DEVELOPMENT AND PERFORMANCE EVALUATION OF BIOLUBRICANT ENHANCED WITH NANOPARTICLES FOR SUSTAINABLE MACHINING APPLICATION

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DEDICATION

This thesis is dedicated to the loving memory of my beloved mother and my adorable brother who passed away to the great beyond in 2020. They were full of prayers and support for me but have to answer the call of the Most high at the appointed time. May Allah have mercy on their souls and repose them in Aljanat Firdaus, Ameen thuma Ameen.

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ABSTRACT

Vegetable oil-based biolubricants usage for lubrication and cooling in the industry has been encouraged due to their pleasant attributes of being environmentally benign, lower volatility, biodegradable, good lubricity, and high-quality viscosity index, amongst others. However, failure of biolubricants at elevated temperatures which is attributed to the thermal degradation hinders their efficient performance. The performance limitations may be ameliorated using nano-additives to enhance the oxidation and thermal stability as well as improve the thermophysical and anti-wear properties of biolubricants. However, thermal stability evaluation of vegetable oil-based nano-enhanced biolubricants has not been reported in any literature. The research aimed to formulate and evaluate performance characteristics of nano-enhanced biolubricants dispersed with exfoliated graphene nanoplatelets and maghemite nanoparticles at varying volume concentrations of 0.1%, 0.2% and 0.3% in coconut oil as base biolubricants; denoted by XGO1, XGO2 and XGO3 for graphene enhanced and MGO1, MGO2 and MGO3 for maghemite enhanced nanolubricants. The developed nanolubricants and the base biolubricants were evaluated for thermal stability, thermophysical and tribological properties tests. The volume fraction 0.2% of the XGO and the MGO concentrations indicated best performance in terms of tribological properties. End milling machining experiment was performed using the best concentrations from the XGO and MGO nanolubricants (0.2% volume fractions) based on the outcome of tribological properties evaluation and compared with the base lubricant. From the results, thermogravimetric analysis (TGA) reveals that the addition of nanoparticles improves the oxidation onset temperature for all concentrations, which implies improved thermal stability. The oxidation onset temperature in the presence of nano-additives can be delayed by 36.3 °C and 14.5 °C for XGO1 and MGO1, respectively, at 0.1% volume concentration. An improvement of thermal conductivity for all concentration levels was observed with a maximum enhancement ratio of 15.5% and 7.9% at 0.3% volume concentration, respectively, for the graphene and maghemite enhanced nanolubricants. The tribological studies revealed a significant reduction in coefficient of friction and wear scar diameter. For graphene enhanced nanolubricants, maximum friction and wear reduction were 17.6% and 7.55%, while for maghemite enhanced nanolubricants were 7.39% and 6.25%, respectively. Machinability performance was performed on machining titanium alloy (Ti-6Al-4V) under the minimum quantity lubrication (MQL) technique to supply lubricants. The results reveal that the enhanced biolubricants indicated superior performance over pure biolubricants in cutting force, tool life, and surface roughness. The graphene nanoplatelets enhanced lubricant was better over the maghemite enhanced lubricant. The research has shown a comprehensive understanding of oxidation stability and thermal degradation process of enhanced nano-biolubricants which are rarely investigated. Thermophysical and tribological properties evaluated indicated significant improvement. Furthermore, the developed non-linear correlation will avail researchers and industry operators the opportunity of selecting potential nanoparticles for nano-enhanced biolubricants formulation. This will reduce downtime and save resources as blends of nanoparticle and vegetable oils can be formulated without performance testing, thus promoting the United Nations Sustainable Development Goals (SDGs) to ensure sustainable consumption and production pattern.

