



Faculty of Mechanical Engineering

**PHYSIOCHEMICAL EVALUATION AND TRIBOLOGICAL
PROPERTIES OF ECO-FRIENDLY BIODEGRADABLE NOVEL
BIOLUBRICANT FROM BANANA PEEL WASTES**

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PEEL WASTES**

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**A thesis submitted
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DECLARATION

I declare that this thesis entitled “Physiochemical Evaluation and Tribological Properties of Eco-friendly Biodegradable Novel Biolubricant from Banana Peel Wastes” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.


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APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Doctor of Philosophy in Mechanical Engineering.

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Date : 30/10/2017

DEDICATION

To my beloved husband, Siddiq Hafiz bin Hassan, my adorable son, Yusof Rifqi Benjamin and not forgotten my beloved parent Tuan Haji A. Hamid bin Haji Mamat & Puan Hajjah Rosnah binti Haji Ali, my siblings (Rozi Hartini, Hasbi, Hafiz, Ibtisam & Hakimi) and my beloved in-laws, Tuan Haji Hassan Bin Unir & Puan Hajjah Ruzaimah binti Ibrahim.

ABSTRACT

Banana peel, which known as a waste product was shown to have a potential sources of plant/vegetable oils due to slippery effect from the existence of hemicellulose of follicular gels contained in its epicarps. From the literature surveys, it was noted that, limited scientific studies are available on banana peels and has not been given great attention by the researchers. The present study is to extract the oil form banana peel waste of *Musa Aliminata Balbisiana* (MBS), characterize the physical, physiochemical properties, chemical compositional and functional group analysis, fatty acids profile and wettability analysis of the oil, investigate the tribological behavioural of MBS Oil at different temperatures and loads, investigate the effect of biodegradable additive of Ethyl Cellulose (EC) and Ethylene Vinyl Acetate (EVA) on tribological performance of MBS oil, analyse the predominant wear mechanism of MBS Oil under wet sliding condition and formulate a mathematical equation for friction and wear of MBS Oil with the presence of EC and EVA using ANOVA Analysis. The peel waste of MBS contained 86.57% of moisture and 0.57% of volatile content. Surface morphology analysis revealed that the existence of follicular gel in the epicarp of peel waste of MBS that was functioned to trapped the oil constituents thus create the slippery effect. A pale brownish of novel bio-oil from banana peel waste of MBS was extracted using *soxhlet* extraction method. In this methods, *n*-hexane was used as solvent of extractor at the optimum condition at the temperature of 68 °C for 7 hour. The pale brownish oil exhibit a density at 40 °C of 0.8502 kg/m³ and specific gravity of 0.8536. Physiochemical analysis revealed that, the MBS oil exhibited a refractive index (RI) of 1.74 and 3.29 cSt of viscosity at 40 °C. Besides, MBS oil contained 2.64 of Free Fatty Acids (FFAs), 0.77 g of I₂/100 g of oil Iodine Values (IV), and 7.46 mg KOH/g Saponification Values (SV), Total Acid Number (TAN) and Total Base Number (TBN) of 0.52 mg KOH/g and 0.77 mg KOH/g respectively. FT-IR analysis showed that, the oil contained an ester of carbonyl functional group, C=O (1733.06 cm⁻¹), C-H of *cis*-di-substituted olefins (1644.42 cm⁻¹), =C-H aliphatic groups (1453.39 cm⁻¹), C-O ester group (1244.10 cm⁻¹, 1173.29 cm⁻¹, 1094.79 cm⁻¹, 1039.58 cm⁻¹ and 982.34 cm⁻¹) and -CH₂ (721.11 cm⁻¹). MBS oil exhibited a wettability analysis at the contact angle values of 157.55°. Fatty acid analysis revealed that MBS Oil composed of 46% of saturated fatty acids (SFAs), 46% of monounsaturated fatty acids (MUFAs) and 4% of polyunsaturated fatty acids (PUFAs). Fatty acids compositional analysis showed that, MBS oils was dominated by Butyric Acid (C4:0), Caproic Acid (C6:0), Myristic Acid (C14:1), Ginkgolic acid (C15:1), Gondoic Acid (C20:1), Methyl Nerranoic Acid (C24:1), Gamma-Linolenic Acid (C18:3N6) and *cis*-8,11,14-Eicosatrienoic Acid (C20:3N6). MBS oil showed a lower coefficient of friction, COF values at the load of 80N at 27 °C, 40 °C and 100 °C with the ranged of 0.05 to 0.13. Wear Scar Diameter, WSD analysis ranged from 251 μm to 1233 μm and Wear rate, *Ws* analysis shows that, it in the ranged from 0.016 x 10⁻¹⁵ to 4.0396 x 10⁻¹⁵ mm³/Nmm. The presences of EC and EVA at difference concentration were lead to reduction in COF values for MBS oil due to the synergism effects between EC and EV with the existences of fatty acid compounds in MBS Oil. The predominant wear mechanisms in the tribological study of MBS oil with the presence of EC and EVA were adhesive and abrasive wear which proved by the SEM micrograph and SEM EDX mapping. A mathematical equation for friction and wear were formulated.

