



Faculty of Engineering

**Experimental Investigation on Electrical and Physicochemical Properties
of POME-Based TiO₂-ZnO HNF Under Transesterification Process and
Thermal Ageing**

Ernieza Musa

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Experimental Investigation on Electrical and Physicochemical Properties of
POME-Based TiO₂-ZnO HNF Under Transesterification Process and
Thermal Ageing

Ernieza Musa

A thesis submitted

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DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgements have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Ernieza Musa
.....

Signature

Name: Ernieza Musa

Matric No.: 22020204

Faculty of Engineering

Universiti Malaysia Sarawak

Date : 28 April 2025

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ABSTRACT

Mineral-based oil is a staple in the transformer industry due to its outstanding dielectric properties as an insulation system and its ability to allow electrical power network operation. The transition towards a more sustainable insulating oil in transformers instead of traditional mineral-based oil has been the subject of study for many years. Alternatives to a biodegradable resource, particularly palm oil, have been explored due to its abundance and properties that can mimic those of mineral-based oil. Recent developments have shown improvements in electrical insulation by combining palm oil's benefits and nanoparticles, known as monotype nanofluid (MNF). While ongoing studies on MNFs have yielded major findings, the investigation of hybrid nanofluid (HNF) has not yet become common. This research focuses on the production of POME-based HNFs, which involves three-phase experimental procedures (POME preparation using transesterification, HNF preparation using a two-step method involving TiO₂ and ZnO NPs with concentrations of 0.05 g/L, 0.10 g/L, 0.15 g/L, and 0.20 g/L, and accelerated thermal ageing at 135 °C at 0-hr, 50-hrs, 500-hrs, and 1000-hrs). Compared to other concentrations, 0.05 g/L of TiO₂-ZnO HNF showed the steadiest increase for AC BDV from 26.8 to 61.4 kV as the ageing time increased, as well as an increment from 36.4 to 290.8 %. The densities and viscosities of the POME-based TiO₂-ZnO HNFs fall in the acceptable range of 0.86 to 0.89 g/cm³ and 6.86 to 8.00 cSt at 40 °C, as per ASTM D7777 and ASTM D445, respectively. Based on FTIR spectroscopy, the molecular structure and chemical bonding of the pure POME and POME-based TiO₂-ZnO HNFs and UV-Vis spectroscopy on the variation concentrations on the stability of TiO₂-ZnO HNFs did not significant change. These results provide insight into the performance of TiO₂-ZnO HNFs as insulation in an actual transformer.

Keywords: Transformers, biodegradable, transesterification, palm oil, palm oil methyl ester, TiO₂ NP, ZnO NP, hybrid nanofluid, ageing

***Penyiasatan Eksperimen tentang Sifat Elektrik dan Fisikokimia TiO₂-ZnO HNF
Berasaskan POME Di Bawah Proses Transesterifikasi dan Penuaan Termal***

ABSTRAK

Minyak mineral merupakan bahan penting dalam industri transformer kerana ia mempunyai sifat penebat elektrik yang baik dan mampu memastikan sistem kuasa elektrik berjalan lancar. Peralihan kepada penggunaan minyak penebat yang mampan dalam transformer telah menjadi subjek kajian selama bertahun-tahun. Alternatif kepada sumber terbiodegradasi, seperti minyak kelapa sawit, telah diterokai kerana sumber ini banyak dan dikatakan boleh meniru sifat dielektrik pada minyak mineral. Perkembangan baru-baru ini dalam pencarian alternatif penebat elektrik telah menunjukkan peningkatan untuk gabungan jenis minyak kelapa sawit dan nanopartikel, yang lebih dikenali sebagai cecair nano jenis mono. Walaupun kajian yang berlangsung kepada cecair nano ini telah menunjukkan hasil yang baik, namun penyiasatan terhadap cecair nano jenis hibrid belum luas. Maka, penyelidikan ini memberi tumpuan kepada penghasilan HNF berasaskan POME yang melibatkan prosedur penyediaan tiga fasa (POME melalui transesterifikasi, penyediaan cecair nano jenis hybrid yang menggunakan kaedah dua langkah, dengan menggabungkan nanopartikel TiO₂ dan ZnO dengan kepekatan 0.05 g/L, 0.10 g/L, 0.15 g/L, dan 0.20 g/L, dan penuaan termal pada suhu 135 °C untuk tempoh 0 jam, 50 jam, 500 jam, and 1000 jam. Apabila dibandingkan dengan kepekatan lain, 0.05 g/L TiO₂-ZnO HNF menunjukkan peningkatan yang lebih stabil untuk AC BDV daripada 26.8 kepada 61.4 kV apabila masa penuaan, meningkat, serta peningkatan daripada 36.4 kepada 290.8%. Ketumpatan dan viskositi TiO₂-ZnO HNFs berasaskan POME jatuh dalam julat yang boleh diterima dari 0.86 hingga 0.89 g/cm³ dan 6.86 hingga 8.00 cSt pada 40 °C, mengikut nilai standard ASTM D7777 dan ASTM D445, masing-masing. Berdasarkan analisis spektrografi FTIR,

struktur molekul dan ikatan kimia TiO₂-ZnO HNF berbanding pure POME manakala UV-Vis pula menentukan variasi kepekatan pada kestabilan TiO₂-ZnO HNF tidak berubah secara signifikan. Temuan ini memberikan pandangan tentang prestasi TiO₂-ZnO HNFs sebagai penebat dalam transformer sebenar.