ABSTRAK

Penggunaan biopelincir berasaskan minyak sayuran untuk pelinciran dan penyejukan dalam industri telah meluas kerana sifat-sifatnya yang baik iaitu mesra alam sekitar, kadar turun-naik lebih rendah, terbiodegradasi, pelinciran yang baik, dan indeks kelikatan tinggi, berbanding pelincir lain. Walau bagaimanapun, prestasi kecekapannya berkurang apabila beroperasi pada suhu yang tinggi disebabkan oleh degradasi haba bahan pelincir tersebut. Prestasi bahan pelincir mungkin boleh diperbaiki dengan menggunakan bahan tambahan bersaiz nano yang bertujuan untuk meningkatkan pengoksidaan dan kestabilan haba serta menambah baik sifat termofizik dan anti-haus biopelincir. Walau bagaimanapun, ia tidak dilaporkan dalam mana-mana literatur. Penyelidikan ini bertujuan untuk merumuskan bahan pelincir dengan penambahan nanoplatelet *exfoliated graphene* dan nano zarah *maghemite* pada kepekatan yang berbeza-beza pada pecahan isipadu 0.1%, 0.2% dan 0.3% ke dalam minyak kelapa yang diwakili dengan XGO1, XGO2 dan XGO3 untuk *graphene* manakala MGO1, MGO2 dan MGO3 untuk *maghemite*. Nanopelincir yang dibangunkan dan cecair tulen telah dinilai berdasarkan kestabilan haba, sifat termofizik dan tribologi. Isipadu dengan campuran 0.2% untuk XGO dan MGO menunjukkan prestasi yang terbaik dari segi sifat tribologinya. Pemesinan kisar akan dilakukan dengan menggunakan nanopelincir campuran kepekatan XGO dan MGO (kepekatan isipadu 0.2%) berdasarkan hasil penilaian sifat tribologi dan juga turut dibandingkan dengan sifat pelincir tulen. Daripada hasil keputusan, analisis termogravimetrik (TGA) mendedahkan bahawa penambahan nanozarah meningkatkan suhu permulaan pengoksidaan untuk semua tahap kepekatan, dalam masa yang sama menunjukkan kestabilan haba yang lebih baik. Suhu permulaan pengoksidaan dengan kehadiran bahan tambahan nano boleh dilengah sebanyak 36.3 °C dan 14.5 °C untuk XGO1 dan MGO1, pada kepekatan isipadu 0.1%. Peningkatan kekonduksian haba untuk semua tahap kepekatan dapat diperhatikan dengan nisbah peningkatan maksimum 15.5% dan 7.9% pada kepekatan isipadu 0.3%, untuk kedua-dua nanopelincir *graphene* dan *maghemite*. Hasil kajian tribologi mendapati pengurangan ketara dalam pekali geseran dan diameter parut kehausan. Bagi nonpelincir campuran *graphene*, geseran maksimum dan pengurangan kadar kehausan ialah 17.6% dan 7.55%, manakala nanopelincir *maghemite* ialah 7.39% dan 6.25%. Prestasi kebolehmesinan dilakukan dengan pemesinan aloi titanium (Ti-6Al-4V) di bawah teknik pelinciran kuantiti minimum (MQL) sebagai medium pembekalan pelincir. Hasilnya mendapati bahawa nanopelincir menunjukkan prestasi yang lebih baik berbanding pelincir tulen berdasarkan daya pemotongan, jangka hayat mata alat dan kekasaran permukaan pemotongan. Nanopelincir dengan campuran nanoplatelet *graphene* adalah lebih baik berbanding nanopelincir campuran *maghemite*. Hasil penyelidikan telah menunjukkan pemahaman yang komprehensif tentang kestabilan pengoksidaan dan proses degradasi haba bagi campuran nanopelincir yang jarang dikaji. Sifat termofizik dan tribologi yang dinilai menunjukkan peningkatan yang ketara. Tambahan pula, korelasi bukan linear yang dibangunkan akan memberi peluang kepada penyelidik dan pengguna untuk memilih jenis nano yang berpotensi untuk perumusan biopelincir nano. Ini akan mengurangkan masa dan menjimatkan sumber kerana campuran nano zarah dan minyak sayuran boleh dirumus tanpa ujian prestasi, sekali gus menggalakkan Matlamat Pembangunan Lestari (SDGs) Pertubuhan Bangsa-Bangsa Bersatu untuk memastikan penggunaan dan corak pengeluaran yang mampan.

