UNIVERSITI PUTRA MALAYSIA

TRANSESTERIFICATION OF PALM-BASED METHYL ESTER AND TRIMETHYLOLPROPANE FOR TRIMETHYLOLPROPANE ESTERS PRODUCTION USING MICROWAVE-ASSISTED HEATING

NUR ATIQAH BINTI MOHAMAD AZIZ

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By

NUR ATIQAH BINTI MOHAMAD AZIZ

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Chair : Prof Robiah binti Yunus, PhD
Faculty : Engineering

Malaysia being the second largest palm oil producer in the world, is currently promoting environmentally friendly or green products including palm oil-based lubricants. Biolubricants have shown good potential as an alternative to mineral-based lubricants and exhibit superior lubricating properties. Trimethylolpropane triester (TMPTE) is one of the synthetic base oils for biolubricants and can be derived from palm oil. TMPTE has good thermal and oxidative stability, density, viscosity index, and pour point, and wear properties. TMPTE has been produced using conventional thermal-heating transesterification process which consumes more energy due to a long production time.

In this study, microwave heating has been chosen as an alternative synthesis method in the production of TMPTE as it is known to provide a more efficient heating. The processing parameters for the microwave reaction were temperature (90 – 150˚C), catalyst amount (0.2 – 1.0 wt.%), reaction time (3 – 25 minutes), molar ratio TMP-to-PME (1:3 – 1:4.5), and pressure (10 – 50 mbar). The highest composition of TMPTE produced by microwave heating was found to be 69 % (98 % of total esters) obtained only in 10 minutes, at 130°C, 10 mbar pressure, using 0.6 wt.% catalyst, and 1:4 molar ratio of TMP-to-PME. Furthermore, sample preheating to 130℃ for microwave-assisted significantly reduced to 3 minutes as compared to 30 to 40 minutes required with conventional heating.

To understand the mechanism of the microwave-assisted transesterification between PME and TMP, kinetics study of the reaction was conducted at 110, 120, 130 and 140°C. The reaction kinetics was successfully modelled in MATLAB using the second-order reversible reaction, and the results correlated well with the experimental data. The fastest reaction rate for TMPTE production

occurred at 130℃, with activation energies ranged from 17.0 -24.7 kcal/mol, clearly lower than the conventional method. When compared to conventional heating method the lower activation energy reflects an energy savings of 68.4 %.

The distribution of electric fields in the microwave-assisted transesterification associated with the hotspots, temperature profile and power absorbed were simulated using COMSOL Multiphysics 4.2. As some part of the microwave was absorbed by the sample, the electric field and heating profile showed an alignment of intense heating at the top corner of the sample. The electric field and power absorbed by the sample were 1.92×10^4 V/m and 1.38×10^7 W/m^3, respectively. Overall, microwave heating has been proven successful in accelerating the production of TMPTE.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

TRANSESTERIFIKASI METIL ESTER BERASASKAN KELAPA SAWIT BAGI MENGHASILKAN TRIMETILOLPROPANA METIL ESTER MENGGUNAKAN PEMANASAN GELOMBANG MIKRO

Oleh

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Malaysia sebagai negara kedua terbesar pengeluar minyak kelapa sawit dunia menggalakkan penggunaan produk mesra alam dan hijau termasuklah bio-pelincir berasaskan kelapa sawit. Bio-pelincir berpotensi sebagai alternatif pelincir berasaskan mineral, menunjukkan prestasi pelinciran yang lebih baik. Trimetilpropana triester (TMPTE) ialah asas minyak sintetik bagi bio-pelincir yang diperolehi berasaskan minyak kelapa sawit. TMPTE mempunyai kestabilan suhu, pengoksidaan, ketumpatan, indeks kelikatan, takat tuang dan ciri tahan haus yang baik. TMPTE yang dihasilkan secara konvensional, iaitu pemanasan suhu bagi proses transesterifikasi menggunakan lebih banyak tenaga kerana panjang tempoh penghasilannya. Penyelidikan ini memilih pemanasan gelombang mikro sebagai alternatif pemanasan menghasilkan TMPTE kerana ia diketahui memanaskan dengan lebih berkesan. Faktor pemprosesan bagi tindak balas menggunakan pemanasan gelombang mikro adalah suhu (90 – 150˚C), kuantiti pemangkin (0.2 – 1.0 wt. %), tempoh tindak balas (3 – 25 minit), nisbah trimetilolpropana (TMP) kepada metil ester minyak sawit (PME) (1:3 – 1:4.5), dan tekanan (10 – 50 mbar). Penghasilan TMPTE tertinggi menggunakan pemanasan gelombang mikro adalah sebanyak 69 % (dengan jumlah ester 98 %) hanya dalam 10 minit, pada 130°C, 10 mbar tekanan, menggunakan 0.6 wt.% pemangkin, dan 1:4 nisbah TMP kepada PME. Tempoh memanaskan sample juga hanya 3 minit berbanding 30 – 40 minit bagi pemanasan biasa.

