



UNIVERSITI PUTRA MALAYSIA

**ANTIOXIDANT EFFECT OF *NIGELLA SATIVA* L. EXTRACTS ON
PHYSICOCHEMICAL PROPERTIES OF DEEP FRIED OIL**

ZEINAB SOLATI

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By
ZEINAB SOLATI

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

May 2011

DEDICATION

This work is dedicated to my parents and my beloved grandmother



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Abstract of thesis presented to the senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Master of Science

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Chairman: Professor Madya Badlishah Sham Baharin

Faculty: Food Science and Technology

The main objective of this study was to investigate the effect of *Nigella sativa* L. extract as a natural antioxidant on the physicochemical changes of frying oil. Two different techniques namely, supercritical CO₂ extraction (SC-CO₂) and solvent extraction were utilized to extract the oil from *Nigella sativa* L. seeds. These two techniques were also compared in terms of extraction yield, thymoquinone content and antioxidant activity of the extracted oil. Initially, influence of SC-CO₂ variables namely, pressure, temperature and dynamic extraction time on the extraction efficiency of *Nigella sativa* L. oil was studied by using full factorial design. The main goal was to determine the most suitable SC-CO₂ condition in order to obtain antioxidant rich *Nigella sativa* L. oil. The response variables studied were yield, antioxidant activity (DPPH method) and thymoquinone (TQ) quantity of the extracts. The results indicated that the highest extraction yield was obtained at 350 bar, 60 °C and 120 min, the highest antioxidant activity was achieved at 350 bar, 50 °C and 60 min and the highest thymoquinone quantity was obtained at 150 bar, 40 °C and 120 min for extraction

pressure, temperature and dynamic extraction time respectively. As shown in the results from gas chromatography analysis, the major fatty acid composition of *Nigella sativa* L. seed oil were: linoleic acid (C18:2), oleic acid (C18:1) and palmitic acid (C16:0).

The results indicated that there was no significant ($p > 0.05$) difference in the fatty acid composition of oil extracted by SC-CO₂ and solvent extraction. The SC-CO₂ extract showed higher thymoquinone concentration (4.09 mg/ml) than the extract from solvent extraction (1.06 mg/ml). The higher extraction yield was obtained by soxhlet (33.00 %) than by SC-CO₂ (23.20 %). Radical scavenging activity (DPPH method) of the extracts obtained by SC-CO₂ and solvent showed the IC₅₀ of 2.59 and 8.26 (mg/ml) respectively. Effect of *Nigella Sativa* L. oil extracted by using SC-CO₂ (with the highest antioxidant activity) on the frying performance of two different frying oils namely, sunflower and RBD palm olein was investigated by adding different concentration levels of 12000 and 10000 ppm of *Nigella Sativa* L. oil to sunflower and RBD palm olein respectively. It should be noted that two samples containing 0 ppm *Nigella sativa* L. oil (Control) and 200 ppm BHT were prepared to check the performance of *Nigella Sativa* L. oil compared to a commonly used antioxidant (i.e. BHT). The physicochemical properties (i.e. Fatty Acid Composition (FAC), Free Fatty Acids (FFA), Peroxide Value (PV), Anisidine Value (AV), Totox Value (TV), Iodine Value (IV), Total Polar Content (TPC), ultra violet absorbance at 232 and 268 nm, C_{18:2}/C_{16:0} ratio, viscosity and color) of frying oils were determined during five consecutive days of frying. Results have shown that the addition of *Nigella sativa* L. oil to both frying oils improved their oxidative stability during the frying process. Moreover, the control sample showed the highest degree of deterioration as compared to those containing BHT and NO (*Nigella sativa* L. Oil). In addition, both frying oils containing 10,000 and 12,000 ppm of *Nigella*

sativa L. extract for palm olein and sunflower oil respectively were able to stabilize the frying oil during the frying process when compared to control. From the results it could be indicated that the stabilizing effect of antioxidants were in the order of BHT > NO. These results were concluded according to the PV, TPC, C18:2/C16:0 ratio, absorbance at 232 and 268 nm and viscosity which were the best indicators of oil deterioration extent. RBD palm olein was found to be more stable than sunflower oil based on the ratio of linoleic acid (C18:2) to palmitic acid (C16:0) and fatty acid composition.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN ANTIOKSIDAN DARI EKSTRAK *NIGELLA SATIVA L.* KEATAS SIFAT-SIFAT FISIKOKIMIA MINYAK GORENG

