



UNIVERSITI PUTRA MALAYSIA

***MICROENCAPSULATED *Nigella sativa* L. OIL AS FUNCTIONAL
INGREDIENT FOR NON-DAIRY CREAMER***

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FSTM 2018 5



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By

NAMEER KHAIRULLAH MOHAMMED

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

March 2018

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DEDICATION

To

My beloved father for his selfless and endless love

The soul of my mother, may Allah forgive her and grant her His highest paradise
(Ameen).

My brothers and sister for the kindness and support

Abstract of thesis presented to the Senate of Universiti Putra Malaysia, in fulfillment of the requirement for the degree of Doctor of Philosophy

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March 2018

Chairman : Associate Professor Anis Shobirin Meor Hussin, PhD
Faculty : Food Science and Technology

The main objective of this research was to produce *Nigella sativa* oil (NSO) based non-dairy creamer (NDC) via microencapsulation and agglomeration process. Two extraction methods namely, supercritical fluid extraction (SFE) and cold press (CP) were used to extract the oil from *Nigella sativa* seed. The microencapsulation using spray dryer was used and the effectiveness of three different independent variables namely, oil concentration (10-30%), wall materials content (10-30%) and inlet air temperature (150-190°C) were optimized using response surface methodology (RSM). The effects of accelerated storage time (24 days) at 65°C on the stability of microencapsulated *Nigella sativa* oil (MNSO) compared to the NSO without encapsulation were evaluated. Total oil was recovered from the powder of MNSO and evaluated in every 6 days along with the NSO. Optimization of the fluidized bed dryer process conditions in terms of drying time (20-60 min), drying temperature (20-50°C) and feed flow rate (1-2.5 mL/min) were conducted using RSM to obtain the non-dairy creamer (NDC) by agglomeration the microencapsulated oil. The NDC was characterized based on antioxidant activity and physical properties. It was found that the oil obtained by SFE showed high content of thymoquinone (TQ) and total phenolic content (TPC) compared to the oil obtained by CP. In addition, antioxidant activity measured by DPPH and ferric reducing antioxidant power (FRAP) activity showed higher activity for SFE oil. The optimal conditions of microencapsulation were 30% wall material, 10% concentration of oil, and 160°C drying inlet air temperature. The properties of oil without encapsulation has undergone many changes with a reduction in oxidative stability, bioactive compounds content, antioxidant activity, and fatty acid composition alteration. Microencapsulated oil indicated a higher stability and resistance under the same storage conditions. The optimum conditions of the fluidized bed agglomeration were: inlet air temperature (50°C), drying time (25 min), and feed flow rate (1 mL/min). This process resulted in further improvement of the powder properties with high solubility, particle size, and glass transition temperature (T_g), as

well as lower moisture content, water activity (a_w), wettability, hygroscopicity, and bulk density compared to the spray dried powder with acceptable results of sensory evaluation. In conclusion, the SFE represents suitable method for NSO. The encapsulation extended the shelf life of the NSO and the agglomeration improved the instant properties and palatable taste. The developed NSO-based NDC can be used as an alternative to the saturated fat and/or hydrogenated oils based NDC.

