

UNIVERSITI PUTRA MALAYSIA

OPTIMIZATION, KINETIC MODELING, AND CHARACTERIZATION OF HYDRO-DISTILLATION AND SUBCRITICAL WATER EXTRACTION OF AQUILARIA MALACCENSIS LAMK LEAVES AND WOOD ESSENTIAL OIL

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By

MAHTAB SAMADI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

February 2017



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DEDICATION

To my great family: my lovely parents, my unique husband, and my kind mother-inlaw

Words cannot express how grateful I am for all their kindness and support. I am forever in their debt.

 My lovely parents (Ali Asghar Samadi and Maryam Kamali), whose smile means a world to me

My father is my kind-hearted hero. From the day that I remember, he has been trying to make the life better for all of his girls. He is the greatest and kindest dad of all. What can I say about my mother now that truly express my gratitude. She is my beautiful kind-hearted angel, whose biggest wish is the success and happiness of me and my sweet sisters. Her friendly, warm, and welcoming voice is the essential element of my daily life. My parents have always wanted the best for their children and have raised us with pure love. Their only dream is the success and happiness of their children and they have sacrificed everything to make that happen. Words cannot express their pure kindness. My dream has always been to make my parents happy. They're the best and I'm so lucky to be their daughter. Their endless love has always given me the strength no matter how far they were away from me.

My unique, talented, kind, and supportive husband (Dr. Amir Masoud Tabatabei Yazdi), whose love is the greatest gift of all and I'm so grateful for that

He is the love of my life. His kind support is beyond the words. I've tasted the sweet and precious love with him. He has been always beside me in sadness and happiness. He is the best of the bests, my best friend, and kindest of all. He has supported me during the saddest and hardest times of my life. His warm hug and kind voice is always soothing. I'm so grateful for his unconditional love and support. The truth is that without his love and support, I would have never been able to continue and finish my study. Even though our field of study is totally different, he never hesitated to help me. He owns my heart and his presence is the reason of my heart beat. Words cannot express my feeling and my appreciation. I only can say that I love him from the bottom of my heart, and I'm so thankful for all the things that he has done for me.

My lovely mother in-law (Dr. Vida Samimi), who is not only like my mother, but also she is my kind, and supportive friend

She has always been supportive with her beautiful smile. Her flawless-pure love and support is priceless. She is the best mother in-law and I can't be luckier to have someone like her in my life. I love her like my own mother and her happiness is my happiness. She has always wanted the best for us and has sacrificed a lot for that. I want to express my gratitude for all the sacrifices that she has done. Appreciate all the love and support that she has been given us unconditionally. I hope that someday I can be able to make it up to her for all her kindness

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Chair: Associate Professor Zurina Zainal Abidin, PhD Faculty: Engineering

Aquilaria malaccensis (gaharu), which is an Indo-Malaysian evergreen tree, is one of the most important higher-plants in south East Asia. Because of the high-demand for this valuable tree especially for its resinous wood oil, *Aquilaria malaccensis* is considered as an endangered species. On the other hand, the current extraction method (distillation) in gaharu industries not only requires lots of wood material for only small amount of essential oil, but also, it consumes lots of time and energy. Therefore, finding an efficient alternative to traditional distillation method such as Subcritical Water Extraction (SCWE; an extraction method that uses water at subcritical conditions) can make a significant difference in saving the materials, time, and energy. Thereby, the primary goal of this study was to assess whether SCWE can be used as an alternative to HD for extraction of *A. malaccensis* wood essential oil.

In addition, this study, for the first time, aimed to analyze *A. malaccensis* leaves oil since not only there had been some indications that the leaves of this evergreen tree may be as valuable as its wood, but also, the positive outcome would put a stop to the waste of *A. malaccensis* leaves.

