TRIBOLOGICAL PERFORMANCE ENHANCEMENT OF RAPESEED BIO-LUBRICANT USING MODIFIED EICHHORNIA CRASSIPES NANOPARTICLES ADDITIVE

ANTHONY CHUKWUNONSO OPIA

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Faculty of Engineering
Universiti Teknologi Malaysia

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DEDICATION

To my beloved parents, brothers, sisters and friends

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ABSTRACT

Lubricant additives are substances that are used in reducing friction and wear between two or more moving components. They are chemically, partially, or mechanically produced with the aim of controlling friction and wear during sliding contact. Various functional vegetable lubricants have been developed with narrow views of the environmental impact issue due to the use of fossil-based additives in the formula. These lubricants can be made more sustainable by using all environmentally friendly and renewable base stock items. However, few researchers, who took these factors into account, emphasised the use of biomaterials in the lubricant additive formulations mainly because of the challenges in their physicochemical characteristics. Eichhornia Crassipes (EC) have been used for industrial applications such as sorbent and bio-fuel, but application in lubrication additive is rarely reported in the literature. Therefore, in this study, EC, a pure aqua bio-plant, was used to enhance the base lubricant performance for lubrication application, based on the most desirable lubricant additive qualities validated by characteristic tests and good compatibility due to their similar functional groups. For the development of EC carbon nanotubes (EC-CNT) that provide anti-wear service, the formulation uses a mechanical approach of ball milling and a cyclic heating method. Synthetic technique was used to develop the EC Carboxymethyl cellulose (EC-CMC) polymer. The novel EC-additives were formulated to improve to improve the tribological qualities and shear stability requirements of the base lubricant. The tribological studies were conducted using a high frequency reciprocating test rig and a unidirectional ball on disc tribo-tester. The final EC-additive package was chosen after optimal concentration study. This indicated that 1.2 wt. % EC-CNTs + 0.4 wt. % EC-CMC yielded the best performance. The formed nanofluid, which contains 98.4 % base lubricant and 1.6 wt.% EC-additives (1.2 wt.% EC-CNTs + 0.4 wt.% EC-CMC) as the additive package, has a coefficient of friction (COF) of 0.08 and average wear scar diameter (WSD) of 54.1 μm on High Frequency Reciprocating Rig (HFRR), better than unidirectional mode. EC-additive results under HFRR were compared to commercial Zinc Dialkyl Dithiophosphate (ZDDP) as a benchmark (COF = 0.07, WSD = 48.3 nm), which were found to be substantially similar. The good performance of EC-additive under HFRR mode was due to little starvation of lubricant at the contact region. Under HFRR mode, better surface protection was achieved while under unidirectional mode yielded severe surface wear due to much lubricant starvation at the contact surface. Shear stability tests of the new EC-additive and the commercial ZDDP were performed for a range of 10 to 1500 1/s shear rate 25^{o}C to 75^{o}C temperature. The test results showed that the novel EC-additive has better shear stability than ZDDP, which could be attributed to the presence adequate polymer. Repassed oil blended EC-additives that can improve tribological performance of a vegetable lubricant during sliding contact lubrication have been successfully developed. This research proved that the performance of the base lubricant can be enhanced by using the novel EC-additive formulated from a pure aqua bio-plant. This means that EC-additive has the potential to effectively replace fossil-based additives for lubrication operation within the tested capacity, resulting in an ecologically sustainable, renewable and good tribological performance.