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

MIKROKAPSUL MINYAK *Nigella sativa* L. SEBAGAI FUNGSI KRIMER BUKAN TENUSU

Oleh

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Objektif utama kajian ini adalah untuk menghasilkan minyak *Nigella sativa* (NSO) berasaskan krimer bukan tenusu melalui proses mikroenkapsulasi dan proses pengaglomeratan. Dua kaedah ekstraksi iaitu pengseksarikan bendalir lampau genting (SFE) dan tekan sejuk (CP) digunakan untuk mendapatkan minyak dari biji benih *Nigella sativa*. Proses mikroenkapsulasi menggunakan pengering sembur dan keberkesanan tiga pemboleh ubah bebas yang berlainan iaitu, kepekatan minyak (10-30%), kandungan bahan dinding (10-30%) dan suhu udara masuk (150-190°C) dioptimumkan menggunakan metodologi tindak balas permukaan (response surface technology-RSM). Kesan masa penyimpanan yang dipercepatkan (24 hari) pada (65°C) pada kualiti dan kestabilan mikrokapsul minyak *Nigella sativa* (MNSO) berbanding dengan NSO tanpa enkapsulasi juga dikaji. Minyak diperolehi dari serbuk MNSO dan NSO (kawalan) diperiksa setiap 6 hari. Pengoptimuman keadaan proses “pengering lapisan terbendalir” dari segi masa pengeringan (20-60 min), suhu pengeringan (20-50°C) dan kadar aliran suapan (1-2.5 mL/min) telah dijalankan dengan menggunakan RSM untuk mendapatkan krimer bukan tenusu (NDC) oleh pengaglomeratan minyak mikroenkapsulasi. NDC dicirikan berdasarkan aktiviti antioksidan dan sifat-sifat fizikal. Didapati bahawa minyak yang diperolehi dari SFE menunjukkan kepekatan tinggi thymoquinone (TQ) dan jumlah kandungan fenolik (TPC) dibandingkan dengan minyak diperolehi dari proses CP. Aktiviti antioksidan yang diukur oleh DPPH dan penurunan kuasa ferik antioksidan (FRAP) menunjukkan aktiviti yang tinggi bagi minyak SFE. Keadaan minyak mikrokapsul yang optimum ialah 30% bahan dinding, 10% kepekatan minyak, dan suhu udara masuk sebanyak 160°C. Minyak tanpa proses enkapsulasi telah mengalami banyak perubahan pada sifatnya seperti pengurangan kestabilan oksidatif, kandungan bahan bioaktif, aktiviti antioksidan, dan perubahan komposisi asid lemak. Minyak mikroenkapsulasi menunjukkan kestabilan dan rintangan yang lebih tinggi di bawah

keadaan simpanan yang sama. Keadaan yang optimum daripada proses pengaglomeratan “lapisan terbendalir” adalah: suhu udara masuk (50°C), masa pengeringan (25 min), dan kadar aliran suapan (1 mL/min). Proses ini menghasilkan peningkatan sifat kecepatan serbuk dengan keterlarutan yang tinggi, saiz zarah dan suhu peralihan kaca (T_g), serta kandungan lembapan yang lebih rendah, aktiviti air (a_w), kebolehbasaan, kebolehan menyerap lembapan dari udara, dan ketumpatan pukal berbanding dengan semburan serbuk kering dengan keputusan penilaian deria boleh terima. Kesimpulannya, penemuan kajian ini menunjukkan SFE adalah kaedah sesuai bagi NSO. Proses enkapsulasi ini memanjangkan jangka hayat NSO dan proses pengaglomeratan meningkatkan sifat segera dan rasa enak. NSO berasaskan NDC yang dibangunkan boleh digunakan sebagai alternatif lemak tepu atau minyak-minyak terhidrogen yang berasaskan NDC.

ACKNOWLEDGEMENTS

In the name of Allah, The Most Graceful, The Most Merciful, The most Compassionate.

All praise is to Almighty ALLAH (S.W.T) and peace is upon His messenger, prophet Mohammad (PBUH).

I would like to express my gratitude to my humble supervisor Associate Professor Dr. Anis Shobirin Meor Hussin, for her general supervision coupled with excellent attention, patience, valuable suggestions and motherly care offered throughout the course of this work and most of all, for her faith towards me.

I also want to give my warm appreciation and acknowledge the efforts of my supervisor committee members, Professor Dato' Dr. Mohd Yazid Abd Manap and Professor Dr. Tan Chin Ping for their attention, kindness and patience. Also for their professional guidance, helpful comments and generous advices throughout preparing this project and thesis, I consider myself very lucky to be given this honour to work with them.

I would like to thank the Faculty of Food Science and Technology, Universiti Putra Malaysia, lecturers and staff those who involved directly and indirectly in the preparation of my thesis.

