UNIVERSITI TEKNOLOGI MARA

OPTIMIZATION OF DIHYDROXYSTEARIC ACID SYNTHESIS FROM PALM KERNEL OIL BASED OLEIC ACID USING TAGUCHI'S METHOD

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

Faculty of Chemical Engineering

November 2018

ABSTRACT

This study looks to establish the process factor that will contribute to the enhancement of the reaction rate and yield of dihydroxy stearic acid (DHSA) formation from palm kernel oil based crude oleic acid. Previous studies showed high quality of DHSA can be produced at satisfactory yield in batches. However, details on the influence of reaction parameters and reaction optimization that associate with the degradation of oxirane ring in water are lacking. For this purpose, the crude oleic acid was first epoxidized in a CSTR and optimized using the traditional one factor at a time (OFAT). The optimized conditions include reaction temperature (55-75 °C), agitation speed (200-300 rpm) and sulfuric acid catalyst loading (0.5-2.0g). It was found that the epoxidation yield resulted around 56% at agitation speed of 300 rpm, reaction temperature of 55°C and sulfuric acid catalyst loading of 2.0g. The FTIR analysis confirmed the formation of epoxy functional groups at 1210-1320 cm^{-1} absorption band region whereas the IV value recorded at 33.44 are comparable to previous epoxidation studies of vegetable oils. Subsequently, hydrolysis reaction on the pre-prepared epoxidized oleic acid produces dihydrostearic acid (DHSA). The formation of hydroxyl compound which characterized the DHSA was confirmed by the FTIR spectrum which showed the absorption band of hydroxyl group at the region 3200-3600cm^{-1}. In addition, the IV value of the DHSA was found to be at 10.4 which is within the range commonly reported value for DHSA. The application of Taguchi optimization method that supported by ANOVA statistical analysis allow for determination of optimum hydrolysis reaction conditions. It was found that all four reaction factors selected (reaction temperature, oil to solution ratio, catalyst type and agitation speed) were all significant in influencing the course of hydrolysis reaction. The optimum DHSA formation as indicated by the reduction in RCO(%) was achieved at the shortest reaction time of around 5 hours at reaction temperature of 80°C (level 3), oil to solution ratio of 0.75 (level 3), H_2SO_4 catalyst (level 2) and agitation speed of 650 rpm (level 1). This study also highlighted the kinetics of the hydrolysis reaction that is fundamental in designing a commercial scale DHSA production facility. The oxirane ring cleavage (hydrolysis) reaction rate constant K, were examined at reaction temperature of 55°C, 70°C, 80°C and 90°C. The corresponding kinetics analysis showed a linear relationship with reaction time, suggesting a pseudo first order reaction with respect to oxirane concentration. On the other hand, the reaction order examined at three water concentrations of 13.86, 27.76 and 38.88 $molL^{-1}$ revealed that the oxirane cleavage reaction was second order with respect to water concentration. It was determined that the DHSA synthesized through oxirane ring cleavage in water follows a kinetic model $r=k[EP][H_2O]^2$. Finally, the activation energy in the oxirane cleavage reaction was found to be 50.32 $kJmol^{-1}$. Overall, this work demonstrated that DHSA produced from palm kernel based oleic acid is commercially appealing not only because of the unique structure and properties that it possess but also because of other inherent advantages as they are derived from renewable and biodegradable source and hence reduces our dependency on petroleum and animal based oils.

ACKNOWLEDGEMENT

Alhamdulillah praise to Allah for the chances He gave me to complete this thesis. This thesis become reality with the kind support and help of many individuals. I would like to extend my sincere thanks to all of them.

I would like to express my gratitude towards my parents, En. Mohamed Bin Mat Din and for their endless encouragement in completion this thesis. My beloved and supportive husband, Shukur who is always by my side and my lovable children, Alfateh and Aqilah who served as my inspiration to pursue this undertaking.

I would like to express my special gratitude and appreciation to my supervisor, Assoc. Prof. Ir. Dr. Ahmad Rafizan Bin Mohamad Daud for his guidance, suggestion and imparting knowledge as well as patience. Thank you for not giving up upon me and always gave continuous supports. A sincere gratitude also goes to my co-supervisor, Cik Siti Khatijah Binti Jamaludin for her suggestions and imparting her expertise in this study. I am deeply indebted to both of you.

Towads this long journey, I also would like to express my appreciation to my suppportive friends and colleage that help me with their suggestions, sharing knowledge, moral supports and also encouragement to finish this journey. They were Izni Mariah, Hidayu, Arman, Nazri, Ezan and Hikmah.

I also take this opportunity to record our sincere thanks to Faculty of Chemical Engineering, UiTM for their guidance and constant supervision as well as providing necessary information regarding to this research.

I humbly extend my thanks to all concerned person who co-operated with me directly or undirectly in this journey to the birth of this thesis.

Thank you very much

NOORFAZLIDA MOHAMED, 2018

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CHAPTER ONE
INTRODUCTION

1.1 Background of Research

The production of palm oil worldwide has increased over the years due to its extensive use in foods as an alternative to other fat products with a high content of trans fatty acids, and due to its use by the bio-fuel industry. The statistic shows the production volume of palm oil worldwide from 2012/13 to 2016/17 will increase from 56.38 million metric tons to 66. 86 million metric tons (The Statistic Portal, 2018)

Traditionally 80% of palm oil is for edible use and 20% for non edible use such as oleochemical manufacture (Barison and Weng, 2004). The non-food application is only 20% but significant because of the high value product can be produced. Palm oil can be used directly or indirectly as oleochemicals. The storage oils obtained from oil palm fruits divided into palm oil from the mesocorp and palm kernel oil from the kernel. The fruit consisting of a hard kernel (seed) within a shell (endocarp) which in turn is surrounded by a fleshy mesocorp as illustrated in Figure 1.1 below.

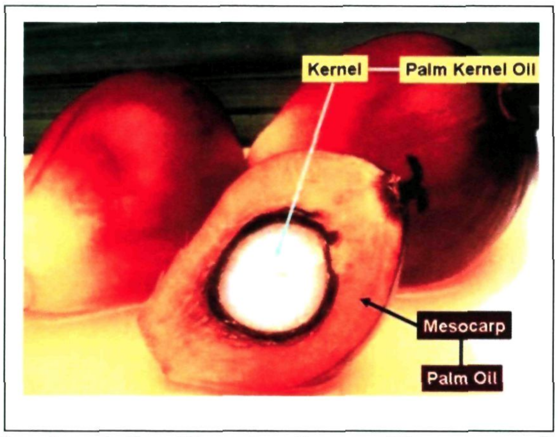

Figure 1.1 Cross Sectional Cut Showing InternalStructure of an Oil Palm Fruit

The mesocorp is made up of about 49% oil and about 50% kernel. The two oils (palm oil and palm kernel oil) have very different compositions. Palm oil (from the mesocorp) contains mainly palmitic acid (C16:0) and oleic acid (C18:1); the two most