For comparing both extraction methods (i.e. SCWE and HD), at first, the optimum conditions for both methods were identified by assessing the effect of different influential parameters (temperature/heating-power, sample-to-solvent ratio, and time). Hydro-distillation experiments conducted using clevenger apparatus, while SCWE was done using a batch subcritical reactor. Yield comparison between the essential oils extracted by HD and SCWE at optimum conditions showed that the yield of both leaves and wood essential oils extracted by SCWE (30 min) were almost 2.5 times the yield of HD (4-16 hrs). Additionally, the characterization tests were carried out on the essential oils and the leaves/wood samples. GC/MS results provided evidence that the quality of *A. malaccensis* wood and leave oils extracted by SCWE is significantly better compared to that extracted by HD as the amount of oxygenated and sesquiterpenoid compounds was found to be higher in oils extracted by SCWE. The essential oils extracted by SCWE also contained several value-added compounds useful in medicine such as furfural and guaiacol. FTIR, SEM, and BET/BJH on the wood and

leaves samples provided further evidence for better performance of SCWE, since the sample's pores, cell walls, cellulose, and hemicellulose were more damaged compared to those in HD sample.

Furthermore, in this study, kinetic modelling was conducted in order to provide a better understanding of both HD's and SCWE's mechanism. The result of kinetic modelling indicated that the unsteady-state diffusion model is the best model for describing HD, whereas, two-site kinetic desorption and second-order model were found to be the best models for explaining SCWE of wood and leaves essential oils respectively.

In short, the result of this study showed that SCWE is a better extraction method in terms of time, efficiency and quality. Both *A. malaccensis* leaves and wood oils were found to contain beneficial compounds useful for producing pesticides and medicine.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGOPTIMUMAN, PEMODELAN KINETIC, DAN PENCIRIAN HYDRO-PENYULINGAN DAN AIR PERAHAN SUBGENTING DARIPADA AQUILARIA MALACCENSIS LAMK DEDAUNAN AND WOOD MINYAK ESSENTIAL

Oleh

MAHTAB SAMADI

Februari 2017

Pengerusi: Profesor Madya Zurina Zainal Abidin, PhD Fakulti: kejuruteraan

Pada masa kini, kepentingan untuk produk semula jadi telah meningkat dengan ketara di seluruh dunia kerana produk semula jadi 'kurang kesan sampingan dan faedah yang lebih tinggi. Tinggi-tumbuhan, yang mengandungi pelbagai jenis metabolit sekunder dengan kepelbagaian struktur yang tinggi, telah digunakan sebagai salah satu sumber utama produk semula jadi untuk beberapa dekad. Aquilaria malaccensis (gaharu), yang merupakan pokok malar hijau Indo-Malaysia, adalah salah satu yang paling penting yang lebih tinggi-kilang di selatan Asia Timur. Oleh kerana permintaan tinggi untuk pokok ini berharga terutama bagi minyak kayu resin itu, Aquilaria malaccensis dianggap sebagai spesies terancam. Sebaliknya, kaedah semasa pengekstrakan (penyulingan) dalam industri gaharu bukan sahaja memerlukan banyak bahan kayu untuk jumlah hanya kecil minyak penting, tetapi juga, ia menggunakan banyak masa dan tenaga. Oleh itu, mencari alternatif yang cekap kepada kaedah penyulingan tradisional boleh membuat perbezaan yang signifikan dalam menyelamatkan bahanbahan, masa, dan tenaga. pengambilan air subgenting (SCWE) adalah salah satu kaedah yang selamat, cekap dan mesra alam. Oleh itu, matlamat utama kajian ini adalah untuk menilai sama ada SCWE boleh digunakan sebagai alternatif kepada HD untuk pengekstrakan A. malaccensis minyak pati kayu. Di samping itu, kajian ini, buat kali pertama, bertujuan untuk menganalisis A. malaccensis meninggalkan minyak. Menganalisis A. malaccensis meninggalkan minyak pati adalah penting kerana bukan sahaja terdapat beberapa tanda-tanda bahawa daun pokok malar hijau ini mungkin yang berharga sama seperti kayu, tetapi juga, hasil positif akan menghentikan pembaziran A. malaccensis meninggalkan apabila pokok gaharu yang ditebang.