ABSTRAK

Kulit pisang yang dikenali sebagai bahan buangan telah terbukti mempunyai sumber potensi minyak tumbuhan/sayur-sayuran disebabkan oleh kesan licin daripada kewujudan hemiselulosa folikel gel yang terkandung dalam epicarps. Hasil daripada kajian ilmiah yang lepas, ianya telah dinyatakan bahawa, terdapat kajian saintifik yang terhad ke atas kulit pisang dan tidak diberi perhatian yang besar oleh para penyelidik. Kajian ini dijalankan untuk mengekstrak minyak dari sisa kulit pisang *Musa Aliminata Balbisiana* (MBS), untuk mencari sifat-sifat fizikal, psikokimia, komposisi kimia dan analisa kumpulan berfungsi, sifat-sifat asid lemak dan ujian kebolehasan minyak disamping mengkaji sifat tribologi minyak MBS pada suhu dan beban yang berbeza. Selain itu, kesan penambah terurai etil selulosa (EC) dan etilena vinil asetat (EVA) ke atas ujian tribologi minyak MBS telah dijalankan bagi menganalisa mekanisma kehausan utama minyak dibawah keadaan gelongsor basah disamping merumuskan persamaan matematik untuk geseran dan kehausan minyak MBS dengan kehadiran EC dan EVA melalui analisa ANOVA. Hasil kajian ini menunjukkan sisa kulit MBS mengandungi 86.57% kelembapan dan nilai pemeruawap sebanyak 0.57%. Kajian morfologi keatas permukaan epikap sisa kulit pisang MBS menunjukkan kewujudan folikel gel yang berfungsi untuk memerangkap minyak dan mewujudkan kesan licin. Minyak nobel yang berwarna coklat pucat telah berjaya diekstrak dengan menggunakan teknik 'soxhlet'. Dalam teknik ini, n-heksana telah digunakan sebagai pelarut untuk mengekstrak pada keadaan optimum iaitu pada suhu 68°C untuk 7 jam. Minyak yang berwarna coklat pucat itu telah menunjukkan ketumpatan pada suhu 40°C sebanyak 0.8502 kg/m³ dan nilai graviti tertentu sebanyak 0.8536. Kajian psikokimia telah menunjukkan minyak MBS mempunyai Indek Biasan (RI) sebanyak 1.74 dan kelikatan pada suhu 40 °C sebanyak 3.29 cSt. Disamping itu, minyak MBS juga mengandungi asid lemak bebas (FFAs) sebanyak 2.64, 0.77 g I₂/100 g nilai iodine (IV), 7.46 mg KOH/g nilai saponifikasi (SV), jumlah nilai asid (TAN) dan jumlah nilai alkali (TBN) masing-masing sebanyak 0.52 mg KOH/g dan 0.77 mg KOH/g. Analisa FT-IR menunjukkan minyak MBS mengandungi kumpulan berfungsi karbonil ester, C=O (1733.06 cm⁻¹), C-H olefins cis-di-digantikan (1644.42 cm⁻¹), =C-H kumpulan alifatik (1453.39 cm⁻¹), C-O kumpulan ester (1244.10 cm⁻¹, 1173.29 cm⁻¹, 1094.79 cm⁻¹, 1039.58 cm⁻¹ and 982.34 cm⁻¹) and -CH₂ (721.11 cm⁻¹). Ujian kebolehasan minyak MBS menunjukkan nilai sudut capahan sebanyak 157.55°. Kajian asid lemak menunjukkan minyak MBS mengandungi 46% asid lemak tepu (SFAs), 46% asid lemak monotaktepu (MUFAs) dan 4% asid lemak politaktepu (PUFAs). Manakala, ujian komposisi asid lemak telah menunjukkan minyak MBS mengandungi asid butirik (C4:0), asid kaprik (C6:0), asid miristic (C14:1), asid Gingkolik (C15:1), asid Gondoik (C20:1), asid metil nerranoik (C24:1), asid gamma linolenik (C18:3N6) dan asid cis-8-11-14-Eikosatrienoik (C20:3N6). Siasatan ke atas sifat tribologi minyak MBS menunjukkan nilai pekali geseran, COF yg rendah pada beban 80N pada 27 °C, 40 °C dan 100 °C iaitu dalam lingkungan 0.05 - 0.13. Ujian kehausan ukur lilit (WSD) menunjukkan nilai dalam lingkungan 251µm - 1233 µm dan ujian kadar kehausan, Ws menunjukkan nilai di antara 0.016 x 10⁻¹⁵ - 4.0396 x10⁻¹⁵ mm³/Nmm. Mekanisma kehausan utama di dalam kajian tribologi minyak MBS dengan kehadiran EC and EVA adalah perlekatan kehausan dan kehausan kasar yang dibuktikan dengan mikrograf SEM dan pemetaan SEM EDX.

