



**UNIVERSITI PUTRA MALAYSIA**

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

**NAMEER KHAIRULLAH MOHAMMED**

**FSTM 2018 5**



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

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**

## COPYRIGHT

All material contained within the thesis, including without limitation text, logos, icons, photographs and all other artwork, is copyright material of Universiti Putra Malaysia unless otherwise stated. Use may be made of any material contained within the thesis for non-commercial purposes from the copyright holder. Commercial use of material may only be made with the express, prior, written permission of Universiti Putra Malaysia.

Copyright © Universiti Putra Malaysia



## DEDICATION

To

My beloved father for his selfless and endless love

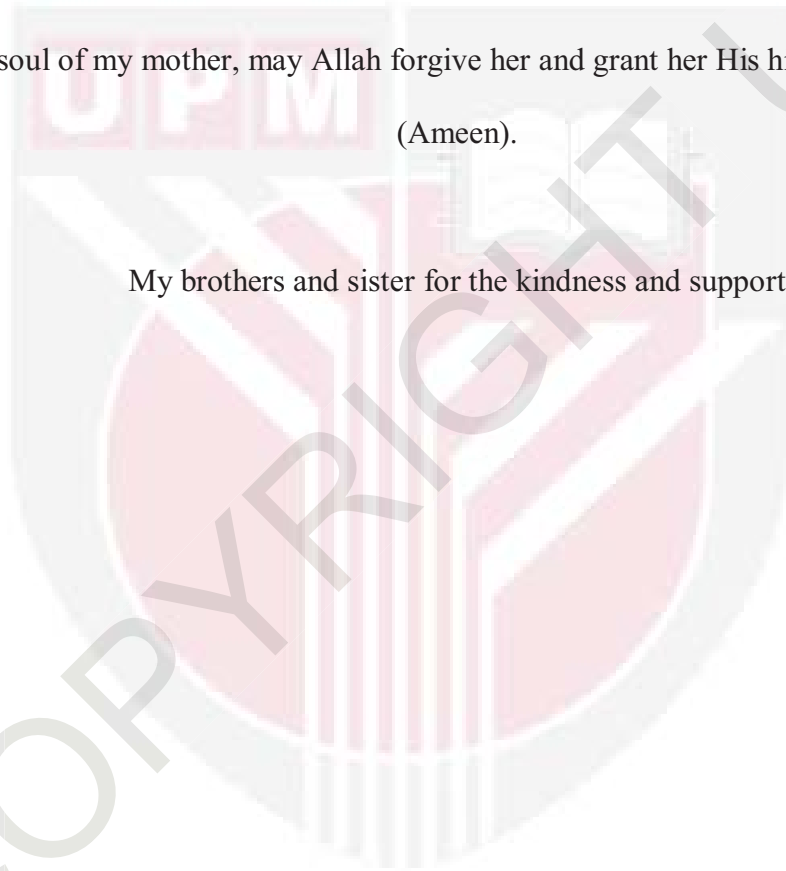
The soul of my mother, may Allah forgive her and grant her His highest paradise

(Ameen).

My brothers and sister for the kindness and support



COPYRIGHT



UPM

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

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

By

**NAMEER KHAIRULLAH MOHAMMED**

**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

**NAMEER KHAIRULLAH MOHAMMED**

**Mac 2018**

**Pengerusi : Profesor Madya Anis Shobirin Binti Meor Hussin, PhD**  
**Fakulti : Sains dan Teknologi Makanan**

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 pengsekrakan 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 mikro kapsul 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 mikro kapsul 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 ( $50^{\circ}\text{C}$ ), 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 ( $a_w$ ), kebolehbasahan, 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.

I certify that a Thesis Examination Committee has met on 26 March 2018 to conduct the final examination of Nameer Khairullah Mohammed on his thesis entitled "Microencapsulated *Nigella sativa* L. Oil as Functional Ingredient for Non-Dairy Creamer" 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.

Members of the Thesis Examination Committee were as follows:

**Yaya Rukayadi, PhD**

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

**Luqman Chuah Abdullah, PhD**

Professor  
Faculty of Engineering  
Universiti Putra Malaysia  
(Internal Examiner)

**Yus Aniza binti Yusof, PhD**

Associate Professor  
Faculty of Engineering  
Universiti Putra Malaysia  
(Internal Examiner)

**Christofora Hanny Wijaya, PhD**

Professor  
Bogor Agricultural University  
Indonesia  
(External Examiner)



---

**NOR AINI AB. SHUKOR, PhD**  
Professor and Deputy Dean  
School of Graduate Studies  
Universiti Putra Malaysia

Date: 26 April 2018

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:

**Anis Shobirin Meor Hussin, PhD**

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

**Dato' Mohd Yazid Abd Manap, PhD**

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

**Tan Chin Ping, PhD**

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

---

**ROBIAH BINTI YUNUS, PhD**

Professor and Dean  
School of Graduate Studies  
Universiti Putra Malaysia

Date:

## Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
- quotations, illustrations and citations have been duly referenced;
- this thesis has not been submitted previously or concurrently for any other degree at any other institutions;
- intellectual property from the thesis and copyright of thesis are fully-owned by Universiti Putra Malaysia, as according to the Universiti Putra Malaysia (Research) Rules 2012;
- written permission must be obtained from supervisor and the office of Deputy Vice-Chancellor (Research and Innovation) before thesis is published (in the form of written, printed or in electronic form) including books, journals, modules, proceedings, popular writings, seminar papers, manuscripts, posters, reports, lecture notes, learning modules or any other materials as stated in the Universiti Putra Malaysia (Research) Rules 2012;
- there is no plagiarism or data falsification/fabrication in the thesis, and scholarly integrity is upheld as according to the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) and the Universiti Putra Malaysia (Research) Rules 2012. The thesis has undergone plagiarism detection software.

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Name and Matric No.: Nameer Khairullah Mohammed, GS39414

## 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) were adhered to.

Signature: \_\_\_\_\_  
Name of  
Chairman of  
Supervisory  
Committee: Associate Professor Dr. Anis Shobirin Meor Hussin

Signature: \_\_\_\_\_  
Name of  
Member of  
Supervisory  
Committee: Professor Dato' Dr. Mohd Yazid Abd Manap.

Signature: \_\_\_\_\_  
Name of  
Member of  
Supervisory  
Committee: Professor Dr. Tan Chin Ping

## TABLE OF CONTENTS

	<b>Page</b>
<b>ABSTRACT</b>	i
<b>ABSTRAK</b>	iii
<b>ACKNOWLEDGEMENTS</b>	v
<b>APPROVAL</b>	vi
<b>DECLARATION</b>	viii
<b>LIST OF TABLES</b>	xvi
<b>LIST OF FIGURES</b>	xviii
<b>LIST OF ABBREVIATIONS</b>	xxi
 <b>CHAPTER</b>	
<b>1 INTRODUCTION</b>	<b>1</b>
1.1 Problem Statements	2
1.2 Hypothesis	3
1.3 Objectives	3
1.3.1 General Objective	3
1.3.2 Specific Objectives	3
<b>2 LITERATURE REVIEW</b>	<b>4</b>
2.1 Background	4
2.2 Classification of <i>Nigella sativa</i>	4
2.3 Common Names of <i>Nigella sativa</i>	5
2.4 Morphology of <i>Nigella sativa</i>	5
2.5 Origin and Cultivation of <i>Nigella sativa</i>	6
2.6 Traditional Uses of <i>Nigella sativa</i>	6
2.7 Chemical Composition of <i>Nigella sativa</i>	7
2.7.1 Fat	7
2.7.1.1 Triglycerides	7
2.7.1.2 Sterols	7
2.7.2 Protein	8
2.7.3 Carbohydrates	8
2.7.4 Vitamins and Minerals	8
2.7.5 Bioactive Compounds	8
2.8 Extraction Methods for <i>Nigella sativa</i> Oil	10
2.8.1 Cold Press (CP)	10
2.8.2 Soxhlet Extraction	10
2.8.3 Supercritical Fluid Extraction	10
2.9 Antioxidant Activity of <i>Nigella sativa</i> Oil	11
2.10 Considerations Associated with Extracted <i>Nigella sativa</i> Oil	13
2.11 Microencapsulation	13
2.12 Techniques of Microencapsulation	15
2.12.1 Spray Drying	15
2.12.2 Freeze Drying	18

2.12.3	Extrusion	19
2.12.4	Spray-chilling and Spray-cooling	20
2.12.5	<i>In situ</i> Polymerization	21
2.12.6	Complex Coacervation	22
2.12.7	Organic Phase Separation	22
2.12.8	The Liposome Entrapment Method	22
2.13	Microencapsulation Process Steps via Spray Drying	23
2.14	Optimization of the Microencapsulation Operating Conditions	25
2.15	Wall Materials for Oil Encapsulation by Spray Drying	28
2.15.1	Carbohydrates	29
2.15.2	Maltodextrins	30
2.15.3	Sodium Caseinate	30
2.15.4	Lecithin	31
2.15.5	Particle Size	31
2.16	Food Applications of Encapsulated Oils	32
2.17	Agglomeration	33
2.18	Fluidized-bed-coating	35
2.19	Operating Conditions of the Fluidization Procedure	36
2.20	Non-dairy Creamer (NDC)	38
<b>3</b>	<b>THE EFFECTS OF DIFFERENT EXTRACTION METHODS ON CHARACTERISITIC OF <i>Nigella sativa</i> L. OIL</b>	<b>40</b>
3.1	Introduction	40
3.2	Materials and Methods	41
3.2.1	Materials and Analytical Samples	41
3.2.2	Chemical Properties of <i>Nigella sativa</i> L. Seeds	41
3.2.2.1	Ash content	41
3.2.2.2	Protein content	41
3.2.2.3	Moisture content	42
3.2.2.4	Fat content	42
3.2.2.5	Carbohydrate content	42
3.2.3	Oil Extraction	42
3.2.3.1	Cold Pressing (CP)	42
3.2.3.2	Supercritical Fluid Extraction	43
3.2.4	Physicochemical Properties of <i>Nigella sativa</i> Oil	44
3.2.5	Composition Profiling by GC-MS	44
3.2.6	Quantification of Thymoquinone	44
3.2.7	1, 1-Diphenyl-2-picrylhydrazyl (DPPH) Radical Scavenging Activity Assay	45
3.2.8	Ferric Reducing Ability Power (FRAP) Assay	45
3.2.9	Total Phenolic Content	46
3.2.10	FT-IR Spectroscopy	46
3.2.11	Thermal Behavior DSC	46
3.2.12	Thermogravimetric Analysis (TGA)	46
3.2.13	Statistical Analysis	47
3.3	Results and Discussion	47
3.3.1	Physicochemical Properties of <i>Nigella sativa</i> L. Seed	47
3.3.2	Composition Profiling by GC-MS Studies	49

3.3.3	Thymoquinone Concentration in <i>Nigella sativa</i> Oil	52
3.3.4	1, 1-Diphenyl-2-picrylhydrazyl (DPPH) Radical Scavenging Activity	53
3.3.5	Ferric Reducing Ability Power (FRAP)	54
3.3.6	Total Phenolic Content	54
3.3.7	Fourier Transform Infrared (FTIR) Spectra of <i>Nigella sativa</i> Oils	55
3.3.8	Thermal Behavior of <i>Nigella sativa</i> L. Oil	56
3.4	Conclusion	59
<b>4</b>	<b>OPTIMIZATION OF PROCESS CONDITIONS FOR SPRAY DRYING MICROENCAPSULATION OF <i>Nigella sativa</i> OIL</b>	<b>60</b>
4.1	Introduction	60
4.2	Materials and Methods	61
4.2.1	Materials and Analytical Samples	61
4.2.2	Oil Extraction	62
4.2.3	Emulsion Preparation	62
4.2.4	Emulsion characterization	63
4.2.4.1	Emulsion stability	63
4.2.4.2	Emulsion viscosity	63
4.2.4.3	Emulsion droplet size	63
4.2.5	Process of Spray Drying for Microencapsulation	63
4.2.6	Analytical Methods	64
4.2.6.1	Moisture Content	64
4.2.6.2	Solubility	64
4.2.6.3	Microencapsulation Efficiency (MEE)	64
4.2.6.4	Microencapsulated Oil Particle Size	65
4.2.6.5	Total Oil Extraction	65
4.2.6.6	1, 1-Diphenyl-2-picrylhydrazyl (DPPH) Radical Scavenging Activity Test	65
4.2.6.7	Total Phenolic Content	65
4.2.7	Quantification of Thymoquinone	66
4.2.8	Scanning Electron Microscopy	66
4.2.9	Assortment of the Factor Ranges	66
4.2.10	Statistical Analysis	66
4.3	Results and Discussion	67
4.3.1	Emulsion Characterization	67
4.3.1.1	Emulsion Stability, Viscosity and Droplet Size	67
4.3.2	Optimization of NSO Microencapsulation Using Response Surface Methodology	68
4.3.3	Moisture Content (Y1)	72
4.3.4	Solubility (Y2)	73
4.3.5	Microencapsulation Efficiency (Y3)	75
4.3.6	Particle Size (Y4)	77
4.3.7	DPPH-IC <sub>50</sub> (Y5)	79
4.3.8	Total phenolic content TPC (Y6)	80
4.3.9	Optimization and Validation Conditions	81
4.3.10	Availability of Thymoquinone (TQ) using HPLC	83