Oleh

ZEINAB SOLATI

Mei 2011

Pengerusi: Profesor Madya Badlishah Sham Baharin

Fakulti: Fakulti Sains dan Teknologi Makanan

Objektif utama dari penyelidikan ini adalah untuk menyelidik kesan ekstrak *Nigella sativa* L. sebagai antioksidan semulajadi terhadap perubahan fisikokimia minyak goreng. Terdapat dua teknik yang berbeza iaitu, ekstraksi superkritikal karbon dioksida dan ekstraksi pelarut yang digunakan untuk mengekstrak minyak dari *Nigella sativa* L. biji. Kedua-dua teknik ini dibandingkan berdasarkan hasil ekstraksi, kadar thymoquinone dan aktiviti antioksidan oleh minyak yang terhasil dari ekstraksi. Pada permulaannya, pengaruh pembolehubah SC-CO₂ iaitu, tekanan, suhu dan masa ekstraksi dinamik terhadap keberkesanan ekstraksi minyak *Nigella sativa* L. dikaji dengan menggunakan faktorial penuh dengan rancangan rawak lengkap (RAL). Tujuan utama adalah untuk menentukan keadaan SC-CO₂ yang paling sesuai bagi mendapatkan antioksidan yang kaya dengan minyak *Nigella sativa* L.. Hasil respon yang dikaji adalah hasil ekstraksi, aktiviti antioksidan (kaedah DPPH) dan kuantiti ekstrak thymoquinone (TQ). Keputusan kajian menunjukkan bahawa hasil ekstraksi tertinggi diperoleh pada 350 bar, 60 °C dan 120 minit manakala aktiviti antioksidan tertinggi dicapai pada 350 bar, 50 °C dan 60 minit dan kuantiti thymoquinone tertinggi

diperoleh pada 150 bar, 40 °C dan 120 menit untuk tekanan ekstraksi, suhu dan masa ekstraksi dinamik masing-masing. Seperti yang ditunjukkan dalam keputusan, komposisi asid lemak utama minyak biji *Nigella sativa* L. adalah: linoleat (C18:2), (C18:1) oleik dan palmitat (C16:0).

Keputusan kajian menunjukkan bahawa tidak ada ($p > 0.05$) perbezaan dalam komposisi asid lemak minyak yang diekstrak oleh SC-CO₂ dan pelarut ekstraksi. Kaedah pengekstrakan SC-CO₂ menunjukkan kuantiti thymoquinone lebih tinggi (4.09 mg / ml) berbanding ekstrak dari ekstraksi pelarut (1.06 mg / ml). Hasil ekstraksi yang lebih tinggi telah diperoleh daripada soxhlet (33.00%) berbanding daripada SC-CO₂ (23,20%). Aktiviti scavenging radikal (kaedah DPPH) dari ekstrak yang diperoleh daripada SC-CO₂ dan pelarut menunjukkan IC₅₀ sebanyak 2.59 dan 8.26 (mg / ml) masing-masing. Kesan-kesan minyak *Nigella Sativa* L. yang diekstrak dengan menggunakan SC-CO₂ (dengan aktiviti antioksidan tertinggi) terhadap prestasi menggoreng dengan menggunakan dua minyak goreng yang berbeza iaitu, minyak bunga matahari dan minyak kelapa sawit RBD olein, dikaji dengan menambahkan tahap kepekatan yang berbeza iaitu 12000 dan 10000 ppm dari minyak *Nigella Sativa* L. ke minyak bunga matahari dan minyak goreng sawit RBD masing-masing. Oleh yang demikian, dua sampel yang mengandungi 0 ppm minyak *Nigella sativa* L. (Control) dan 200 ppm BHT disediakan untuk mengkaji prestasi minyak *Nigella Sativa* L. bagi membandingkan dengan antioksidan yang biasa digunakan (iaitu BHT). Sifat fisikokimia (iaitu Komposisi Asid Lemak (FAC), Asid Lemak (FFA), penilaian Peroxide (PV), penilaian Anisidine (AV), penilaian Totox (TV), penilaian Iodine (IV), jumlah kandungan polar (TPC), penyerapan ultra violet pada 232 dan 268 nm, C18: 2/C16: 0 nisbah, kelikatan dan warna) minyak goreng ditentukan selama lima hari berturut-turut

