UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF PALM FATTY HYDRAZIDE DERIVATIVES

NORASHIKIN BINTI AHMAD

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SYNTHESIS AND CHARACTERIZATION OF PALM FATTY HYDRAZIDE DERIVATIVES

By

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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

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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

SYNTHESIS AND CHARACTERIZATION OF PALM FATTY HYDRAZIDE DERIVATIVES

By

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December 2012

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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^{o}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

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Terbitan hidrazida telah disintesis daripada metil ester, hidrazon dan minyak sayur-sayuran. 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^{o}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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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.

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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.

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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

KOH : Potassium hydroxide

$CHCl_3$: Chloroform

THF : Tetrahydrofuran

CMC : Critical Micelle Concentration

MeCN : Acetonitrile

HPLC : High Performance Liquid Chromatography

DMSO-d_6 : Dimethyl sulfoxide-d_6

Tween 20 : Polyoxyethylene (20) sorbitan monolaurate

BSTFA : *N,O*-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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