Finally, I would like to thank my family and friends, especially Dr. Belal Jamal Muhidin and Dr. Ahmed Mediani for their entire support and encouragement throughout the process of completing this research.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
CCD	Central Composite Design
CP	Cold Press
a_w	Water activity
DE	Dextrose Equivalent
DB	Dry Base
DPPH	1, 1-Diphenyl-2-picrylhydrazyl
DSC	Differential Scanning Calorimeter
DTG	Derivative Thermograms Analysis
$D_{[3,2]}$	Surface Mean Diameters
$D_{[4,3]}$	Volume Mean Diameter
FTIR	Fourier-Transform Infrared
FRAP	Ferric Reducing Antioxidant Power
GC-MS	Gas Chromatography Mass Spectrometry
GAE	Galic Acid Equivalent
HPLC	High Performance Liquid Chromatography
IC ₅₀	Inhibition Concentration by 50%
meq	Milliequivalent
MNSO	Microencapsulated <i>Nigella sativa</i> Oil
MD	Maltodextrin
MW	Molecular Weight
MEE	Microencapsulation Efficiency

NSO	<i>Nigella sativa</i> oil
NDC	Non-dairy Creamer
OD	Optical Density
PV	Peroxide Value
RSM	Response Surface Methodology
SDS	Sodium Dodecyl Sulfate
SEM	Scanning Electron Microscope
TGA	Thermogravimetric Analysis
TPC	Total Phenolic Content
TQ	Thymoquinone
T _g	Glass Transition Temperature
X ₁	Independent Variables
Y ₁	Dependent Responses

CHAPTER 1

INTRODUCTION

Recently, with the increase of awareness among individuals, consumers are demanding for health improvement food and beverages supplementations. With the high market demand, manufacturers and scientists have considered essential oils for their biological activities which further benefit human health. Most of the edible seed oils, particularly, *Nigella sativa* L. seed oil (NSO) is considered as a novel natural source of antioxidants and has an important function in nutrition and human health due to its biological activities such as antioxidant, anti-inflammation and anti-microbial activity (Ramadan, Asker, & Tadros, 2012a). According to Ramadan, Kroh, and Mörsel (2003), the NSO is rich in monounsaturated fatty acid and contains functional compounds including thymoquinone (TQ) which is the main active compound. Also, in human and rodents studies indicate that daily oral doses of thymoquinone between 5 mg kg⁻¹ and 90 mg kg⁻¹ can be therapeutic and nontoxic (Tisserand & Young, 2013).

Methods used for oil extraction may alter minor constituents that have functional properties and contribute to oxidation stability. Studies have been conducted on a solvent extract of NSO using different types of conventional extraction such as Soxhlet (Khoddami, Ghazali, Yassoralipour, Ramakrishnan, & Ganjloo, 2011), cold-pressing (Lutterodt, Luther, Slavin, Yin, Parry, Gao, & Yu, 2010), and Microwave assisted (Kiralan, Özkan, Bayrak, & Ramadan, 2014). Alternatively, supercritical fluid extraction (SFE) is one of the most recent techniques for the extraction of plant oils. This method offers some favorable features over the traditional techniques that have been used in the essential oil industries. The CO₂ used in SFE as a solvent for many reasons such as, nontoxic, non-flammable, inexpensive, and friendly environmental solvent which helps to produce high-quality natural product. Furthermore, higher oil yield can be obtained by using SFE with less damage to the bioactive compounds and free organic solvent.

After the extraction, NSO is associated with few major issues that limit its use as food supplement. Firstly, NSO is sensitive to oxidation by external factors such as heat, light, and oxygen with long storage period. In food industry, the lipid oxidation is a crucial quality criterion, as the oxidized products formed not only causes rancidity but also degrades the nutritional quality and safety of the lipid products (Kanazawa, Sawa, Akaike, & Maeda, 2002). Secondly, the peppery and bitter taste of NSO resulted in limited application as a food supplement. Finally is due to the difficulties of handling liquid oils and it is favored to dry powder that is easier to handle and have longer shelf life (Tan, Tuyen, Parks, Stathopoulos, & Roach, 2015).

