High Yield Dihydroxystearic Acid (DHSA) Based on Kinetic Model from Epoxidized Palm Oil

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Abstract
In recent years, studies related to the epoxidation of fatty acids have garnered much interest due to the rising demand for eco-friendly epoxides derived from vegetable oils. From the epoxidation reaction, there is a side reaction involving epoxide and water. This reaction produces a by-product – dihydroxystearic acid (C18H36O4, DHSA). DHSA is one of the chemical precursors in the production of cosmetic products. Therefore, a kinetic model was developed to determine the optimised epoxidation process and concentration of DHSA, where each of the reactions was identified. The kinetic rate, k parameters obtained were: k1 = 6.6442, k2 = 11.01185, k3 = 0.1026 for epoxidation palm oleic acid, and k41 = 0.0021, k51 = 0.0142 in degradation process. The minimum error of the simulation was 0.0937. In addition, DHSA yield optimisation was done through Taguchi method, and the optimum conditions obtained were H2O2/oleic acid – OA unsaturation molar ratio 1 : 1 (level 2), formic acid – FA/OA unsaturation molar ratio 0.5 : 1 (level 1), temperature 35 °C (level 1), and agitation speed 100 rpm (level 1). A high yield of DHSA can be achieved under these conditions.

Keywords
Epoxidation, kinetic rate, MATLAB, DHSA, Taguchi

1 Introduction
The growth in vegetable oil demand has increased compared to the petroleum-based polymer. Since petroleum causes environmental pollution concerns, vegetable oil has become an alternative in the production of the epoxy. In the epoxidation reaction of vegetable oil, oxirane ring-opening, known as epoxide ring degradation, will occur. The oxirane ring-opening depletes the yield and can cause high peroxide values of epoxidized vegetable oils, so the opening needs to be minimised. The commercial production of palm oil is quite high compared to that of other vegetable oils. The low cost of palm oil makes studies on the synthesis and oxirane cleavage of palm oil more practical and economical. Epoxidized palm oil (EPO) can be obtained by reacting to the double bond of oil with peroxy acid that is generated in situ by reacting with concentrated hydrogen peroxide (H2O2) and formic acid (CH2O2, FA) in the presence of mineral salt as a catalyst. The EPO can be used as a raw material in the manufacture of a wide range of products, such as paint, plastic, and adhesives. Besides, EPO is obtained from renewable resources, and can be regarded as biodegradable and non-toxic. Hence, it is suitable in replacing petroleum, since petroleum is toxic and harmful to the environment.

In this study, the oxirane-ring degradation of epoxidation palm oil was determined through MATLAB simulation. Ode45 is one of the tools in MATLAB designed to work with differential equations. The benefit of this tool is its ability to determine the reaction rate of the epoxidation and degradation of EPO. Nevertheless, the epoxy that reacts with water produces DHSA. The physical appearance of DHSA is white and tasteless with an acid odour, and is non-irritating to the skin. It is suitable for cosmetic ingredients. As the cosmetic industry is gradually increasing, it is suitable to produce a large amount of DHSA. However, literature on the optimisation of DHSA production is still lacking and has only a few references. Thus, in this paper, the optimisation of DHSA production will be studied by the Taguchi method, and by referring the data from the simulation.

2 Methodology
2.1 Kinetic model
Two main reactions involved in epoxidation of palm oil can be illustrated in Eqs. 1 and 2, while, the degradation of epoxidation is described in Eqs. 3, 4, and 5.