Bagi memahami mekanisma tindak balas menggunakan gelombang mikro, kajian kinetik bagi penghasilan TMPTE pada suhu 110, 120, 130 dan 140°C telah dijalankan. Model kinetik dibangunkan daripada tindak balas berturutan dan secara berbalik. Model terpilih menunjukkan ketepatan yang tinggi berikutan dengan ralat standard yang rendah dan hubungan model yang baik bagi profil taburan produk antara simulasi dengan data eksperimen, menggunakan MATLAB. Pada 130°C, kadar tindak balas adalah paling laju bagi penghasilan

TMPTE, dan tenaga pengaktifan bagi menghasilkan TMPTE adalah 17 – 24.7 kcal/mol, yang mana terbukti lebih rendah berbanding kaedah pemanasan tradisional. Tenaga pengaktifan ini berhubungkait dengan penjimatan tenaga 68.4% berbanding menggunakan kaedah pemanasan konvensional.

Untuk memahami interaksi antara sampel dan gelombang mikro, simulasi telah dijalankan dengan menggunakan perisian COMSOL Multiphysics 4.2. Taburan medan elektrik di dalam ketuhar bagi titik panas, profil suhu dan tenaga yang diserap oleh sampel dinilai dengan jelas. Oleh kerana sebahagian daripada gelombang mikro diserap oleh sampel, medan elektrik dan profil pemanasan menunjukkan pemanasan ketara dan setempat di sudut atas sampel. Medan elektrik dan daya yang diserap oleh sampel masing-masing adalah 1.92 x 104 V/m dan 1.38 x 107 W/m^3. Secara keseluruhan, pemanasan gelombang mikro telah terbukti berjaya dalam mempercepat pengeluaran TMPTE.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

BSTFA	N,O-Bis(trimethylsilyl)trifluoroacetamide
DE	Diester
DSC	Differential Scanning Calorimetric
emw	Electromagnetic Wave
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acids
FTIR	Fourier Transform Infrared Spectroscopy
GC	Gas Chromatography
ht	Heat Transfer
ME	Monoester
Met	Methanol
NPG	Neopentylglycol
ODE	Ordinary Differential Equation
PAO	Poly Alpha Olefin
PE	Pentaerythritol
PFAD	Palm Fatty Acid Distillate
PI	Prediction Interval
PME	Palm oil Methyl Ester
PWM	Pulse width modulation
re	Reaction Engineering
SD	Standard Deviation
SE	Standard Error
TE	Triester
TGA	Thermogravimetric Analysis
TMP	Trimethylolpropane
TMPDE	Trimethylolpropane Diester
TMPE	Trimethylolpropane Ester

TMPME	Trimethylolpropane Monoester
TMPTE	Trimethylolpropane Triester

LIST OF NOTATIONS

ρ	Density
C_p	Heat capacity
D	Diameter
E_{Heat}	Energy consumption by heating
E_{Vacuum}	Energy consumption by vacuum
E_R	Energy absorbed by reaction system
ε'	Dielectric constant
ε"	Loss factor
k	Reaction rate constant
$tan\ \delta$	Loss tangent
$T_{reaction}$	Reaction temperature
T_{ref}	Reference temperature
A	Pre-exponential value
C_A	Concentration of A
X_A	Mass fraction of A
MW	Molecular weight
ΔG	Gibbs energy
ΔH	Enthalpy change
ΔS	Entropy change
wo	Oven width
do	Oven depth
ho	Oven height
wg	Waveguide width
dg	Waveguide depth
hg	Waveguide height
rf	Mixture radius
T0	Initial temperature

E	Electric field
H	Magnetic field
B	Magnetic Induction
j	Conduction current
Q_{MW}	Heat source from microwave

LIST OF EQUATIONS

$\mathrm{RCOOR'} + \mathrm{R''OH} \leftrightarrow \mathrm{RCOOR''} + \mathrm{R'OH}$ Eq. (1)

$TMP + PME \leftrightarrow TMPME + Met$ Eq. (2)

$TMPME + PME \leftrightarrow TMPDE + Met$ Eq. (3)

$TMPDE + PME \leftrightarrow TMPTE + Met$ Eq. (4)

$\mathrm{TMP} + 3\ \mathrm{PME} \leftrightarrow \mathrm{TMPTE} + 3\ \mathrm{Met}$ Eq. (5)

$\mathrm{R'COOR} + \mathrm{H_2O} \leftrightarrow \mathrm{R'COOH} + \mathrm{ROH}$ Eq. (6)

$\mathrm{R'COOH} + \mathrm{NaOH} \leftrightarrow \mathrm{RCOONa} + \mathrm{H_2O}$ Eq. (7)

$\mathrm{RCOOCH3} + \mathrm{H\text{-}OH} \rightarrow \mathrm{RCOOH} + \mathrm{CH3OH}$ Eq. (8)

$\mathrm{RCOOH} + \mathrm{CH3ONa} \rightarrow \mathrm{RCOONa} + \mathrm{CH3OH}$ Eq. (9)

