

**ENHANCEMENT IN TRIBOLOGICAL
PERFORMANCES OF ADVANCED
MICROWAVES SYNTHESISED
MoS₂ NANOPARTICLES AS NANO ADDITIVES
IN MILITARY DIESEL-BASED ENGINE OIL**

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TITLE

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ABSTRACT

The automobile industry has a strong emphasis on environmentally friendly, high-quality, long-lasting, and energy-efficient features. Friction and wear are the primary causes of energy loss and mechanical failure. The application of lubricant additives is one of the most effective ways to reduce friction and wear under boundary lubrication. The use of nanoparticles/nanomaterials as lubricant additives in lubricants such as engine oil is referred to as nanolubricants. However, nanoparticles addition in military diesel-based engine oil remains largely unexplored. In this dissertation, optimised Molybdenum disulfide (MoS_2) nanoparticle was synthesised via advanced microwave synthesis method using Response Surface Methodology. The physicochemical parameters of the optimised MoS_2 nanoparticles have been thoroughly studied. The nanoparticle was dispersed in SAE 20W50 military-grade diesel engine oil to formulate the nanolubricant. The tribological, oxidation and thermal conductivity properties of the nanolubricant have been investigated using a wide range of analytical methods. FESEM images confirmed the nanosheet morphology of the MoS_2 with sizes approximately 150nm-300nm and the high-resolution EDS elemental mapping has shown the uniform and homogeneous distribution of molybdenum and sulfur. The broad XRD diffraction peaks of MoS_2 implied that the crystalline size is very small and confirmed that the crystal structure is pure MoS_2 . The FT-IR spectra reveals the required functional groups, which further confirms the formation of MoS_2 . The visual observation confirms that clearly indicates there was no sign of sedimentation after 21 days in the nanolubricants. The zeta potential value of the MoS_2 nanolubricant with 0.05wt. %, 0.01wt. % and 0.005wt. % is to be extremely stable. The nanolubricant with 0.01 wt.% concentration of MoS_2

nanoparticle showed the reduction of Coefficient of Friction (COF) and Average Wear Scar Diameter (WSD) with 19.24 % and 19.52 % decrement compare to the base oil due to the formation of tribofilm and the mending effect. The nanolubricant with 0.05 wt.% concentration of MoS₂ nanoparticle shown the enhancement of 65.68 % in Oxidation Induction Time (OIT) compare to the base oil. The synergistic effect of MoS₂ nanoparticles and Zinc dialkyldithiophosphates (ZDDP) can exhibit good oxidation stability, which enhanced the antioxidant properties. The addition of MoS₂ within the base oil demonstrates an improvement in thermal conductivity with ~10 % enhancement compared to the base oil. This due to the percolation mechanism, which may increase the thermal conductivity. This study has essentially provided a thorough understanding of a revolutionary advanced microwave synthesised MoS₂ based nanolubricant. This research's integrated approach to understanding tribological, oxidation, and thermal conductivity mechanisms is expected to lead to novel strategies for building superior nanolubricants for military vehicles in the future.

ABSTRAK

Industri automobil mempunyai keutamaan pada ciri mesra alam, berkualiti tinggi, tahan lama, dan penggunaan tenaga dengan cekap. Geseran dan kehausan adalah penyebab utama kehilangan tenaga dan kegagalan mekanikal. Penggunaan bahan tambahan pelincir adalah salah satu kaedah paling berkesan untuk mengurangkan geseran. Penggunaan nanopartikel / nanomaterial sebagai bahan tambahan pelincir dalam pelincir seperti minyak enjin disebut sebagai bahan pelincir nano. Walau bagaimanapun, penambahan nanopartikel dalam minyak enjin gred tentera berasaskan diesel masih belum diterokai luas. Dalam disertasi ini, nanopartikel Molybdenum disulfide (MoS_2) yang dioptimumkan telah disintesis melalui kaedah Sintesis Gelombang Mikro Moden menggunakan Metodologi Permukaan Respons. Parameter fizikokimia nanopartikel MoS_2 yang dioptimumkan telah dikaji secara menyeluruh. Nanopartikel tersebut diserakkan dalam minyak enjin gred tentera berasaskan diesel SAE 20W50 untuk memformulasikan bahan pelincir nano. Sifat tribologi, pengoksidaan dan kekonduksian terma bahan pelincir nano telah diselidiki menggunakan pelbagai kaedah analisis. Imej FESEM mengesahkan morfologi nanosheet MoS_2 dengan ukuran kira-kira 150nm-300nm dan pemetaan elemen EDS beresolusi tinggi telah menunjukkan taburan molibdenum dan sulfur yang seragam dan homogen. Puncak difraksi XRD luas MoS_2 menyiratkan bahawa ukuran kristal sangat kecil dan mengesahkan bahawa struktur kristal adalah MoS_2 tulen. Spektrum FT-IR mengungkapkan kumpulan fungsional yang diperlukan, yang selanjutnya mengesahkan pembentukan MoS_2 . Pemerhatian visual jelas menunjukkan tiada tanda pemendapan setelah 21 hari di bahan pelincir nano. Nilai potensi zeta bahan pelincir nano MoS_2 dengan berkepekatan 0.05wt. %, 0.01wt. % dan 0.005wt. % menunjukkan