	4.3.11 Morphology of powder by SEM	84
4.4	Conclusion	85
<b>5</b>	<b>STABILITY CHANGES OF MICROENCAPSULATED <i>Nigella sativa</i> OIL UPON ACCELERATED STORAGE</b>	<b>87</b>
5.1	Introduction	87
5.2	Materials and methods	88
	5.2.1 Chemicals and Materials	88
	5.2.2 <i>Nigella sativa</i> Oil Extraction	88
	5.2.3 Preparation of the Emulsion	89
	5.2.4 Spray Drying Process	89
	5.2.5 Accelerated Storage Condition	89
	5.2.6 Microencapsulation Efficiency (MEE)	89
	5.2.7 Peroxide Value Determination	89
	5.2.8 Antioxidant Stability of the Microcapsule During Accelerated Storage	90
	5.2.9 Estimation of Total Phenolic Content	90
	5.2.10 Changes in Thymoquinone Quantification Upon Accelerated Storage	90
	5.2.11 Fatty Acid Methyl Esters (FAME)	90
	5.2.12 Scanning Electron Microscopy	90
	5.2.13 Evaluation of Color	91
	5.2.14 Statistical Analysis	91
5.3	Results and Discussion	91
	5.3.1 Microencapsulation Efficiency	91
	5.3.2 Total Phenolic Content	92
	5.3.3 Evaluation of Antioxidant Activity Using 1,1-diphenyl-2-picrylhydrazyl (DPPH) Radical Scavenging Assay	92
	5.3.4 Fatty Acid Methyl Esters (FAME) Compositional Analyses	93
	5.3.5 Oxidative Stability	95
	5.3.6 Quantification of Thymoquinone Content Upon Accelerated Storage	97
	5.3.7 Morphology of Microcapsules Upon Accelerated Storage by Scanning Electron Microscopy (SEM)	98
	5.3.8 Color Determination	100
5.4	Conclusion	101
<b>6</b>	<b>DEVELOPMENT OF FUNCTIONAL NON-DAIRY CREAMER FROM <i>Nigella sativa</i> OIL WITH IMPROVED PHYSICAL PROPERTIES AND SENSORY ATTRIBUTE</b>	<b>102</b>
6.1	Introduction	102
6.2	Material and Methods	104
	6.2.1 Preparation of the Emulsion	104
	6.2.2 Process of Spray Drying for Microencapsulation	104
	6.2.3 Experimental design	104
	6.2.4 Fluidized Bed Drying	106

6.2.5	Physicochemical and instrumental analyses	107
6.2.5.1	Moisture Content	107
6.2.5.2	Solubility Index Measurement	107
6.2.5.3	Radical Scavenging Activity by DPPH Assay	107
6.2.5.4	Total Phenolic Compounds	107
6.2.5.5	Quantification of Thymoquinone	107
6.2.5.6	Powder Bulk Density	107
6.2.5.7	Powder Wettability and Dispersibility	108
6.2.5.8	Water activity	108
6.2.5.9	Hygroscopicity	108
6.2.5.10	Colour analysis	108
6.2.5.11	Glass Transition Temperature	108
6.2.5.12	Particle Size of the Creamer Powders	109
6.2.5.13	Particle Morphology using SEM	109
6.2.5.14	Fourier Transform Infrared (FTIR) Analysis	109
6.2.5.15	Proximate Analysis and Nutrition Value	109
6.2.5.16	Coffee Creamer Evaluation	109
6.2.6	Sensory Evaluation	109
6.3	Results and Discussion	110
6.3.1	Response surface methodology (RSM)	110
6.3.1.1	Moisture content and solubility	112
6.3.1.2	Antioxidant activity of the NDC of the spray-dried powder (DPPH and TPC)	114
6.3.2	Physico-chemical Analysis	116
6.3.2.1	Water Activity and Moisture Content	116
6.3.2.2	Solubility	118
6.3.2.3	Availability of Thymoquinone (TQ) using HPLC	118
6.3.2.4	Reconstituting properties (wettability and dispersibility)	120
6.3.2.5	Proximate analyses of Non-dairy Creamer (NDC) of <i>Nigella sativa</i> oil	120
6.3.3	Hygroscopicity	121
6.3.3.1	Bulk density	122
6.3.3.2	Particle Size of the Creamer Powders	122
6.3.3.3	Colour Determination of Creamer Powders	123
6.3.3.4	Particle Morphology by SEM	124
6.3.3.5	Glass transition by Differential Scanning Calorimetry (DSC)	125
6.3.3.6	Fourier Transform Infrared Spectroscopy (FTIR) Analysis	126
6.3.3.7	Coffee Creamer Evaluation (Colour, pH, and viscosity for the mixture of coffee and creamers)	127
6.3.3.8	Sensory Attributes of the Creamer Samples	129
6.4	Conclusion	130

<b>7</b>	<b>SUMMARY, CONCLUSION AND RECOMMENDATION FOR FUTURE RESEARCH</b>	131
7.1	Summary	131
7.2	Conclusion	132
7.3	Recommendation for Future Studies	132
	<b>REFERENCES</b>	134
	<b>APPENDICES</b>	163
	<b>BIODATA OF STUDENT</b>	169
	<b>LIST OF PUBLICATIONS</b>	170



## LIST OF TABLES

Table		Page
2.1	Taxonomic classification of <i>Nigella sativa</i>	5
2.2	Common Names of <i>Nigella sativa</i>	5
2.3	Recent published works for the antioxidant activity of <i>Nigella sativa</i> oil	12
2.4	Types of microencapsulation techniques	15
2.5	Microencapsulated oils from different oil sources by different encapsulation technique	17
2.6	Selected previous researches carried out on spray drying process	27
2.7	Selected wall materials used in spray drying process	29
2.8	Summary of some encapsulation techniques used by the food industry	32
2.9	Application of microencapsulated oil in several functional food products	33
2.10	Selected agglomeration conditions used in fluidized bed drying process.	37
3.1	Chemical properties of the selected <i>Nigella sativa</i> seed in comparison with previous studies	47
3.2	Oil physiochemical properties obtained using two extraction methods	48
3.3	GC–MS identification of chemical composition for <i>Nigella sativa</i> oil extracted by supercritical fluid and cold press	51
3.4	Antioxidant activities of <i>Nigella sativa</i> oil determined by DPPH radical scavenging and FRAP ferrous reducing, TPC total phenolic content with Thymoquinone content	54
3.5	The Main Peaks in the FTIR Spectrum of <i>Nigella sativa</i> oils extracted by supercritical fluid and cold press with their assignment	56
4.1	The experimental data for response surface analyzes of the effect of processing conditions on the MNSO	62
4.2	Droplet size $d_{43}$ ( $\mu\text{m}$ ), phase separation (24 h), and viscosity (Pa s) of emulsion produced with different initial solid and oil percentages	67
4.3	Matrix of the CCD cubic design and response variables (moisture content, solubility, MEE, particle size, DPPH, TPC) for MNSO	69

4.4	Parameters of regression coefficient of MNSO for non-reduced models	70
4.5	Parameters of regression coefficient of MNSO for reduced models	71
4.6	Experimental and fitted value of responses and <i>p</i> -value of validation (six-sample t-test)	83
5.1	Total oil (TO), surface oil (SO), and microencapsulation efficiency (MEE) of microencapsulated <i>Nigella sativa</i> oil (MNSO) under accelerated storage	91
5.2	Antioxidant activity (DPPH), total phenolic content (TPC), thymoquinone content (TQ) of uncapsulated (NSO) and microencapsulated (MNSO) under accelerated storage	93
5.3A	Relative percentage composition (%) of fatty acids in uncapsulated (NSO) under accelerated storage	94
5.3B	Relative percentage composition (%) of fatty acids in microencapsulated MNSO under accelerated storage	95
5.4	Color (Hunter Lab $L^* a^* b^*$ ) value MNSO for five different samples	101
6.1	Matrix of the central cubic design (CCD) and response variables. (moisture content, solubility, DPPH, TPC) for NDC	105
6.2	Parameters of regression coefficient of NDC for non-reduced models	111
6.3	Parameters of regression coefficient of NDC for reduced models	115
6.4	Experimental and fitted value of responses and <i>p</i> -value of validation (four-sample t-test)	116
6.5	Physical properties of agglomerated NDC produced from spray-dried <i>Nigella sativa</i> oil and two commercial creamers Moisture content ( $X_w$ ), Solubility ( $X_s$ ), water activity ( $a_w$ ), surface mean diameters ( $D_{[3,2]}$ ), volume mean diameter ( $D_{[4,3]}$ ), wettability	118
6.6	Proximate analyzes (% w/w, dry basis) of non-dairy creamer (NDC), commercial non-dairy creamer (C-NDC) and commercial dairy-creamer (C-DC)	121
6.7	Physical properties of mixed coffee with non-dairy creamer (NDC), commercial non-dairy creamer (C-NDC), commercial dairy-creamer (C-DC) and coffee	128

## LIST OF FIGURES

Figure	Page
2.1 <i>Nigella sativa</i> (whole Plant, flower, and seeds)	6
2.2 Structure of thymoquinone	9
2.3 Idealized Phase Diagram	11
2.4 Composition of an oil microcapsule in simplified form	14
2.5 Different types of microcapsules; (a) simple microcapsule, (b) matrix (microsphere), (c) irregular microcapsule, (d) multicore microcapsule, (e) multiwall microcapsule, and (f) assembly of microcapsule	14
2.6 Schematic representation of the spray-dryer. The dried powder is collected in a cyclone at the end	16
2.7 Schematic diagram of a typical laboratory freeze dryer	18
2.8 Phase diagram showing the critical point of freeze drying	19
2.9 Schematic diagram of a melt extruder	20
2.10 Schematic diagram of a spray chilling	21
2.11 Schematic representation of the microencapsulation process by spray-drying	24
2.12 Schematic representation of the particle formation in a fluid bed agglomeration	34
2.13 Schematic representation of the particle formation in a fluid bed agglomeration	36
3.1 Schematic representation of the cold-press machine	43
3.2 Supercritical Carbon Dioxide (SC-CO <sub>2</sub> ) Extraction Apparatus Source:(Herrero et al., 2010).	43
3.3 GC-MS chromatography analyzes of <i>Nigella sativa</i> oil extracted by; (A), supercritical fluid and (B), cold press	50
3.4 Thymoquinone concentration of <i>Nigella sativa</i> oil as determined by HPLC; (A), supercritical fluid extraction (B), cold press extraction and (C), Thymoquinone standard	53
3.5 FTIR spectra of <i>Nigella sativa</i> L. oil scanned at 4,000 - 650 cm <sup>-1</sup> ; (A), supercritical fluid and (B), cold press	55

3.6	Differential scanning calorimetry (DSC) thermal behavior of <i>Nigella sativa</i> oil; (A), supercritical fluid and (B), cold press	57
3.7	TGA and DTG curves of weight loss of <i>Nigella sativa</i> oil; (A), supercritical fluid and (B), cold press	58
4.1	Response surface for moisture content of the MNSO: with 10% oil concentration and 30% wall material	73
4.2	Response surface for solubility content of the MNSO: (A) with 10% oil concentration and 30% wall material (B) with 10% oil concentration and 160°C inlet air temperature	74
4.3	Response surface for MEE of the MNSO: (A) with 30% wall material and with 10% oil concentration (B) with 30% wall material and 160°C inlet air temperature (C) with 10% oil concentration and 160°C inlet air temperature	76
4.4	Response surface for Particle size of the MNSO: (A) with 30% wall material and 10% oil concentration (B) with 30% wall material and 160°C inlet air temperature (C) with 10% oil concentration and 160°C inlet air temperature	79
4.5	Response surface for TPC of the MNSO: (A) with 30% wall material and 160 °C inlet air temperature	81
4.6	Response optimizer for the optimum conditions of spray-dried MNSO as determined by RSM	82
4.7	Thymoquinone concentration (mg/mL) of extracted <i>Nigella sativa</i> oil of MNSO as determined by HPLC	84
4.8	Scanning electron micrographs of spray dried powder (A) internal microstructures, (B) external microstructure	85
5.1	Peroxide value of microencapsulated <i>Nigella sativa</i> oil (MNSO) and <i>Nigella sativa</i> oil (NSO) upon accelerated storage at 65°C	96
5.2	Micrographs of the surface topology of microencapsulated <i>Nigella sativa</i> oil (MNSO), internal (on the left) and external (on the right) micro structures of powders under accelerated storage: (A) day 0, (B) day 6, (C) day 12, (D) day 18 and (E) day 24	100
6.1	Schematic diagram of the fluidized bed drying	106
6.2	Three-dimensional (3D) response surface and response optimizer plots (A–E) showing the significant ( $p < 0.05$ ) interaction effect of fluidized bed drying variables on the moisture, solubility, DPPH and TPC	113
6.3	Thymoquinone (TQ) concentration (mg/mL) of <i>Nigella sativa</i> oil (A) agglomerated NDC, (B) spray dried powder as determined by HPLC	119

6.4	Droplets size distribution of spray dried of NSO (A), non-dairy creamer NDC of NSO (B) commercial dairy creamer (C), commercial non-dairy creamer (D)	123
6.5	Morphology of spray dried NSO (A), non-dairy creamer of NSO (B), commercial non-dairy creamer (C) and commercial dairy creamer (D)	125
6.6	Glass transition temperature ( $T_g$ ) of spray dried NSO (A), non-dairy creamer of NSO (B), commercial non-dairy creamer (C) and commercial dairy creamer (D)	126
6.7	FTIR of microencapsulated <i>Nigella sativa</i> oil (MNSO) before agglomeration and non-dairy creamer of <i>Nigella sativa</i> oil after agglomeration (NDC)	127
6.8	Coffee mixed with nondairy creamer (A), commercial nondairy creamers (B) and commercial dairy creamer (C)	128
6.9	Sensory scores of creamer samples (1 reflected very low preference and 9 very high preference)	130



## 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 <sub>50</sub>	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 <sub>g</sub>	Glass Transition Temperature
X <sub>1</sub>	Independent Variables
Y <sub>1</sub>	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<sup>-1</sup> and 90 mg kg<sup>-1</sup> 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 CO<sub>2</sub> 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.