$\tau_R = \frac{v\eta}{kT}\xi C + \tau_0$ Eq. (10)

$\tau_0 \approx \frac{2\pi\sqrt{\frac{I}{kT}}}{9}$ Eq. (11)

$\eta = \frac{Nh}{V}\exp\left(\frac{\Delta G}{RT}\right)$ Eq. (12)

$\tau = \frac{4\pi r^3 \mu}{k_b Temp}$ Eq. (13)

$tan\ \delta = \frac{\varepsilon''}{\varepsilon'}$ Eq. (14)

$c = f\lambda$ Eq. (15)

$Q = \pi f \varepsilon_0 \varepsilon''_{eff} |E|^2$ Eq. (16)

$$TMP + PME \underset{k1r}{\overset{k1}{\rightleftarrows}} \text{ME + Met}$$ Eq. (17)

$$ME + PME \underset{k2r}{\overset{k2}{\rightleftarrows}} \text{DE + Met}$$ Eq. (18)

$$DE + PME \underset{k3r}{\overset{k3}{\rightleftarrows}} \text{TE + Met}$$ Eq. (19)

$$TMP + 3\,PME \underset{kor}{\overset{ko}{\rightleftarrows}} \text{TE + 3 Met}$$ Eq. (20)

$$[PME] = [PME_0] - ([TMP_0]-[TMP])$$ Eq. (21)

$$[TE] = 100 - [PME]-[TMP]-[ME]-[DE]$$ Eq. (22)

$$\frac{dC_{TE}}{dt} = k_3\, C_{DE}\, C_{PME} - k_{3r}\, C_{TE}\, C_M$$ Eq. (23)

$$C_A = \frac{X_a \cdot \rho_T}{MW_A}$$ Eq. (24)

$$\frac{dC_A}{dt} = \frac{dX_A \cdot \rho_T}{dt \cdot MW_A}$$ Eq. (25)

$$\frac{dX_{TE}}{dt} = k_3 X_{DE}\, X_{PME} \left(\frac{MW_{TE} \cdot \rho_T}{MW_{DE} \cdot MW_{PME}}\right) - k_{3r} X_{TE} X_M \left(\frac{\rho_T}{MW_M}\right)$$ Eq. (26)

$$\ln (1 - X) = - kt$$ Eq. (27)

$$log_{10}(k) = -\frac{E_a}{2.303RT} + C$$ Eq. (28)

$$A = \gamma\lambda^2\Gamma$$ Eq. (29)

$$\Delta G = \Delta H - T\Delta S$$ Eq. (30)

$$ln\frac{k}{T} = -\frac{\Delta H}{RT} + ln\frac{k_b}{h} + \frac{\Delta S}{R}$$ Eq. (31)

$$TMP + PME \underset{k1r}{\overset{k1}{\rightleftarrows}} TMPME + Met$$ Eq. (32)

$$TMPME + PME \underset{k2r}{\overset{k2}{\rightleftarrows}} TMPDE + Met$$ Eq. (33)

$$TMPDE + PME \underset{k3r}{\overset{k3}{\rightleftarrows}} TMPTE + Met$$ Eq. (34)

$$TMP + 3\,PME \underset{kor}{\overset{ko}{\rightleftarrows}} TMPTE + 3\,Met$$ Eq. (35)

$$r_{TMP} = -k_1\,C_{TMP}\,C_{PME} + k_{1r}\,C_{TMPME}\,C_{Met}$$ Eq. (36)

$$r_{TMPME} = -k_2\,C_{TMPME}\,C_{PME} + k_{2r}\,C_{TMPDE}C_{Met} + k_1\,C_{TMP}\,C_{PME} - k_{1r}\,C_{TMPME}\,C_{Met}$$ Eq. (37)

$$r_{TMPDE} = k_2\,C_{TMPME}\,C_{PME} - k_{2r}\,C_{TMPDE}\,C_{Met} - k_3\,C_{TMPDE}\,C_{PME} + k_{3r}\,C_{TMPTE}\,C_{Met}$$ Eq. (38)

$$r_{TMPTE} = k_3\,C_{TMPDE}\,C_{PME} - k_{3r}\,C_{TMPTE}\,C_{Met}$$ Eq. (39)

$$r_{PME} = -k_1\,C_{TMP}\,C_{PME} + k_{1r}\,C_{TMPME}\,C_{Met} - k_2\,C_{TMPME}\,C_{PME} + k_{2r}\,C_{TMPDE}\,C_{Met} - k_3\,C_{TMPDE}\,C_{PME} + k_{3r}\,C_{TMPTE}\,C_{Met}$$ Eq. (40)

$$r_{Met} = k_1\,C_{TMP}\,C_{PME} - k_{1r}\,C_{TMPME}\,C_{Met} + k_2\,C_{TMPME}\,C_{PME} - k_{2r}\,C_{TMPDE}\,C_{Met} + k_3\,C_{TMPDE}\,C_{PME} - k_{3r}\,C_{TMPTE}\,C_{Met}$$ Eq. (41)