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LIST OF ABBREVIATIONS

ADDC	-	Antimony dialkyldithiocarbamate
AW	-	Antiwear
BHA	-	Butylated hydroxyl anisole
BHT	-	Butylated hydroxyl toluene
CB	-	Carbon Black
CBN	-	Cubic boron nitride
CCNG	-	Cold nitrogen gas
CCNGOM	-	Compressed cold nitrogen gas and oil mist
CNT	-	Carbon Nanotube
COF	-	Coefficient of Friction
CuO	-	Copper Oxide
CVD	-	Chemical vapour deposition
CWMJ	-	Coldwater mist jet
DBP	-	Dibutyl 3.5-di-t-butyl-hydroxy benzyl phosphate
DDSA	-	Dodecenyl Succunic Anhydride
DTC	-	Dithiocarbamates
DTG	-	Differential thermogravimetric
DTP	-	Zincdithiophosphates
EC	-	Ethyl cellulose
EDS	-	Energy Dispersion Spectroscopy
EDX	-	Energy dispersive Xray
EP	-	Extreme Pressure
EVA	-	Ethylene-vinyl acetate
FESEM	-	Field Emission Scanning Electron Microscopy
FFA	-	Free Fatty Acid
FMs	-	Friction modifiers
GNPs	-	Graphene Nanoparticles
hBN	-	Hexagonal Boron Nitride
HPC	-	High-pressure cooling
HSM	-	High Speed Machining

LN2	-	Liquid Nitrogen
MoS2	-	Molybdenum Sulphide
MQL	-	Minimum Quantity Lubrication
MWCNT	-	Multiwalled Carbon Nanotubes
NDM	-	Near dry machining
NECO	-	Nanoparticle-Enhanced Coconut Oil
PAG	-	Polyalkylene glycol
PAOs	-	Polyalphaoleifins
PCBN	-	Polycrystalline cubic boron nitride
PCD	-	Polycrystalline diamond
PG	-	Propyl gallate
POME	-	Palm oil methyl ester
PPD	-	Pour point depressant
Ra	-	Surface roughness
SCO	-	Soluble Coconut Oil
SEM	-	Scan Electron Microscope
SFA	-	Saturated fatty acid
TBHQ	-	Mon-tert-butyl-hydroquinone
TCP	-	Tricresyl phosphate
TEM	-	Transmission Electron Microscopy
TGA	-	Thermogravimetric analysis
TMP	-	Trimethylolpropane
USFA	-	Unsaturated fatty acid
WSD	-	Wear scar diameter
WSD	-	Wear scar diameter
XRD	-	X-Ray Diffraction
ZDDC	-	Zinc diamy dithiocarbamate
ADDC	-	Antimony dialkyldithiocarbamate

LIST OF SYMBOLS

η	-	dynamic viscosity, Pas
T	-	force per unit area of shear stress acting on the lubricant, Pa
γ	-	shear rate i.e., velocity gradient normal to the shear stress, s^{-1}
υ	-	kinematic viscosity, cst or mm^2/s
ρ	-	density of lubricant, g/cm^3
w_1	-	weight of pycnometer filled with sample
w_0	-	weight of empty pycnometer
$vol.$	-	volume of pycnometer (25 ml)
k_p	-	solid particle thermal conductivity,
k_{bf}	-	thermal conductivity of the base fluid
k_{nl}	-	thermal conductivity of nano-enhanced biolubricant
k_r	-	thermal conductivity ratio
k_a	-	thermal conductivity of agglomerates
μ_{nl}	-	Viscosity of nano-enhanced biolubricant, mPa s
μ_{bf}	-	Viscosity of pure biolubricant, mPa s
ϕ	-	stands for the volume concentration of particles, %.
Φ_a	-	volume fraction of particles.
ra	-	Chip thickness ratio,
to	-	Undeformed chip thickness
tc	-	Deformed chip thickness
α	-	Rake angle
Fc	-	cutting force
Ft	-	thrust force
F	-	friction force
N	-	normal force to friction
Fs	-	shear force
N	-	rotational speed of the spindle per minute (rpm)
M_p	-	mass of nanoparticle in g
ρ_p	-	density of nanoparticles in g/cm^3

