

UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF PALM FATTY HYDRAZIDE DERIVATIVES

NORASHIKIN BINTI AHMAD

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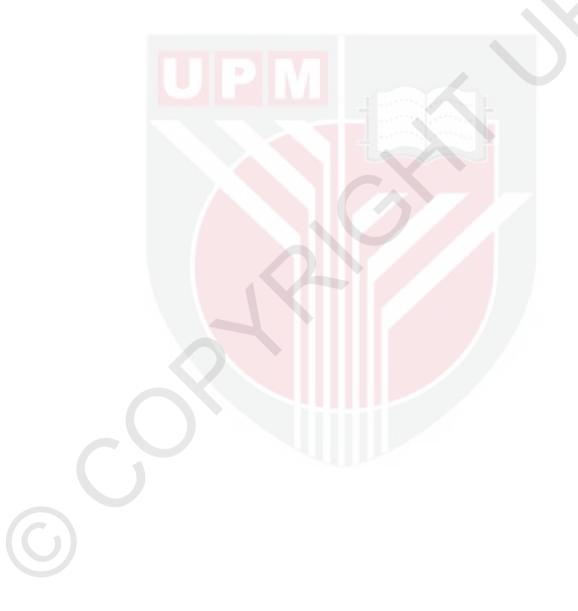
Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master Science

December 2012

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

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December 2012

Chairman : Mansor Hj Ahmad @ Ayob, PhD

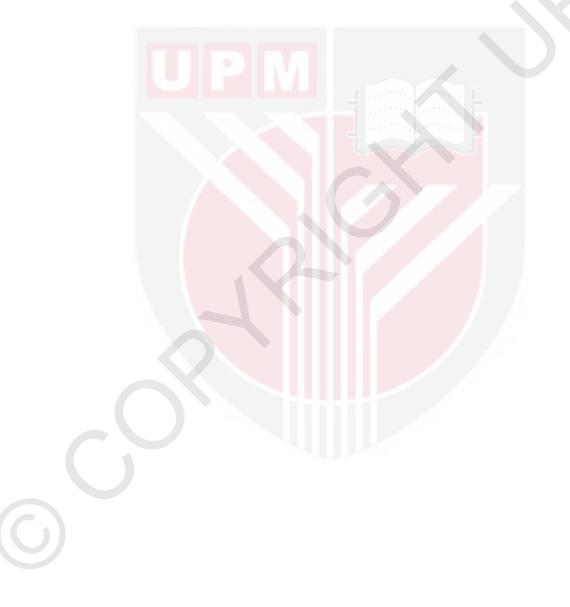
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Hydrazide derivatives have been synthesized from methyl esters, hydrazones and vegetable oils. They are important due to their diverse applications in pharmaceutical products, detergents as well as in oil and gas industries. The chemical synthesis of fatty hydrazides is well-established; however, few publications described the synthesis of fatty hydrazide derivatives, particularly, when produced from refined, bleached and deodorized palm olein. Here, the synthesis and characterization of fatty hydrazide derivatives are reported. Besides, palmityl hydrazide derivative synthesized from palmityl hydrazide was used as reference. The fatty hydrazides derivatives were characterized using a Fourier transform infrared (FTIR), gas chromatography (GC) and nuclear magnetic resonance (NMR) spectroscopy. The synthesis of *N*,*N*-dimethyl fatty hydrazides was carried out at 100° C in dioxane for 6 hours with the molar ratio between hydrazide, dimethyl sulfate and potassium hydroxide was 1:1:1. The proton NMR confirmed the product obtained was *N*,*N*-dimethyl fatty hydrazides. Other fatty hydrazide derivatives were cationic surfactants



prepared using *N*,*N*-dimethyl fatty hydrazides. The surface tensions of these cationic surfactants vary according to the types of acyl chlorides. Lower surface tension was recorded for cationic surfactants prepared with acyl chloride with phenyl group. In addition, cationic surfactant can also be prepared from reaction between acyl chlorides and fatty hydrazides. Hence, fatty hydrazide derivatives from palm olein were successfully prepared and characterized.



