

UNIVERSITI TEKNOLOGI MARA

**OPTIMIZATION OF
DIHYDROXYSTEARIC ACID (DHSA)
PRODUCTION VIA *IN SITU*
HYDROLYSIS OF EPOXIDIZED
OLEIC ACID USING TAGUCHI
ORTHOGONAL ARRAY DESIGN**

ISMAIL BIN MD RASIB

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(Chemical Engineering)

College of Engineering

June 2023

ABSTRACT

Concern regarding the setback of dependency on using fossil fuels as the main resources as the precursor for many derivatives had drawn an attention to further study on production of dihydroxystearic acid (DHSA) by in-situ hydrolysis of epoxidized oleic acid with reaction rate estimate by MATLAB by using 27 data from previous experiment which provide all the parameter involved and optimum parameter later decided by Taguchi method, which are more resource friendly due to low number of experiment need to be done and can further be used as a raw material in cosmetic industry. Epoxidized oleic acid are produced by using *in situ* formed performic acid (HCOOOH), which are the mixture of formic acid (HCOOH) as the oxygen carrier with hydrogen peroxide (H₂O₂) as the oxygen donor. *Ode 45* method by MATLAB provide the kinetic simulation of the oxirane oxygen ring degradation throughout the epoxidation process with the expected concentration of DHSA produce. Taguchi method further propose the most optimum parameter for DHSA production as in H₂O₂ / oleic acid molar ratio 1.5, HCOOH / oleic acid molar ratio 0.5, reaction temperature at 35 °C with agitation speed at 200 RPM. This setup produce crude DHSA with hydroxyl value of 267 mg KOH/g and further prove by lower hydroxyl value quantity present from non-optimum parameter by Taguchi method where parameters are changed to H₂O₂ / oleic acid molar ratio 0.5 and reaction temperature at 50 °C show lower hydroxyl value content at 172.9 and 263.1 mg KOH/g respectively. DHSA produce, which known as crude DHSA are further purify order for it to be use in cosmetic industry. Physicochemical properties of crude and purify DHSA are compared, where hydroxyl value for purify is higher as compared to crude with 333.1 mg KOH/g and 267 mg KOH/g respectively. Others properties such as higher iodine value in crude compared to purified also proven with value 8.9 to 3.7 respectively, and the form or particle size which show the most significance properties where it is semi solid for crude DHSA and white powder form with particle size of 125-63 μm for purify DHSA. Overall, purify DHSA was successfully produce from oleic acid with low number of running experiment due to prediction by MATLAB and Taguchi method on the reaction rate and the most optimum parameter for epoxidation process.

ACKNOWLEDGEMENT

Firstly, I wish to thank God for giving me the opportunity to embark on my Msc and for completing this long and challenging journey successfully. My gratitude and thanks go to my supervisor Dr Mohd Jumain bin Jalil and my co-supervisor Dr Sh Mohd Firdaus bin Sh Abdul Nasir. Both of them really increase my motivation by sharing their expertise in order for me to finish this journey.

My appreciation goes to the faculty school of chemical engineering and technician of the UiTM Pulau Pinang chemical lab who provided the facilities and assistance during my study. Special thanks to my colleagues and friends for helping me with this project. Not to forget, third party lab where I send my sample for testing.

Finally, this thesis is dedicated to the loving memory of my very dear parents for the vision and determination to educate me. This piece of victory is dedicated to both of you. Alhamdulillah.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF SYMBOLS	xi
LIST OF ABBREVIATIONS	xii
CHAPTER ONE: INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement	3
1.3 Objectives of the Study	4
1.4 Scope of the Study	5
1.5 Significance of the Study	6
CHAPTER TWO: LITERATURE REVIEW	7
2.1 Palm Oil as Raw Renewable Material	7
2.1.1 Palm Oil Industry	8
2.1.2 Fatty Acid Composition of Palm Oil	10
2.2 Epoxidation by Peracid Mechanism	11
2.2.1 Homogenous Catalyst in Epoxidation Process	13
2.3 Oxirane Ring Opening	14
2.4 Oxirane Ring by Hydrolysis to Form DHSA	16
2.5 Dihydroxystearic Acid (DHSA)	17
2.6 Difference Between Crude and Purify DHSA	18
2.6.1 Application of Purify DHSA	19
2.7 Taguchi Optimization	20

2.7.1	Orthogonal Array and ANOVA Analysis	21
2.8	Kinetic Study of Epoxidation	23
2.8.1	Development Kinetic Modelling	24
2.9	Concluding Remarks	27
 CHAPTER THREE: RESEARCH METHODOLOGY		28
3.1	Overview	28
3.2	Materials for Producing Crude DHSA	30
3.3	Experiment Setup	30
3.3.1	Setup and Procedure for Production of Crude DHSA	30
3.3.2	Purification of Crude DHSA	32
3.3.3	Relative Conversion Oxirane	32
3.4	Analytical Procedure	33
3.4.1	Iodine Value	33
3.4.2	Acid Value	34
3.4.3	Hydroxyl Value	34
3.5	Physico-Chemical Properties of Crude and Purify DHSA	35
3.5.1	Fourier Transform Infrared Spectroscopy FTIR	35
3.5.2	Nuclear Magnetic Resonance NMR	36
3.6	Kinetic Model of Degradation of Epoxy ring	37
3.7	Optimization using the Taguchi Method	40
3.8	Concluding Remarks	42
 CHAPTER FOUR: RESULTS AND DISCUSSION		44
4.1	Kinetic Model	44
4.1.1	Validation of Kinetic Model	45
4.2	Optimization of Process Parameters	47
4.2.1	Signal-to-Noise Ratio	48
4.2.2	Analysis of Variance	48
4.2.3	Contour Plot of Response	49
4.2.4	Confirmation Test of Taguchi Optimization	50
4.3	Synthesis of DHSA via <i>In Situ</i> Hydrolysis based on Optimized Parameters	52
4.3.1	Fourier Transform Infrared Spectroscopy	53