



UNIVERSITI PUTRA MALAYSIA

***COMPARISON OF PHYSICOCHEMICAL PROPERTIES OF DATE SEED
OIL FROM SOLVENT EXTRACTION AND SUPERCRITICAL FLUID
EXTRACTION***

ABDUL AFIQ BIN MOHD JAIH

IPPH 2014 6



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By

ABDUL AFIQ BIN MOHD JAIH

**Thesis Submitted to the School of Graduate Studies,
Universiti Putra Malaysia, in Fulfillment of the Requirements
for the Degree of Master of Science**

December 2014

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

COMPARISON OF PHYSICOCHEMICAL PROPERTIES OF DATE SEED OIL FROM SOLVENT EXTRACTION AND SUPERCRITICAL FLUID EXTRACTION

By

ABDUL AFIQ BIN MOHD JAIH

December 2014

Chair : Professor Russly Abdul Rahman, PhD

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Date seed is a by-product of date fruit industry, which is normally being discarded, or traditionally used as animal feed ingredient or turned into non-caffeinated coffee by the Arabs. In this study, oil samples were extracted from the seed of five varieties of mature date fruit namely Mabroum, Rotab, Mariami, Safawi and Sufri through Soxhlet extraction method. The extracted oil were analysed on its Iodine value (IV), fatty acid content (FAC), triacylglycerol (TAG) composition, as well as its thermal properties. The antioxidant properties in date seed oils (DSOs) were determined through total phenolic content (TPC) analysis, radical scavenging activity analysis and reducing power assay. Other than that, the DSO also was extracted through supercritical fluid extraction (SFE) technique. From the findings, it was found that Rotab seed has the highest oil content (9.75 ± 0.04 % of the date seed), while Mariami seed has the lowest oil content (8.04 ± 0.01 % of the date seed). The IVs of the five DSOs were ranged from 48.7 to 55.5 g I₂/100g. The major fatty acids that were found in Mariami, Safawi, Rotab and Sufri seed oils were oleic acid, followed by lauric acid, while opposite major fatty acid content was found in Mabroum seed oil. The major TAG found in those five DSOs were LaOO (La: lauric acid; O: oleic acid). The differences in FAC, TAG and IV between the five varieties of DSO lead to variation in melting and solidification properties of the oils. Analysis of antioxidant content showed that the total phenolic contents in the five DSOs were in the range of 7.962 to 17.724 mg GAE/g oil. The analysis on antioxidant activity found that its capability to terminate the radical activity of DPPH that were expressed as 50 % effective concentration (EC₅₀) ranged from 5.170 to 17.175 mg/ml. Moreover, the reducing power analysis found that the highest reducing activity was obtained at concentration 4 mg/ml. In SFE technique, the

combination of 40 °C and 20 MPa of the extraction parameters resulted in the highest oil yield. The oil yield obtained through this technique ranged from 4.28 to 8.10 %. Oleic acid was found as the only major fatty acid present in the DSO extracted through SFE technique. Overall, the findings of the present study suggest that the DSOs are potentially to be developed as a new functional oil product due to its properties and SFE can be a technique in producing halalan thoyyiban oil product.



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**PERBANDINGAN SIFAT FIZIKOKIMIA MINYAK BIJI KURMA DARIPADA
PENGEKSTRAKAN PELARUT DAN PENGEKSTRAKAN
SUPERKRITIKAL BENDALIR**

Oleh

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Biji kurma adalah hasil sampingan industri buah kurma yang kebiasaannya dibuang, atau secara tradisional dijadikan sebagai ramuan makanan binatang atau kopi bebas kafein oleh orang Arab. Dalam kajian ini, sampel minyak diekstrak daripada lima jenis kurma matang iaitu Mabroum, Rotab, Mariami, Safawi dan Sufri melalui kaedah pengekstrakan *Soxhlet*. Minyak yang diekstrak kemudiannya dianalisis nilai iodin (IV), kandungan asid lemak (FAC), komposisi triasilgliserol (TAG), dan sifat habanya. Sifat antioksidan dalam minyak biji kurma (DSO) ditentukan melalui analisis jumlah kandungan fenolik (TPC), analisis memerangkap aktiviti radikal dan analisis pengurangan kuasa. Selain itu, DSO juga diekstrak melalui teknik pengekstrakan bendalir superkritikal (SFE). Berdasarkan penemuan kajian, biji Rotab didapati mempunyai kandungan minyak tertinggi (9.75 ± 0.04 % daripada biji kurma), manakala biji Mariami mempunyai kandungan minyak terendah (8.04 ± 0.01 % daripada biji kurma). IV untuk kelima-lima DSO tersebut adalah dalam lingkungan 48.7 hingga 55.5 g I₂/100g. Asid lemak major yang terdapat dalam minyak biji Mariami, Safawi, Rotab dan Sufri adalah asid oleik, diikuti asid laurik sementara kandungan asid lemak yang sebaliknya ditemui dalam minyak biji Mabroum. TAG major yang terdapat dalam kelima-lima DSO adalah LaOO (La: asid laurik; O: asid oleic). Perbezaan dalam FAC, TAG dan IV antara kelima-lima jenis DSO telah menyebabkan perbezaan dalam sifat peleburan dan pemejalan minyak tersebut. Analisis antioksidan menunjukkan bahawa jumlah kandungan fenolik yang terdapat dalam kelima-lima DSO tersebut adalah dalam lingkungan 7.962 hingga 17.724 mg GAE/g minyak. Analisis terhadap aktiviti antioksidan mendapati kebolehannya untuk menyekat aktiviti radikal DPPH yang ditunjukkan sebagai 50 % kepekatan efektif (EC₅₀) adalah dalam lingkungan