In the search for solution to the above issues, microencapsulation is a recommended by researchers to improve the oils stability and enhance the overall acceptability. Microencapsulation is a rapidly expanding technology which is a unique way to package solids, liquids, or gaseous materials in the form of micro-particles that can release their contents at controlled rates under specific conditions. In addition, it has been well developed and acknowledged within the pharmaceutical, chemical, food and many other industries (Fang & Bhandari, 2010). Spray drying is the most common process in the food industry for the microencapsulation of oils. The spray drying method was proven to be more efficient than freeze drying in oil encapsulations, the cost of the spray-drying is 30–50 times cheaper, and it produces good quality particles (Ng, Wong, Tan, Long, & Nyam, 2013b).

In order to use microencapsulated oil as non-dairy coffee creamer, it should meet certain criteria including instant and rapid dissolving in hot water without separation due to the fat content in the powder as well as providing a good whitening ability after adding to hot coffee (Kelly, Oldfield, & O'Kennedy, 1999). Furthermore, review of literatures showed limited works on the development of microencapsulated *Nigella sativa* oil using spray drying technique followed by agglomeration and the application of resulted powder as non-dairy coffee creamer.

1.1 Problem Statements

Conventional extraction methods involved several steps including pressing filtration and evaporation which required longer time that resulted in oxidation reactions and low bioactivity of the obtained oil (Khoddami et al., 2011; Kiralan et al., 2014). The oils in liquid form are prone to oxidation, and degradation due to external factors such as heat, light, and oxygen leading to the formation of unpleasant tastes and odors and, consequently lead to reduction of product's shelf life. In addition, direct application of *Nigella sativa* oil in processed foods is problematic due to the low solubility of oils in water, handling and the bitterness. To overcome with previous mentioned issues, microencapsulation is recommended to produce a stable product. Thus, evaluation of the stability and functionality of the microencapsulated oil during storage considered as a crucial stage to be used in food application (Ahn, Kim, Lee, Seo, Lee, & Kim, 2008; Ramadan et al., 2003; Tan et al., 2015; Tonon, Grosso, & Hubinger, 2011).

Nowadays, due to growing public demand for functional food, such as, most of the coffee creamers are considered as unhealthy products because they contain high amount of saturated fats and/or hydrogenated oils as well as lactose intolerance which are avoided by some consumers who are with allergic potential (Hedayatnia, Mirhosseini, Amid, Sarker, Veličkovska, & Karim, 2016a; Krupa & Patel, 2011). In this sense, there is a need to develop NDC that could cater such growing demands.

1.2 Hypothesis

Microencapsulation of the NSO is entrapped in the wall materials consist of maltodextrin and sodium caseinate as well as the soy lecithin as an emulsifier agent will successfully produce microcapsules with antioxidant activity and improved characteristics. The microencapsulation of NSO in terms of the core/wall material percentage as well as the temperature of the spray dryer will be successfully optimized using response surface methodology. Furthermore, the oxidative stability of the microencapsulated oil will be more stable compared to the oil without encapsulation under accelerated storage. Moreover, agglomeration of the microencapsulated oil using optimized conditions of fluidized bed dryer by response surface methodology to obtain instant non-dairy creamer will be successfully applied. The non-dairy creamer with antioxidant activity will be successfully produced.

1.3 Objectives

1.3.1 General Objective

The purpose of this research is to produce *Nigella sativa* oil based-nondairy creamer with antioxidant activity, low fat and improved physical properties.

1.3.2 Specific Objectives

- 1- To investigate the effect of different extraction methods (supercritical fluid and cold press) on characteristic of *Nigella sativa* oil.
- 2- To optimize the process conditions for spray drying microencapsulation of *Nigella sativa* oil.
- 3- To investigate the stability changes of microencapsulated *Nigella sativa* oil upon accelerated storage.
- 4- To characterize a functional non-dairy creamer of *Nigella sativa* oil with improved physical properties and sensory attributes.

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