## REFERENCES

- Abdullah, E., & Geldart, D. (1999). The use of bulk density measurements as flowability indicators. *Powder Technology*, 102(2), 151-165.
- Abedi, A.S., Rismanchi, M., Shahdoostkhany, M., Mohammadi, A., & Hosseini, H. (2016). Microencapsulation of *Nigella sativa* seeds oil containing thymoquinone by spray-drying for functional yogurt production. *International Journal of Food Science & Technology*, 51(10), 2280-2289.
- Abrahamsson, V., Rodriguez-Meizoso, I., & Turner, C. (2015). Supercritical fluid extraction of lipids from linseed with on-line evaporative light scattering detection. *Analytica Chimica Acta*, 853, 320-327.
- Adams, R.P. (2007). *Identification of essential oil components by gas chromatography/mass spectrometry*, 4<sup>th</sup> Ed. Allured publishing corporation. Carol Stream, USA
- Ahlatci, A., Kuzhan, A., Taysi, S., Demirtas, O.C., Alkis, H.E., Tarakcioglu, M., & Cinar, K. (2014). Radiation-modifying abilities of *Nigella sativa* and thymoquinone on radiation-induced nitrosative stress in the brain tissue. *Phytomedicine*, 21(5), 740-744.
- Ahmad, I., Muneer, K.M., Tamimi, I.A., Chang, M.E., Ata, M.O., & Yusuf, N. (2013). Thymoquinone suppresses metastasis of melanoma cells by inhibition of nlrp3 inflammasome. *Toxicology & Applied Pharmacology*, 270(1), 70-76.
- Ahmad, Z., Ghafoor, A., Ochatt, S., & Jain, S. (2007). *Nigella sativa*-a potential commodity in crop diversification traditionally used in healthcare. *Breeding of neglected and under-utilized crops, spices and herbs*, Eds: Ochatt, A. and S.M. Jain, Science Publishers, Enfield, NH, USA, pp. 215-230.
- Ahn, J.-H., Kim, Y.-P., Lee, Y.-M., Seo, E.-M., Lee, K.-W., & Kim, H.-S. (2008). Optimization of microencapsulation of seed oil by response surface methodology. *Food Chemistry*, 107(1), 98-105.
- Akanda, M.J.H., Sarker, M.Z.I., Ferdosh, S., Manap, M.Y.A., Ab Rahman, N.N.N., & Ab Kadir, M.O. (2012). Applications of supercritical fluid extraction (SFE) of palm oil and oil from natural sources. *Molecules*, 17(2), 1764-1794.
- Akhtar, M., Maikiyo, A.M., Khanam, R., Mujeeb, M., Aqil, M., & Najmi, A.K. (2012). Ameliorating effects of two extracts of *Nigella sativa* in middle cerebral artery occluded rat. *Journal of Pharmacy & Bioallied Sciences*, 4(1), 70.
- Al-Ali, A., Alkhawajah, A. A., Randhawa, M. A., & Shaikh, N. A. (2008). Oral and intraperitoneal LD50 of thymoquinone, an active principle of *Nigella sativa*, in mice and rats. *J Ayub Med Coll Abbottabad*, 20(2), 25-27

- Al-Bukhari, M., & Sahi, A.-B. (1976). The collection of authentic sayings of prophet mohammad (peace be upon him), division 71 on medicine, 2<sup>nd</sup> ed. *Hilal Yayinlari, Ankara, Turkey*.
- Al-Jishi, S., & Abuho Hozaifa, B. (2003). Effect of *Nigella sativa* on blood hemostatic function in rats. *Journal of Ethnopharmacology*, 85(1), 7-14.
- Al-Gaby, A. M. A. (1998). Amino acid composition and biological effects of supplementing broad bean and corn proteins with *Nigella sativa* (black cumin) cake protein. *Molecular Nutrition & Food Research*, 42(05), 290-294.
- Alemi, M., Sabouni, F., Sanjarian, F., Haghbeen, K., & Ansari, S. (2013). Anti-inflammatory effect of seeds and callus of *Nigella sativa* L. Extracts on mix glial cells with regard to their thymoquinone content. *Journal of the American Association of Pharmaceutical Scientists*, 14(1), 160-167.
- Alhosin, M., Ibrahim, A., Boukhari, A., Sharif, T., Gies, J.-P., Auger, C., & Schini-Kerth, V.B. (2012). Anti-neoplastic agent thymoquinone induces degradation of  $\alpha$  and  $\beta$  tubulin proteins in human cancer cells without affecting their level in normal human fibroblasts. *Investigational New Drugs*, 30(5), 1813-1819.
- Alves, A.I., Rodrigues, M.Z., Pinto, M.R.M.R., Vanzela, E.S.L., Stringheta, P.C., Perrone, I.T., & Ramos, A.M. (2017). Morphological characterization of pequi extract microencapsulated through spray drying. *International Journal of Food Properties*(just-accepted).
- Andreola, K., Butzge, J., da Silva, C., Kis, L., Rocha, S., & Taranto, O. (2015). *Effect of operating conditions on the agglomeration and drying of hydrolyzed collagen in a fluidized bed*. Paper presented at the First Nordic Baltic Drying Conference.
- Anwar, F., Rashid, U., Shahid, S.A., & Nadeem, M. (2014). Physicochemical and antioxidant characteristics of kapok (*Ceiba pentandra* Gaertn.) seed oil. *Journal of the American Oil Chemists' Society*, 91(6), 1047-1054.
- AOAC International. (2005). *Official methods of analyzes of AOAC international*: AOAC International.
- AOAC International (1995). *Official Methods of Analyzes*, 15th ed. Association of Official Analytical Chemist. Washington, DC.
- AOAC. (2000). *Official Methods of Analyzes of AOAC International (17th ed.)*. Maryland, USA.
- Arana-Sánchez, A., Estarrón-Espinosa, M., Obledo-Vázquez, E., Padilla-Camberos, E., Silva-Vázquez, R., & Lugo-Cervantes, E. (2010). Antimicrobial and antioxidant activities of mexican oregano essential oils (*Lippia graveolens*

HBK) with different composition when microencapsulated in $\beta$ -cyclodextrin. *Letters in Applied Microbiology*, 50(6), 585-590.

- Aris, N., Norhuda, I., & Adeib, I. (2013). Extraction of Phoenix Dactylifera (Mariami) seeds oil using supercritical carbon dioxide (SC-CO<sub>2</sub>). *International Journal*, 4(1).
- Arteaga, G., Li-Chan, E., Vazquez-Arteaga, M., & Nakai, S. (1994). Systematic experimental designs for product formula optimization. *Trends in Food Science & Technology*, 5(8), 243-254.
- Ashour, A.E., Abd-Allah, A.R., Korashy, H.M., Attia, S.M., Alzahrani, A.Z., Saquib, Q., & Rishi, A.K. (2014). Thymoquinone suppression of the human hepatocellular carcinoma cell growth involves inhibition of il-8 expression, elevated levels of trail receptors, oxidative stress and apoptosis. *Molecular & Cellular Biochemistry*, 389(1-2), 85-98.
- Ashraf, S.S., Rao, M.V., Kaneez, F.S., Qadri, S., Al-Marzouqi, A.H., Chandranath, I.S., & Adem, A. (2011). *Nigella sativa* extract as a potent antioxidant for petrochemical-induced oxidative stress. *Journal of Chromatographic Science*, 49(4), 321-326.
- Atta, M.B. (2003). Some characteristics of nigella (*Nigella sativa* L.) seed cultivated in Egypt and its lipid profile. *Food Chemistry*, 83(1), 63-68.
- Augustin, M.A., & Hemar, Y. (2009). Nano-and micro-structured assemblies for encapsulation of food ingredients. *Chemical Society Reviews*, 38(4), 902-912.
- Augustin, M.A., Sanguansri, L., & Bode, O. (2006). Maillard reaction products as encapsulants for fish oil powders. *Journal of Food Science*, 71(2), E25-E32.
- Aulton, M. E. (2013). Powder flow. *Pharmaceutics. The design and manufacture of medicines, 4th edn. Edinburgh: Churchill Livingstone*, 187-199.
- Avaltroni, F., Bouquerand, P., & Normand, V. (2004). Maltodextrin molecular weight distribution influence on the glass transition temperature and viscosity in aqueous solutions. *Carbohydrate Polymers*, 58(3), 323-334.
- Bae, E., & Lee, S. (2008). Microencapsulation of avocado oil by spray drying using whey protein and maltodextrin. *Journal of Microencapsulation*, 25(8), 549-560.
- Bakry, A.M., Abbas, S., Ali, B., Majeed, H., Abouelwafa, M.Y., Mousa, A., & Liang, L. (2016). Microencapsulation of oils: A comprehensive review of benefits, techniques, and applications. *Comprehensive Reviews in Food Science & Food Safety*, 15(1), 143-182.



- Baldwin, A.J., & Truong, G.N.T. (2007). Development of insolubility in dehydration of dairy milk powders. *Food & Bioprocess Technology*, 85(3), 202-208.
- Bansal, Vikas, Sharma, Harish Kumar, & Nanda, Vikas. (2014). Optimization of spray drying process parameters for low-fat honey-based milk powder with antioxidant activity. *International Journal of Food Science & Technology*, 49(4), 1196-1202.
- Barbosa-Cánovas, G., & Juliano, P. (2005). Physical and chemical properties of food powders. In *Onwulata C (ed) Encapsulated and powdered foods*. Taylor & Francis, New York, 39-71
- Bastioğlu, A.Z., Koç, M., Yalçın, B., Ertekin, F.K., & Ötleş, S. (2017). Storage characteristics of microencapsulated extra virgin olive oil powder: Physical and chemical properties. *Journal of Food Measurement & Characterization*, 1-17.
- Benelli, L., Cortés-Rojas, D.F., Souza, C.R.F., & Oliveira, W.P. (2015). Fluid bed drying and agglomeration of phytopharmaceutical compositions. *Powder Technology*, 273, 145-153.
- Benzie, I., & Strain, J. (1999). Ferric reducing/antioxidant power assay: Direct measure of total antioxidant activity of biological fluids and modified version for simultaneous measurement of total antioxidant power and ascorbic acid concentration. *Methods in Enzymology*(299), 15-27.
- Beristain, C., Azuara, E., & Vernon-Carter, E. (2002). Effect of water activity on the stability to oxidation of spray-dried encapsulated orange peel oil using mesquite gum (*Prosopis juliflora*) as wall material. *Journal of Food Science*, 67(1), 206-211.
- Bhandari, B.R., D'Arc, B.R., & Padukka, I. (1999). Encapsulation of lemon oil by paste method using  $\beta$ -cyclodextrin: Encapsulation efficiency and profile of oil volatiles. *Journal of Agricultural & Food Chemistry*, 47(12), 5194-5197.
- Binsi, P., Nayak, N., Sarkar, P., Jeyakumari, A., Ashraf, P.M., Ninan, G., & Ravishankar, C. (2017). Structural and oxidative stabilization of spray dried fish oil microencapsulates with gum Arabic and sage polyphenols: Characterization and release kinetics. *Food Chemistry*, 219, 158-168.
- Blois, M.S. (1958). Antioxidant determinations by the use of a stable free radical. *Nature*, 181, 1199-1200.
- Bourgou, S., Pichette, A., Marzouk, B., & Legault, J. (2010). Bioactivities of black cumin essential oil and its main terpenes from Tunisia. *South African Journal of Botany*, 76(2), 210-216.