5.170 hingga 17.175 mg/ml. Tambahan lagi, analisis kuasa penurunan tertinggi didapati berlaku pada kepekatan 4 mg/ml. Dalam teknik SFE, kombinasi parameter pengekstrakan pada 40 °C dan 20 MPa telah berjaya menghasilkan jumlah minyak yang tertinggi. Jumlah penghasilan semua jenis minyak yang diekstrak melalui teknik ini adalah dalam lingkungan 4.28 hingga 8.10 %. Asid oleik adalah satu-satunya asid lemak major yang terdapat dalam DSO yang diekstrak melalui kaedah SFE. Keseluruhannya, hasil kajian ini mendapati bahawa DSO adalah berpotensi untuk dibangunkan sebagai satu produk minyak fungsian berdasarkan kepada ciri-cirinya dan SFE boleh dijadikan satu teknik untuk mengekstrak produk minyak yang halal dan thoyyiban.



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APPROVAL SHEET 1

I certify that a Thesis Examination Committee has met on 4th December 2014 to conduct the final examination of Abdul Afiq Bin Mohd Jaih on his thesis entitled "Comparison of Physicochemical Properties of Date Seed Oil from Solvent Extraction and Supercritical Fluid Extraction (SFE)" 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 Masters of Science.

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LIST OF ABBREVIATIONS

AU	Absorbance Units
DPPH	1,1-diphenyl-2-picrylhydrazyl
DSC	Differential Scanning Calorimetry
DSO	Date Seed Oil
ECN	Equivalent Carbon Number
FAC	Fatty Acid Composition
FAME	Fatty Acid Methyl Ester
FID	Flame Ionization Detector
FRAP	Ferric Reducing Antioxidant Power
GA	Gallic Acid
GAE	Gallic Acid Equivalent
GC	Gas Chromatography
HDL	High Density Lipoprotein
HPLC	High Performance Liquid Chromatography
LDL	Low Density Lipoprotein
R ²	Coefficient of Determination
RID	Refractive Index Detector
SAFA	Saturated Fatty Acid
SFE	Supercritical Fluid Extraction
TAG	Triacylglycerol
TPTZ	2,4,6-tris(2-pyridyl)-s-triazine
USFA	Unsaturated Fatty Acid
VCO	Virgin Coconut Oil
VLDL	Very Low Density Lipoprotein

CHAPTER 1

INTRODUCTION

1.1 General introduction

Date fruit (*Phoenix dactylifera* L.) is an important fruit in some countries as a source of nutrition and for economics (Al-Qarawi *et al.*, 2003; Awad 2007; Briones *et al.*, 2011). Date fruit consists of 73-79 % carbohydrates, 14-18 % total dietary fibers, 2.5 % ash, 2.1-3.0 % protein (Elleuch *et al.*, 2008), and 2.0-3.2 % fat (Al-Farsi *et al.*, 2007), depending on the variety of the date fruit.

About 11-18 % of the date fruit weight comes from the seed (Besbes *et al.*, 2004a; Nehdi *et al.*, 2010; Amira *et al.*, 2011). The Food and Agriculture Organization of the United Nations (FAO, 2010) reported that during 2010, the total world production of date fruits have exceeded 7 million tons. From that amount, approximately more than 1 million tons of date seeds were produced during that year. The date seed have been used traditionally as animal feed or grinded into smaller size and being roasted to turn it into caffeine-free coffee substitute, which have been commercialised by the Arabs in two types, whether plain or mixed with coffee (Rahman *et al.*, 2007; Al-Farsi and Lee, 2011). Other than that, the date seed has no other application and normally being removed as waste by the date fruit industry.