ABSTRAK

Bahan tambah pelincir ialah bahan yang digunakan dalam mengurangkan geseran dan haus antara dua atau lebih komponen yang bergerak. Ia dihasilkan secara kimia, sebahagian atau mekanikal dengan tujuan mengawal geseran dan haus semasa sentuhan gelongsor. Pelbagai pelincir sayuran berfungsi telah dibangunkan dengan pandangan sempit tentang isu kesan alam sekitar kerana penggunaan bahan tambahan berasaskan fosil dalam formula. Minyak pelincir ini boleh dibuat lebih mampan dengan menggunakan semua item stok asas yang mesra alam dan boleh diperbaharui. Walau bagaimanapun, beberapa penyelidik, yang mengambil kira faktor-faktor ini, menekankan penggunaan bahan-bio dalam formulasi bahan tambah pelincir terutamanya kerana cabaran dalam ciri fizikokimia mereka. Eichhornia crassipes (EC) telah digunakan dalam industri seperti bahan mengerap dan bahan api-bio, namnu penggunaan dalam bahan tambah pelincir sangat jarang dilaporkan dalam literatur. Oleh itu, dalam kajian ini, EC, tumbuhan bio aqua tulen, telah digunakan untuk meningkatkan prestasi pelincir asas untuk aplikasi pelinciran, berdasarkan kualiti bahan tambah pelincir yang paling diingini yang disahkan oleh ujian kecirian dan keserasian yang baik kerana persamaannya. kumpulan berfungsi. Untuk pembangunan tiub karbon nano EC (EC-CNT) yang menyediakan perkhidmatan anti-haus, formulasi menggunakan pendekatan mekanikal pengilangan bebola dan kaedah pemanasan kitaran. Teknik sintetik digunakan untuk membangunkan polimer EC Carboxymethyl cellulose (EC-CMC). Bahan tambah EC yan baharu telah dirumus untuk menambah baik kualiti tribologi dan keperluan kestabilan ricih pelincir asas. Kajian tribologi telah dijalankan menggunakan pelantar ujian salingan frekuensi tinggi dan bola satu arah pada penguji tribo cakera. Pakej bahan tambah EC akhir telah dipilih selepas kajian kepekatan optimum. Ini menunjukkan bahawa 1.2 wt. % EC-CNTs + 0.4 wt. % EC-CMC menghasilkan prestasi terbaik. Bendalir nano yang terbentuk, yang mengandungi 98.4 % pelincir asas dan 1.6 wt.% bahan tambah EC (1.2 wt.% EC-CNTs + 0.4 wt.% EC-CMC) sebagai pakej bahan tambah, mempunyai pekali geseran (COF) sebanyak 0.08 dan purata diameter parut haus (WSD) sebanyak 54.1 µm pada Rig Salingan Frekuensi Tinggi (HFRR), lebih baik daripada mod satu arah. Kekurangan bahan tambah EC di bawah HFRR dibandingkan dengan Zinc Dialkyl Dithiophosphate (ZDDP) komersial sebagai penanda aras (COF = 0.07, WSD = 48.3 nm), yang didapati hampir sama. Prestasi baik bahan tambah EC di bawah mod HFRR adalah disebabkan oleh sedikit kekurangan pelincir di kawasan sentuhan. Di bawah mod HFRR, perlindungan permukaan yang lebih baik telah dicapai manakala di bawah mod satu arah menghasilkan kehausan permukaan yang teruk akibat kekurangan pelincir pada permukaan sentuhan. Ujian kestabilan ricih bagi aditif EC baharu dan ZDDP komersial telah dilakukan untuk julat 10 hingga 1500 1/s kadar ricih 25°C hingga 75°C suhu. Keputusan ujian menunjukkan bahawa bahan tambah EC baharu mempunyai kestabilan ricih yang lebih baik daripada ZDDP, yang boleh dikaitkan dengan kehadiran polimer yang mencukupi. Bahan tambah EC campuran minyak yang dilalui semula yang boleh meningkatkan prestasi tribologi pelincir sayuran semasa pelinciran sentuhan gelongsor telah berjaya dibangunkan. Penyelidikan ini membuktikan bahawa prestasi pelincir asas boleh dipertingkatkan dengan menggunakan bahan tambahan EC baharu yang dirumus daripada tumbuhan bio akua tulen. Ini bermakna bahan tambah EC mempunyai potensi untuk menggantikan bahan tambahan berasaskan fosil dengan berkesan untuk operasi pelinciran dalam kapasiti yang diuji dalam menghasilkan prestasi tribologi yang mampan dari segi ekologi, boleh diperbaharui dan baik.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

EC	-	Eichhornia crassipes
EC-NPs		Eichhornia crassipes nanoparticles
EC-CNTs	-	Eichhornia Crassipes carbon nanotubes
EC-CMC	-	Eichhornia Crassipes Carboxymethyl cellulose
RO	-	Rapeseed oil
MO	-	Mineral oil
ZDDP		Zinc dialkyldithiophosphates
WSD	-	Wear surface diameter
COF	-	Coefficient of friction
AFM	-	Atomic force machine
SEM	-	Scanning Electron Microscopy
EDX	-	Energy-dispersive X-ray
XPS		X-ray photoelectron spectroscopy