M_{bf}	-	mass of base fluid in g
ρ_{bf}	-	density of base fluid g/cm^3

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Manufacturing in industries involves several procedures and processes towards achieving the desired goals. Metal cutting operation plays a critical role in manufacturing varieties of human needs ranging from household items to industrial components for various applications in aerospace, machine elements, automotive, marine industries, and others. Lubricants are applied to conduct heat away from contacting surfaces, reduce friction and wear of the contacting surfaces in relative motion (Ahmed & Nassar, 2013). Conventionally, mineral oil-based lubricants form the bulk of the lubricating oils utilized to facilitate the ever-increasing machining operations to attain good dimensional accuracy, better surface finish, and prolong the life of machine elements. The detrimental effects or impacts of mineral oil or petroleum-based lubricants when not properly disposed of after use are the degradation of the soil, contamination of underground water, and loss of aquatic life. Besides, the vast cost is expended for proper disposal of these lubricants to reduce their impact on the environment (Chetan et al., 2015; Madanhire & Mbohwa, 2016b).

Preservation of the environment and ensuring the quality of human life in manufacturing activities are critical features of a sustainable manufacturing practice. Manufacturing performances are evaluated not by the economic viability alone but, the impact of manufacturing activities on the social and environmental aspects (Abdul-Rashid et al., 2017). Manufacturing is integral to a sustainable society, and there is a need to balance environmental performance with economic and social objectives. According to Jawahir et al. 2015 (Jawahir et al., 2015), critical elements of sustainable manufacturing include energy consumption, manufacturing cost, environmental impact, operational safety, the health of personnel and waste reduction. For a manufacturing process to be termed sustainable, the products, the processes or procedures and the system must ensure the satisfaction of the six critical elements of sustainable manufacturing without compromise of product improvement and process quality. Conventional lubricants' use attracted grave concern because of their adverse effects on the environment, workplace, safety of personnel, and high cost of disposal. Thus, the clamour for utilising alternative lubricant sources gains prominence, considering depletion of crude oil reserve, high crude oil prices, and regulations by several environmental regulatory bodies to achieve the manufacturing process's sustainability.

Biolubricants (vegetable oils, animal fats and plant-based) known to be biodegradable, environmentally friendly, non-toxic, high viscosity index, higher flash point and high lubricity are considered as a replacement for the petroleum-based lubricants (Mobarak et al., 2014; Mosarof et al., 2016). In addition to their comparable or superior performance over conventional lubricants in varied application areas, they offer significant benefits such as biodegradability, improved personnel safety, and lower manufacturing cost thereby satisfying process sustainability. Key Performance constraints of biolubricants such as low-temperature property and oxidative stability are improved upon using additives, modification of the chemical structure of the oils, and or genetic alteration of the seed crop (Shashidhara & Jayaram, 2010). Dispersion of nanoparticles is one of the methods employed in enhancing biolubricants' performance.

Many studies investigated the tribological properties of vegetable oil-based nano-enhanced biolubricants and reported improved performance over the past decades(Jayadas et al., 2007; Kashyap & Harsha, 2016; Su et al., 2015). Their application for metal cutting operations recorded tremendous improvement in cutting force reduction, tool life elongation and improving surface finish. The effect of base fluid types with nanographene inclusion on cutting force and temperature was examined by (Y. Su et al., 2016) during cylindrical turning of AISI 1045. Under same cutting condition, vegetable oil LB200 dispersed with nanographene indicated better performance over ester oil dispersed with nanographene in terms of cutting force reduction and temperature due to its better lubricity. (Park et al., 2011) reported reduced tool wear condition during the ball milling of AISI 1045 with nanographene enhanced biolubricant at 0.1 wt.% using coated carbide inserts when compared to pure base vegetable oil. Dispersion of gamma-Al_2O_3 nanoparticles at 1% and 2% volume fractions into vegetable oil was reported to improve surface roughness of machined surface by 15% and 25% respectively during the end milling of AISI D3 steel compared to pure vegetable oil lubrication (Hadi & Atefi, 2015).