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LIST OF ABBREVIATIONS

Adj SS	-	Adjusted Sum of Square
ANOVA	-	Analysis of Variance
AOCS	-	American Oil Chemist's Society
AUA	-	Anhydrouronic Acids
ASTM	-	American Society of Testing and Materials
AW	-	Anti wear
BC	-	Before century
BMSO	-	<i>Bauhinia monandra</i> seed oil
BMME	-	<i>Bauhinia Monandra</i> Methyl Ester
COF	-	Coefficient of Friction
DE	-	Degree of Esterification
DOF	-	Degree of Freedom
EC	-	Ethyl Cellulose
EDS	-	Energy Dispersive X-ray Spectrometer
EHD	-	Elastohydrodynamic
EP	-	Extreme Pressure
EVA	-	Ethylene Vinyl Acetate
EVOO	-	Extra Virgin Olive Oil
F	-	Frictional Force
FAME	-	Fatty Acids Methyl Ester
FFAs	-	Free Fatty Acids

FTIR	-	Fourier Transmittance Infra Red
GC-FID	-	Gas Chromatography –Flame Ionisation Detector
GC-MS	-	Gas Chromatography Mass Spectroscopy
H ₂ SO ₄	-	Sulphuric Acid
HCl	-	Hydrochloric Acid
HD	-	Hydrodynamic
IC	-	Internal Combustion
ISO	-	International Standard Organization
KHC ₈ H ₄ O ₄	-	Potassium hydrogen phthalate Solution
KI	-	Potassium Iodide Solution
KIO ₃	-	Potassium Iodate Solution
KOH	-	Potassium Hydroxide Solution
L	-	Sliding Distance
MBS	-	<i>Musa Aluminata Balbisiana</i>
MCS	-	<i>Musa Acuminata Cavendish</i> Subgroup
Mc	-	Moisture Content
MES	-	<i>Musa Acuminata Colla</i>
MUFAs	-	Monounsaturated Fatty Acids
NaOH	-	Sodium Hydroxide Solution
Na ₂ S ₂ O ₃	-	Sodium Thiosulfate Solution
Nf	-	Critical Number
OH	-	Hydroxyl group
PO	-	Palm Oil
PUFAs	-	Polyunsaturated Fatty Acids
R	-	Radius of the hemisphere pin
RI	-	Refractive Index
SBS	-	Styrene-butadiene-styrene

SEM	-	Scanning Electron Microscope
Seq MS	-	Sequential Mean Square
Seq SS	-	Sequential Sum of Square
SFAs	-	Saturated Fatty Acids
SI	-	Standard International Units
S/N	-	Signal to Noise Ratio
SV	-	Saponification Values
TAG	-	Triacylglycerol
TAN	-	Total Acid Number
TBN	-	Total Base Number
TMP	-	Trimethylolpropane
UV-VIS	-	Ultra Violet-Visible Spectroscopy
V_c	-	Volatile Content
VI	-	Viscosity Index
W	-	Applied Load
W_s	-	Wear Rate
WSD	-	Wear Scar Diameter
WVO	-	Waste Vegetable Oil
XRD	-	X-ray Powder Diffraction