LIST OF SYMBOLS

μ	-	Coefficient of friction
D, d	-	Diameter
F	-	Force
L	-	Load
a	-	Contact area
δ	-	Maximum deflection
r	-	Radius
$R^/$		Radius of the curvature
$E^/$		Reduced young modulus
λ		Lambda
Ra	-	Surface roughness
Rz		Roughness depth
Rq		Root mean square
B1		Rapeseed oil
B2		Mineral oil
A1		RO + EC-bio-additive
A2		MO+ EC-bio-additive
A3		RO + ZDDP

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

The study on the tribological performance enhancement of bio lubricant (rapeseed oil) using eichhornia crassipes nanoparticles (EC-NPs) bio-additives is introduced in this chapter. The overall background describes the operation and function of lubricant action, the impact of bio-additives as substitute to synthetic and inorganic based additives on living health, and climatic effect. Other related issues on conventional petroleum derived lubricant application in mechanism sliding contact were explained. The idea to propose eichhornia crassipes as lubricant additives both for synthetic and bio-lubricants for contact lubrication is based on it potentials towards world sustainability strive [1,2]. Thus, this chapter also includes the research problem statements, research objectives, research questions, scope of the work. The significant of the research with critical understanding on the tribological behavior of bio additives (eichhornia crassipes) in lubricants for sliding contact are elucidated.

1.2 Background of Study

Lubricating oil and grease grant protection to the rubbing machine components against friction and wear. The operation and functions are not limited to cooling and lubrication, but they also contribute in extending the lifespan of the components in contact with the fluid. Almost all moving engine components needs lubrication to minimize and maintain temperature required. Tribological improvement on lubricants has been a serious concern in the automobile industry. The engineer responsible for developing low friction lubricants requires knowledge of the engine as a whole [3–6], its subsystems and constituent components. The mechanism of sliding contacts generates great chemical energy (heat) which further converted into rotary motion via mechanical energy, thus needs adequate cooling medium [7–9]. In doing this, it is important to adopt system that will not affect the operation of the machine, environment as well as to maintain the ecosystem.

Today, most of lubricants together with its additives applied in most sliding mechanisms are petroleum (synthetic) derived products [10–12]. The product generated during the mechanical operation of these lubricants and additives (petroleum), turns to be a serious threat to our ecosystem through their emission, especially the greenhouse gas [4,13–15]. Green technology appears to be a viable alternative to this environmental threat. These bio lubricants and additives, which are carefully prepared for existing engines with or without modifications, considerably exhibit good lubrication in hopes of helping the world's sustainability initiative [13,16,17]. It is a serious concern in the industry that the purpose of inclusion of additive to overcome friction and the expected engine output are not fully achieved, perhaps due to poor or inadequate additive/lubricant applied [3,18–20].

Friction therefore, is the resistant force of relative motion of solid surfaces, fluid layers, and metal elements under an applied load. In thermodynamics, the amount of energy utilized to overcome friction is not available for useful work [3]. Bio-additives and bio-lubricants have better affinity to metal surfaces in terms of tribological performance, provides better anti-corrosion than petroleum derived type [21,22]. Bio-additives are active green substitute to toxic inorganic compound constitutes like sulphur (S) and Phosphorus (P) based additives. It also has capacity in synergetic enhancement with conventional ZDDP and other high additives [23,24].

Global warming, in general, is a disaster produced by human activities as a result of rising global temperatures, primarily due to high levels of carbon dioxide and other greenhouse gases in the atmosphere [4,25,26]. It is an established fact that additives influence the ash formulation and toxicity characteristics of a lubricant formulation [16]. The conventional additives both synthetic and inorganic compounds contain toxic elements ranging from phosphorus, ashes, and sulphur (PAS), also multiples of pollutants are recorded from the use of synthetic mineral oil base lubricants. The chemical characteristics of by-products of bio-additives are found low risk to flora, fauna and the ecosystem [24,27,28]. As bio-additives discovered and found considerable alternatives to synthetic and inorganic compounds, similarly non-toxic bio-lubricants would be required [9,16,29]. Eco-friendly lubricant can complement bio-additives in mitigate the release of sulphur, carbon oxides, methane, nitrous oxides and other greenhouse gases [11,16,23].