$$\frac{dX_{TMP}}{dt} = -k_1\,X_{TMP}\,X_{PME}\left(\frac{\rho_T}{MW_{PME}}\right) + k_{1r}\,X_{TMPME}\,X_{Met}\left(\frac{\rho_T\,MW_{TMP}}{MW_{TMPME}MW_{Met}}\right)$$ Eq. (42)

$$\frac{dX_{TMPME}}{dt} = +k_1 X_{TMP} X_{PME} \left(\frac{MW_{TMPME}\rho_T}{MW_{TMP}MW_{PME}}\right) - \quad \text{Eq. (45)}$$
$$k_{1r} X_{TMPME} X_{Met} \left(\frac{\rho_T}{MW_{Met}}\right) - k_2 X_{TMPME} X_{PME} \left(\frac{\rho_T}{MW_{Met}}\right) +$$
$$k_{2r} X_{TMPDE} X_{Met} \left(\frac{MW_{TMPME}\rho_T}{MW_{TMPDE}MW_{Met}}\right) \quad \text{Eq. (43)}$$

$$\frac{dX_{TMPDE}}{dt} = +k_2 X_{TMPME} X_{PME} \left(\frac{MW_{TMPDE}\rho_T}{MW_{PME}MW_{TMPME}}\right) -$$
$$k_{2r} X_{TMPDE} X_{Met} \left(\frac{\rho_T}{MW_{Met}}\right) - k_3 X_{TMPDE} X_{PME} \left(\frac{\rho_T}{MW_{PME}}\right) +$$
$$k_{3r} X_{TMPTE} X_{Met} \left(\frac{MW_{TMPDE}\rho_T}{MW_{TMPTE}MW_{Met}}\right) \quad \text{Eq. (44)}$$

$$\frac{dX_{TMPTE}}{dt} = k_3 X_{TMPDE} X_{PME} \left(\frac{MW_{TMPTE}\rho_T}{MW_{TMPDE}MW_{PME}}\right)$$
$$- k_{3r} X_{TMPTE} X_{Met} \left(\frac{\rho_T}{MW_{Met}}\right)$$

$$\frac{dX_{PME}}{dt} = -k_1 X_{TMP} X_{PME} \left(\frac{\rho_T}{MW_{TMP}}\right) \quad \text{Eq. (46)}$$
$$+ k_{1r} X_{TMPME} X_{Met} \left(\frac{MW_{TMP}\rho_T}{MW_{TMPME}MW_{Met}}\right)$$
$$- k_2 X_{TMPME} X_{PME} \left(\frac{\rho_T}{MW_{TMPME}}\right)$$
$$- k_{2r} X_{TMPDE} X_{Met} \left(\frac{MW_{PME}\rho_T}{MW_{TMPDE}MW_{Met}}\right)$$
$$- k_3 X_{TMPDE} X_{PME} \left(\frac{\rho_T}{MW_{TMPDE}}\right)$$
$$- k_{3r} X_{TMPTE} X_{Met} \left(\frac{MW_{PME}\rho_T}{MW_{TMPTE}MW_{Met}}\right)$$

$$\frac{dX_{Met}}{dt} = k_1 X_{TMP} X_{PME} \left(\frac{MW_{Met}\rho_T}{MW_{TMP}MW_{PME}}\right) - \quad \text{Eq. (47)}$$
$$k_{1r} X_{TMPME} X_{Met} \left(\frac{\rho_T}{MW_{TMPME}}\right) +$$
$$k_2 X_{TMPME} X_{PME} \left(\frac{MW_{Met}\rho_T}{MW_{TMPME}MW_{PME}}\right) -$$
$$k_{2r} X_{TMPDE} X_{Met} \left(\frac{\rho_T}{MW_{TMPDE}}\right) +$$
$$k_3 X_{TMPDE} X_{PME} \left(\frac{MW_{Met}\rho_T}{MW_{TMPDE}MW_{PME}}\right) - k_{3r} X_{TMPTE} X_{Met} \left(\frac{\rho_T}{MW_{TMPTE}}\right)$$

$$SD_i = \sqrt{\frac{1}{N-1}\sum\left(X_{exp,i} - X_{simulated,i}\right)^2} \quad \text{Eq. (48)}$$

$$SE_i = \frac{SD_i}{\sqrt{N}} \quad \text{Eq. (49)}$$

$$Total\ SE = SE_{TMP} + SE_{TMPME} + SE_{TMPDE} + SE_{TMPTE} \quad \text{Eq. (50)}$$

$$PI_i = X_{simulated,i} \pm z(SE_i)$$ Eq. (51)

$$Total\ energy\ consumption = E_{Heat} + E_{Vacuum} - E_R$$ Eq. (52)

$$Yield\ of\ TMPTE = \frac{Experimental\ mass\ of\ TMPTE}{Theoretical\ mass\ of\ TMPTE} \times 100\%$$ Eq. (53)