\[ \text{FA} + \text{H}_2\text{O}_2 \xrightarrow{k_{11}} \text{PFA} + \text{H}_2\text{O} \quad (1) \]
\[ \text{PFA} + \text{OA} \rightarrow \text{EPOXY} + \text{FA} \quad (2) \]
\[ \text{EPOXY} + \text{H}_2\text{O} \rightarrow \text{DHSA} \quad (3) \]
\[ \text{EPOXY} + \text{H}_2\text{O}_2 \rightarrow \text{DEG1} \quad (4) \]
\[ \text{EPOXY} + \text{FA} \rightarrow \text{DEG2} \quad (5) \]
where FA, H₂O₂, PFA, H₂O, OA, EPOXY, and DHSA are formic acid, hydrogen peroxide, performic acid, water, oleic acid, epoxide vegetable oil, and dihydroxystearic acid, respectively. From the given epoxidation and degradation process, the differential equations that describe each species are further derived as follows:

\[
\frac{d[FA]}{dt} = -k_{11}[FA][H_2O] + k_{12}[PFA][H_2O] + i_{21} + k_{21}[PFA][OA] - k_{22}[EPOXY][FA] - k_{31}[EPOXY][FA] + k_{32}[DEG2]
\]

\[
\frac{d[H_2O]}{dt} = -k_{11}[FA][H_2O] + k_{12}[PFA][H_2O] - k_{21}[PFA][OA] + k_{22}[EPOXY][FA]
\]

\[
\frac{d[PFA]}{dt} = k_{11}[FA][H_2O] - k_{12}[PFA][H_2O] - k_{21}[PFA][OA] + k_{22}[EPOXY][FA]
\]

\[
\frac{d[H_2O]}{dt} = k_{11}[FA][H_2O] - k_{12}[PFA][H_2O] - k_{31}[H_2O][EPOXY] + k_{32}[DHSA]
\]

\[
\frac{d[OA]}{dt} = -k_{21}[PFA][OA] + k_{22}[EPOXY][FA]
\]

\[
\frac{d[EPOXY]}{dt} = k_{21}[PFA][OA] - k_{22}[EPOXY][FA] - k_{31}[EPOXY][H_2O] + k_{32}[EPOXY][H_2O]
\]

\[
\frac{d[DHSA]}{dt} = k_{31}[EPOXY][H_2O] - k_{32}[DHSA]
\]

\[
\frac{d[DEG1]}{dt} = k_{41}[EPOXY][H_2O] - k_{42}[DEG1]
\]

\[
\frac{d[DEG2]}{dt} = k_{51}[EPOXY][FA] - k_{52}[DEG2]
\]

To determine the rate coefficient numerically, parametric studies were conducted. There were two computing processes involved, which solved a set of differential equations (Eqs. 6–14) numerically, and computed the errors between the experimental and the simulation. The Ode45 function of MATLAB was used to solve the differential equation by numerical integration using the fourth-order Runge-Kutta method. The parameter values were predicted using a genetic algorithm in MATLAB software. The algorithm can employ a set of orthogonal arrays; with reaction parameters, optimisation is performed using the lowest possible number of experimental runs. A few parameters, such as reaction temperature, FA to OA unsaturation molar ratio, H₂O₂ to OA unsaturation molar ratio, and agitation speed can affect the reaction of epoxidation. All these parameters were explored with the diversity of levels, as shown in Table 1. Therefore, the optimal reaction conditions of these vital reaction temperatures were analysed based on a DOE approach. The diversity of factors was studied by crossing the orthogonal array of the control parameters. The result obtained was further analysed manually using the signal to noise (S/N) ratio and analysis of variance (ANOVA). The optimum combination of reaction conditions projected by the Taguchi method was then tested and validated by running a confirmation reaction at the optimal predicted reaction conditions.

Table 1 – Design of experiment (DOE) using four parameters and three levels

| Parameters                      | Levels |
|---------------------------------|--------|
| A H₂O₂/OA unsaturation molar ratio | 0.5 1 1.5 |
| B FA/OA unsaturation molar ratio | 0.5 1 1.5 |
| C Temperature/°C                | 35 55 75 |
| D Agitation speed/rpm           | 100 200 300 |
Choosing the parameters was conducted according to the suggestion given by D. B. Wijayasekara. For instance, 100 rpm, 200 rpm, and 300 rpm were the agitation speeds tested. The reaction temperatures (35, 55, 75 °C) were the reaction parameters, unsaturation molar ratio (0.5, 1.0, 1.5) of FA to OA, and unsaturation molar ratio (0.5, 1.0, 1.5) of H$_2$O$_2$ to OA, as shown in Table 1. Optimisation was conducted using the analytical Taguchi method to find the optimum conditions for every parameter.