Following world innovation, conventional additives contain anti-wear (AW) additives, zinc diakyldithio-phosphate (ZDDP) [11,30,31]. The package (ZDDP) has the potential to work with other brand additives that contribute necessary characteristics to the fully developed lubricant [31,32]. Many class additives in a complete engine lubricant contain viscosity modifiers (VM or VI), friction modifier (FM), extreme pressure agents, corrosion inhibitors, dispersants, detergents, antioxidants, pour point depressants and hydrophobic tendency [11,30,33–35]. For optimum friction and wear reduction, additives with constituents of S and P are considered to replace with nonylphenol (NP) due to their excellent performance during service [36,37]. It is generally desirable that all the lubricating materials should maintain its anti-wear and antifriction performance for a long period of time in order to keep the moving parts of the machine functional both in lubrication and cooling.

With this, the friction together with other negative occurrences can be mild and may not cause misadventure failure. Additives as well as the fundamental oil base stock used in producing most of these lubricants are petroleum-derived or synthetic [12,38]. In the study of synthetic produced lubricants from cycloaliphatic group, like polyalphaolefins (PAOs) and gas to liquid (GTLs) base stocks, they are an important class of lube base stocks with many good lubricating properties, including viscosity, but have low polarity [39]. This low polarity leads to low solubility and dispersancy for polar additives and sludge generated during service. These base stocks require the use of co-base stocks additives to improve lubricant behavior and deposit solubility [24].

Eichhornia crassipes plant additives (solid/powder phase) of infinitesimal sizes below 100nm, with high property in carbon nanomaterials for nanotubes additive (friction reduction) [40,41] and formulated carboxymethyl polymer (viscosity modifier) with oil lubricant affinity, thereby would establish homogenous and stable colloidal scattering behavior [34,42]. Biomass appears to be an adequate attractive feedstock because of its potentials in abundance, renewability and practical environmental impacts resulting in no net release of carbon dioxide and very low sulphur content [43–45]. Nanomaterials are distinct from traditional bulk materials because of their extremely small size and strong specific surface area [45,46]. In this regard, lubricants have mixed with nanoscale additives to form homogeneous colloidal dispersion mixture, which significantly applied in mitigating most of friction and wear occurrences in fluid lubricated systems [47,48].

However, in order to lift the demand and tribological properties, synthetic formulated additive borates and boronate esters derived from boric acids or boronic acids were adopted and vigorously studied [49]. The borates and boronate ester formulated additive provides less ashes, almost phosphorus free and reduce sulphur. But many effects were recorded ranging from toxicity, poor degradability and highly flammable and do require special handling care [49]. Consequently, aryl amine and phenol additives provides a synergistic enhanced performance but considered beyond cost, owing to the restrictive high cost of its synthetic oil [50,51].

Therefore, there is a need for good sorption additives that provide appropriate solubility and dispersibility for the oil lubricants and control sludge generated during service of lubrication. The expectation of plants additives in the formulation of bio lubricants had been indicated by some bio lubricant researchers [52–54]. The achievability's of accepted plant for additive formulation where based on potential alkalinity levels and total base number and some other physical properties that have been reported by bio researchers [55–57].

1.3 Problem Statement

The global environmental challenges on pollution that are associated with human activities through industrial operations are still a major concern. Additives are widely used in petroleum (non-renewable) derived lubricants to enhance lubrication performance. However, those lubricants together with additives still contains some toxic constituents, has poor biodegradability and contributes significantly to eco-pollution. Petroleum additives have been studied, most product contain very high percentage content of mineral oil, implying a possible high level of toxicity and highly poor biodegradability. To be considered suitable for consumption, the lubricant base oil together the inclusive additives are expected to possess little or no toxic constituents as not to affect human health and environment. As a result of these, bio lubricant with features similar to the conventional mineral oil with non-toxic effect, need to be considered as alternative in the area of sliding contact lubrication.