kestabilan yang tinggi. Bahan pelincir nano dengan berkepekatan 0.01wt. % menunjukkan pengurangan Pekali Geseran (COF) dan Purata Diameter Kehausan (WSD) dengan penurunan 19.24% dan 19.52% dibandingkan dengan minyak asas kerana pembentukan tribofilm dan kesan penyembuhan. Bahan pelincir nano dengan kepekatan 0.05 wt.% nanopartikel MoS₂ menunjukkan peningkatan 65.68% dalam Masa Induksi Pengoksidaan (OIT) berbanding dengan minyak asas. Kesan sinergistik nanopartikel MoS₂ dan Zink dialkyldithiophosphates (ZDDP) dapat menunjukkan kestabilan pengoksidaan yang baik, yang meningkatkan sifat antioksidan. Penambahan MoS₂ dalam minyak asas menunjukkan peningkatan kekonduksian terma dengan peningkatan ~ 10% berbanding dengan minyak asas. Ini disebabkan oleh mekanisme perkolasi, yang dapat meningkatkan kekonduksian terma. Kajian ini pada asasnya memberikan pemahaman menyeluruh tentang bahan pelincir nano MoS₂ berasaskan Sintesis Gelombang Mikro Moden. Pendekatan bersepadu penyelidikan ini adalah untuk memahami mekanisme kekonduksian, pengoksidaan, dan kekonduksian termal dengan penghasilan bahan pelincir nano yang unggul untuk kenderaan tentera di masa hadapan.

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LIST OF ABBREVIATION

MoS₂	Molybdenum Disulphide
COF	Coefficient of Friction
WSD	Average Wear Scar Diameter
OIT	Oxidation Induction Time
ZDDP	Zinc Dialkyldithiophosphates
RSM	Response Surface Methodology
SAPS	Sulphated Ash, Phosphorous and Sulphur
API	American Petroleum Institute
PAO	Polyalphaolefin
PAG	Polyalkyleneglycol
MWCNTs	Multi-Walled Carbon Nanotubes
SAE	Society of Automotive Engineers
TMC	Transition Metal Chalcogenide
CCD	Central Composite Design
MoDTC	Molybdenum Dithiocarbamate
UHMWPE	Ultra-High-Molecular-Weight Polyethylene
STRIDE	Science and Technology Research Institute for Defence
ILSAC	International Lubricant Specification Advisory Committee
DOE	Design of Experiments
ANOVA	Analysis of Variance
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy-Dispersive X-Ray Spectroscopy
XRD	X-ray Diffractometer

FTIR	Fourier-Transform Infrared Spectroscopy
ASTM	American Society for Testing and Materials
HP-DSC	High-Pressure Differential Scanning Calorimeter
LFA	Laser Flash Analysis
DOE	Design of Experiment
JCPDS	Joint Committee on Powder Diffraction Standards
et al.	(et alia): and others

CHAPTER 1

INTRODUCTION

1.1 Background

Friction and wear are the primary causes of energy dissipation and mechanical outage. As a result, reducing friction and wear is crucial for extending the life of mechanical equipment while also conserving energy and lowering emissions[1]. Lubrication is one of the most productive strategies for reducing friction and wear, which is essential for energy saving, environmental protection, and emission decrement[2]. Mineral oil, bio lubricant, and synthetic oil are the most common lubricants. Synthetic oil provides several advantages over mineral oils, including the ability to lubricate at extremely low or high temperatures and better wear resistance. Synthetic oils also have several benefits, including decreased energy usage maintenance costs and increased energy efficiency[3].