- Bringas-Lantigua, M., Expósito-Molina, I., Reineccius, G.A., López-Hernández, O., & Pino, J.A. (2011). Influence of spray-dryer air temperatures on encapsulated mandarin oil. *Drying Technology*, 29(5), 520-526.
- Burits, M., & Bucar, F. (2000). Antioxidant activity of *Nigella sativa* essential oil. *Phytotherapy Research*, 14(5), 323-328.
- Bush, L., Stevenson, L., & Lane, K.E. (2017). The oxidative stability of omega-3 oil-in-water nanoemulsion systems suitable for functional food enrichment: A systematic review of the literature. *Critical Reviews in Food Science & Nutrition* (just-accepted), 00-00.
- Butt, M.S., & Sultan, M.T. (2010). *Nigella sativa*: Reduces the risk of various maladies. *Critical Reviews in Food Science & Nutrition*, 50(7), 654-665.
- Cai, Y., & Corke, H. (2000). Production and properties of spray-dried amaranthus betacyanin pigments. *Journal of Food Science*, 65(7), 1248-1252.
- Cal, K., & Sollohub, K. (2010). Spray drying technique. I: Hardware and process parameters. *Journal of Pharmaceutical Sciences*, 99(2), 575-586.
- Calvo, P., Castaño, Á.L., Hernández, M.T., & González-Gómez, D. (2011). Effects of microcapsule constitution on the quality of microencapsulated walnut oil. *European Journal of Lipid Science & Technology*, 113(10), 1273-1280.
- Calvo, P., Castaño, Á.L., Lozano, M., & González-Gómez, D. (2012). Influence of the microencapsulation on the quality parameters and shelf-life of extra-virgin olive oil encapsulated in the presence of bht and different capsule wall components. *Food Research International*, 45(1), 256-261.
- Calvo, P., Hernández, T., Lozano, M., & González-Gómez, D. (2010). Microencapsulation of extra-virgin olive oil by spray-drying: Influence of wall material and olive quality. *European Journal of Lipid Science & Technology*, 112(8), 852-858.
- Campbell, I.J., Lips, A., & Morley, W.G. (1994). *U.S. Patent No. 5,290,581*. Washington, DC: U.S. Patent and Trademark Office.
- Cano-Chauca, Milton, Stringheta, PC, Ramos, AM, & Cal-Vidal, J. (2005). Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innovative Food Science & Emerging Technologies*, 6(4), 420-428.
- Carneiro, H.C., Tonon, R.V., Grosso, C.R., & Hubinger, M.D. (2013). Encapsulation efficiency and oxidative stability of flaxseed oil microencapsulated by spray drying using different combinations of wall materials. *Journal of Food Engineering*, 115(4), 443-451.

- Celli, G.B., Ghanem, A., & Brooks, M.S. (2015). Bioactive encapsulated powders for functional foods—a review of methods and current limitations. *Food & Bioprocess Technology*, 8(9), 1825-1837.
- Cheikh-Rouhou, S., Besbes, S., Hentati, B., Blecker, C., Deroanne, C., & Attia, H. (2007). *Nigella sativa* L.: Chemical composition and physicochemical characteristics of lipid fraction. *Food Chemistry*, 101(2), 673-681.
- Chen, C., Shao, Y., Tao, Y., & Wen, H. (2015). Optimization of dynamic microwave-assisted extraction of *Armillaria polysaccharides* using RSM, and their biological activity. *LWT-Food Science and Technology*, 64(2), 1263-1269.
- Chen, X.D., & Patel, K.C. (2008). Manufacturing better quality food powders from spray drying and subsequent treatments. *Drying Technology*, 26(11), 1313-1318.
- Chevallier, A. (1996). *The Encyclopedia of Medicinal Plants*. Dorling Kindersley Limited, London (1996) pp. 336.
- Chiou, D., & Langrish, T. (2007). Crystallization of amorphous components in spray-dried powders. *Drying Technology*, 25(9), 1427-1435.
- Cho Ping, N., Hashim, N.H., & Hasan Adli, D.S. (2014). Effects of *Nigella sativa* (habbatus sauda) oil and nicotine chronic treatments on sperm parameters and testis histological features of rats. *Evidence-Based Complementary & Alternative Medicine*, 2014.
- Chung, M.S., Ruan, R., Chen, P., Chung, S.H., Ahn, T.H., & Lee, K.H. (2000). Study of caking in powdered foods using nuclear magnetic resonance spectroscopy. *Journal of Food Science*, 65(1), 134-138.
- Chung, S.K., Seo, J.Y., Lim, J.H., Park, H.H., Yea, M.J., & Park, H.J. (2013). Microencapsulation of essential oil for insect repellent in food packaging system. *Journal of Food Science*, 78(5), E709-E714.
- Cocks, L.V., & van Rede, C. (1966). *Laboratory handbook for oil and fat analyzts*. Academic Press, London.
- Codex, A. C. (1982). Recommended internal standard for edible fats and oils. *Edn, 1*, 1-179.
- Coronel-Aguilera, C.P., & San Martín-González, M.F. (2015). Encapsulation of spray dried  $\beta$ -carotene emulsion by fluidized bed coating technology. *LWT-Food Science & Technology*, 62(1), 187-193.
- da Silva, F.C., da Fonseca, C.R., de Alencar, S.M., Thomazini, M., de Carvalho Balieiro, J.C., Pittia, P., & Favaro-Trindade, C.S. (2013). Assessment of production efficiency, physicochemical properties and storage stability of

spray-dried propolis, a natural food additive, using gum arabic and osa starch-based carrier systems. *Food & Bioproducts Processing*, 91(1), 28-36.

- da Silva, Felipe C, Favaro-Trindade, Carmen S, de Alencar, Severino M, Thomazini, Marcelo, & Balieiro, Julio C C. (2011). Physicochemical properties, antioxidant activity and stability of spray-dried propolis. *Journal of ApiProduct & ApiMedical Science*, 3(2), 94-100.
- Dacanal, G., Hirata, T., & Menegalli, F. (2013). Fluid dynamics and morphological characterization of soy protein isolate particles obtained by agglomeration in pulsed-fluid bed. *Powder Technology*, 247, 222-230.
- Dacanal, G., & Menegalli, F. (2010). Selection of operational parameters for the production of instant soy protein isolate by pulsed fluid bed agglomeration. *Powder Technology*, 203(3), 565-573.
- Daud, W.R.W. (2008). Fluidized bed dryers—Recent advances. *Advanced Powder Technology*, 19(5), 403-418.
- Daukšas, E., Venskutonis, P., Povilaityte, V., & Sivik, B. (2001). Rapid screening of antioxidant activity of sage (*Salvia officinalis* L.) extracts obtained by supercritical carbon dioxide at different extraction conditions. *Food/Nahrung*, 45(5), 338-341.
- de Barros Fernandes, R.V., Marques, G.R., Borges, S.V., & Botrel, D.A. (2014). Effect of solids content and oil load on the microencapsulation process of rosemary essential oil. *Industrial Crops & Products*, 58, 173-181.
- Descamps, N., Palzer, S., Roos, Y.H., & Fitzpatrick, J.J. (2013). Glass transition and flowability/caking behavior of maltodextrin de 21. *Journal of Food Engineering*, 119(4), 809-813.
- Dhanalakshmi, K., Ghosal, S., & Bhattacharya, S. (2011). Agglomeration of food powder and applications. *Critical Review in Food Science & Nutrition*, 51(5), 432-441.
- Dobry, Dan E, Settell, Dana M, Baumann, John M, Ray, Rod J, Graham, Lisa J, & Beyerinck, Ron A. (2009). A model-based methodology for spray-drying process development. *Journal of Pharmaceutical Innovation*, 4(3), 133-142.
- Domian, E., Sulek, A., Cenkier, J., & Kerschke, A. (2014). Influence of agglomeration on physical characteristics and oxidative stability of spray-dried oil powder with milk protein and trehalose wall material. *Journal of Food Engineering*, 125, 34-43.
- Drusch, S. (2007). Sugar beet pectin: A novel emulsifying wall component for microencapsulation of lipophilic food ingredients by spray-drying. *Food Hydrocolloids*, 21(7), 1223-1228.

- Dweck, J., & Sampaio, C. M. S. (2004). Analyzes of the thermal decomposition of commercial vegetable oils in air by simultaneous TG/DTA. *Journal of thermal analyzes and calorimetry*, 75(2), 385-391.
- Edris, A.E., Kalembe, D., Adamiec, J., & Piątkowski, M. (2016). Microencapsulation of *Nigella sativa* oleoresin by spray drying for food and nutraceutical applications. *Food Chemistry*, 204, 326-333.
- El-Dakhakhny, M. (1963). Studies on the chemical constitution of Egyptian *Nigella sativa*. *Planta Medica*, 11(04), 465-470.
- El-Khouly, D., El-Bakly, W.M., Awad, A.S., El-Mesallamy, H.O., & El-Demerdash, E. (2012). Thymoquinone blocks lung injury and fibrosis by attenuating bleomycin-induced oxidative stress and activation of nuclear factor kappa-b in rats. *Toxicology*, 302(2), 106-113.
- Ellinger, R.H. (1972). *Phosphates as food ingredients*. Cleveland, Ohio, CRC Press.
- Ersus, Seda, & Yurdagel, Unal. (2007). Microencapsulation of anthocyanin pigments of black carrot (*Daucus carota* L.) by spray drier. *Journal of Food Engineering*, 80(3), 805-812.
- FAO. (2015). *FAO Statistical Pocketbook Coffee 2015*. Food and Agriculture Organization of the United Nations, Rome, p 16.
- Fäldt, P., & Bergenståhl, B. (1995). Fat encapsulation in spray-dried food powders. *Journal of the American Oil Chemists' Society*, 72(2), 171-176.
- Fang, Z., & Bhandari, B. (2010). Encapsulation of polyphenols—A review. *Trends in Food Science & Technology*, 21(10), 510-523.
- Fang, Z., & Bhandari, B. (2012). Spray drying, freeze drying and related processes for food ingredient and nutraceutical encapsulation. *Encapsulation technologies and delivery systems for food ingredients and nutraceuticals*, WP Woodhead Publishing, Oxford, pp. 73-109.
- Fernandes, L. P, Turatti, Izabel C.C. Lopes, N, P. Ferreira, J, C, Candido, R, C. & Oliveira, W, P. (2008). Volatile retention and antifungal properties of spray-dried microparticles of *Lippia sidoides* essential oil. *Drying Technology*, 26(12), 1534-1542.
- Fernandes, R.V.D.B., Borges, S.V., & Botrel, D.A. (2013). Influence of spray drying operating conditions on microencapsulated rosemary essential oil properties. *Food Science & Technology (Campinas)*, 33, 171-178.
- Fernandes, R. V. D. B., Botrel, D. A., Silva, E. K., Pereira, C. G., Carmo, E. L. D., Dessimoni, A. L. D. A., & Borges, S. V. (2017a). Microencapsulated ginger

oil properties: Influence of operating parameters. *Drying Technology*, 35(9), 1098-1107.

- Fernandes, R. V. D. B., Guimarães, I. C., Ferreira, C. L. R., Botrel, D. A., Borges, S. V., & Souza, A. U. (2017b). Microencapsulated Rosemary (*Rosmarinus officinalis*) Essential Oil as a Biopreservative in Minas Frescal Cheese. *Journal of food processing and preservation*, 41(1).
- Ferreira, C.D., da Conceição, E.J.L., Machado, B.A.S., Hermes, V.S., de Oliveira Rios, A., Druzian, J.I., & Nunes, I.L. (2016). Physicochemical characterization and oxidative stability of microencapsulated crude palm oil by spray drying. *Food & Bioprocess Technology*, 9(1), 124-136.
- Filkova, I., & Mujumdar, A.S. (1995). Industrial spray drying systems. *Handbook of industrial drying*, Majumdar AS (ed), Marcel Dekkar Inc, New York, p 263–307
- Finco, A.M.D.O., Mamani, L.D.G., Carvalho, J.C.D., de Melo Pereira, G.V., Thomaz-Soccol, V., & Soccol, C.R. (2017). Technological trends and market perspectives for production of microbial oils rich in omega-3. *Critical Reviews in Biotechnology*, 37(5), 656-671.
- Fitzpatrick, J., Hodnett, M., Twomey, M., Cerqueira, P., O'Flynn, J., & Roos, Y. (2007). Glass transition and the flowability and caking of powders containing amorphous lactose. *Powder Technology*, 178(2), 119-128.
- Forny, L., Marabi, A., & Palzer, S. (2011). Wetting, disintegration and dissolution of agglomerated water soluble powders. *Powder Technology*, 206(1), 72-78.
- Frascareli, E., Silva, V., Tonon, R., & Hubinger, M. (2012). Effect of process conditions on the microencapsulation of coffee oil by spray drying. *Food & Bioproducts Processing*, 90(3), 413-424.
- Fries, L., Antonyuk, S., Heinrich, S., Niederreiter, G., & Palzer, S. (2014). Product design based on discrete particle modeling of a fluidized bed granulator. *Particuology*, 12, 13-24.
- Fuchs, M., Turchiuli, C., Bohin, M., Cuvelier, M., Ordonnaud, C., Peyrat-Maillard, M., & Dumoulin, E. (2006). Encapsulation of oil in powder using spray drying and fluidised bed agglomeration. *Journal of Food Engineering*, 75(1), 27-35.
- Gallardo, G., Guida, L., Martinez, V., López, M.C., Bernhardt, D., Blasco, R., & Hermida, L.G. (2013). Microencapsulation of linseed oil by spray drying for functional food application. *Food Research International*, 52(2), 473-482.
- Gamboa, O.D., Gonçalves, L.G., & Grosso, C.F. (2011). Microencapsulation of tocopherols in lipid matrix by spray chilling method. *Procedia Food Science*, 1, 1732-1739.