$$Heat\ duty\ of\ reaction, E_R = \sum n_{out}\widehat{H}_{out} - \sum n_{in}\widehat{H}_{in}$$ Eq. (54)

$$E_R = \sum n_{out}\left[C_P\left(T_{reaction} - T_{ref}\right) + \Delta\widehat{H}_f^o\right] - \sum n_{in}\left[C_P\left(T_{reaction} - T_{ref}\right) + \Delta\widehat{H}_f^o\right]$$ Eq. (55)

$$Enthalpy\ for\ water\ and\ methanol = \int CpdT + Hf + \Delta Hv$$ Eq. (56)

$$Enthalpy\ for\ TMP = \Delta Hfusion + \int CpdT + Hf$$ Eq. (57)

$$Others: Total\ enthalpy = \int CpdT + Hf$$ Eq. (58)

$$Fatty\ soap = \frac{Mass\ of\ solid\ material\ (mg)}{Total\ mass\ of\ reactant\ (g)} \times 100\%$$ Eq. (59)

$$\nabla \times \left(\frac{1}{\mu_r}\nabla \times \frac{1}{\mu_r}\boldsymbol{E}\right) - k_o^2\left(\varepsilon_r - \frac{j\sigma}{w\varepsilon_o}\right)\boldsymbol{E} = 0$$ Eq. (60)

$$\nabla \times \boldsymbol{E} = -j\omega\mu\boldsymbol{H}$$ Eq. (61)

$$\nabla \times \boldsymbol{H} = (\sigma + \mathrm{j}\omega\varepsilon)\boldsymbol{E}$$ Eq. (62)

$$\nabla \cdot \boldsymbol{E} = 0$$ Eq. (63)

$$\nabla \cdot \boldsymbol{B} = 0$$ Eq. (64)

$$\varepsilon_r = \varepsilon' - j\varepsilon''$$ Eq. (65)

$$\tan\theta = \frac{\varepsilon''}{\epsilon\prime} = \frac{\sigma}{\omega\varepsilon}$$ Eq. (66)

$$\frac{P}{V} = 2\pi f\varepsilon_o E^2\varepsilon''\mathrm{mix}$$ Eq. (67)

$$E = \frac{3E_o}{\varepsilon' mix+2} \quad \text{Eq. (68)}$$

$$\varepsilon' mix = x \cdot \varepsilon' TMP + x \cdot \varepsilon' PME \quad \text{Eq. (69)}$$

$$\varepsilon'' mix = x \cdot \varepsilon'' TMP + x \cdot \varepsilon'' PME \quad \text{Eq. (70)}$$

$$\frac{P}{V} = \frac{Cp \cdot \rho w \cdot \Delta T}{\Delta t} \quad \text{Eq. (71)}$$

$$\frac{P}{V} = 2\pi f \varepsilon_o E^2 \tan\delta \, K \quad \text{Eq. (72)}$$

$$E_o = \frac{K+2}{3} E \quad \text{Eq. (73)}$$

$$Q_{MW} = \begin{cases} \pi f \varepsilon_0 \varepsilon''_r |E|^2 & \text{if } 0 \le t < 12 \\ 0 & \text{if } 12 \le t \le 20 \end{cases} \quad \text{Eq. (74)}$$

$$\rho c_p \frac{\partial T}{\partial t} + \rho c_p u \cdot \nabla T = \nabla \mathrm{T} \cdot (k \nabla T) + Q_{MW} + Q_{\Delta H} \quad \text{Eq. (75)}$$

$$Q_{\Delta H} = \Delta H_r \dot{R} \quad \text{Eq. (76)}$$

$$\frac{dc_i}{dt} = R_i = v_i r \quad \text{Eq. (77)}$$

$$r_j = k_j^f \prod_{i=1}^{Q_r} c_i^{-v_{ij}} - k_j^r \prod_{i=1}^{Q_p} c_i^{v_{ij}} \quad \text{Eq. (78)}$$

$$k^f = A^f \left(T/T_{ref}\right)^{n^f} exp\left(\frac{-E^f}{R_g T}\right), T_{ref} = 1K \quad \text{Eq. (79)}$$

$$k^r = k^f / K_{eq_0} \quad \text{Eq. (80)}$$

$$k = Ae^{-E_A/RT} \quad \text{Eq. (81)}$$

$$\ln k = -E_A/(RT) + \ln A \quad \text{Eq. (82)}$$

CHAPTER 1

INTRODUCTION

1.1 Background

Transesterification reaction has been investigated by numerous researchers, mainly for the production of esters for biodiesel and biolubricant base stocks. Transesterification reaction involves a reaction between an alcohol and another ester to produce a new ester and is usually catalysed by acid or alkaline catalyst. In the production of polyol esters for lubricant base oils, the reaction uses a high amount of energy due to the high-temperature process. At high temperatures, many side reactions could take place and may affect the product yield. Using vacuum in the transesterification process and alkaline catalyst has successfully shortened the reaction time to 1 hour (Yunus and Idris, 2003a) as compared to 10 hours reaction without vacuum technology (Uosukainen et al., 1998). However, the use of 0.8 – 0.9 wt.% of sodium methoxide alkaline catalyst by Yunus and Idris (2003a) contributed to competing saponification, reducing the product yield. Chang et al. (2012) has used only 0.3 %w/w calcium methoxide – a heterogeneous alkaline catalyst to reduce the soap formation. Chang et al. (2012) achieved a high yield of 98% of the product with 9.2 mg soap/g compared to 46 mg soap/g when 0.9 wt.% sodium methoxide was used. However, it took 8 hours to complete the reaction.