However, less attention has is paid to thermo-physical properties enhancement and thermal stability despite being a crucial area of research in the use of nano-enhanced biolubricants. There is a need to understand the resistance of the novel nano-enhanced biolubricants to thermal degradation over increasing temperature applications. Similarly, developments of an accurate theoretical model for predicting thermal conductivity of nano-enhanced biolubricants based on their experimental responses will reduce wastages as trial formulations are eliminated, thereby saving time and resources.

1.2 Problem Statement

Vegetable oils (biolubricants) have been regarded as a suitable alternative for petroleum-based mineral oil lubricants due to their pleasant attributes, including environmentally benign, good lubricity, higher viscosity index, lower volatility, less toxic and biodegradable. The efficient performance of biolubricants is hampered at elevated temperatures due to thermal degradation as they become thermally unstable (Quinchia et al., 2014; Salih et al., 2013). The use of nano-additives has been reported to improve the performance of vegetable oil-based nanolubricants. However, the level of improvement on the thermo-oxidative stability can be appreciated through thermogravimetric analysis (TGA) study, an area with less attention in evaluating vegetable oil-based enhanced nanolubricants.

Several studies have been conducted on the thermal stability of nanolubricants. Magnetite nanoparticle coated with stearic acid was reported to improve the thermal stability of polyalphaolefin synthetic base oil (PAO8) (Zuin et al., 2017). The synthetic base lubricant was dispersed with weight fractions of the additives and evaluated for thermal stability under an inert and oxidizing agent. They further affirm that the improvement of thermal stability influence positively the oil retention capacity for effective tribological performance. In a related study, Zuin et al. (Zuin et al., 2020) evaluated the nano-enhanced lubricants for thermal stability and tribological performance. It was noted that the dispersed magnetite nanoparticles coated with stearic acid in commercial engine oil improve thermal stability and influence efficient lubrication property of the enhanced nanolubricants in lowering friction and wear of the contacting surface. It was reported by Ali et al. (Ali & Hou, 2020) that oxidation onset temperature and the burn-out temperature, which are critical parameters in evaluating the thermal stability of lubricants, were delayed longer using hybrid nano-additives. The improved thermal stability by nano-enhanced engine oil translated to improved brake thermal efficiency and heat transfer characteristics compared to the base lubricant. The resultant effects of the enhanced thermo-oxidation stability of nano-enhanced biolubricants on heat transfer capacity and tribological performance make it an important study area. However, researchers have yet to explore the thermo-oxidation stability of vegetable oil-based nano-enhanced biolubricants. There is no report from the literature on the thermal stability evaluation of graphene nanoplatelets (xGnP) and gamma Iron oxide ($\gamma Fe2O3$) dispersed in coconut oil as nano-enhanced biolubricants.

In addition, the preparation of nano-enhanced biolubricants involves several trials to attain the desired property enhancements, which result in wastages in terms of material, time and resources. Theoretical models have been developed to serve as guide in reproduction of nano-enhanced lubricants in previous studies (Afrand, 2017; Nabil et al., 2017; Sharif et al., 2016). Due to the difference in base fluid's thermophysical properties, a theoretical model implemented for a particular base fluid may not suit other lubricants. Developing an accurate theoretical model based on measured properties for thermal conductivity of nano-enhanced biolubricants will aid researchers and industry operators in selecting potential nanoparticles and vegetable oils (biolubricants) to formulate nano-enhanced biolubricants for future applications without preparation trials, thereby reducing downtime and saving resources.

1.3 Aims and Objectives

This current study aims to conduct a comprehensive investigation of the thermal degradation of lubricants for understanding the decomposition mechanisms of nano-enhanced biolubricants when subjected to elevated temperatures. Similarly, thermophysical properties over a range of temperature and nanoparticle concentrations will be evaluated. A theoretical model will be developed based on the response of bio-based nanolubricants thermal conductivity for a future blending of nanolubricants without performance testing. Therefore, the objectives of this research are:

i. To investigate the thermal degradation of nano-enhanced biolubricants at elevated temperature in comparison with pure biolubricants.
ii. To characterize the thermo-physical properties of nano-enhanced biolubricants in addition to developing a non-linear theoretical model for prediction of thermal conductivity.
iii. To access the tribological properties of bio-based nanolubricants and the based coconut oil.
iv. To evaluate machinability performance of titanium alloy under minimum quantity lubrication (MQL) technique using nano-enhanced biolubricants.