- Gardiner, D.S. (1977). *U.S. Patent No. 4,046,926*. Washington, DC: U.S. Patent and Trademark Office.
- Ghafoor, K., Park, J., & Choi, Y.-H. (2010). Optimization of supercritical fluid extraction of bioactive compounds from grape (*Vitis labrusca* B.) peel by using response surface methodology. *Innovative Food Science & Emerging Technologies*, 11(3), 485-490.
- Gharsallaoui, A., Roudaut, G., Chambin, O., Voilley, A., & Saurel, R. (2007). Applications of spray-drying in microencapsulation of food ingredients: An overview. *Food Research International*, 40(9), 1107-1121.
- Ghasemzadeh, A., Jaafar, H.Z., & Rahmat, A. (2010). Antioxidant activities, total phenolics and flavonoids content in two varieties of Malaysia young ginger (*zingiber officinale roscoe*). *Molecules*, 15(6), 4324-4333.
- Gholamnezhad, Z., Keyhanmanesh, R., & Boskabady, M.H. (2015). Anti-inflammatory, antioxidant, and immunomodulatory aspects of *Nigella sativa* for its preventive and bronchodilatory effects on obstructive respiratory diseases: A review of basic and clinical evidence. *Journal of Functional Foods*, 17, 910-927.
- Ghosheh, O.A., Houdi, A.A., & Crooks, P.A. (1999). High performance liquid chromatographic analyzes of the pharmacologically active quinones and related compounds in the oil of the black seed (*Nigella sativa* L.). *Journal of Pharmaceutical & Biomedical Analyzes*, 19(5), 757-762.
- Ghosheh, O.A., Houdi, A.A., & Crooks, P.A. (1999). High performance liquid chromatographic analyzes of the pharmacologically active quinones and related compounds in the oil of the black seed (*Nigella sativa* L.). *Journal of Pharmaceutical & Biomedical Analyzes*, 19(5), 757-762.
- Gianfrancesco, A., Turchiuli, C., & Dumoulin, E. (2008). Powder agglomeration during the spray-drying process: Measurements of air properties. *Dairy Science & Technology*, 88(1), 53-64.
- Gibbs, Selim Kermasha, Inteaz Alli, Catherine N. Mulligan, B. (1999). Encapsulation in the food industry: a review. *International journal of food sciences and nutrition*, 50(3), 213-224.
- Gloria, H., & Aguilera, J.M. (1998). Assessment of the quality of heated oils by differential scanning calorimetry. *Journal of Agricultural & Food Chemistry*, 46(4), 1363-1368.
- Gökmen, V., Mogol, B.A., Lumaga, R.B., Fogliano, V., Kaplun, Z., & Shimoni, E. (2011). Development of functional bread containing nanoencapsulated omega-3 fatty acids. *Journal of Food Engineering*, 105(4), 585-591.

- Gora, J., Lis, A., Kula, J., Staniszevska, M., & Wołoszyn, A. (2002). Chemical composition variability of essential oils in the ontogenesis of some plants. *Flavor and Fragrance Journal*, 17(6), 445-451.
- Goreja, W. (2003). *Black seed: Nature's miracle remedy*: Amazing Herbs Press, New York, NY.
- Gouin, S. (2004). Microencapsulation: Industrial appraisal of existing technologies and trends. *Trends in Food Science & Technology*, 15(7), 330-347.
- Goula, A.M., & Adamopoulos, K.G. (2005). Spray drying of tomato pulp in dehumidified air: Ii. The effect on powder properties. *Journal of Food Engineering*, 66(1), 35-42.
- Goula, A.M., & Adamopoulos, K.G. (2008). Effect of maltodextrin addition during spray drying of tomato pulp in dehumidified air: In. Powder properties. *Drying Technology*, 26(6), 726-737.
- Guadarrama-Lezama, A. Y., Dorantes-Alvarez, L., Jaramillo-Flores, M. E., Pérez-Alonso, C., Niranjana, K., Gutiérrez-López, G. F., & Alamilla-Beltrán, L. (2012). Preparation and characterization of non-aqueous extracts from chilli (*Capsicum annum* L.) and their microencapsulates obtained by spray-drying. *Journal of Food Engineering*, 112(1), 29-37.
- Gupta, P.K., Jadhav, S.B., & Singhal, R.S. (2015). Development of shrikhand premix using microencapsulated rice bran oil as fat alternative and hydrocolloids as texture modifier. *Food Hydrocolloids*, 48, 220-227.
- Hadad, G.M., Abdel Salam, R.A., Soliman, R.M., & Mesbah, M.K. (2012). High-performance liquid chromatography quantification of principal antioxidants in black seed (*Nigella sativa* L.) phytopharmaceuticals. *Journal of AOAC International*, 95(4), 1043-1047.
- Hadnađev, M., Hadnađev, T.D., Torbica, A., Dokić, L., Pajin, B., & Krstonošić, V. (2011). Rheological properties of maltodextrin based fat-reduced confectionery spread systems. *Procedia Food Science*, 1, 62-67.
- Hamilton, R., Kalu, C., McNeill, G., Padley, F., & Pierce, J. (1998). Effects of tocopherols, ascorbyl palmitate, and lecithin on autoxidation of fish oil. *Journal of the American Oil Chemists' Society*, 75(7), 813-822.
- Hardas, N., Danviriyakul, S., Foley, J., Nawar, W., & Chinachoti, P. (2000). Accelerated stability studies of microencapsulated anhydrous milk fat. *LWT-Food Science and Technology*, 33(7), 506-513.
- Hedayatnia, S., Mirhosseini, H., Amid, B.T., Sarker, Z.I., Veličkovska, S.K., & Karim, R. (2016a). Effect of different fat replacers and drying methods on thermal



behavior, morphology and sensory attributes of reduced-fat coffee creamer. *LWT-Food Science & Technology*, 72, 330-342.

- Hedayatnia, S., Mirhosseini, H., Tamnak, S., & Golpira, F. (2016b). Improvement of glass transition and flowability of reduced-fat coffee creamer: Effect of fat replacer and fluidized bed drying. *Food & Bioprocess Technology*, 9(4), 686-698.
- Hedges, A., & McBride, C. (1999). Utilization of  $\beta$ -cyclodextrin in food. *Cereal Foods World*, 44(10), 700-704.
- Hee, Y.Y., Tan, C.P., Abdul Rahman, R., Mohd Adzahan, N., Lai, W.T., & Chong, G.H. (2015). Influence of different wall materials on the microencapsulation of virgin coconut oil by spray drying. *International Journal of Food Engineering*, 11(1), 61-69.
- Herbst, S. (1995). Wang. 1995. Encyclopedia: London: Barron's Educational Series.
- Herkes, J., Grubinger, V., Schumacher, J. & Thompson, J. (2015). Mechanical extraction processing technology for biodiesel. Retrieved from <http://articles.extension.org/pages/26911/mechanical-extraction-processing-technology-for-biodiesel>.
- Herrero, M., Mendiola, J. A., Cifuentes, A., & Ibáñez, E. (2010). Supercritical fluid extraction: recent advances and applications. *Journal of Chromatography A*, 1217(16), 2495-2511.
- Hirata, T., Dacanal, G., & Menegalli, F. (2013). Effect of operational conditions on the properties of pectin powder agglomerated in pulsed fluid bed. *Powder Technology*, 245, 174-181.
- Hogan, S.A., McNamee, B.F., O'Riordan, E.D., & O'Sullivan, M. (2001). Emulsification and microencapsulation properties of sodium caseinate/carbohydrate blends. *International Dairy Journal*, 11(3), 137-144.
- Hogekamp, S., & Schubert, H. (1999). Agglomeration and agglomerator systems. *Wiley Encyclopedia of Food Science and Technology*, Wiley & Sons, New York, 13-18.
- Hooda, S., & Jood, S. (2005). Organoleptic and nutritional evaluation of wheat biscuits supplemented with untreated and treated fenugreek flour. *Food Chemistry*, 90(3), 427-435.
- Hosseinnia, M., Khaledabad, M.A., & Almasi, H. (2017). Optimization of *Ziziphora clinopodiodes* essential oil microencapsulation by whey protein isolate and pectin: A comparative study. *International Journal of Biological Macromolecules*, 101, 958-966.

- Huidong, Z.J.L.Y.L., & Yuanchang, J. (2005). The enhancement and encapsulation of agaricus bisporus flavor. *Journal of Chinese Institute of Food Science & Technology*, 4, 026.
- Huynh, T.V., Caffin, N., Dykes, G.A., & Bhandari, B. (2008). Optimization of the microencapsulation of lemon myrtle oil using response surface methodology. *Drying Technology*, 26(3), 357-368.
- Islam, M., Kitamura, Y., Yamano, Y., & Kitamura, M. (2016). Effect of vacuum spray drying on the physicochemical properties, water sorption and glass transition phenomenon of orange juice powder. *Journal of Food Engineering*, 169, 131-140.
- Ismail, M., Al-Naqeeb, G., Chan, K. W., & Adnan, R. N. E. (2013). *U.S. Patent No. 8,501,250*. Washington, DC: U.S. Patent and Trademark Office.
- Ismail, M., Al-Naqeeb, G., & Chan, K.W. (2010). *Nigella sativa* thymoquinone-rich fraction greatly improves plasma antioxidant capacity and expression of antioxidant genes in hypercholesterolemic rats. *Free Radical Biology & Medicine*, 48(5), 664-672.
- Ixtaina, V.Y., Vega, A., Nolasco, S.M., Tomás, M.C., Gimeno, M., Bárzana, E., & Tecante, A. (2010). Supercritical carbon dioxide extraction of oil from mexican chia seed (*Salvia hispanica* L.): Characterization and process optimization. *The Journal of Supercritical Fluids*, 55(1), 192-199.
- Jafari, S.M., Assadpoor, E., He, Y., & Bhandari, B. (2008). Encapsulation efficiency of food flavors and oils during spray drying. *Drying Technology*, 26(7), 816-835.
- Janfaza, S., & Janfaza, E. (2012). The study of pharmacologic and medicinal valuation of thymoquinone of oil of *Nigella sativa* in the treatment of diseases. *Annals of Biological Research*, 3, 1953-1957.
- Jaya, S, & Das, H. (2004). Effect of maltodextrin, glycerol monostearate and tricalcium phosphate on vacuum dried mango powder properties. *Journal of Food Engineering*, 63(2), 125-134.
- Jin, Y., Perrie, C., Zhang, W., Van Diepen, C., Curtis, J., & Barrow, C.J. (2007). Microencapsulation of marine lipids as a vehicle for functional food delivery. *Marine nutraceuticals and functional foods*, 115-155.
- Jinapong, N., Suphantharika, M., & Jamnong, P. (2008). Production of instant soymilk powders by ultrafiltration, spray drying and fluidized bed agglomeration. *Journal of Food Engineering*, 84(2), 194-205.
- Jones, M.G., Kimsey, I.M., Morrison, A., Wong, J.Y., & Heteren, J.V. (1994). *U.S. Patent No. 5,336,514*. Washington, DC: U.S. Patent & Trademark Office.

- Jrah Harzallah, H., Kouidhi, B., Flamini, G., Bakhrouf, A., & Mahjoub, T. (2011). Chemical composition, antimicrobial potential against cariogenic bacteria and cytotoxic activity of Tunisian *Nigella sativa* essential oil and thymoquinone. *Food Chemistry*, 129(4), 1469-1474.
- Jyothi, N.V.N., Prasanna, P.M., Sakarkar, S.N., Prabha, K.S., Ramaiah, P.S., & Srawan, G. (2010). Microencapsulation techniques, factors influencing encapsulation efficiency. *Journal of Microencapsulation*, 27(3), 187-197.
- Kagami, Y., Sugimura, S., Fujishima, N., Matsuda, K., Kometani, T., & Matsumura, Y. (2003). Oxidative stability, structure, and physical characteristics of microcapsules formed by spray drying of fish oil with protein and dextrin wall materials. *Journal of Food Science*, 68(7), 2248-2255.
- Kanazawa, A., Sawa, T., Akaike, T., & Maeda, H. (2002). Dietary lipid peroxidation products and DNA damage in colon carcinogenesis. *European Journal of Lipid Science & Technology*, 104(7), 439-447.
- Karlsson, S., Rasmuson, A., Björn, I.N., & Schantz, S. (2011). Characterization and mathematical modelling of single fluidised particle coating. *Powder Technology*, 207(1), 245-256.
- Kaushik, P., Dowling, K., Barrow, C. J., & Adhikari, B. (2015). Microencapsulation of omega-3 fatty acids: A review of microencapsulation and characterization methods. *Journal of functional foods*, 19, 868-881.
- Kazemi, M. (2015). Chemical composition and antioxidant properties of the essential oil of *Nigella sativa* L. *Bangladesh Journal of Botany*, 44(1), 111-116.
- Kelly, P., Oldfield, D., & O'kenedy, B. (1999). The thermostability of spray dried imitation coffee whiteners. *International Journal of Dairy Technology*, 52(3), 107-113.
- Kha, T.C., Nguyen, M.H., Roach, P.D., & Stathopoulos, C.E. (2014). Microencapsulation of gac oil by spray drying: Optimization of wall material concentration and oil load using response surface methodology. *Drying Technology*, 32(4), 385-397.
- Khan, M.A. (1999). Chemical composition and medicinal properties of *Nigella sativa* Linn. *Inflammopharmacology*, 7(1), 15-35.
- Khan, R.S., Grigor, J., Winger, R., & Win, A. (2013). Functional food product development—opportunities and challenges for food manufacturers. *Trends in Food Science & Technology*, 30(1), 27-37.
- Khare, C. (2004). Encyclopedia of Indian medicinal plants. *New York: Springer-Verlag*.