3 Results and discussion

3.1 Reaction rate of DHSA production

Kinetic model for catalytic epoxidation of palm oleic acid based on palm oil (PO) was developed by MATLAB simulation. The kinetic data for the epoxidation and degradation of palm oil corresponded to the initial concentration. The Genetic Algorithm (GA) method was used to fit the experimental data and the Runge-Kutta fourth-order method was applied using the Ode45 tool to solve the system of differential equations. There were 27 experiments run in the simulation, in order to ensure the optimal value of epoxidation. The experimental data was obtained from the previous experiments, and the simulation was based on that data. The initial concentration of formic acid (FA), hydrogen peroxide (H$_2$O$_2$), and oleic acid (OA) from previous experimental data was chosen as a reference to find the kinetic rate constant, $k$. On the other hand, the kinetic rate was used to determine the concentration of DHSA in oxirane cleavage. This occurred when the epoxide reacted with water. It corresponded to the objective of this study.

From the 27 experiments that were run (Table 2), experiment 11 was chosen as the best to display the optimised epoxidation process, as it had the lowest error (0.0937%) compared to others. The reaction rate constants for this experiment were: $k_{11} = 6.6442$ mol l$^{-1}$ min$^{-1}$, $k_{12} = 11.0185$ mol l$^{-1}$ min$^{-1}$, $k_{13} = 0.1026$ mol l$^{-1}$ min$^{-1}$.