Untuk membandingkan kedua-dua kaedah pengekstrakan (iaitu SCWE dan HD), pada mulanya, keadaan optimum untuk kedua-dua kaedah telah dikenalpasti dengan menilai kesan parameter berpengaruh berbeza. Hasil perbandingan antara minyak pati diekstrak oleh HD dan SCWE pada keadaan optimum menunjukkan bahawa hasil daripada kedua-dua daun dan minyak pati kayu telah meningkat kepada hampir 2.5 kali oleh SCWE Masa perahan yang jauh lebih pendek (kurang dari 30 min). Selain itu, keputusan GC / MS memberikan bukti bahawa kualiti *A. malaccensis* kayu dan

meninggalkan minyak yang diekstrak oleh SCWE adalah jauh lebih baik berbanding yang diekstrak oleh HD kerana ia mengandungi sebatian nilai tambah. Hasil ujian pencirian (FTIR, SEM, dan BET / BJH) memberikan bukti lanjut untuk prestasi yang lebih baik daripada SCWE sejak liang sampel tersebut, dinding sel, selulosa, hemiselulosa dan selepas menjalani SCWE, lebih rosak berbanding sampel HD.

Tambahan pula, dalam kajian ini, model kinetik telah dijalankan untuk memberi pemahaman yang lebih baik daripada kedua-dua ini HD dan mekanisme SCWE ini. Hasil daripada model kinetik menunjukkan bahawa model penyebaran -State tak mantap adalah model yang terbaik untuk menerangkan HD, manakala, dua tapak kinetik desorption dan kedua untuk model didapati model yang terbaik untuk menerangkan SCWE kayu dan daun minyak pati masing-masing .

Pendek kata, hasil daripada kajian ini menunjukkan bahawa SCWE adalah kaedah pengekstrakan yang lebih baik dari segi masa, kecekapan dan kualiti. Kedua-dua *A. malaccensis* daun dan minyak kayu didapati mengandungi sebatian bermanfaat berguna untuk menghasilkan racun perosak dan perubatan.

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APPROVAL

I certify that a Thesis Examination Committee has met on 28th February 2017 to conduct the final examination of Mahtab Samadi on her thesis entitled "Optimization, kinetic modeling, and characterization of hydro-distillation and subcritical water extraction of *Aquilaria malaccensis* lamk leaves and wood essential oil" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
BET	Brunauer Emmett Teller
BJH	Barrett-Joyner-Halenda
CCD	Central Composite Design
EO	Essential Oil
FT-IR	Fourier Transform-Infrared
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectroscopy
HD	Hydro-Distillation
RSM	Response Surface Methodology
SCWE	Sub-Critical Water Extraction
SEM	Scanning Electron Microscopy
WHO	World Health Organization



(C)

CHAPTER 1

INTRODUCTION

1.1 Background

Aromatic herbs and plants are proven to be quiet beneficial to human being as they have diverse range of applications in food, medicine, and etc. (Bonjar, 2004; Bakkali et al., 2008; Soran et al., 2015). For instance, the vast range of vegetables, herbs, fruits, and aromatic plants have been used in traditional medicine as they contain a rich source of biological active compounds (Nikolić, et al., 2014). It is not surprising that the interest for natural and organic products has increased significantly worldwide as they are considered to be less harmful in comparison with non-organic products (Jiao, et al., 2013). Nowadays, there are numerous natural products in the market for antiparasitical, bactericidal, fungicidal, pesticides, cosmetic, perfumery, food additives, and medicinal purposes (Gechev et al., 2014; Sarker et al., 2006).

Lots of the above-mentioned organic products contain essential oils extracted from aromatic plants (Jiao et al., 2013). Essential oil, which is the key property of aromatic plants, is an intricate combination of volatile compounds usually exists in low amount within the plant (Reverchon, 1997; Hui, 1992; Lucchesi et al., 2004; Baser & Buchbauer, 2009). However, the amount of essential oil as well as chemical composition existing in essential oil varies significantly in each method of extraction (Jiao et al., 2013; Baser & Buchbauer, 2009). Therefore, given the aforementioned importance of essential oil for natural products, finding the highly efficient isolation method of essential oils as well as understanding and identifying the chemical composition of the essential oil extracted by each method are vitally important.