Microwave is an attractive approach to intensify a process reaction, improvises a technique that promises a higher product yield as compared to the conventional method. Any method that reduces the processing time, increase energy consumption (Hincapié et al., 2014; Tippayawong and Sittisun, 2012), improve the quality and quantity of the produced product, reduces the amount of the byproducts and provide reliable sustainable technology (Martinez-guerra and Gude, 2016; Zhang and Zhang, 2011; Zhang et al., 2010) has a great potential to be explored. Microwave application is proposed not only to increase the rate of reaction but to enhance product selectivity (Lin et al., 2014). Recent studies showed a lot of application of microwave radiation in various fields such as food, polymer, rubber, and plastic industries, curing, drying, the extraction process, biodiesel, and biolubricant.

Lokman et al. (2015) reported a reaction time of 15 minutes to produce 96% palm fatty acid distillate with microwave assistance as compared to 120 minutes for the same conversion under a similar condition without a microwave. This condition was due to the increases potential of methanol to react with palm fatty acid distillate (PFAD) molecules by the increased vibration frequency of the atoms present in the reaction interface. Tippayawong and Sittisun (2012) has shown that only 30 seconds were required for transesterification reaction with sodium methoxide to produce 96% yield of biodiesel from jatropha oil which conventional heating required 24 to 120 minutes for 90-99% of biodiesel yield.

Transesterification of waste cooking oil in the presence of 1 wt.% sodium methoxide using microwave heating produced 98.87% conversion in 5 minutes of reaction time (Azcan and Yilmaz, 2013).

The interaction between the electric field and the properties of the material influenced the sample in a microwave-assisted reaction. The dielectric properties of the material govern this effect. The dielectric properties affect the interaction of electromagnetic fields to a material at the molecular level, which means it defines the amount of energy transferred to the material (Thostenson and Chou, 1999). The microwave can penetrate the materials and transfer the energy so that the heat is generated inside the materials. A dielectric constant represents the ability of a material to be polarized by the electric field, thus stores electrical potential energy. A dielectric loss factor is related to materials dissipation of electromagnetic energy, which takes place through either dipolar rotation or ionic conduction. When dipoles and ions interact with the oscillating electrical field, inter- and intra-molecular friction is formed. Heat is generated from the interaction, which heats up the whole volume of the material (Campos et al., 2014). High conductivity materials are preferable by conventional heating methods, as heat is transmitted effectively, whereas, for microwaves, materials with moderate dielectric loss factor in conductivity range are preferred.

1.2 Problem statement

Numerous studies have been conducted on the transesterification of oil and trimethylolpropane (TMP) to produce polyol ester as a lubricant base oil. The operating parameters covered in the conventional heating method are temperature, catalyst amount, reaction time, reactants molar ratio, and pressure. The operating conditions for transesterification of PME and TMP for the conventional heating method vary from 80 – 150 ˚C, 20 - 50 mbar, 0.28 – 2 wt.% of an alkaline catalyst, 1:3.2 – 1:4.5 of polyol ester and PME molar ratio. The main issue with the current transesterification reaction is the extended reaction time, which took 53 minutes to 10 hours for a production yield of 37 – 99%. Additionally, with longer production time, high operating temperature, and a large amount of sodium-based catalyst, the saponification reaction between the sodium and fatty acid to produce a sodium soap will likely occur. Consequently, most of the catalyst will be consumed in the reaction, and less available for the main transesterification reaction. Lack of catalyst will affect the reaction conversion and product yield significantly.

So far, the microwave application in the transesterification of oil and alcohol was mainly researched for biodiesel production. The reaction was successfully conducted under microwave heating for 30 seconds to 30 minutes to complete as compared to around 60 minutes for the conventional process. The range of operating conditions employed were 60 - 120˚C under atmospheric pressure, using 1 – 4 wt.% of an acidic or alkaline catalyst, molar ratio of oil to alcohol 1: 3 – 1: 60 with a production yield of 80 – 99%. In view of that, it is envisaged that microwave heating has the potential to improve the current transesterification

reaction of a polyol, TMP and methyl ester, PME in terms of reaction time and less soap production. The microwave heating and the corresponding polarisation effect generates heat and would accelerate any reaction. In addition, the interaction between the dielectric properties and material properties with microwave parameters can be modelled and simulated using the simulation software namely Comsol. To date, there is a limited study on using the microwave to produce TMP ester. Available studies focused on utilizing simple alcohols, such as methanol and ethanol in the transesterification with acid or ester.