- Khazdair, M.R. (2015). The protective effects of *Nigella sativa* and its constituents on induced neurotoxicity. *Journal of Toxicology*, 1–7.
- Khoddami, A., Ghazali, H.M., Yassoralipour, A., Ramakrishnan, Y., & Ganjloo, A. (2011). Physicochemical characteristics of nigella seed (*Nigella sativa* L.) oil as affected by different extraction methods. *Journal of the American Oil Chemists' Society*, 88(4), 533-540.
- Kim, T. S., Kim, J.Y., Kim, K. H., Lee, S., Choi, D., Choi, I. G., & Choi, J.W. (2012). The effect of storage duration on bio-oil properties. *Journal of Analytical & Applied Pyrolysis*, 95, 118-125.
- Kiralan, M., Özkan, G., Bayrak, A., & Ramadan, M.F. (2014). Physicochemical properties and stability of black cumin (*Nigella sativa*) seed oil as affected by different extraction methods. *Industrial Crops & Products*, 57, 52-58.
- Kiralan, M., Ulaş, M., Özyaydin, A., Özdemir, N., Özkan, G., Bayrak, A., & Ramadan, M.F. (2016). Blends of cold pressed black cumin oil and sunflower oil with improved stability: A study based on changes in the levels of volatiles, tocopherols and thymoquinone during accelerated oxidation conditions. *Journal of Food Biochemistry*, 41(1).
- Kolanowski, W., Laufenberg, G., & Kunz, B. (2004). Fish oil stabilisation by microencapsulation with modified cellulose. *International Journal of Food Sciences and Nutrition*, 55(4), 333-343.
- Kolanowski, W., & Laufenberg, G. (2006). Enrichment of food products with polyunsaturated fatty acids by fish oil addition. *European Food Research & Technology*, 222(3-4), 472-477.
- Kosikowski, F.V., & Jimenez-Flores, R. (1987). *U.S. Patent No. 4,689,245*. Washington, DC: U.S. Patent & Trademark Office.
- Krupa, H., & Patel, H. (2011). Synergy of dairy with non-dairy ingredients or product: A review. *African Journal of Food Science*, 5(16), 817-832.
- Langa, E., Cacho, J., Palavra, A., Burillo, J., Mainar, A., & Urieta, J. (2009). The evolution of hyssop oil composition in the supercritical extraction curve: Modelling of the oil extraction process. *The Journal of Supercritical Fluids*, 49(1), 37-44.
- Lee, E., & Choe, E. (2012). Changes in oxidation-derived off-flavor compounds of roasted sesame oil during accelerated storage in the dark. *Biocatalysis & Agricultural Biotechnology*, 1(1), 89-93.
- Lim, H.K., Tan, C.P., Bakar, J., & Ng, S.P. (2012). Effects of different wall materials on the physicochemical properties and oxidative stability of spray-dried

microencapsulated red-fleshed pitaya (*Hylocereus polyrhizus*) seed oil. *Food & Bioprocess Technology*, 5(4), 1220-1227.

Liolios, C.C, Gortzi, O., Lalas, S., Tsaknis, J., & Chinou, I. (2009). Liposomal incorporation of carvacrol and thymol isolated from the essential oil of *Origanum dictamnus* L. and in vitro antimicrobial activity. *Food Chemistry*, 112(1), 77-83.

Liu, H., Wang, L., Yang, T., Zhang, G., Huang, J., Sun, J., & Huo, J. (2016). Optimization and evaluation of fish oil microcapsules. *Particuology*, 29, 162-168.

Liu, X.-D., Atarashi, T., Furuta, T., Yoshii, H., Aishima, S., Ohkawara, M., & Linko, P. (2001). Microencapsulation of emulsified hydrophobic flavors by spray drying. *Drying Technology*, 19(7), 1361-1374.

Lloyd, R.J., Dong Chen, X., & Hargreaves, J.B. (1996). Glass transition and caking of spray-dried lactose. *International Journal of Food Science & Technology*, 31(4), 305-311.

Lutterodt, H., Luther, M., Slavin, M., Yin, J. J., Parry, J., Gao, J. M., & Yu, L.L. (2010). Fatty acid profile, thymoquinone content, oxidative stability, and antioxidant properties of cold-pressed black cumin seed oils. *LWT-Food Science & Technology*, 43 (9), 1409-1413.

LutLutterodt, H., Slavin, M., Whent, M., Turner, E., & Yu, L.L. (2011). Fatty acid composition, oxidative stability, antioxidant and antiproliferative properties of selected cold-pressed grape seed oils and flours. *Food Chemistry*, 128(2), 391-399.

Machado, V., Hirata, T., & Menegalli, F. (2014). Agglomeration of soy protein isolate in a pulsed fluidized bed: Experimental study and process optimization. *Powder Technology*, 254, 248-255.

Machmudah, S., Shiramizu, Y., Goto, M., Sasaki, M., & Hirose, T. (2005). Extraction of *Nigella sativa* L. Using supercritical co<sub>2</sub>: A study of antioxidant activity of the extract. *Separation Science & Technology*, 40(6), 1267-1275.

Madene, A., Jacquot, M., Scher, J., & Desobry, S. (2006). Flavor encapsulation and controlled release—a review. *International Journal of Food Science & Technology*, 41(1), 1-21.

Mannan, A., & Kahvic, M. (2010). Ibn sina: A tribute. *The Gulf Journal of Oncology* (7), 60-63.

McClements, D.J. (2011). Edible nanoemulsions: Fabrication, properties, and functional performance. *Soft Matter*, 7(6), 2297-2316.

- Meste, M.L., Champion, D., Roudaut, G., Blond, G., & Simatos, D. (2002). Glass transition and food technology: A critical appraisal. *Journal of Food Science*, 67 (7), 2444-2458.
- Mirhosseini, H., Tan, C.P., Hamid, N.S., Yusof, S., & Chern, B.H. (2009). Characterization of the influence of main emulsion components on the physicochemical properties of orange beverage emulsion using response surface methodology. *Food Hydrocolloids*, 23(2), 271-280.
- Mirhosseini, H., Tan, C.P., Hamid, N.S.A., & Yusof, S. (2007). Modeling the relationship between the main emulsion components and stability, viscosity, fluid behavior,  $\zeta$ -potential, and electrophoretic mobility of orange beverage emulsion using response surface methodology. *Journal of Agricultural & Food Chemistry*, 55(19), 7659-7666.
- Mohideen, F.W., Stine, J., Bechtel, P.J., Solval, K.M., Bankston, J.D., & Sathivel, S. (2015). Effects of blueberry (*Vaccinium corymbosum*) juice on lipid oxidation during spray drying of microencapsulated menhaden oil. *International Journal of Food Properties*, 18(5), 1139-1153.
- Mollet, B., & Rowland, I. (2002). Functional foods: At the frontier between food and pharma: *Current Opinion in Biotechnology*, 13, 483-485.
- Montgomery, D.C. (2017). *Design and analyzes of experiments*: John Wiley & Sons.
- Morales-Medina, R., Tamm, F., Guadix, A., Guadix, E., & Drusch, S. (2016). Functional and antioxidant properties of hydrolysates of sardine (*S. Pilchardus*) and horse mackerel (*T. mediterraneus*) for the microencapsulation of fish oil by spray-drying. *Food Chemistry*, 194, 1208-1216.
- Mounir, S., & Allaf, K. (2008). Three-stage spray drying: New process involving instant controlled pressure drop. *Drying Technology*, 26(4), 452-463.
- Mozafari, M.R., Khosravi-Darani, K., Borazan, G.G., Cui, J., Pardakhty, A., & Yurdugul, S. (2008). Encapsulation of food ingredients using nanoliposome technology. *International Journal of Food Properties*, 11(4), 833-844.
- Munin, A., & Edwards-Lévy, F. (2011). Encapsulation of natural polyphenolic compounds; a review. *Pharmaceutics*, 3(4), 793-829.
- Murthy, Z., & Joshi, D. (2007). Fluidized bed drying of aonla (*Emblica officinalis*). *Drying Technology*, 25(5), 883-889.
- Murugesan, R., & Orsat, V. (2012). Spray drying for the production of nutraceutical ingredients—a review. *Food & Bioprocess Technology*, 5(1), 3-14.
- Nagar, P., Chauhan, I., & Yasir, M. (2011). Insights into polymers: Film formers in mouth dissolving films. *Drug Invention Today*, 3(12), 280-289.

- Nep, E.I., & Conway, B.R. (2011). Physicochemical characterization of grewia polysaccharide gum: Effect of drying method. *Carbohydrate Polymers*, 84(1), 446-453.
- Nergiz, C., & Ötleş, S. (1993). Chemical composition of *Nigella sativa* L. Seeds. *Food Chemistry*, 48(3), 259-261.
- Ng, S.K., Choong, Y.H., Tan, C.P., Long, K., & Nyam, K.L. (2014). Effect of total solids content in feed emulsion on the physical properties and oxidative stability of microencapsulated kenaf seed oil. *LWT-Food Science & Technology*, 58(2), 627-632.
- Ng, S.K., Jessie, L.Y.L., Tan, C.P., Long, K., & Nyam, K.L. (2013a). Effect of accelerated storage on microencapsulated kenaf seed oil. *Journal of the American Oil Chemists' Society*, 90(7), 1023-1029.
- Ng, S.K., Wong, P.Y., Tan, C.P., Long, K., & Nyam, K.L. (2013b). Influence of the inlet air temperature on the microencapsulation of kenaf (*Hibiscus cannabinus* L.) seed oil. *European Journal of Lipid Science & Technology*, 115(11), 1309-1318.
- Niamnuy, C., Nachaisin, M., Laohavanich, J., & Devahastin, S. (2011). Evaluation of bioactive compounds and bioactivities of soybean dried by different methods and conditions. *Food Chemistry*, 129(3), 899-906.
- Nicoli, MC, Anese, M, & Parpinel, M. (1999). Influence of processing on the antioxidant properties of fruit and vegetables. *Trends in Food Science & Technology*, 10(3), 94-100.
- Nireesha, G. R., Divya, L., Sowmya, C., Venkateshan, N., Babu, M. N., & Lavakumar, V. (2013). Lyophilization/freeze drying-an review. *International journal of novel trends in pharmaceutical sciences*, 3(4), 87-98.
- Novy, P., Kloucek, P., Rondevaldova, J., Havlik, J., Kourimska, L., & Kokoska, L. (2014). Thymoquinone vapor significantly affects the results of *Staphylococcus aureus* sensitivity tests using the standard broth microdilution method. *Fitoterapia*, 94, 102-107.
- Obón, J., Castellar, M., Alacid, M., & Fernández-López, J. (2009). Production of a red–purple food colorant from *opuntia stricta* fruits by spray drying and its application in food model systems. *Journal of Food Engineering*, 90(4), 471-479.
- O'brien, R.D. (2008). *Fats and oils: Formulating and processing for applications*: CRC press.

- Oktaý, M., Gülçin, İ., & Küfreviođlu, Ö.İ. (2003). Determination of in vitro antioxidant activity of fennel (*Foeniculum vulgare*) seed extracts. *LWT-Food Science & Technology*, 36(2), 263-271.
- Oliveira, D.M., Clemente, E., Afonso, M.R.A., & da Costa, J.M.C. (2013). Hygroscopic behavior of lyophilized powder of grugru palm (*Acrocomia aculeata*). *American Journal of Analytical Chemistry*, 4(10), 1.
- Omran, O.M. (2013). Effects of thymoquinone on stz-induced diabetic nephropathy: An immunohistochemical study. *Ultrastructural Pathology*, 38(1), 26-33.
- Ortega-Rivas, E., Juliano, P., & Yan, H. (2006). *Food powders: Physical properties, processing, and functionality*: Springer. New York (2005) p. 372.
- Otsuka, T., Iwao, Y., Miyagishima, A., & Itai, S. (2011). Application of principal component analyzes enables to effectively find important physical variables for optimization of fluid bed granulator conditions. *International Journal of Pharmaceutics*, 409(1), 81-88.
- Padhye, S., Banerjee, S., Ahmad, A., Mohammad, R., & Sarkar, F.H. (2008). From here to eternity-the secret of pharaohs: Therapeutic potential of black cumin seeds and beyond. *Cancer Therapy*, 6(b), 495.
- Palacios, L.E., & Wang, T. (2005). Egg-yolk lipid fractionation and lecithin characterization. *Journal of the American Oil Chemists' Society*, 82(8), 571-578.
- Parikh, D.M., Bonck, J.A., & Mogavero, M. (1997). Batch fluid bed granulation. *Drugs and The Pharmaceutical Sciences*, 81, 227-302.
- Park, J.S., Shin, H.S., & Hong, J.P. (2013). *U.S. Patent Application No. 13/873,312*.
- Peroni-Okita, F.H., Simão, R.A., Cardoso, M.B., Soares, C.A., Lajolo, F.M., & Cordenunsi, B.R. (2010). In vivo degradation of banana starch: Structural characterization of the degradation process. *Carbohydrate Polymers*, 81(2), 291-299.
- Peleg, M. (1983). Physical characteristics of food powders. In M. Peleg, & E. B. Bagley (Eds.), *Physical properties of foods* (pp. 293–323). Westport, Connecticut: AVI
- Peroni-Okita, F. H., Simão, R. A., Cardoso, M. B., Soares, C. A., Lajolo, F. M., & Cordenunsi, B. R. (2010). In vivo degradation of banana starch: Structural characterization of the degradation process. *Carbohydrate polymers*, 81(2), 291-299.
- Peter, K.V. (2006). *Handbook of herbs and spices* (Vol. 3): Woodhead Publishing, London; CRC Press.



