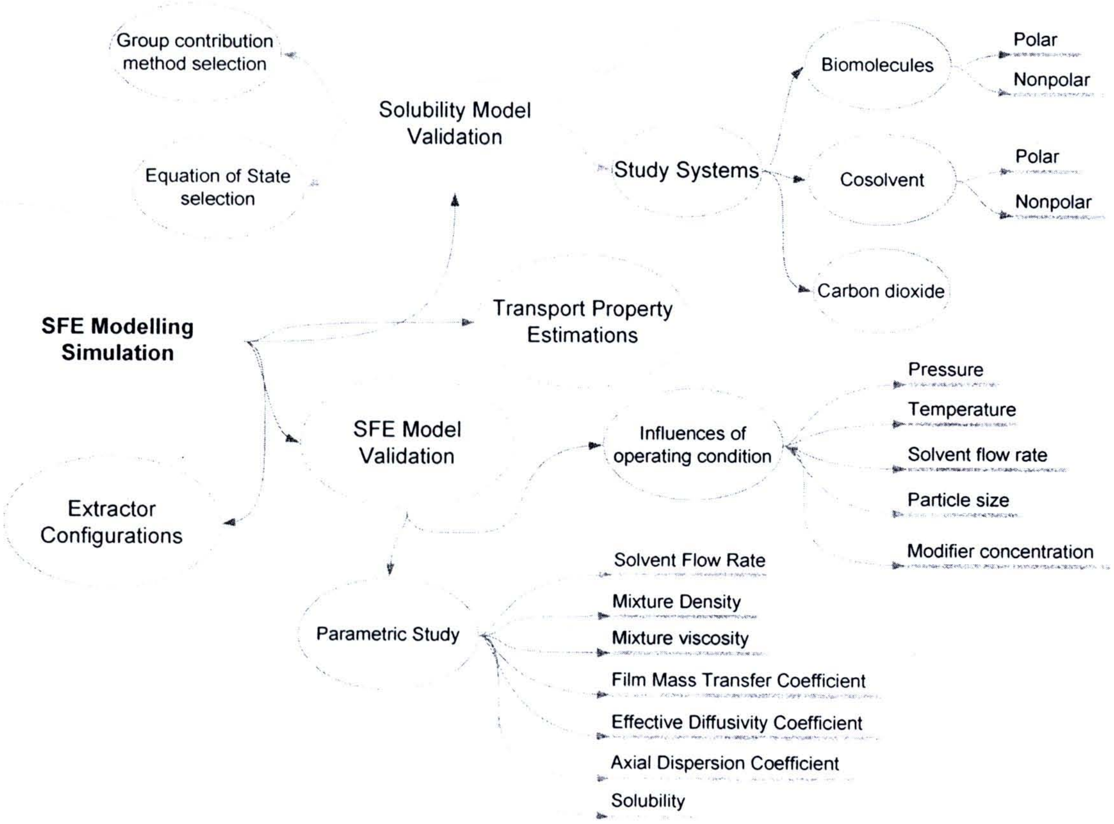

Figure 1.1 Conceptual framework of supercritical fluid extraction model.