Generally, for vegetable oil extraction process, the solvent, gas and pressing techniques are normally applied, prior to several treatments to the oil. However, recently the awareness among the consumers regarding the oil that has been extracted through the conventional solvent extraction process has increase. This situation has affected the oil industry that has practice the conventional solvent extraction technique in their extraction process. In fact, although the solvent used already has been removed after the extraction process, consumers are still trying to avoid from using those kind of products due to the possibility of solvent contamination in the final products.

Supercritical fluid extraction (SFE) is a technique that has been developed as another way of extraction technique, including for fat and oil products. It has been recognised as a better extraction technique than the solvent and enzyme extraction method, in terms of originality and naturality of the extracted products.

In Islam, the followers are required to take only halal things and it is a compulsory for every Muslims. Halal covers all aspects in Muslim's daily life such as belief, business transaction, recreation and play, social relation,

clothing, adornment, food and drink, working, entertainment, and many others. Halal is defined as lawful and permitted, with respect to which no restriction exists, and the doing of which Allah has allowed.

There are 11 elements of principles pertaining to halal and haram that have been listed by Yusuf Al-Qaradawi (1960). In Al-Quran, the word of thoyyiban always has been mentioned together with the word of halal, indicating the importance of thoyyiban aspect for every Muslims. Thoyyiban means wholesome and good. In terms of food, thoyyiban means a food that is good for the health. Eventhough a food is considered as halal, it is not an assurance that the food also is thoyyiban. For example, stearic acid is a saturated fatty acid that present in palm oil. In this case, although the palm oil is considered as halal, however the high consumption of the stearic acid is bad for health, as it could result in developing of several diseases to human. Moreover, the thoyyiban aspect not only related to the food itself, but also could be related to the processing method of the food product. For instance, the use of chemical-solvent in extraction of several types of plant oil, as there is possibilities of product contamination either by chemical solvent or incomplete chemical solvent removal due to poor evaporation process.

In this study, the oil being studied was extracted from the date seed. There were two extraction technique that have been applied in the present study. SFE technique was performed as alternative to Soxhlet extraction technique. The oil obtained through this technique was studied and compared to the oil obtained through Soxhlet extraction technique. Nevertheless, we found that there is no study that has been conducted yet regarding the application of SFE technique in extraction of date seed oil. Eventhough the halal aspect is not a big issue in plant oil products, in contrast the thoyyiban aspect of plant oil could be disputed as explained earlier.

1.2 Problem statement

As mentioned before, there was about 1 million tons of date seed produced from 7 million tons of date fruits from the whole world in 2010. From that amount, it is only traditionally applied as animal feed and caffeine-free coffee substitute in small scale. Until today, there is no industry reported of using date seed for the purpose of human benefit. From previous study, it was found that there are several beneficial content present in date seed, including oil.

Previous studies on date seed oil shows that it can be a potential seed oil to be developed for various purposes particularly for health beneficial products. However, previous studies have only conducted solvent extraction method, which involved the use of chemical-solvent in date seed oil extraction process. As well known, the application of chemical-solvent in oil extraction

method is not a good practice as there might be a possibility of solvent contamination into the final product or incomplete evaporation process. As a result, it may cause short-term or long-term effect to the user.

Because of that, recently, users' awareness regarding what is good for their health have increase, hence they tend to use high beneficial products although they have to pay at a higher price compared to normal products. Fats and oil that have special features are normally being applied in beneficial products such as confectionaries, cosmetics, personal care and functional foods. Medium chain fatty acid such as lauric acid, and essential fatty acid such as linoleic and linolenic acids, are good for health in terms of prevention of cardiovascular disease (Gilmore *et al.*, 2011) and skin care products (Vermaak *et al.*, 2011). The same trend is also observed on products that are high in antioxidant contents.

Moreover, beneficial product is also a product that contains no chemical-solvent and free from the application of chemical-solvent in its processing method. Thus, in order to produce a beneficial product that is free from any chemical-solvent usage, an alternative to the solvent extraction method used on date seed oil was applied in this study, namely supercritical fluid extraction method.

To sum up, this study is significance to benefit the date seed into better and beneficial products rather than only traditionally being treated as animal feed or turned into non-caffeinated coffee. The extracted oil from the seed might become as a new halalan thoyyiban product as its application is determined. Moreover, the application of supercritical fluid extraction technique in extraction of date seed oil as performed in this study is the first that has been reported and is a new technology applied for this oil.