1.2 Problem statement

1.2.1 Why Aquilaria malaccensis?

Aquilaria malaccensis, also known as Agarwood or Gaharu, is one of the valuable Aquilaria species, Aquilaria species (23 species) is one of the important medicinal plant genuses that have been used for centuries by both people and industries in southeast Asia. Aquilaria is a genus in the family Thymeleceae and class magnoliopsida (Dashet al., 2008; Barden et al., 2000). For more than 2000 years, Aquilaria malaccensis/Agarwood has been considered precious as it has been used for perfumery, cosmetic, medicine and others (Barden et al., 2000). The importance of this evergreen tree is mostly because of its wood resin, which is produced as a natural immune response of the tree towards fungal attack. Despite the fact that 23 species of Aquilaria have been identified so far, only a few Aquilaria species- including Aquilaria malaccensis- produce this resin (Donovan & Puri, 2004; Barden et al., 2000). Thanks to its resin, Aquilaria malaccensis' wood has many useful medicinal applications and it has been used in traditional medicine to treat pain, fever, rheumatism, and asthma. Additionally, researches on non-traditional medicine revealed that the wood of A. malaccensis also has remarkable anticancer activity (Gunasekera et al., 1981). In the recent years, Aquilaria malaccensis has been widely used -resulting in over-harvestingand hence it has been enlisted in red list of threatened trees (Barden et al., 2000).

1.2.2 Why new extraction method is needed?

Hydro-distillation (HD) and Steam distillation (SD) are two common methods used by industries for extracting essential oil from wood of Aquilaria malaccensis. Yet, these conventional methods are extremely time-consuming (Desai et al., 2014; Yoswathana et al., 2012; Sulaiman et al., 2015). Prior to extraction, these methods require one week for soaking (Fazila & Halim, 2012; Tajuddin & Yusoff et al., 2010). Additionally, they take about 16 hours to extract essential oil from the wood of Aquilaria malaccensis. Moreover, beside the disadvantages on the time consumption, HD and SD methods have other drawbacks as well. The loss of volatile compounds, low extraction efficiency, degradation of unsaturated compounds, and high energy consumption are the examples of these problems (Tam et al., 2007). To address these drawbacks, other extraction methods have been employed. Yet, other extraction methods were also found to be problematic or inefficient. For instance, Supercritical Fluids Extraction (SFE) method was developed to reduce the time consumption and also to improve the extraction efficiency (i.e. higher yield; Ibrahim et al., 2011). But, the carbon dioxide emitted during the extraction process is not environmental friendly as it has greenhouse effect (Jiménez-Carmona et al., 1999). Additionally, recently soxhlet extraction and Accelerated Solvent Extraction (ASE) methods have been used to improve the efficiency of essential oil extracted from A. malaccensis (Sulaiman et al., 2015). Despite the fact that both the soxhlet and ASE methods seem to improve the yield of essential oil, these methods still are not desirable since these methods use organic solvents (e.g. n-hexane), which are toxic, hazardous, and uneconomic. Thus, still a new extraction method for addressing aforementioned problems of HD and SD is required.

Subcritical Water Extraction (SCWE) is a new promising extraction method, which is safe, fast, economic, and environmental friendly in comparison with other new methods. Extraction of essential oil using SCWE uses water as solvent, which is cheap and environmental friendly (Ayala & Luquede Castro, 2001). Furthermore, SCWE has shown to require significantly less extraction time (around 2-3 times) and utilize lower amount of raw material, and it produces higher quality and quantity of essential oil (Herrero, Cifuentes, & Ibanez, 2006). The SCWE is rapidly emerging as an alternative for the extraction of essential oils compounds in the world (Luquede Castro et al., 1998).

Yoswathana et al. (2012) provided evidence for the suitability of SCWE method for extracting essential oil from *Aquilaria crassna* (i.e. one of *Aquilaria*/agarwood species). By using the SCWE for extracting essential oil from A. crrasna (i.e. one of the species from *Aquilaria* family) in a very limited range of temperatures (i.e. 100, 125, and 150°C) and constant time, Yoswathana et al. (2012) concluded that in comparison with HD, Subcritical Water Extraction (SCWE) method resulted in higher yield of A. crrasna oil in shorter time period. Hence, there are some indications that SCWE may be a better alternative for HD and SD methods in terms of time consumption, yield, and the quality *A. malaccensis*' oil.

