UNIVERSITI TEKNOLOGI MARA

SYNTHESIS OF DIHYDROXYSTEARIC ACID FOR PALM OIL-DERIVED OLEIC ACID VIA SUSTAINABLE EPOXIDATION AND SUBSEQUENT RING-OPENING USING AMBERLITE IR-120H ION EXCHANGE RESIN CATALYST

SITI MARIAM BINTI A. RAHMAN

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

College of Engineering

November 2023

ABSTRACT

Vegetable oils have high content of unsaturated bond that can be converted into epoxidized oleic acid thus they are considered sustainable, renewable, and also an environmentally friendly. Many commercial epoxides in the market are mainly based on petroleum and animals which are not environmentally friendly. At present, there are limited studies on the production of epoxidized oleic acid using eco-friendly ion exchange resin method. Consequently, the objective of this study is to optimise the reaction conditions of epoxidation oleic acid using ion exchange resin (amberlite IR-120H) as a catalyst and subsequent ring-opening by hydrolysis for production dihydroxystearic acid (DHSA). Epoxidized oleic acid was produced by using in situ formed performic acid. Performic acid was formed by mixing formic acid as the oxygen carrier with hydrogen peroxide as the oxygen donor with applied amberlite IR-120H as catalyst. The oxirane rings are highly reactive to opening, especially through hydrolysis process to produced DHSA. The results showed maximum relative conversion of oleic acid to oxirane was achieved using the optimum conditions with up to 85% under following conditions: (1) concentration of hydrogen peroxide: 50%, (2) reaction temperature: 75°C, (3) type of catalyst: ion exchange resin, (4) formic acid to oleic acid molar ratio: 1.0, and (5) hydrogen peroxide to oleic acid molar ratio: 1.0 and, (6) catalyst concentration: 0.6 wt%. The optimum epoxidation reaction parameters were used to study the DHSA yield produced via in situ and ex situ hydrolysis of epoxidized oleic acid. Fourier transform infrared spectroscopy showed an absorption peak at a wavelength of 1200 cm-1 and 3300 cm-1, indicating the presence of a oxirane ring and hydroxyl group respectively. Overall, DHSA with a high hydroxyl value (182 mg KOH/g), was successfully produced from oleic acid using in situ hydrolysis of epoxidized oleic acid.

ACKNOWLEDGEMENT

Bismilahirrohmanirrohim. Alhamdulillah, all praises to Allah for His blessing, the opportunity and strength in completing this thesis. First and foremost, I am immensely grateful to my thesis supervisor, Ir. Dr Mohd Jumain bin Jalil for his guidance, expertise, and unwavering support. His profound knowledge, insightful feedback, and dedication have been instrumental in shaping this research and enhancing my academic growth.

I would also like to extend my heartfelt appreciation to the faculty members of School of Chemical Engineering UiTM Pulau Pinang who have provided me with an exceptional learning environment and valuable insights that have greatly contributed to the development of this thesis input, perspectives, and experimental assistance have been invaluable in refining the research questions and analyzing the results.

Lastly, I would like to acknowledge my late father A. Rahman bin Debok, my mum , my siblings, friends, and loved ones for their continuous encouragement, understanding, and patience throughout this challenging journey. Their unwavering belief in me has been a constant source of motivation and inspiration.

Although I have endeavored to mention everyone who played a significant role in this thesis, I apologize if any omissions have occurred unintentionally. To those names left unmentioned but indirectly contributed to the birth of this thesis, thank you very much from the bottom of my heart.

TABLE OF CONTENTS

		Page
CO]	NFIRMATION BY PANEL OF EXAMINERS	ii
AU	ΓHOR'S DECLARATION	iii
ABS	STRACT	iv
AC	KNOWLEDGEMENT	V
TABLE OF CONTENTS LIST OF TABLES LIST OF FIGURES LIST OF SYMBOLS LIST OF ABBREVIATIONS		vi
		ix
		x xi
CH	APTER 1 INTRODUCTION	1
1.1	Background of the Study	1
1.2	Problem Statement	2
1.3	Objectives	3
1.4	Scope and Limitations of the Study	4
1.5	Significance Contributions of the Study	5
1.6	Outline of the Thesis	5
CHAPTER 2 LITERATURE REVIEW		7
2.1	Palm Oil Production	7
	2.1.1 Fatty Acid Composition Saturated and Unsaturated Palm oil	9
2.2	Epoxidation of Unsaturated Fatty Acid	12
2.3	Epoxidation Process	14
	2.3.1 Reaction of Hydrogen Peroxide and Formic Acid	15
	2.3.1.1 Performic Acid Mechanism	15
2.4	Epoxidation Applied Amberlite IR-120H as Catalyst	16
2.5	Ring Opening of Epoxide	18
	2.5.1 Addition of Water	19
2.6	Overview of Epoxidation of Vegetables Oil	20

CHAPTER 1 INTRODUCTION

1.1 Background of the Study

In the modern era nowadays, palm oil is commonly used due to many benefits and advantages as a renewable resource rather than using petroleum that also in general and will be decrease sooner in the future. Palm oil has increased in demand for various products and become one of the largest exportations in the world. Amount of monthly production from year 2018 to year 2019 has been increasing which is from 13,897,927 tonnes to 15,190,569 tonnes respectively meanwhile crude palm kernel oil also increased from 1,634,626 tonnes to 1,765,714 tonnes from year 2018 to 2019 [1]. This shows that the production of palm oil keeps on demanding every year. The overall development of Malaysia's industry in palm oil is growing rapidly and the country has the most mature oil palm industry in the world [2]. Most of palm oil is not only used in food production but also in non-food applications such as surfactants, agrochemicals, candles and many more as the palm oil is an oleo chemical product that are available as another alternative being taken instead of petroleum products [3][4].

Epoxidation of palm oil is known as the carbon atoms adjacent to the alkyl group that are joined to the same oxygen atom in the structure and these products are named as epoxides or oxiranes [5]. In the epoxidation process, there are two methods known in industrial such as peracetic acid and performic acid. These two methods are most common used in epoxidation. Another epoxidation of vegetable oils are carried out with different method such as epoxidation with organic and inorganic peroxides, epoxidation with molecular oxygen and also epoxidation with halohydrins using hypohalous acids [6]. The application of epoxide has been widely used in industries such as stabilizers or plasticizers in plastic industry, finishing polyacrylate and as starting materials for the production of polyols [7]. Other than that, epoxide can be used to replace the volatile organic solvent in paints [8]. Generally, epoxide can be obtained by reacting of double bond of oil with peracid that generated in situ by reacting it with concentrated hydrogen peroxide and formic acid with the presence of catalyst [9]. Epoxide is obtained from renewable resources and it can be regarded as biodegradable and non-toxic [10]. Hence,it