

**SYNTHESIS OF PALM BIODIESEL USING DIRECT APPLICATION
OF PURE SODIUM METHOXIDE AS THE CATALYST**

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DEDICATED TO

MY WIFE, DAUGHTER, PARENTS with love

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

SYNTHESIS OF PALM BIODIESEL USING DIRECT APPLICATION OF PURE SODIUM METHOXIDE AS THE CATALYST

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Palm biodiesel (methyl ester) was successfully synthesized from refined bleached deodorized palm oil (RBDPO) by transesterification reaction. The alkali catalyst was selected for this reaction, and the effects of operating variables such as molar ratio, reaction temperature, reaction time, and quantity of catalyst were investigated. The reaction was carried out under atmospheric pressure. The reaction temperatures were varied among 55, 60, 65 and 70°C, while the reaction times were between 50, 60, 70, 80 and 90 min respectively. The effects of two alkali catalysts namely sodium methoxide (NaOCH₃) and sodium hydroxide (NaOH) at catalyst amounts of 1.0% w/w and molar ratios of methanol to RBDPO at 3:1, 4:1, 5:1, and 6:1 were investigated on reaction yield. The optimum conditions for NaOCH₃ catalyst are as follows: reaction temperature is 65°C, reaction time is 60 minutes, molar ratio is 6:1, and catalyst amount, 1.0% w/w. The reaction conversion was almost 99%. While using NaOH catalyst, the conversion was 94%. The low pour point palm biodiesel was produced through winterization and vacuum distillation process. The vacuum

distillation operated under pressure and temperature were between 5.2-6.0 mbar and 156-158°C. Basically, the lower the content of saturated components in a biodiesel, a better pour point biodiesel would be produced. However, in this experiment, the lowest of saturated component achievable was 16% w/w, and the lowest pour point attainable for palm biodiesel was at 3°C. Besides that, the kinetics study on transesterification of RBDPO with methanol established that the reaction occurred via two stepwise and irreversible elementary reactions. The rate constants for the formation of intermediate diglycerides and the final product palm oil methyl esters were determined at various temperatures. The conversion of triglycerides (TG) and diglycerides (DG) appeared to be following the second order reaction. The values of k_{TG} were between 0.005 – 0.013 and the values of k_{DG} were between 0.019 – 0.027. The activation energies for stepwise reaction in transesterification of palm based oils with methanol ranged from 6.87 to 11.45 kcal/mol.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

**SINTESIS BIODIESEL KELAPA SAWIT MENGGUNAKAN APLIKASI
LANGSUNG NATRIUM METOKSIDA TULIN SEBAGAI PEMANGKIN**

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Sintesis bahan bakar diesel (biodiesel) kelapa sawit telah berjaya dijalankan melalui tindakbalas transesterifikasi kimia minyak kelapa sawit yang telah ditapis, diluntur dan dienyahbau (RBDPO) Pemangkin alkali dipilih pada tindakbalas ini, dan kesan pembolehubah kendali iaitu: nisbah mol, suhu tindakbalas, masa tindakbalas, dan kuantiti pemangkin dikaji. Tindakbalas dijalankan dibawah tekanan atmosfera. Suhu tindakbalas diubah antara 55, 60, 65 dan 70°C, sedangkan masa tindakbalas antara 50, 60, 70, 80 dan 90 minit. Kesan dua pemangkin alkali iaitu natrium metoksida (NaOCH_3) dan natrium hidroksida (NaOH) pada kuantiti pemangkin 1% w/w dan nisbah mol metanol kepada minyak RBDPO pada 3:1, 4:1, 5:1, dan 6:1 dikaji terhadap hasil tindakbalas. Keadaan optimum dengan menggunakan pemangkin NaOCH_3 adalah seperti berikut: suhu tindakbalas 65°C, masa tindakbalas 60 minit, nisbah mol 6:1, dan kuantiti pemangkin yang digunakan adalah 1% w/w. Penukaran

tindakbalas juga hampir 99%. Sedangkan dengan menggunakan pemangkin NaOH tindakbalas adalah 94%. Takat tuang yang rendah diperolehi melalui kaedah “winterisasi” dan kaedah penyulingan vakum. Penyulingan vakum beroperasi pada tekanan 5.2-6.0 mbar dan suhu 156-158°C. Pada asasnya, semakin rendah kandungan komponen tepu di dalam biodiesel, semakin baik takat tuang biodiesel yang diperolehi. Walaubagaimanapun, pada ujikaji ini kandungan tepu terendah yang dihasilkan adalah 16% w/w, dan nilai takat tuang terendah yang diperolehi adalah 3°C. Disamping itu juga, kajian kinetik keatas transesterifikasi minyak RBDPO dengan metanol menunjukkan tindakbalas berlaku secara turutan dan melibatkan dua tindakbalas asas tak berbalik. Kadar pemalar untuk pembentukan digliserida perantaraan dan produk akhir metil ester minyak kelapa sawit (bioidesel) dikaji pada beberapa suhu. Penukaran trigliserida (TG) dan digliserida (DG) didapati berada pada kadar kedua pada keseluruhan masa tindakbalas. Nilai-nilai k_{TG} berada pada julat 0.005–0.013 dan k_{DG} adalah diantara 0.014–0.027. Tenaga keaktivifan bagi tindakbalas sintesis transesterifikasi tersebut berada diantara 6.87–11.45 kcal/mol.

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Putra Malaysia or other institutions.

AZHARI

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NOVEMBER 2006

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BSTFA	N,O-Bis(Trimethylsilyl)trifluoroacetamide
CP	Cloud Point
CFC	Chloro Fluoro Carbon
DF	Diesel Fuel
DG	Diglyceride
ELSD	Evaporative Light Scattering Detector
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acid
FID	Flame Ionization Detection
GC	Gas Chromatography
HPLC	High Performance Liquid Chromatography
HPSEC	High Performance Size-Exclusion Chromatography
ID	Inside Diameter
K	Kilo
ME	Methyl Ester
MG	Monoglyceride
POME	Palm Oil Methyl Ester
PORIM	Palm Oil Research Institute Malaysia
PP	Pour Point
RBDPO	Refined Bleached Deodorized Palm Oil
TG	Triglyceride
UV	Ultra Violet

US	United State
Al ₂ O ₃	Aluminium Oxide
CO ₂	Carbon Dioxide
NaOCH ₃	Natrium Methoxide
NaOH	Natrium Hydroxide
SiO ₂	Silicon Dioxide
H ₂ SO ₄	Sulfuric Acid
D2	No.2 Diesel
cal	calorie
g	gram
h	hour
mL	milliliter
min	minute
nm	nanometer
rpm	rotation per minute