1.2.3 Gap

As mentioned earlier, finding a better alternative for HD process can be quite useful for the industry. Additionally, assessing the extraction mechanism as well as the optimization is very beneficial for controlling and fine-tuning the extraction process. Yet, to the best of the author's knowledge, so far, no study on the extraction mechanism and optimization of subcritical water extraction (SCWE) for extraction of *A. malaccensis* oil has been carried out. Additionally, the study on the extraction mechanism and optimization of hydro-distillation (HD) of *A. malaccensis* is very limited (only one study exists; Sulaiman, et al., 2015). So, more investigation on the essential oil of *Aquilaria malaccensis*' wood is needed.

There are some indications that *Aquilaria malaccensis*' leaves can also be useful for variety of purposes. For instance, numerous studies (e.g. Suresh et al., 2008; Ibrahim et al., 2011; Morris et al., 2011; Dash et al., 2008; and Gurib Fakim, 2006 maintained that in higher plants (e.g. Agarwood), there is a good chance of finding rich amount of secondary metabolites, aromatic substances, microorganisms, and high structural diversity compounds in all of its parts. These materials have significant application in medicine, cosmetic, and pesticides (Gurib Fakim, 2006; Sarker et al., 2006). By taking into consideration that Agarwood has lots of green leaves throughout the year, finding secondary metabolites, aromatic substances, and microorganisms in Agarwood's leaves can be quite useful for providing necessary material for producing medicinal, cosmetic, and pesticide products.

Moreover, there are other indications that investigating on Aquilaria malaccensis' leaves are likely to be fruitful. For example, Huda et al. (2009), Khalil et al. (2013) and Wil et al. (2014) noted that solvent extraction of A. malaccensis' leaves shows significant antioxidant activity, and therefore, it can be considered as a natural antioxidant. Begum (2015), also provided evidences for both antibacterial and antioxidant activity of solvent extract of A. malaccensis' leaves. In spite of all the above-mentioned indications for the possible applications of A. malaccensis' leaves, surprisingly, no study on the essential oil of A. malaccensis' leaves or its chemical compounds has been conducted. So, by studying A. malaccensis' leave oil through characterization, this study aimed to fill this void.

1.3 Significance of the study/Practical Contribution

The significance of this research is threefold. First, by testing Subcritical Water Extraction (SCWE) method in terms of time consumption, yield, and the quality of product, this study tried to determine whether SCWE can be used as a better alternative for hydro-distillation (HD) and steam distillation (SD). By identifying SCWE as a better extraction method for *Aquilaria malaccensis*, this research can be quite beneficial for industries, in which *Aquilaria malaccensis*' essential oil is extracted, and help them to save a lot of time and money.

Second, by finding useful components from *Aquilaria malaccensis*' leaves such as secondary metabolites, aromatic substances, and microorganisms, which are useful for many industries (e.g. health care and agriculture), this study can take a big step in providing information for producing organic products. Finding useful components from *Aquilaria malaccensis*' leaves is importance since not only there are huge amount of leaves on *Aquilaria malaccensis* throughout the years that can be used, but also it will find alternative usage for the leaves of *Aquilaria malaccensis* which is already cut down (better usage of the whole tree).

Finally, by conducting a kinetic modelling on the extraction process using *Aquilaria malaccensis*, this study provides necessary information required for better controlling and optimizing the extraction process. This information can also help organizations to enhance the efficiency of extraction process and consequently improve their revenue (Paunović et al., 2014; Cassel et al., 2009). In addition, the result of kinetic modelling is essential for comparing traditional and modern extraction methods, scaling up purposes, and the design of unit operation (Chan et al., 2014; Silva et al., 2008; Minozzo, et al., 2012).