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

SINTESIS DAN PENCIRIAN TERBITAN HIDRAZIDA LEMAK SAWIT

Oleh

NORASHIKIN BINTI AHMAD

Disember 2012

Pengerusi : Mansor Hj Ahmad @ Ayob, PhD

Fakulti : Sains

Terbitan hidrazida telah disintesis daripada metil ester, hidrazon dan minyak sayursayuran. Ianya penting disebabkan oleh kepelbagaian penggunaan dalam produk farmaseutikal, bahan pencuci dan juga dalam industri minyak dan gas. Sintesis kimia lemak hidrazida telah lama dibangunkan; namun hanya terdapat beberapa penerbitan sahaja yang menerangkan sintesis terbitan lemak hidrazida khususnya menggunakan olein sawit yang dinyahbau, luntur dan ditapis. Di sini, melaporkan sintesis dan pencirian terbitan lemak hidrazida. Selain itu, terbitan palmitil hidrazida yang disintesis daripada palmitil hidrazida telah digunakan sebagai rujukan. Pencirian terbitan lemak hidrazida telah dijalankan dengan menggunakan spektroskopi fourier transform inframerah (FTIR), kromatografi gas (GC) dan spektroskopi resonans magnetik nuklear (NMR). Sintesis *N,N*-dimetil lemak hidrazida telah dijalankan pada suhu 100°C di dalam dioksana selama 6 jam pada nisbah molar 1:1:1 bagi hidrazida, dimetil sulfat dan kalium hidroksida. NMR proton mengesahkan bahawa produk



yang diperolehi adalah *N*,*N*-dimetil lemak hidrazida. Terbitan lemak hidrazida yang lain adalah surfaktan kationik yang dihasilkan menggunakan *N*,*N*-dimetil lemak hidrazida. Ketegangan permukaan untuk surfaktan kationik ini berbeza-beza bergantung kepada jenis asid klorida. Ketegangan permukaan yang lebih rendah telah dicatatkan untuk surfaktan kationik yang dihasilkan dengan asid klorida yang mempunyai kumpulan fenil. Di samping itu, surfaktan kationik juga boleh dihasilkan daripada tindak balas antara asid klorida dan lemak hidrazida. Oleh itu, penghasilan dan pencirian terbitan lemak hidrazida daripada olein sawit telah berjaya.



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vi

I certify that a Thesis Examination Committee has met on 28 December 2012 to conduct the final examination of Norashikin Binti Ahmad on her thesis entitled "Synthesis and characterization of palm fatty hydrazide derivatives" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

Mohd Aspollah Hj Md Sukari, PhD

Professor Faculty of Science Universiti Putra Malaysia (Chairman)

Kamaliah Sirat, PhD

Senior Lecturer Faculty of Science Universiti Putra Malaysia (Internal Examiner)

Siti Mariam Mohd Nor, PhD

Senior Lecturer Faculty of Science Universiti Putra Malaysia (Internal Examiner)

Mohd Ambar Yarmo, PhD

Professor Faculty of Science and Technology Universiti Kebangsaan Malaysia Malaysia (External Examiner)

NORITAH OMAR, PhD

Associate Professor and Deputy Dean School of Gaduate Studies Universiti Putra Malaysia

Date: 26 June 2013

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

Mansor Hj Ahmad @ Ayob, PhD

Associate Professor Faculty of Science Universiti Putra Malaysia (Chairman)

Nor Azowa Ibrahim, PhD

Senior Lecturer Faculty of science Universiti Putra Malaysia (Member)

Hazimah Abu Hassan, PhD

Director of Advanced Oleochemical Technology Division (AOTD) Malaysian Palm Oil Board (Member)

Yeong Shoot Kian, PhD

Head of Synthesis Product Development Unit Advanced Oleochemical Technology Division (AOTD) Malaysian Palm Oil Board (Member)

Wan Md Zin Wan Yunus, PhD

Professor Chemistry Department Centre for Defence Foundation Studies National Defence University of Malaysia

BUJANG BIN KIM HUAT, PhD

Professor and Dean School of Gaduate Studies Universiti Putra Malaysia

Date:

DECLARATION

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

NORASHIKIN BINTI AHMAD

Date: 28 December 2012

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LIST OF ABBREVIATIONS

RBDPOo	: Refined, Bleached and Deodorized Palm olein
RBD	: Refined, Bleached and Deodorized
MPOB	: Malaysian Palm Oil Board
КОН	: Potassium hydroxide
CHCl ₃	: Chloroform
THF	: Tetrahydrofuran
СМС	: Critical Micelle Concentration
MeCN	: Acetonitrile
HPLC	: High Performance Liquid Chromatography
DMSO- d_6	: Dimethyl sulfoxide- d_6
Tween 20	: Polyoxyethylene (20) sorbitan monolaurate
BSTFA	: <i>N</i> , <i>O</i> -bis(trimethylsilyl) trifluoroacetamide with 1% trimethylsilyl chloride
FTIR	: Fourier Transform Infrared
GC	: Gas Chromatography
GC/MS	: Gas Chromatography/Mass Spectrometry
NMR	: Nuclear Magnetic Resonance

TMS : Trimethylsilyl



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