SOLUBILITY OF *SWIETENIA MAHAGONI* SEED IN SUPERCRITICAL CARBON DIOXIDE EXTRACTION

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To my beloved wife and family

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ABSTRACT

Swietenia mahagoni (*S. mahogany*) is a group of plant that is known to have an insecticidal activity as well as medicinal uses such as hypertension, diabetes and malaria. The main purpose of this research was to study the effect of temperature, pressure, and solvent flow rate on extraction yield of *S. mahagoni* as well as to establish the solubility data for *S. mahagoni*. In this study, supercritical carbon dioxide (SC-CO_2) extraction was applied for extraction of *S. mahagoni* oil from seeds at different temperatures (40 °C to 60 °C) and pressures (15 MPa to 35 MPa). The effects of temperature and pressure on the extraction yield and solubility of oil were determined. The current results showed that an increase of temperature and pressure increased the extraction of oil. In addition, the morphological of *S. mahagoni* structure before and after the extraction process was observed by scanning electron microscopy (SEM). For solubility study of *S. mahagoni*, a dynamic method of extraction was applied and a density based-model was used in comparison with experimental solubility data. The experimental solubility data for *S. mahagoni* was correlated with the Chrastil model with the lowest average absolute percent deviation (AAPD) value of 0.3315 with the values of parameter *k*, *a* and *b* as 4.7040, -6368.4400 and -10.9353, respectively. The optimization process was conducted using a commercial response surface methodology software. Analysis of variance (ANOVA) showed that the value of R^2 was 0.9391 implies that 93.91% of the total variance is explained by the model, indicating a good correlation and agreement between the experimental and the predicted values.

ABSTRAK

Swietenia mahagoni (*S. mahogany*) merupakan sekumpulan tumbuhan yang dikenali mempunyai aktiviti pembunuh serangga serta kegunaan untuk perubatan seperti tekanan darah tinggi, kencing manis dan demam malaria. Tujuan utama kajian ini adalah untuk mengkaji kesan suhu, tekanan dan kadar aliran pelarut terhadap hasil pengekstrakan *S. mahagoni* serta menerbitkan data keterlarutan untuk *S. mahagoni*. Bendalir genting lampau karbon dioksida (SC-CO_2) telah digunakan untuk mengekstrak minyak *S. mahagoni* dari benihnya pada suhu antara 40 °C hingga 60 °C dan tekanan antara 15 MPa hingga 35 MPa untuk menentukan kesan suhu dan tekanan ke atas hasil ekstrak dan keterlarutan minyak. Keputusan eksperimen menunjukkan bahawa peningkatan suhu dan tekanan akan meningkatkan hasil pengekstrakan minyak. Keadaan struktur *S. mahagoni* sebelum dan selepas proses pengekstrakan dikaji dengan menggunakan mikroskop elektron imbasan (SEM). Bagi kajian keterlarutan *S. mahagoni*, kaedah pengekstrakan dinamik digunakan, dan model berasaskan ketumpatan telah digunakan sebagai perbandingan dengan data keterlarutan daripada eksperimen. Data keterlarutan bagi *S. mahagoni* daripada eksperimen berjaya dikaitkan dengan model Chrastil berdasarkan nilai purata mutlak peratus sisihan (AAPD) yang rendah iaitu 0.3315 dengan nilai-nilai parameter *k, a* dan *b* yang masing-masing adalah 4.7040, -6368.4400 dan -10.9353. Proses pengoptimuman telah dijalankan dengan menggunakan perisian kaedah permukaan sambutan. Analisis varians (ANOVA) menunjukkan bahawa nilai pekali penentuan, R^2 iaitu 0.9391 membuktikan bahawa 93.91% daripada jumlah varians yang diterangkan oleh model, menunjukkan hubungkait yang baik antara nilai sebenar daripada eksperimen dan nilai-nilai ramalan.

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LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
BBD	-	Box-Behnken Design
CO_2	-	Carbon Dioxide
COM	-	Cost of Manufacturing
GCMS	-	Gas Chromatography Mass Spectroscopy
GRAS	-	Generally Recognized as Safe
M-T	-	Mendez-Santiago and Teja
MOX	-	Malaysian Oxygen
PAF	-	Plattelet-Activating Factor
PT	-	Pressure-Temperature
RSM	-	Response Surface Methodology
SC-CO_2	-	Supercritical Carbon Dioxide
SCF	-	Supercritical Fluid
SEM	-	Scanning Electron Microscope
SFE	-	Supercritical Fluid Extraction

LIST OF SYMBOLS

a, *b*, *c*	-	Constants in the Density Based Model
k	-	Associated Number
M_A	-	Molecular weight of the solute
M_B	-	Molecular weight of the gas solvent
MPa	-	Mega Pascal
K	-	Kelvin
P	-	Pressure
R	-	Gas constant
S	-	Solubility of the compound
T	-	Temperature
Y*	-	Solute solubility in solvent
°C	-	Degree celcius
ρ	-	Density

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Swietenia mahagoni (Mahogany) or the other name is West Indies Mahogany is known as a large tropical tree, belonging to Meliaceae family. Newton *et al*. (1993) reported that Meliaceae family includes some of the most valuable tropical timber including Mahogany (*Swietenia mahagoni),* African mahagony (*Khaya* sp.) and Spanish Cedar (*Cedrela odorata L.*).