- Peterson, D.M. (2001). Oat antioxidants. *Journal of Cereal Science*, 33(2), 115-129.
- Piatkowski, M., & Zbicinski, I. (2007). Analyzes of the mechanism of counter-current spray drying. *Transport in Porous Media*, 66(1-2), 89-101.
- Piras, A, Rosa, A, Marongiu, B, Porcedda, S, Falconieri, D, Dessi, MA, & Koca, U. (2013). Chemical composition and *in vitro* bioactivity of the volatile and fixed oils of *Nigella sativa* L. extracted by supercritical carbon dioxide. *Industrial Crops & Products*, 46, 317-323.
- Pisecky, J. (1997). *Handbook of milk powder manufacture*. Copenhagen: Niro
- Pokorny, J., Yanishlieva, N., & Gordon, M. H. (Eds.). (2001). *Antioxidants in food: practical applications*. CRC press.
- Pordy, W.T. (1994). *U.S. Patent No. 5,366,751*. Washington, DC: U.S. Patent & Trademark Office.
- Prakash, N. S., Combes, M.-C., Somanna, N., & Lashermes, P. (2002). AFLP analyzes of introgression in coffee cultivars (*Coffea arabica* L.) derived from a natural interspecific hybrid. *Euphytica*, 124(3), 265-271.
- Pua, C.K., Hamid, N.S.A., Tan, C.P., Mirhosseini, H., Rahman, R.B.A., & Rusul, G. (2010). Optimization of drum drying processing parameters for production of jackfruit (*Artocarpus heterophyllus*) powder using response surface methodology. *LWT-Food Science & Technology*, 43(2), 343-349.
- Quispe-Condori, S., Saldaña, M.D., & Temelli, F. (2011). Microencapsulation of flax oil with zein using spray and freeze drying. *LWT-Food Science & Technology*, 44(9), 1880-1887.
- Ramadan, M.F. (2007). Nutritional value, functional properties and nutraceutical applications of black cumin (*Nigella sativa* L.): An overview. *International Journal of Food Science & Technology*, 42(10), 1208-1218.
- Ramadan, M.F., & Mörsel, J.T. (2004). Oxidative stability of black cumin (*Nigella sativa* L.), coriander (*Coriandrum sativum* L.) and niger (*Guizotia abyssinica* Cass.) crude seed oils upon stripping. *European Journal of Lipid Science & Technology*, 106(1), 35-43.
- Ramadan, M.F., Asker, M.M.S., & Tadros, M. (2012a). Antiradical and antimicrobial properties of cold-pressed black cumin and cumin oils. *European Food Research & Technology*, 234(5), 833-844.
- Ramadan, M.F., & Wahdan, K.M.M. (2012b). Blending of corn oil with black cumin (*Nigella sativa*) and coriander (*Coriandrum sativum*) seed oils: Impact on functionality, stability and radical scavenging activity. *Food Chemistry*, 132(2), 873-879.

- Ramadan, M.F., Kroh, L.W., & Mörsel, J.-T. (2003). Radical scavenging activity of black cumin (*Nigella sativa* L.), coriander (*Coriandrum sativum* L.), and niger (*Guizotia abyssinica* Cass.) crude seed oils and oil fractions. *Journal of Agricultural & Food Chemistry*, 51(24), 6961-6969.
- Randhawa, M.A., & Alghamdi, M.S. (2011). Anticancer activity of *Nigella sativa* (black seed)—A review. *The American Journal of Chinese Medicine*, 39 (06), 1075-1091.
- Raybaudi-Massilia, R.M., & Mosqueda-Melgar, J. (2012). Polysaccharides as carriers and protectors of additives and bioactive compounds in foods: In *The Complex World of Polysaccharides*. InTech, Caracas, Venezuela.
- Rayo, L.M., e Carvalho, L.C., Sardá, F.A., Dacanal, G.C., Menezes, E.W., & Tadini, C.C. (2015). Production of instant green banana flour (*Musa cavendishii*, var. *Nanicão*) by a pulsed-fluidized bed agglomeration. *LWT-Food Science & Technology*, 63(1), 461-469.
- Razmkhah, S., Tan, C.P., Long, K., & Nyam, K.L. (2013). Quality changes and antioxidant properties of microencapsulated kenaf (*Hibiscus cannabinus* L.) seed oil during accelerated storage. *Journal of the American Oil Chemists' Society*, 90(12), 1859-1867.
- Reineccius, G.A. (2004). The spray drying of food flavors. *Drying Technology*, 22(6), 1289-1324.
- Ren, G.y., Li, D., Wang, L.j., Özkan, N., & Mao, Z.h. (2010). Morphological properties and thermoanalyzes of micronized cassava starch. *Carbohydrate Polymers*, 79(1), 101-105.
- Rivellese, A.A., Maffettone, A., Vessby, B., Uusitupa, M., Hermansen, K., Berglund, L., & Riccardi, G. (2003). Effects of dietary saturated, monounsaturated and n-3 fatty acids on fasting lipoproteins, ldl size and post-prandial lipid metabolism in healthy subjects. *Atherosclerosis*, 167(1), 149-158.
- Rodea-González, D. A., Cruz-Olivares, J., Román-Guerrero, A., Rodríguez-Huezo, M. E., Vernon-Carter, E. J., & Pérez-Alonso, C. (2012). Spray-dried encapsulation of chia essential oil (*Salvia hispanica* L.) in whey protein concentrate-polysaccharide matrices. *Journal of Food Engineering*, 111(1), 102-109.
- Rodríguez-Hernández, G., González-García, R., Grajales-Lagunes, A., Ruiz-Cabrera, M., & Abud-Archila, M. (2005). Spray-drying of cactus pear juice (*Opuntia streptacantha*): Effect on the physicochemical properties of powder and reconstituted product. *Drying Technology*, 23(4), 955-973.
- Rohman, A., & Ariani, R. (2013). Authentication of *Nigella sativa* seed oil in binary and ternary mixtures with corn oil and soybean oil using FTIR spectroscopy

coupled with partial least square. *The Scientific World Journal*, Article ID 740142.

- Ronsse, F., Pieters, J., & Dewettinck, K. (2008). Modelling side-effect spray drying in top-spray fluidised bed coating processes. *Journal of Food Engineering*, 86(4), 529-541.
- Roos, Y., & Karel, M. (1990). Differential scanning calorimetry study of phase transitions affecting the quality of dehydrated materials. *Biotechnology Progress*, 6(2), 159-163.
- Roos, Y. H. (2002). Importance of glass transition and water activity to spray drying and stability of dairy powders. *Le Lait*, 82(4), 475-484.
- Roos, Y.H. (2010). Glass transition temperature and its relevance in food processing. *Annual Review of Food Science & Technology*, 1 (1), 469-496.
- Rosida, D.F., Mulyani, T., & Septalia, L.R. (2016). A comparative study of non-dairy cream based on the type of leguminosae protein source in terms of physico chemical properties and organoleptic. *Agriculture & Agricultural Science Procedia*, 9, 431-439.
- Rutz, J.K., Borges, C.D., Zambiasi, R.C., Crizel-Cardozo, M.M., Kuck, L.S., & Noreña, C.P. (2017). Microencapsulation of palm oil by complex coacervation for application in food systems. *Food Chemistry*, 220, 59-66.
- Sagit, M., Korkmaz, F., Akcadag, A., & Somdas, M.A. (2013). Protective effect of thymoquinone against cisplatin-induced ototoxicity. *European Archives of Oto-Rhino-Laryngology*, 270 (8), 2231-2237.
- Sahak, M.K.A., Kabir, N., Abbas, G., Draman, S., & Adli, D.S.H. (2016). The role of *Nigella sativa* and its active constituents in learning and memory. *Evidence-Based Complementary & Alternative Medicine 1-6*. Article ID 6075679.
- Şahin-Nadeem, H., Dinçer, C., Torun, M., Topuz, A., & Özdemir, F. (2013). Influence of inlet air temperature and carrier material on the production of instant soluble sage (*Salvia fruticosa* Miller) by spray drying. *LWT-Food Science and Technology*, 52 (1), 31-38.
- Salim, E.I. (2010). Cancer chemopreventive potential of volatile oil from black cummin seeds, *Nigella sativa* L., in a rat multi-organ carcinogenesis bioassay. *Oncology Letters*, 1(5), 913-924.
- Salim, L.Z.A., Mohan, S., Othman, R., Abdelwahab, S.I., Kamalidehghan, B., Sheikh, B.Y., & Ibrahim, M.Y. (2013). Thymoquinone induces mitochondria-mediated apoptosis in acute lymphoblastic leukaemia *in vitro*. *Molecules*, 18(9), 11219-11240.

- Sanchez, V., Baeza, R., Galmarini, M.V., Zamora, M.C., & Chirife, J. (2013). Freeze-drying encapsulation of red wine polyphenols in an amorphous matrix of maltodextrin. *Food & Bioprocess Technology*, 6(5), 1350-1354.
- Santos, J., Santos, I., Conceição, M., Porto, S., Trindade, M. F. S. A., Souza, A., ... & Araújo, A. (2004). Thermoanalytical, kinetic and rheological parameters of commercial edible vegetable oils. *Journal of thermal analyzes and calorimetry*, 75(2), 419-428.
- Sawale, P.D., Patil, G.R., Hussain, S.A., Singh, A.K., & Singh, R.R.B. (2017). Release characteristics of polyphenols from microencapsulated terminalia arjuna extract: Effects of simulated gastric fluid. *International Journal of Food Properties*, 1-9.
- Saxena, S., Rathore, S., Diwakar, Y., Kakani, R., Kant, K., Dubey, P., & John, S. (2017). Genetic diversity in fatty acid composition and antioxidant capacity of *Nigella sativa* L. Genotypes. *LWT-Food Science & Technology*, 78, 198-207.
- Scher, H. (1983). Human welfare and the environment. *IUPAC pesticide chemistry*, 295-300.
- Schrooyen, P. M., van der Meer, R., & De Kruif, C. G. (2001). Microencapsulation: its application in nutrition. *Proceedings of the Nutrition Society*, 60(4), 475-479.
- Schubert, H. (1993). Instantization of powdered food products. *International Chemical Engineering*, 33(1), 28-45.
- Schuck, P., Dolivet, A., Méjean, S., Zhu, P., Blanchard, E., & Jeantet, R. (2009). Drying by desorption: A tool to determine spray drying parameters. *Journal of Food Engineering*, 94(2), 199-204.
- Shabana, A., El-Menyar, A., Asim, M., Al-Azzeh, H., & Al Thani, H. (2013). Cardiovascular benefits of black cumin (*Nigella sativa*). *Cardiovascular Toxicology*, 13(1), 9-21.
- Shahidi, F., & Han, X.Q. (1993). Encapsulation of food ingredients. *Critical Reviews in Food Science & Nutrition*, 33(6), 501-547.
- Shaikh, J., Bhosale, R., & Singhal, R. (2006). Microencapsulation of black pepper oleoresin. *Food Chemistry*, 94(1), 105-110.
- Shamaei, S., Seiiedlou, S.S., Aghbashlo, M., Tsotsas, E., & Kharaghani, A. (2017). Microencapsulation of walnut oil by spray drying: Effects of wall material and drying conditions on physicochemical properties of microcapsules. *Innovative Food Science & Emerging Technologies*, 39, 101-112.

- Shang, F., Li, X., & Jiang, X. (2016). Coffee consumption and risk of the metabolic syndrome: a meta-analyses. *Diabetes & metabolism*, 42(2), 80-87.
- Sharma, P., & Gujral, H. S. (2011). Effect of sand roasting and microwave cooking on antioxidant activity of barley. *Food Research International*, 44(1), 235-240.
- Sharma, A., Jana, A.H., & Chavan, R.S. (2012). Functionality of milk powders and milk-based powders for end use applications—A review. *Comprehensive Reviews in Food Science & Food Safety*, 11(5), 518-528.
- Shiga, H., Yoshii, H., Nishiyama, T., Furuta, T., Forssele, P., Poutanen, K., & Linko, P. (2001). Flavor encapsulation and release characteristics of spray-dried powder by the blended encapsulant of cyclodextrin and gum arabic. *Drying Technology*, 19(7), 1385-1395.
- Shu, B., Yu, W., Zhao, Y., & Liu, X. (2006). Study on microencapsulation of lycopene by spray-drying. *Journal of Food Engineering*, 76(4), 664-669.
- Sielicka, M., & Samotyja, U. (2013). Solvent influence on antioxidant activity assay of selected cold-pressed plant oils. *PhD Interdisciplinary Journal*, 1, 67-74.
- Singh, H., Newstead, D., & Fox, P. (1992). Aspects of proteins in milk powder manufacture. *Advanced Dairy Chemistry-1: Proteins*. (Ed. 2), 735-765.
- Singh, S., Das, S., Singh, G., Schuff, C., de Lampasona, M.P., & Catalán, C.A. (2014). Composition, in vitro antioxidant and antimicrobial activities of essential oil and oleoresins obtained from black cumin seeds (*Nigella sativa* L.). *Biomed Research International*, Article ID 918209.
- Singleton, V.L., Orthofer, R., & Lamuela-Raventos, R.M. (1999). analyzes of total phenols and other oxidation substrates and antioxidants by means of folin-ciocalteu reagent. *Methods in Enzymology* (299), 152-178.
- Solati, Z., Baharin, B.S., & Bagheri, H. (2012). Supercritical carbon dioxide (SC-O<sub>2</sub>) extraction of *Nigella sativa* L. Oil using full factorial design. *Industrial Crops & Products*, 36(1), 519-523.
- Solati, Z., Baharin, B.S., & Bagheri, H. (2014). Antioxidant property, thymoquinone content and chemical characteristics of different extracts from *Nigella sativa* L. Seeds. *Journal of the American Oil Chemists' Society*, 91(2), 295-300.
- Soliman, E.A., El-Moghazy, A.Y., El-Din, M.M., & Massoud, M.A. (2013). Microencapsulation of essential oils within alginate: Formulation and *in vitro* evaluation of antifungal activity. *Journal of Encapsulation and Adsorption Sciences*, 3(1), 48.