penggorengan. Keputusan menunjukkan bahawa penambahan antioksidan untuk kedua-dua minyak goreng meningkatkan kestabilan oksidatif mereka semasa proses menggoreng. Tambahan lagi, sampel kawalan menunjukkan tahap tertinggi kerosakan berbanding dengan minyak yang mengandungi BHT dan NO (*Nigella sativa* L. Oil). Selain itu, kedua-dua minyak goreng yang mengandungi 10,000 dan 12,000 ppm ekstrak *Nigella sativa* L. untuk minyak goreng sawit dan minyak bunga matahari masing-masing mampu menstabilkan minyak goreng semasa proses menggoreng bila dibandingkan dengan kawalan. Dari hasil itu boleh menunjukkan bahawa kesan stabilisasi antioksidans berada di urutan BHT > NO. Keputusan ini membuat kesimpulan mengikut nisbah C18:2/C16:0, PV, TPC, absorbansi pada 232 dan 268 nm dan viskositas yang merupakan penunjuk terbaik peringkat kerosakan minyak. RBD olein sawit ditemui lebih stabil dari minyak bunga matahari berdasarkan nisbah asid linoleat kepada asid palmitat dan perubahan komposisi asid lemak.

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APPROVAL

I certify that a Thesis Examination Committee has met on 20 May 2011 to conduct the final examination of Zeinab Solati on her thesis entitled "Antioxidant Effect of *Nigella sativa* L. Extract on Physicochemical Properties of Deep Fried Oils: A comparative study using SC-CO₂ and Soxhlet extraction methods" 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 Master of Science.

Members of the Thesis Examination Committee were as follows:

Mohammad Reza Mozafari, PhD

Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

Faridah binti Abas, PhD

Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

Abdul Aziz bin Ariffin, PhD

Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

Miskandar b Mat Sahri, PhD

Head of Unit Food Technology and Nutrition
Malaysian Palm Oil Board
(External Examiner)

SHAMSUDDIN SULAIMAN, PhD

Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 26 July 2011

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory committee were as follows:

Badlishah Sham Baharin, PhD

Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

Seyed Hamed Mirhosseini, PhD

Lecturer
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

Tan Chin Ping, PhD

Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

HASANAH MOHD GHAZALI, PhD

Professor/Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

ZEINAB SOLATI

Date: 20 May 2011

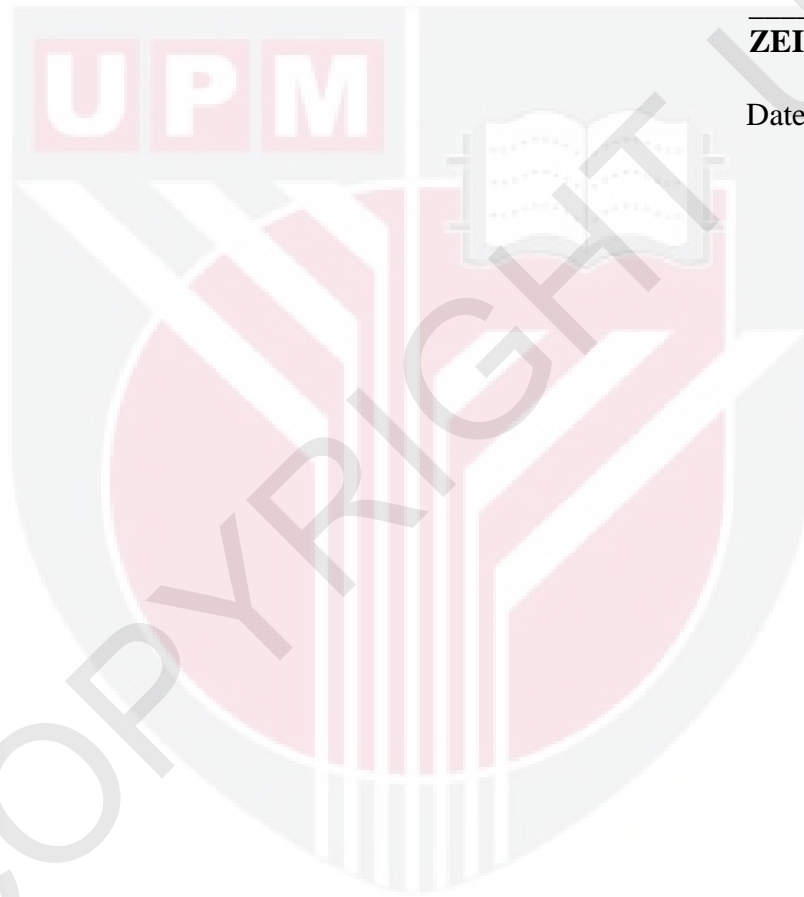


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