There are two types of engine oil in today's cars: gasoline-based engine oil for gasoline-powered vehicles and diesel-based engine oil for diesel-powered vehicles. Due to their prolonged use with heavy ammunitions during the unanticipated conflict, military vehicles are one of the few vehicles that face tremendous heat and pressure. Therefore, it has supplemented with diesel-based engine oil to tackle the problem, as reducing friction in the combustion engine is crucial for efficiency. However, the existing diesel-based engine oils

possess lower tribological, oxidation and thermal performances, which cause damages to engine parts and massive heat energy loss in a short period of usage[4]. The incorporation of nanoparticles in lubricants has opened up to a new class of lubricant, the nanolubricant. Nanolubricants are advantageous over conventional lubricants in the sense that they provide significant enhancement in tribological properties[5, 6]. However, there has been no research that has reported on the tribological characteristics of military diesel-based engine oil used by the Malaysian Armed Forces. Furthermore, the concept of nanolubricant is relatively new in the Malaysian context and globally as well.

The base oil used in this study is military diesel-based engine oil to improve its tribological qualities. First, the MoS₂ nanoparticle-based nanolubricant were synthesised and characterised. This research is the first to attempt the synthesise using an advanced microwave synthesis route for tribological application. This research focuses on optimising the synthesis of MoS₂ nanoparticles using an advanced microwave synthesis method, which saves time, energy and produces better tribological, oxidation and thermal conductivity properties than the traditional hydrothermal method. The physicochemical parameters of the optimised MoS₂ nanoparticles were then determined, and the nanoparticles were dispersed in Military diesel-based engine oil to develop a novel nanolubricant. Following that, the tribological, oxidation, and thermal characteristics were investigated. The primary goal of this study is to create MoS₂ nanoparticles using advanced microwave technology, which have improved tribological, oxidation, and thermal properties when distributed in military diesel engine oil. This study will pave the path for developing new microwave-synthesised MoS₂ nano additives for military diesel engine oil.

1.2 Problem Statement

Engine oil is critical for maintaining the efficiency of vehicle machinery. To avoid friction and damage to the moving contact surfaces, it lubricates the contact points of all gear and wedges in the engine. Due to its extensive use with massive loads amidst unexpected battle, military vehicles are one of the few vehicles that experience tremendous heat and pressure[4]. To improve the engine's performance, it's critical to reduce friction and wear, as well as improve the oxidative and thermal properties of the engine oil. Currently, engine oil used for military vehicles deployed without any nanoparticles. As a result, it possesses lower tribological, oxidation and thermal performances, which cause damages to engine parts and massive heat energy loss in a short period of usage[7].

Furthermore, the lubricant used also tend to oxidise in several months of usage. In that context, it also increases the maintenance costing and vehicles downtime. Besides, the current hydrothermal method of MoS₂ nanoparticles synthesis requires an enormous amount of time and energy[8].

1.3 Objectives

This research aims to achieve a comprehensive understanding of advanced microwave synthesised MoS₂ nanoparticles based nanolubricants. To accomplish this, thermo-physical characterisations are necessary. Furthermore, application studies of tribology, oxidation and thermal conductivity involving the MoS₂ nanoparticles dispersed in military diesel-based engine oil would pave the way for uncovering the interaction of MoS₂ in a combustion engine of a military vehicle. Therefore, the objectives of this study are:

- 1) To synthesise optimised MoS₂ nanoparticles via advanced microwave using Response Surface Methodology (RSM) for tribological application.
- 2) To assess the physicochemical properties of the optimised MoS₂ nanoparticles by using FESEM, EDX, XRD, FTIR and Zeta potential
- 3) To investigate the tribology, oxidation and thermal conductivity performances of the MoS₂ nanolubricant formulation.