- Soto, C., Chamy, R., & Zuniga, M. (2007). Enzymatic hydrolysis and pressing conditions effect on borage oil extraction by cold pressing. *Food Chemistry*, 102(3), 834-840.
- Stein, S., Mirokhin, D., Tchekhovskoi, D., Mallard, G., Mikaia, A., Zaikin, V., & Sparkman, D. (2002). The nist mass spectral search program for the nist/epa/nih mass spectra library. *Standard Reference Data Program of the National Institute of Standards and Technology. Gaithers-burg, MD, US.*
- Subramaniyan, J., Krishnan, G., Balan, R., Divya, M., Ramasamy, E., Ramalingam, S., & Thiruvengadam, D. (2014). Carvacrol modulates instability of xenobiotic metabolizing enzymes and downregulates the expressions of pcna, mmp-2, and mmp-9 during diethylnitrosamine-induced hepatocarcinogenesis in rats. *Molecular and Cellular Biochemistry*, 1-12.
- Suhag, Y., & Nanda, V. (2016). Optimization for spray drying process parameters of nutritionally rich honey powder using response surface methodology. *Cogent Food & Agriculture*, 2(1), 1176631.
- Suhag, Y., & Nanda, V. (2017). Degradation kinetics of ascorbic acid in encapsulated spray-dried honey powder packaged in aluminum laminated polyethylene and high-density polyethylene. *International Journal of Food Properties*, 20(3), 645-653.
- Sultan, M.T., Butt, M.S., Karim, R., Ahmad, A.N., Suleria, H.A.R., & Saddique, M.S. (2014). Toxicological and safety evaluation of *Nigella sativa* lipid and volatile fractions in streptozotocin induced diabetes mellitus. *Asian Pacific Journal of Tropical Disease*, 4, S693-S697.
- Sun-Waterhouse, D., Zhou, J., Miskelly, G., Wibisono, R., & Wadhwa, S. (2011). Stability of encapsulated olive oil in the presence of caffeic acid. *Food Chemistry*, 126(3), 1049-1056.
- Swanson, R.B., Garden, L.A., & Parks, S.S. (1999). Effect of a carbohydrate-based fat substitute and emulsifying agents on reduced-fat peanut butter cookies. *Journal of Food Quality*, 22(1), 19-29.
- Szulc, K., & Lenart, A. (2013). Surface modification of dairy powders: Effects of fluid-bed agglomeration and coating. *International Dairy Journal*, 33(1), 55-61.
- Szulc, K., Nazarko, J., Ostrowska-Ligęza, E., & Lenart, A. (2016). Effect of fat replacement on flow and thermal properties of dairy powders. *LWT-Food Science & Technology*, 68, 653-658.
- Takeungwongtrakul, S., & Benjakul, S. (2015). Wall materials and the presence of antioxidants influence encapsulation efficiency and oxidative stability of

- micro-encapsulated shrimp oil. *European Journal of Lipid Science & Technology*, 117(4), 450-459.
- Takruri, H.R., & Dameh, M.A. (1998). Study of the nutritional value of black cumin seeds (*Nigella sativa*). *Journal of the Science of Food & Agriculture*, 76(3), 404-410.
- Tan, S. P., Tuyen, C. K., Parks, S. E., Stathopoulos, C. E., & Roach, P. D. (2015). Effects of the spray-drying temperatures on the physicochemical properties of an encapsulated bitter melon aqueous extract powder. *Powder Technology*, 281, 65-75.
- Taylor, T.M., Weiss, J., Davidson, P.M., & Bruce, B.D. (2005). Liposomal nanocapsules in food science and agriculture. *Critical Reviews in Food Science and Nutrition*, 45(7-8), 587-605.
- Tembhurne, S., Feroz, S., More, B., & Sakarkar, D. (2014). A review on therapeutic potential of *Nigella sativa* (kalonji) seeds. *Journal of Medicinal Plants Research*, 8(3), 167-177.
- Timilsena, Y.P., Adhikari, R., Barrow, C.J., & Adhikari, B. (2016). Microencapsulation of chia seed oil using chia seed protein isolate-chia seed gum complex coacervates. *International Journal of Biological Macromolecules*, 91, 347-357.
- Tisserand, R., & Young, R. (2013). *Essential Oil Safety-E-Book: A Guide for Health Care Professionals*: Elsevier Health Sciences.
- Tonon, R.V., Brabet, C., & Hubinger, M.D. (2008). Influence of process conditions on the physicochemical properties of açai (*Euterpe oleraceae* Mart.) powder produced by spray drying. *Journal of Food Engineering*, 88(3), 411-418.
- Tonon, R.V., Grosso, C.R., & Hubinger, M.D. (2011). Influence of emulsion composition and inlet air temperature on the microencapsulation of flaxseed oil by spray drying. *Food Research International*, 44(1), 282-289.
- Turchiuli, C., Fuchs, M., Bohin, M., Cuvelier, M.E., Ordonnaud, C., Peyrat-Maillard, M.N., & Dumoulin, E. (2005). Oil encapsulation by spray drying and fluidised bed agglomeration. *Innovative Food Science & Emerging Technologies*, 6(1), 29-35.
- Turchiuli, C., Lemarié, N., Cuvelier, M.E., & Dumoulin, E. (2013). Production of fine emulsions at pilot scale for oil compounds encapsulation. *Journal of Food Engineering*, 115(4), 452-458.
- Tuyen, C.K., Nguyen, M.H., & Roach, P.D. (2010). Effects of spray drying conditions on the physicochemical and antioxidant properties of the gac (*Momordica*

*cochinchinensis*) fruit aril powder. *Journal of Food Engineering*, 98(3), 385-392.

Tuyen, C.K., Nguyen, M.H., Roach, P.D., & Stathopoulos, C.E. (2015). A storage study of encapsulated gac (*Momordica cochinchinensis*) oil powder and its fortification into foods. *Food & Bioproducts Processing*, 96, 113-125.

Tze, N. L., Han, C. P., Yusof, Y. A., Ling, C. N., Talib, R. A., Taip, F. S., & Aziz, M. G. (2012). Physicochemical and nutritional properties of spray-dried pitaya fruit powder as natural colorant. *Food Science and Biotechnology*, 21(3), 675-682.

Umesha, S., Manohar, R.S., Indiramma, A., Akshitha, S., & Naidu, K.A. (2015). Enrichment of biscuits with microencapsulated omega-3 fatty acid (*Alpha-linolenic acid*) rich garden cress (*Lepidium sativum*) seed oil: Physical, sensory and storage quality characteristics of biscuits. *LWT-Food Science & Technology*, 62(1), 654-661.

Umesha, SS, Monahar, B, & Naidu, K Akhilender. (2013). Microencapsulation of  $\alpha$ -linolenic acid-rich garden cress seed oil: Physical characteristics and oxidative stability. *European Journal of Lipid Science & Technology*, 115(12), 1474-1482.

Vega, C., & Roos, Y. (2006). Invited review: Spray-dried dairy and dairy-like emulsions—compositional considerations. *Journal of Dairy Science*, 89(2), 383-401.

Venkatachallam, S. K. T., Pattekan, H., Divakar, S., & Kadimi, U. S. (2010). Chemical composition of *Nigella sativa* L. seed extracts obtained by supercritical carbon dioxide. *Journal of food science and technology*, 47(6), 598-605.

Verdurmen, R.E., Verschuere, M., Gungor, M., Straatsma, H., Blei, S., & Sommerfeld, M. (2005). Simulation of agglomeration in spray dryers: The edecad project. *Le Lait*, 85(4-5), 343-351.

Vilstrup, P. (2001). *Microencapsulation of food ingredients*: Leatherhead Publication, Surrey.

Viuda-Martos, M., Mohamady, M., Fernández-López, J., ElRazik, K.A., Omer, E., Pérez-Alvarez, J., & Sendra, E. (2011). *In vitro* antioxidant and antibacterial activities of essential oils obtained from Egyptian aromatic plants. *Food Control*, 22(11), 1715-1722.

Vongsvivut, J., Heraud, P., Zhang, W., Kralovec, J.A., McNaughton, D., & Barrow, C.J. (2012). Quantitative determination of fatty acid compositions in microencapsulated fish-oil supplements using fourier transform infrared (FTIR) spectroscopy. *Food Chemistry*, 135(2), 603-609.



- Vu, T., Galet, L., Fages, J., & Oulahna, D. (2003). Improving the dispersion kinetics of a cocoa powder by size enlargement. *Powder Technology*, 130(1), 400-406.
- Wajs, A., Bonikowski, R., & Kalemba, D. (2008). Composition of essential oil from seeds of *Nigella sativa* L. Cultivated in Poland. *Flavor & Fragrance Journal*, 23(2), 126-132.
- Wan, Y., Bechtel, P.J., & Sathivel, S. (2011). Physical and nutritional properties of baby food containing menhaden oil (*Brevoortia tyrannus*) and microencapsulated menhaden oil. *LWT-Food Science & Technology*, 44(2), 576-581.
- Wanasundara, U.N., & Shahidi, F. (1998). Antioxidant and pro-oxidant activity of green tea extracts in marine oils. *Food Chemistry*, 63(3), 335-342.
- Wang, Y., Liu, B., Wen, X., Li, M., Wang, K., & Ni, Y. (2017). Quality analyzes and microencapsulation of chili seed oil by spray drying with starch sodium octenylsuccinate and maltodextrin. *Powder Technology*, 312, 294-298.
- Wang, B., & Liu, J. (2014). Trans-free nondairy creamer prepared from enzymatic interesterification of soybean oil and fully hydrogenated soybean oil. *Journal of Food Process Engineering*, 37(4), 339-348.
- Wang, R., Tian, Z., & Chen, L. (2011). A novel process for microencapsulation of fish oil with barley protein. *Food Research International*, 44(9), 2735-2741.
- Wang, G., & Wang, T. (2008). Oxidative stability of egg and soy lecithin as affected by transition metal ions and pH in emulsion. *Journal of Agricultural & Food Chemistry*, 56(23), 11424-11431.
- Wang, W., & Zhou, W. (2012). Characterization of spray-dried soy sauce powders using maltodextrins as carrier. *Journal of Food Engineering*, 109(3), 399-405.
- Warrier, P.K., Nambiar, V., & Ramankutty, C. (1996). *Indian medicinal plants: A compendium of 500 species* (Vol. 5): Orient Longman, Chennai.
- Wilson, N., & Shah, N. (2007). Microencapsulation of vitamins. *ASEAN Food Journal*, 14(1), 1-14.
- Woo, C.C., Kumar, A.P., Sethi, G., & Tan, K.H.B. (2012). Thymoquinone: Potential cure for inflammatory disorders and cancer. *Biochemical Pharmacology*, 83(4), 443-451.
- Wu, K.G., & Xiao, Q. (2005). Microencapsulation of fish oil by simple coacervation of hydroxypropyl methylcellulose. *Chinese Journal of Chemistry*, 23(11), 1569-1572.

- Yackel, W., & Cox, C. (1992). Application of starch-based fat replacers. *Food Technology*, 46 (6), 146-148.
- Ye, A., Cui, J., Taneja, A., Zhu, X., & Singh, H. (2009). Evaluation of processed cheese fortified with fish oil emulsion. *Food Research International*, 42 (8), 1093-1098.
- Ying, D., Ong, Y.L., Cheng, L.J., Sanguansri, L., Shen, Z., & Augustin, M.A. (2016). Compressible extruded granules containing microencapsulated oil powders. *Powder Technology*, 291, 276-283.
- Zahoor, A., Ghaffar, A., & Aslam, M. (2004). *Nigella sativa*; a potential commodity in crop diversification traditionally used in health care. Pakistan: Ministry of Food, Agriculture & Livestock; p. 6–10.
- Zaoui, A., Cherrah, Y., Mahassini, N., Alaoui, K., Amarouch, H., & Hassar, M. (2002). Acute and chronic toxicity of *Nigella sativa* fixed oil. *Phytomedicine*, 9 (1), 69-74.
- Zohary, D., Hopf, M., & Weiss, E. (2012). *Domestication of plants in the old world: The origin and spread of domesticated plants in southwest Asia, Europe, and the Mediterranean basin*: Oxford Science Publications, Oxford.
- Zuidam, N., & Nedovic, V. (2010a). Encapsulation of probiotics for use in food products. In *Encapsulation technologies for active food ingredients and food processing* (pp. 269-302). Springer NY.
- Zuidam, N.J., & Shimoni, E. (2010b). Overview of microencapsulates for use in food products or processes and methods to make them *Encapsulation technologies for active food ingredients & food processing* (pp. 3-29): Springer NY.
- Zuloaga, O., Navarro, P., Bizkarguenaga, E., Iparraguirre, A., Vallejo, A., Olivares, M., & Prieto, A. (2012). Overview of extraction, clean-up and detection techniques for the determination of organic pollutants in sewage sludge: A review. *Analytica Chimica Acta*, 736, 7-29.
- Zúñiga, M., Soto, C., Mora, A., Chamy, R., & Lema, J. (2003). Enzymic pre-treatment of *Gguevina avellana* mol oil extraction by pressing. *Process Biochemistry*, 39 (1), 51-57.