1.4 Research Objectives

This study attempts to fill the mentioned gaps and problem in three ways. First, by extracting and analyzing the essential oil of *Aquilaria malaccensis* from wood and (especially) leaves by both hydro-distillation and subcritical water extraction, this study attempts to identify its chemical compositions in order to provide a foundation for further investigations. Secondly, by conducting the kinetic-modeling for extraction method of the essential oil, this study tries to get fuller understanding of the mechanism of both hydro-distillation and subcritical water extraction processes, and consequently, to identify the optimum condition, in which higher yield of essential oil can be obtained. Finally, given the abundant benefits of finding better extraction methods for different industries, this study aimed to assess whether SCWE method is better than traditional methods (i.e. HD and SD) in terms of time consumption, yield efficiency and quality of the essential oil.

Overall, the objectives are divided into specific objectives as follows, which are the main contribution of the thesis to the body of knowledge:

- To optimize the hydro-distillation and subcritical water extraction of essential oils from Aquilaria malaccensis' leaves and wood.
- To assess kinetic modelling for both hydro-distillation and subcritical-water extraction process using the existing models and the experiment results in order to provide a better understanding of the extraction processes.
- ✤ To assess the effectiveness of hydro-distillation (HD) and subcritical-water extraction (SCWE) on extraction of the essential oil from *A. malaccensis* leaves and wood using GC/MS, SEM, FTIR, and BET results.

1.5 Scope

- In this study, extraction of the essential oils by hydro-distillation Clevenger apparatus method have been done as preliminary study (reference method) and main focus in on the extraction of the essential oils by subcritical water extraction (SCWE).
- ✤ A batch/static subcritical water extraction system, which has been imported from Japan, was employed in this study because not only this method is environmental friendly, also has the advantage of the short time of extraction.
- Response surface methodology (RSM) was used to design the extraction experiments by SCWE from both leaves and wood of *A. malaccensis*. A central composite design was used to optimize operational parameters of the extraction of essential oils by SCWE.
- The optimization of hydro-distillation method was assessed based on the yield of essential oil at different heating powers and sample-to-solvent ratios.
- The optimization of subcritical water extraction method was assessed based on the essential oil yield at different extraction temperatures.
- The effectiveness of HD and SCWE process on quality of the extracted essential oil was evaluated based on the chemical composition of *A. malaccensis* leave and wood essential oils identified by GC/MS
- The effectiveness of HD and SCWE in extraction of the essential oils was assessed based on morphological and structural changes of samples (using the results of SEM, FTIR, and BET on A. malaccensis leave and wood samples before and after extraction).

1.6 Structure of thesis

This thesis is divided into five chapters. Chapter one covers introduction, problem statements, objectives, scope and thesis structure. Chapter two includes descriptions on the essential oil, Aquilaria malaccensis, its benefits and its extraction methods. The advantages and disadvantages of hydro-distillation as current extraction method of A. malaccensis essential oil were explained. Also, Subcritical water extraction was introduced in full description as a possible replacement extraction method for gaharu industries. Furthermore, this chapter consists of the kinetic mechanism of extraction of agarwood oil by HD and SWE. Also, theory of multi-objective design optimization using response surface methodology was explained shortly. In chapter three, both the materials and methods for extracting essential oil from A. malaccensis wood and leave are elaborated. Moreover, the characterization methods (i.e. GC/MS, FTIR, BET, and SEM) for testing A. malaccensis leave and wood oil as well as for assessing leave and wood sample before and after extraction were explained. In chapter four, the results of experiments were thoroughly explained and discussed. Chapter four consists of preliminary study on extraction of essential oil from both leaves and wood of A. malaccensis by HD at optimum condition, which was found by studying the effect of key parameters and kinetic modeling of the process. An experimental design using

Central Composite Design (CCD) was done on the SCWE extraction of oils from wood and leaves of *A. malaccensis*. Then it continues to optimize operational parameters using response surface methodology. Finally, the kinetic study of the SCWE process for oil extraction from *A. malaccensis* was presented to have a better understanding of the process. This chapter also covers the results of several characterization tests reports of both essential oils and dry sample (before and after extraction) of HD and SCWE, in order to do a better comparison of the extraction methods. Finally, the overview of the study, conclusion, implication, and the direction for future studies were presented in Chapter 5.



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