To date, there is no report on the kinetics study of the microwave-assisted transesterification to produce TMP ester or TMP triesters (TMPTE). Hence, very little information is available to understand the reaction pathways under microwave and vacuum condition. Previous kinetics study by Yunus et al. (2005) on TMPTE production used conventional heating at a lower temperature range of 70 to 130 ˚C, which can be used as a reference for the kinetics study of microwave reaction. The fact is the reactions that take place in conventional and microwave-assisted heating are the same, only the reaction rates could change due to the different heating mechanism. Hence, similar assumptions used in conventional heating are applicable for kinetics study in microwave reaction. The information from the kinetics study will provide insights on how the microwave reaction compares to the conventional method in terms of the rate of reaction.

Moreover, the production of TMPTE using microwave should be supported by simulation work to better understand the phenomena of heat transport, the distribution of the electric field in the oven and the reaction mechanism. The lack of this knowledge may cause inconvenience in the up scaling of the reactor and the understanding of the fundamentals related to microwave heating. Numerous microwave simulation work focused on the drying of solid food or biodiesel production in a continuous reactor (Wu et al., 2013; Zhang et al., 2016). None, so far consider a coupled electromagnetic and heat transfer simulation model together with a reaction module. Both the electric field of the cavity and the power absorbed by the sample can be determined from this approach. In addition, the temperature and concentration profile of the production of TMPTE can also be obtained.

1.3 Contribution of research

The main aim of this study is to evaluate the potential of microwave irradiation to improve the rate of transesterification reaction in the production of biolubricant base oil as compared to conventional heating. Microwave is frequently used in biodiesel production because both methanol and ethanol are examples of good microwave radiation absorbance (Martinez-guerra and Gude, 2016). This favourable interaction of microwave and reactant (oil and alcohol-methanol) resulted in a significant reduction in process activation energy (Tangy et al., 2017). This phenomenon is due to an increase in dipolar polarisation that uniquely attributed to microwave response. As a good microwave radiation

absorber, the movement of methanol dipoles reorients and actively aligns with the applied electric field, destroying the two-tier structure of the methanol-oil interface (Lin et al., 2014). The functional group of methanol is reactive, facilitating the formation of micro emulsions (Boffito et al., 2012; Patil et al., 2011; Queiroz et al., 2015; Yu et al., 2010). Choedkiatsakul et al. (2015) and Patil et al. (2012) explained that decreasing in the dielectric constant and the polarity of methanol increases the solubility of methanol and oil, hence increasing the transesterification yield.

Besides that, the application of ionic catalyst such as alkaline catalyst can also provide positive interaction with microwave irradiation. The dielectric properties of the mixtures are strongly affected by the catalyst at high alcohol concentration due to the presence of ion in the mixtures. There is a strong interaction between the counter ion and the solvent molecules. Ionic conduction governs the energy dissipation instead of dipolar rotation. As temperature increases, the dielectric constant increases, the increase in kinetic energy lowers the viscosity, resulting in easier rotation and faster response from changes in the electrical field. Campos et al. (2014) in his study claimed that the use of ionic catalyst at high concentration of alcohol has a stronger effect on the dielectric properties in the production of methyl and ethyl ester due to the high mobility counterions (from sulfuric acid which is associated with high mobility ions and counterions). Under the electric field, the dipole moment of alcohol accelerates causing vigorous movement of ions which disrupts the barrier between alcohol and oil, resulting in the decrease of dielectric constant and alcohol polarity (Nurhidayanti et al., 2021). As stated previously, this effect lowers the dielectric constant of reaction mixture in comparison to the pure alcohol and increases the miscibility of oil and alcohol (Campos et al., 2014). The loss factor also decreases at the end of the reaction due to the generation of less polar compounds such as alkyl ester, diglycerides and monoglycerides. This is an indicator of synergistic effect when ionic catalyst is used under a microwave condition.

On the other hand, the substances which are involved in the production of polyol ester namely TMP and PME have different polarities as compared to methanol or ethanol used in the biodiesel system. TMP and PME have dielectric constants and loss factors of 1.78 and 0.04, and 3.39 and 0.25, respectively, as evaluated in this study. This has introduced challenges to the microwave-assisted transesterification proposed in this study. The use of vacuum to promote forward reaction, presents another obstacle for assessment. Biodiesel production is conducted under atmospheric pressure, not under vacuum condition.