| Exp | Initial concentration/mol l$^{-1}$ | $k_{11}$/mol l$^{-1}$ min$^{-1}$ | $k_{12}$/mol l$^{-1}$ min$^{-1}$ | $k_{13}$/mol l$^{-1}$ min$^{-1}$ | $k_{14}$/mol l$^{-1}$ min$^{-1}$ | $k_{15}$/mol l$^{-1}$ min$^{-1}$ | $k_{16}$/mol l$^{-1}$ min$^{-1}$ | $k_{17}$/mol l$^{-1}$ min$^{-1}$ | Error min | DHSA estimated concentration/mol l$^{-1}$ |
|-----|----------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|----------------------------------|-----------|----------------------------------|
| 1   | 1.4714 1.4714 2.9484             | 0.1174 0.0633 0.1315             | 0.1366 0.0142 0.2945             | 0.733307                         | 0.2694 0.2892 1.017948           |
| 2   | 1.4714 1.4714 2.9484             | 0.1028 0.0070 0.0138             | 0.0166 0.2694 0.700095           |
| 3   | 1.4714 1.4714 2.9484             | 0.0543 0.0075 0.0028             | 0.0074 0.2892 1.017948           |
| 4   | 1.4714 2.9484 2.9484             | 6.0696 0.1572 0.0243             | 0.0131 0.1500 0.404717           |
| 5   | 1.4714 2.9484 2.9484             | 0.0299 0.0107 0.0105             | 0.0068 0.1525 0.467933           |
| 6   | 1.4714 2.9484 2.9484             | 0.0691 0.1153 0.0078             | 0.005 0.2158 0.469234           |
| 7   | 1.4714 4.4226 2.9484             | 0.0391 0.0157 0.0171             | 0.0041 0.1815 1.583085           |
| 8   | 1.4714 4.4226 2.9484             | 0.0168 0.0077 0.0003             | 0.005 0.2258 0.360169           |
| 9   | 1.4714 4.4226 2.9484             | 0.0477 0.1153 0.0078             | 0.005 0.2258 0.360169           |
| 10  | 2.9484 1.4714 2.9484             | 6.4757 10.7925 0.0583             | 0.0171 0.0041 1.583085           |
| 11  | 2.9484 1.4714 2.9484             | 6.6442 11.0185 0.1026             | 0.0238 0.0021 0.0014 0.0937 1.954907 |
| 12  | 2.9484 1.4714 2.9484             | 12.8848 0.1025 0.0122             | 0.1811 0.1136 1.603342           |
| 13  | 2.9484 2.9484 2.9484             | 0.1168 0.0019 0.0058             | 0.1978 1.941024 0.672475         |
| 14  | 2.9484 2.9484 2.9484             | 0.0027 0.119431 0.0196             | 0.0058 0.1978 1.941024           |
| 15  | 2.9484 2.9484 2.9484             | 0.0505 0.1707 0.0147             | 0.0107 0.2550 1.627645           |
| 16  | 2.9484 2.9484 2.9484             | 3.5703 0.0613 0.0135             | 0.0008 0.1331 1.293596           |
| 17  | 2.9484 2.9484 2.9484             | 2.8117 0.0273 0.0203             | 0.0062 0.0723 1.37666             |
| 18  | 2.9484 4.4226 2.9484             | 4.6865 0.11910 0.0106             | 0.0055 0.2295 1.33783            |
| 19  | 2.9484 1.4714 2.9484             | 1.5004 0.0426 0.0203             | 0.0205 0.1239 1.602207           |
| 20  | 2.9484 1.4714 2.9484             | 0.102 0.0095 0.0012             | 0.0365 0.1770 1.532 1.759485     |
| 21  | 4.4226 1.4714 2.9484             | 4.6066 0.0925 0.0233             | 0.0101 0.0062 0.2219 0.3293 1.644378 |
| 22  | 4.4226 2.9484 2.9484             | 0.0128 0.0102 0.0012             | 0.0315 0.1083 1.677388           |
| 23  | 4.4226 2.9484 2.9484             | 8.8191 1.6479 0.0231             | 0.0100 0.0033 0.0166 0.1530 1.719904 |
| 24  | 4.4226 2.9484 2.9484             | 12.3125 6.7678 0.0036             | 9.1681 0.0003 0.2815 0.963317     |
| 25  | 4.4226 4.4226 2.9484             | 5.9174 0.1286 0.0272             | 0.0071 0.0226 0.213 2.420 1.208793 |
| 26  | 4.4226 4.4226 2.9484             | 0.0230 0.0073 0.0014             | 0.0073 0.1164 1.980805           |
| 27  | 4.4226 4.4226 2.9484             | 10.3146 0.6149 0.0414             | 0.0130 0.0361 0.1213 2.482569     |
The “larger-the-better” S/N ratio indicated better performances regardless of the performance characteristics. According to the previous research, the optimum level for all parameters would be the level with the greatest S/N ratio. The 27 experimental runs based on Taguchi method suggested that the optimum DHSA was obtained at $\text{H}_2\text{O}_2$/OA unsaturation molar ratio of 1 : 1 (level 2), FA/OA unsaturation molar ratio 0.5 : 1 (level 1), temperature of 35 °C (level 1), and agitation speed of 100 rpm (level 1). These results were plotted for each parameter in Fig. 2.