1.4 Scope

The research involves the development and performance evaluation of nano-enhanced biolubricants. Selected nanoparticles (xGnP and γFe2O3) at three varying volume concentrations of 0.1%, 0.2% and 0.3% were dispersed in coconut oil as base fluid to formulate the nanolubricants. TEM, FESEM, XRD and zeta potential are characterisation methods employed to characterise nanoparticles and the developed nanolubricants. Thermo-physical and tribological properties evaluation were carried out according to ASTM standard procedure to evaluate their performance characteristics as sustainable lubricants at varying concentrations. Thermogravimetric analysis (TGA) was employed to assess the oxidation and thermal stability of pure and nano-enhanced biolubricants to analyse lubricants' resistance to thermal degradation at elevated temperature under inert gas environment. Machining experiment using the best performing nano-enhanced biolubricants concentration in terms of anti-wear and friction reduction was conducted to study the machinability improvement (cutting force, tool life and surface roughness) during the end milling of titanium alloy (Ti-6Al 4V).

1.5 Significance of the Research

Nanoparticles have been used as additives in variety of base fluids such as water, ethyl glycol, conventional lubricants and biolubricants. Nano-enhanced base fluids and lubricants are applied for industrial cooling, vehicle cooling or transportation, refrigeration, heat exchanging devices and manufacturing processes. Nano-enhanced biolubricants are used to improve tribological performance of friction pairs. Thermophysical properties and thermal degradation analysis are rarely investigated despite their critical importance to performance of nano- enhanced biolubricants. Therefore, the current study will evaluate resistance of the nano-enhanced biolubricants to thermal degradation, thermophysical and tribological properties in addition to development of correlation models. Non-linear correlation models for predicting thermal conductivity based on the experimental responses for future use. These models will help researchers and industry operators formulate vegetable oil-based nanolubricants with nanoparticles dispersion for application, thereby reducing downtime and saving resources as the formulation of blends can be done without performance testing.

1.6 Thesis Outline

The contents of this thesis are divided into five (5) chapters. The first chapter details the introduction to the research work under the sub-section of the study's background, statement of the problem, Aims/objectives, the scope of the research and significance of the study. The second **chapter** tagged literature review evaluated research topics related to current research such as the conventional lubricants, biolubricants and nanoparticles as additives in base fluids. Thermophysical and tribological properties of lubricants and their application in machining operations were discussed. Besides, thermal degradation study of oils and their effect on lubricants' performance under elevated temperature. The third chapter described in detail the materials and methodologies employed to achieve the research work's stated aims and objective. Formulation of nano-enhanced lubricants using two different nanoparticles, characterisation of nanoparticles and the nano-enhanced oils, measurement of relevant properties and the various equipment used. Procedure for machining experiments under MQL lubrication and varying cutting parameters were also presented. The fourth chapter is devoted to results and discussion of findings from the analysis of nano-enhanced biolubricants. Non-linear correlation models for prediction thermal conductivity and dynamic viscosity of nano lubricants were developed. The nano-enhanced biolubricants was further evaluated in a milling machining operation to study Titanium alloy machinability (Ti-6Al-4V) under MQL cooling/lubrication strategy. The last and the fifth chapter is a compendium of the key findings from this research and the future study direction in a potential area for consideration.

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1. Sharif, S., **Sadiq, I. O.**, Yusof, N. M., & Mohruni, A. S. (2017). A review of minimum quantity lubrication technique with nanofluids application in metal cutting operations. *International Journal on Advanced Science, Engineering and Information Technology*, *7*(2), 587-593. (**Indexed by SCOPUS).**
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