S. mahagoni is a large tropical tree, have 40-60 ft of its height, a range of diameter at base height (dbh) between 30 to 105 cm and density of wood with 560-720 kg/m^3 (Hayssam *et al.*, 2011). In tropical region, Malaysia, India and Southern of China is the mainly zones where this species is cultivated. *S. mahagoni (L.) Jacq* seed extract is high containing of lipids, which are basically, were neutral lipids, glycolipids and phospholipids, and the most compound from class of phospholipids is phosphatidycholine. In the other hand, *S. mahagoni* seeds oil also rich in fatty acid composition, namely arachidic acid, palmitic acid, oleic acid, stearic acid and myristic acid (Rahman *et al.*, 2010).

From previous literature, it is reported that *S. mahagoni* have important role for the healing of coughs, chest pain, amoebiasis, cancer and intestinal parasitism (Alrdahe *et al.*, 2010). Fatty acids and tetranortriterpenoids, one of the biologically active ingredients in *S. mahagoni* are considered to be responsible for these therapeutic effects (Sahgal *et al.*, 2009). Besides, it also can be used as a medicine for the hypertension, malaria and diabetes treatment.

Currently, the use of supercritical fluid extraction (SFE) technique is gaining popularity as a replacement of conventional method. El-Aty *et al*. (2008) reported that supercritical fluid extraction is a modern, safe and environmentally friendly if compared to other extraction techniques due to its potential to reduce or remove flammable and hazardous organic solvents. The use of supercritical fluids as a replacement for traditional solvents has been explored in a wide range of fields over the past two decades, including extraction of natural products, fractionation/separation processes, particle design and as reaction media (Perrut, 2000). Previous literature reported that the applications of SFE have focused more on edible oil extraction as examples, sunflower seed (Salgin *et al*., 2006), olive oil (Fornari *et al.*, 2008) and palm kernel oil (Hassan *et al.*, 2000).

In particular, supercritical carbon dioxide (SC-CO_2) has received a great deal of attention due to its many favorable properties, which are low in toxicity, critical temperature and pressure, low cost and inert nature. These properties make SC-CO_2 as an environmentally friendly or attractive green solvent (Wai, Gopalan, & Jacobs, 2003). Carbon dioxide (CO_2) is a linear molecule with no net dipole moment, meaning that it is a poor solvent for polar and ionic species (Raveendran & Wallen, 2003). For these types of species, CO_2 can be used in conjunction with a polar modifier or co-solvent to increase solubility of the solvent.

1.2 Problem Statement

Recently, the application of supercritical fluid extraction of natural plants is extensively research. However the high capital, high operating investment and higher pressure needed in the process are the main problems for the commercialization of this technology. The parameters used for the extraction in this study are temperature and pressure as these two variables play an important role to the overall extraction process performance. Particle size of the seed also affected the total yield of the extraction.

For solubility study, there are many model proposed by many researcher in the past. The example of modeling for solubility behaviour using supercritical fluid extraction includes Chrastil model (Chrastil, 1982), del Valle and Aguilera model (del Valle and Aguilera, 1988), Gordillo model (Gordillo *et al*., 1999), Sovova model (Sovova, 1994), Mendez-Santiago and Teja model (Mendez-Santiago and Teja, 1999), A-L model (Adachi and Lu, 1983) and Yu model (Yu *et al*., 1994). In this research, the modeling is focused on Chrastil model and del Valle and Aguilera model.

Currently, there has been no reported study on solubility data of *S. mahagoni* which is important due to the characteristics which indicates the equilibrium solubility of solutes in supercritical fluids corresponds to the limit of the total amount of solutes that can be extracted at saturation equilibrium. Therefore the experimental solubility of solutes in supercritical fluids has been extensively reported in the literature (Marceneiro *et al.,* 2011). This research is conducted due to the lack of solubility data in previous literature about the supercritical carbon dioxide extraction. This data is very important for future references and also for scale-up production of natural plants. This solubility data can be used to describe the properties of any substance, indicates the polarity of the substances and also very useful in the separating process of any mixtures.

1.3 Research Objectives

This research is conducted according to the aims as follows:

i. To investigate the effects of SC-CO_2 parameters namely pressure, temperature and solvent flow rate on oil yield and solubility of *Swietenia mahagoni* (Mahogany) seed extract.

ii. To establish the solubility data of *Swietenia mahagoni* (Mahogany) seed extract in SC-CO_2 extraction using solvent density based models which are Chrastil and del Valle and Aguilera model.

1.4 Scope of Research

The extraction of *S. mahagoni* seeds was done at selected range of conditions using conventional method (soxhlet extraction) and supercritical carbon dioxide (SC-CO_2) extraction. In order to achieve the objectives stated in section 1.3, the scopes of study are as follows:

i. Comparison between extractions of *S. mahagoni* seed by soxhlet extraction method using different solvents (hexane, ethanol and water) and supercritical fluid extraction (SFE) at different extraction conditions (pressure, temperature, and solvent flow rate).

ii. Determination of the extraction yield and solubility of *S. Mahagoni* seeds at various conditions, namely temperature, pressure and flow rate by experiments.

iii. Correlation of solubility data using density based model which are Chrastil (1982) model and del Valle and Aguilera (1988) model.

iv. Optimization of oil yield using research surface methodology (RSM).

1.5 Thesis Summary

This thesis is divided into five main chapters. Chapter 1 is the introduction of the research that includes background of the research, problem statement, objectives and scope of the research. Chapter 2 includes the overview of *S. mahagoni* (Mahogany), extraction process involved, mathematical modeling and process optimization. Chapter 3 discussed the overall methods used in the research including the procedures of chemicals and raw material preparation, extraction process involved (soxhlet and supercritical carbon dioxide extraction), solubility measurement and modeling, and process optimization. Chapter 4 discussed the results and discussion for overall experiments while the conclusion and some recommendations were discussed in Chapter 5.

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