### Table 3 – Taguchi orthogonal array experimental design for DHSA

| Run | Temperature / °C | FA/OA | $\text{H}_2\text{O}_2$/OA | Agitation speed / rpm | DHSA estimated concentration / mol l$^{-1}$ |
|-----|------------------|-------|-----------------|---------------------|---------------------------------|
| 1   | 1                | 1     | 1               | 1                   | 0.733307                       |
| 2   | 1                | 1     | 1               | 1                   | 0.700095                       |
| 3   | 1                | 1     | 1               | 1                   | 1.017948                       |
| 4   | 1                | 2     | 2               | 2                   | 0.404717                       |
| 5   | 1                | 2     | 2               | 2                   | 0.626330                       |
| 6   | 1                | 2     | 2               | 2                   | 0.482747                       |
| 7   | 1                | 3     | 3               | 3                   | 0.467893                       |
| 8   | 1                | 3     | 3               | 3                   | 0.469234                       |
| 9   | 1                | 3     | 3               | 3                   | 0.360169                       |
| 10  | 2                | 1     | 2               | 3                   | 1.583085                       |
| 11  | 2                | 1     | 2               | 3                   | 1.954907                       |
| 12  | 2                | 1     | 2               | 3                   | 1.603342                       |
| 13  | 2                | 2     | 3               | 1                   | 1.592831                       |
| 14  | 2                | 2     | 3               | 1                   | 1.941024                       |
| 15  | 2                | 2     | 3               | 1                   | 1.627645                       |
| 16  | 2                | 3     | 1               | 2                   | 1.293596                       |
| 17  | 2                | 3     | 1               | 2                   | 1.376660                       |
| 18  | 2                | 3     | 1               | 2                   | 1.337830                       |
| 19  | 3                | 1     | 3               | 2                   | 1.602207                       |
| 20  | 3                | 1     | 3               | 2                   | 1.759485                       |
| 21  | 3                | 1     | 3               | 2                   | 1.644378                       |
| 22  | 3                | 2     | 1               | 3                   | 1.677388                       |
| 23  | 3                | 2     | 1               | 3                   | 1.719904                       |
| 24  | 3                | 2     | 1               | 3                   | 0.963317                       |
| 25  | 3                | 3     | 2               | 1                   | 1.208793                       |
| 26  | 3                | 3     | 2               | 1                   | 1.980805                       |
| 27  | 3                | 3     | 2               | 1                   | 2.482569                       |
List of abbreviations

DEG 1  – degradation 1
DEG 2  – degradation 2
DHSA  – dihydroxystearic acid
FA    – formic acid
OA    – oleic acid
EPO   – epoxidized palm oil
PFA   – performic acid
EPOXY – epoxidized oil
S/N   – signal to noise

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Dihidroksistearinska kiselina (DHSA) visokog prinosa temeljena na kinetičkom modelu iz epoksidiranog palminulja

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Posljednjih godina studije povezane s epoksidacijom masnih kiselina izazvali su veliko zanimanje zbog sve veće potražnje za ekološki prihvatljivim epoksidima dobivenim iz biljnih ulja. Iz reakcije epoksidacije dolazi do nuspojave koja uključuje epoksid i vodu. Tom reakcijom nastaje nusproizvod – dihidroksistearinska kiselina (DHSA). DHSA jedan je od kemijskih prekursora u proizvodnji kozmetičkih proizvoda. Stoga je razvijen kinetički model za određivanje optimiranog procesa epoksidacije i koncentracije DHSA, gdje je identificirana svaka od reakcija. Dobiveni parametri kinetičke brzine, \( k \) bili su: \( k_{11} = 6,6442, k_{12} = 11,0185, k_{21} = 0,1026 \) za epoksida-ciju palmino-oleinsku kiselinu i \( k_{41} = 0,0021, k_{51} = 0,0142 \) u procesu razgradnje. Minimalna pogreška simulacije bila je 0,0937. Uz to, optimizacija prinosa DHSA provedena je Taguchijevom metodom, a dobiveni optimalni uvjeti su molarni omjer nezasićenja H₂O/oleinske kiseline – OA 1:1 (razina 2), molarni omjer nezasićenja mravlje kiseline – FA/OA 0,5:1 (razina 1), temperatura 35 °C (razina 1) i brzina miješanja 100 o min⁻¹ (razina 1). Pod tim se uvjetima može postići visok prinos DHSA.

Ključne riječi
Epoksidacija, kinetika, MATLAB, DHSA, Taguchi

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SAŽETAK

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