The aim of additives inclusion in lubricants is to achieve good shear stability, good tribological properties required to control excessive temperature and some other unwanted conditions. At this point, no advance work has been reported using pure nanoparticle plant as base additive for lubricants. In this work, formulation of lubricant nano-additive using unrefined physio mechanically approach have been proposed. This is based on the convincing information on the prospects of EC material meeting some serious requirements for lubricant additive formulations (bio sorption property [58], and good for carbon nanotubes [23] and polymer [59]), though this may require some previous conventional approaches. Recent findings among bio lubricant researchers have it partially refined, mechanically extracted plant oils, possess high stability, exhibit better response to additives (plant additives) and lubricants and tribological behavior than their refined counterparts [60,61].

Although good number of additives for lubricants enhancement have been used in the past, they still require detail investigation to answer many questions, such as raw/synthetics materials selection, tribological behavior towards withstanding the working condition. Therefore, as advancement pushes with intensity of developing more green technology by tribological manufacturers, the constraint insists researchers to formulate new product that respond more effectively to the final users, also always available and affordable.

1.4 Objectives of the Study

In this research, the aim is to provide solutions to those mentioned worrisome environmental effect, sustainability and cost problems associated with lubricants by formulating additive from Eichhornia Crassipes plant. However, in order to achieve the goal, the following precise objectives were considered.

i. To determine the best concentration (wt.%) of the formulated Eichhornia Crassipes carbon nanotubes (EC-CNTs) and Eichhornia Crassipes Carboxymethyl cellulose (EC-CMC) additives for optimum tribological performance in rapeseed base oil.

ii. To analyse the tribological effect of the optimal formulated EC-additives in rapeseed oil and compare it to mineral oil and ZDDP additives under boundary lubrication regime.

iii. To propose the frictional wear mechanisms of the optimal formulated EC-additives in rapeseed oil and compare it to mineral oil and ZDDP additives under boundary lubrication regime.

iv. To investigate the shear stability of the optimal formulated EC-additives in rapeseed oil and compare it to ZDDP additives.

1.5 Research Questions

The concern task is to produce an economical substitute to petroleum derived additives for lubricants from a productive source, especially a water plant and environmentally friendly type. The appropriate questions that required to be followed in accomplishing this task are:

i. How could formulation of the sorption (lubricants and bio-additives additive) most suitable as optimum base class for lubricant additive achieved?

ii. Why are there significant effects of the formulated EC-NP additives on friction and wear generation during working conditions?

iii. How adequate is the shear stability of the produced additive in meeting the stipulated requirements of international additives?

1.6 Scope of the Study

Lubricant additives application in the field of engineering covers a very wide range. The suitable usage of the additive, however, strongly depends on the mechanism working condition and additive strength. In order to achieve the objective of the study, the following scopes are been outlined.

i. Two types of NPs additives (EC-CNTs and CMC polymer) formulated from Eichhornia Crassipes and inorganic based ZDDP counterpart for reference are used.

ii. Usage of rapeseed bio-lubricant was used as a key study base oil, compared to base mineral oil in respect to compatibility with the new formulated additives.

iii. Application of ASTM G133 ball on flat and ASTM G99 ball on Disc under ISO 6892-1 tribological standard test were carried out on the formulated additives using selected loads under different temperatures (RT and 100 ^{o}C) respectively.

vi. The application of EC-NPs additives with the lubricant is limited on the selected concentration (wt.%) with optimum performance in the initial coefficient of friction and wear analysis conducted. ZDDP was also included at a concentration of 1 wt.% for comparison.

v. Sliding contact and boundary lubrication behavior (friction and wear) of the selected lubricant and the formulated NPs additives.

vi Additives study limitations also include suitable shear stability (viscosity indices, kinematic viscosity) with its good response to lubricants.

1.7 Significance of the Study

Adopting formulated Eichhornia Crassipes nanoparticles as complementary and alternative to non-renewable petroleum derived additives for lubricants, has not been applied by previous researchers. It is, therefore necessary for current research work to explore and examine its additives impact on base lubricants during lubrication. Having known the physiochemical properties of the Eichhornia Crassipes materials and behaviors in lubricants [55,62]. Various classifications of lubricant, functional fluid (base oil) and additives have been examined in furtherance of replace and improved lubricating performance of the petroleum base or inorganic base lubricants, that known as the major additives/lubricants base stock with plants formulated additive type. The sector of applications of these products covers both industrial, automotive and domestic.