1.3 Aims and objectives of study

This study focuses on the characteristics of different cultivar of date seed oils, antioxidant properties and extraction techniques. Therefore, this research embarks on the following specific objectives:

- a. To determine the total oil content, basic physicochemical and antioxidant properties of date seed oils obtained from Soxhlet extraction technique.
- b. To determine the effects of temperature and pressure on the yield and fatty acid composition of date seed oil extracted using supercritical fluid extraction technique.

1.4 Scope of study

The research field of this study falls under fats and oil. The extracted date seed oils through chemical extraction technique were analysed on their physical and chemical properties. In addition, the characteristics of the antioxidant content in the oils also were analysed on their amount and activity. Whilst, supercritical fluid extraction technique was studied as alternative to the conventional solvent extraction. The best temperature and pressure for date seed oil extraction was determined based on the highest amount of oil extracted using this technique.

1.5 Experimental design structure

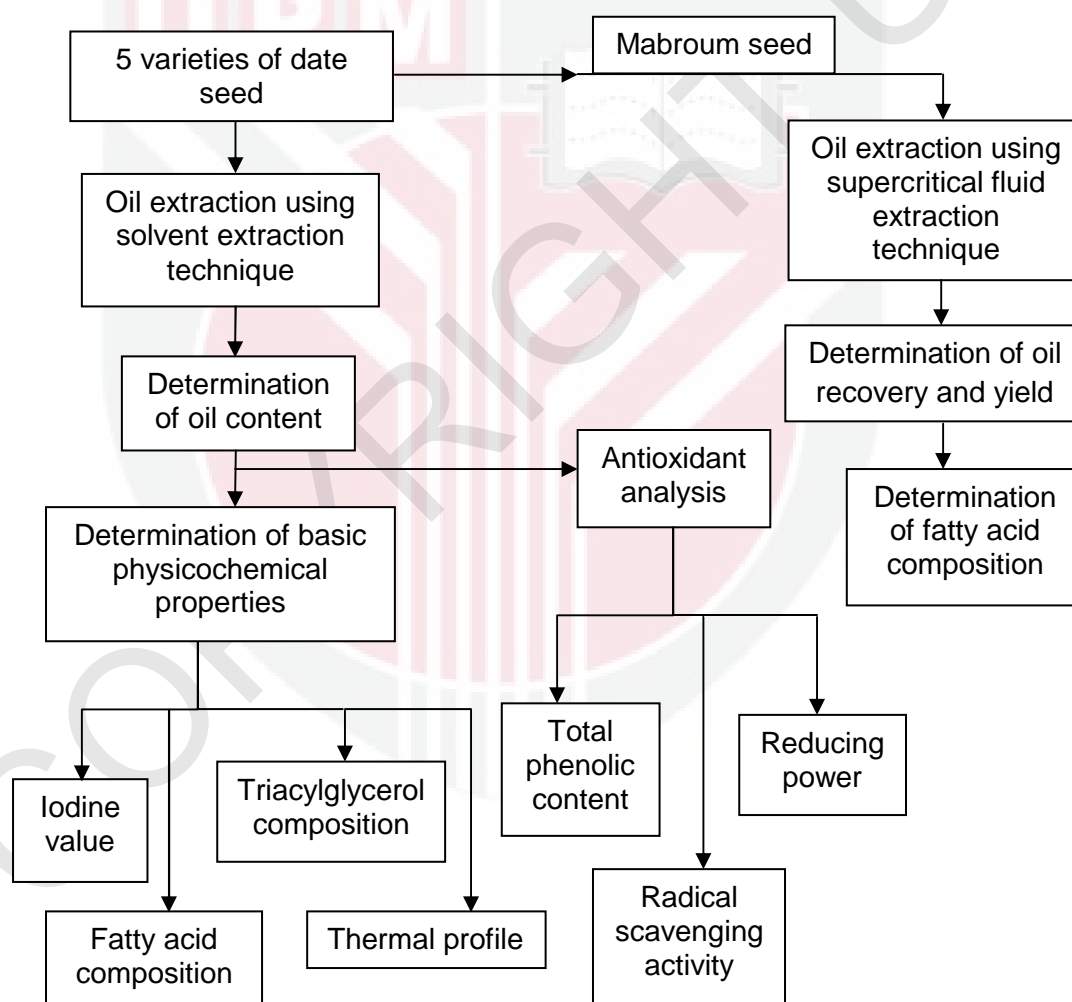


Figure 1.1: The flow diagram of the whole structure of the study

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