The conventional process requires transmission of energy by conduction, convection and heat radiation from the bottom of reactor surface for a reaction to take place. Such a heat transfer is energetically inefficient. Microwave heating by means of which the electromagnetic wave produced travels with the speed of light can intensify the transport through a process of radiation that heats target compounds at the molecular level without heating the entire reactor. This condition saves time and energy. In addition, the conventional method of transesterification to produce polyol ester-based lubricants usually takes hours

to complete. Although using the alkaline-based catalyst under a vacuum condition has successfully shortened the reaction time, competing saponification of the alkaline catalyst has reduced the yield of the product. The microwave technology is envisaged to accelerate the rate of reaction with significantly less catalysts and therefore curtails the saponification reaction. This combined technology would greatly improve the economic viability of biolubricants as an alternative to the conventional lubricant products and preserve the non-renewable resources.

1.4 Research objectives

The objectives of this study are:

i. To investigate the effect of chemical reaction parameters on transesterification of trimethylolpropane (TMP) and palm oil methyl ester (PME) using a microwave-assisted heating.
ii. To evaluate the kinetics of microwave-assisted transesterification reaction of trimethylolpropane (TMP) and palm oil methyl ester (PME).
iii. To simulate the microwave-assisted reaction using a coupled electromagnetic wave-heat transfer-chemical reaction models via numerical modelling software (COMSOL Multiphysics 4.2).

1.5 Scope of study

1.5.1 Optimization of trimethylolpropane triesters (TMPTE) production using microwave-assisted heating

The raw materials chosen to produce TMPTE were palm oil methyl ester (PME) and trimethylolpropane (TMP). An alkaline catalyst: sodium methoxide was used to accelerate the reaction conducted in a modified domestic microwave oven. The reactions were conducted under different operating conditions to find the best range for the operating parameters. The effect of temperature was studied from 110 to 150 ˚C while the effects of catalyst amount, time, TMP-to-PME molar ratio and pressure ranged from 0.2 – 1.0 wt.%, 3 – 25 minutes, 1:3 – 1:4.5, and 10 – 50 mbar, respectively. GC system was chosen to analyse the composition of TMPTE produced. The final product was filtered to remove the excess catalyst and other solid product (soap) produced by the reactions. After that, the sample was fractionated using a distillation process to remove excess PME from the final product. Basic chemical and physical properties such as density, viscosity, cloud and pour point analysis were tested.

The dielectric properties of the raw materials and the product were measured at room temperature at 1- 10 GHz using a network analyser. The network analyser

cable used is not capable of measuring the sample at high temperature, which is the limitation faced during the study. Furthermore, the studies were only carried out at a frequency 2.45 GHz, hence the effect of frequency was not investigated. In addition, no temperature imaging analyser was available during the experimental activity to identify the hotspot temperature or its specific position in the microwave.

1.5.2 Kinetics study of transesterification of TMP and PME via microwave heating

The optimum conditions were selected to conduct the kinetics study at four different temperatures: 110, 120, 130 and 140 ˚C. The kinetics parameters were the reaction rate constant, the activation energy, the pre-exponential value, A which were the main factor in the Arrhenius equation and Gibbs energy. Assuming that all reaction steps are reversible, there are six reaction rate law expressions that need to be solved to achieve the kinetics parameters. MATLAB simulation software was used to solve these expressions using the ordinary differential equation (ODE) method. The simulated versus experimental data curve showed well-fit data with a small sum of standard error (SE).

1.5.3 COMSOL simulation of electromagnetic - heat transfer coupled model

A model to simulate the transesterification reaction of TMP and PME in COMSOL was constructed. Several modules were used in the model such as reaction engineering (*re*), electromagnetic waves, (*emw*) and heat transfer in fluids (*ht*). First, the *emw* was defined accordingly to solve the distribution of the electric field and forward the results to *ht* module. Finally, re module was solved by predicting the concentration profile for the transesterification reaction. The numerical modelling was conducted based on the following sub objectives:

i. Formulate a fully coupled electromagnetic-heat transfer model for the transesterification reaction to obtain a temperature profile.
ii. Determine the electric field inside the oven cavity and sample in order to estimate the power absorbed by the sample.
iii. Investigate the effect of temperature on the concentration of TMPE profile.

Some limitation faced during this part was the current setup is unable to record the live-time changes of the thermophysical (such as density, viscosity, dielectric constant, loss factor, heat capacity, thermal conductivity) of the reaction mixture. The combination of a stationary (electromagnetic wave module) and a rotating frame (to introduce a stirrer for mixing effect) has developed a discontinuity in the interfaces, which could not be solved in this computation.

1.6 Thesis outline

This thesis consists of five chapters that begin with a brief introduction to the study in Chapter 1. The problem statements and the objectives are further explained in this chapter. Chapter 2 is on the literature review, which provides an in-depth study of the previous reports by various researchers. Chapter 2 involves a critical evaluation and reasoning of the process and decision chosen for the project. This includes the selection of materials, the study of process parameters, the improvement of the available simulation method and the selection of simulation data using COMSOL. Detail procedures are elucidated in Chapter 3, based on the study objectives for the reliable repetition of experimental work. Chapter 4 presented all the results obtained for these objectives and provided a scientific evaluation and discussion of the results as compared to other published work. Finally, a concluding remark and some recommendations were detailed in Chapter 5.

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