Conventionally, machine (mechansism) dealers and consumer's curiosity in enhanced motoring comfort, and satisfaction, crave for higher quality of vehicles and bestride will universally escalate the use of lubricants together with consistency in using better quality lubricants, additives among other variables. The World summit on

Social Development identified sustainable development goals, such as economic development, social development and environmental protection [15,25]. The sustainable development consists of balancing local and global efforts to meet basic human needs without destroying or degrading the natural environment. Recent publications forecasted that the expected global numerical strength of light-duty automobile in 2040 will shy by a very small margin from 1.7 billion, rising from 900 million the same year, a near double of the fleet in 2014 [63]. Adding to the global vehicle fleet, covering passenger cars and commercial vehicle, will rise from the current total of 1.2 billion to over 2.4 billion by 2035 [64].

It is a known fact that scientists in 2007 (UNFCC, 2009) concluded that there was at least a 90% probability that atmospheric increase in toxic products, was human-induced, mostly as a result of emissions from fossil and petroleum products [4,15,64]. These by implication, point to more environmental risk in the future if petroleum derived lubricants and additives are not replaced with eco-friendly types [15,24,65]. Confronting environmental risk through lengthen market lubricating oils and additives, makes the present research more relevant. Adopting green economy as an approach of improving human well-being and social equity, evidently will significantly reduce environmental risks.

1.8 Outline of Thesis

The outline of this proposal from chapter 2, started with the issues of friction under sliding lubrication. The lubricant properties relevant to optimum tribological performance were also introduced. The need of formulating eco-friendly additives became necessary in solving catastrophes confronted the ecosystem with constant use of petroleum/synthetic lubricant and additives was developed. The nanoparticle additives positive impact on friction and wear reduction during lubrication mechanism were discussed.

Chapter 3 presented the analytical tools, experimental procedure and the formulation of various components of the EC-NPs additive. The corresponding mixing techniques of developed additives with the suitable tribological test were critically discussed. Chapter 4 showcase the characterization results of the new formulated EC-CNTs and EC-CMC additives. Various tribological test together the optimal concentration of the two additives was conducted as to select the final additive package. The tribological behavior of the new formulated additive in base rapeseed oil were evaluated under different working conditions compared to mineral oil and organic ZDDP additive. The wear surface images analysis and the lubrication phenomena and mechanisms as well as the shear stability features were conducted. Chapter 5 covers the conclusions and discussion of this study, connecting to its contribution in reducing excessive friction and wear during lubrication.

1.9 Summary

A clear background on the lubrication challenges associated with high heat and friction generation and its resultant effect on energy losses were discussed in this chapter. The environmental catastrophe caused by persistent consumption of synthetic/petroleum blended lubricant (base oil and additives) were elucidated. Reliable objectives towards carrying out this study have been developed with a motivated research question towards actualization of the objectives mentioned. Confirmed holistic scope of the study that will not affect the objectives of the work was drawn and followed accordingly.

The next chapter, gives an elaborate review of previous literatures on the research describing friction mechanism, requirements of good additives for optimum lubrication. The ideas extracted from literature works, provides a clear structure towards preparation of EC-nano-additives using simple and cost effective technology as to address the research questions and meeting the objectives aspiration.

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1. **AC. Opia, MKA Hamid, S. Syahrullail, CAN. Johnson, et al.,** (2021). Eichhornia crassipes nanoparticles as a sustainable lubricant additive: Tribological properties optimization and performance under boundary lubrication regime, 175: 114252. Industrial crops and products, (Q1, Index WoS).
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3. **AC. Opia, MKA Hamid, S. Syahrullail, CAN. Johnson, et al.,** (2021). Improving Tribological Properties and Shear Stability of Base Lubricant using Eichhornia Crassipes Carboxylmethyl Polymer under Different Conditions. Industrial crops and products, 175: 114252. Industrial crops and products (Q1, IF: 5.645).
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12. **A. S. Gheidan, Mazlan Abdul Wahid, Fudhail Abdul Munir, AC. Opia.** (2021). Feasibility Study of Bio-fuel as a Sustainable Product of Biomass: An Overview of Its Fundamentals, Application and Environmental Impact, Journal of Advanced Research in Fluid Mechanics and Thermal Sciences, Vol 8 (2) (Index Scopus).