Kata kunci: *Transformer, terbiodegradasi, transesterifikasi, minyak sawit, minyak sawit ester metil, TiO₂ nanopartikel, ZnO nanopartikel, cecair nano hibrid, penuaan termal*

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LIST OF ABBREVIATIONS

2 nd	Second
a.u	Absorbance unit
Al ₂ O ₃	Aluminium Oxide
ASTM	American Society for Testing and Materials
BDV	Breakdown voltage
°C	Degree Celsius
CH ₃ OH	Methanol
CNC	Cellulose nanocrystals
CNT	Carbon Nanotubes
CO	Coconut Oil
CO ₂	Carbon Dioxide
CoO	Corn Oil
cSt	centistokes
CTAB	Cetyl Trimethyl Ammonium Bromide
DI	Deionised
EG	Ethylene Glycol
FAME	Fatty Acid Methyl Ester
FESEM-EDS	Field Emission Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy
FTIR	Fourier Transform Infrared
g	grammes
G	Graphene

GO	Graphene Oxide
H ₂ O	Water
h-BN	hexagonal Boron Nitride
HNF	Hybrid Nanofluid
hr	hour
IEC	International Electrotechnical Commission
KOH	Potassium Hydroxide
L	litre
MEPKO	Methyl Ester Palm Kernel Oil
mL	millimetre
MNF	Monotype Nanofluid
MO	Mineral Oil
MUFA	Monosaturated
MWCNT	Multi-walled carbon nanotubes
NaOH	Sodium Hydroxide
NF	Nanofluid
NP	Nanoparticle
NPME	Palm oil methyl esters-based nanofluid
OA	Oleic Acid
PKME	Palm Kernel Methyl Ester
PKO	Palm Kernel Oil
PO	Palm Oil
POI	Palm Olein
POME	Palm Oil Methyl Ester
PS	Palm Stearin

PUFA	Polysaturated
RBDPO	Refined, Bleached, and Deodorised Palm Oil
RBO	Rice Bran Oil
RO	Rapeseed Oil
RPO	Refined Palm Oil
SDS	Sodium Dodecyl Sulphate
SE	Synthetic ester
SiC	Silicon Carbide
SoO	Soybean Oil
SSEO	Semi synthetic ester oil
SuO	Sunflower Oil
TiO ₂	Titanium dioxide
TMP	Trimethylolpropane
UV-Vis	Ultraviolet-visible
VI	Viscosity Index
W	Water
WCO	Waste Cooking Oil
WCOME	Waste Cooking Oil Methyl Ester
ZnO	Zinc oxide

CHAPTER 1

INTRODUCTION

1.1 Research Background

The electrical power industry began more than a century ago during the 2nd Industrial Revolution and has had a major influence on the evolution of the advancement of technologies involving power generation and green energy integration. This electrical power system, often known as the power grid, is in charge of generating, transmitting, and distributing electricity. The emergence of more reliable and affordable electricity has revolutionised nearly every aspect of human daily life. At home, for instance, it powers lighting and domestic appliances like laundry machines and refrigerators. This electricity is generated using a power plant that runs on natural gas, coal, or water. Another crucial element in the electrical power system is the transformer, which primarily regulates the voltage of electrical energy, either increasing or decreasing it. Reddy (2020) explained that the substation transformer lowers the electrical energy's voltage before it passes through distribution lines and into residential homes. Each residential area may have a transformer mounted on a pole to reduce voltage further. Consequently, it is crucial to tackle concerns about the efficiency and safety of transformers, considering their significant role in electrical power systems.

In response to this matter, researchers and engineers have come together to find remedies that would result in a safer-to-operate transformer and, by extension, lead to more reliable and easier maintenance of the power system grid. Reddy (2020) discovered that a transformer can be considered energy-efficient and reliable if its rating exceeds 98%. When building and operating a transformer, it is necessary to consider certain crucial aspects,

especially its design, the core materials, and the windings. The ongoing discoveries and increased awareness of the role of each transformer component, the insulation is the component regarded as the most significant of the three and is needed in medium and high voltage systems the most (Nainggolan et al., 2017; Saha & Purkait, 2017). On top of that, the longevity of a power transformer is affected by the insulation material used in the device (Razzaq et al., 2019). The research and development of oil insulation systems for power transformers has been going on for more than two centuries, beginning in 1890 with the first oil-cooled transformer (Shen et al., 2021). For this reason, the insulation between the active components of transformer must be adequate and sufficient (Saha & Purkait, 2017).

According to studies conducted by Metwally (2011) and Banovic & Sanchez (2014), transformers can be insulated with either one of three insulating materials: oil-immersed, gas-insulated (commonly SF₆), or dry-type materials. However, only dry-type and oil-immersed-type transformers are in use now, as claimed by Hussain et al. (2022). Paper-type and oil-type insulation, on the other hand, are the two most common types used in high voltage applications, in the opinion of Razzaq et al. (2019), who conducted a review of transformer insulating materials. Oil-type insulation continues to be a popular insulating material and has become the subject of research worldwide due to its excellent thermal and insulating dielectric properties (Amin et al., 2019). In addition, oil-type insulation can also be referred to as insulating oil, which is the same term as liquid dielectric, as well as transformer oil (Muhamad & Razali, 2016). Insulating oil, as its name implies, functions as both an insulator and a coolant for the power transformer (Amin et al., 2019; Hussain et al., 2022; Tlhabologo et al., 2022). Figure 1.1 indicates the sequence of events that led to the invention of transformer oil and the commencement of its usage as insulation material (Shen et al., 2021). The year 1890 marked the construction of the very first commercialized oil-

immersed transformer (Rao et al., 2019; cited in Shen et al., 2021). Since the invention of oil-immersed transformers, transformers have relied on mineral-based oils of paraffin followed by naphthenic type of mineral-based oils as both insulation and cooling agents; the other two notable insulating oils are silicon oil and natural esters-based oil (Asano & Page, 2014; Shen et al., 2021; Xu et al., 2014).

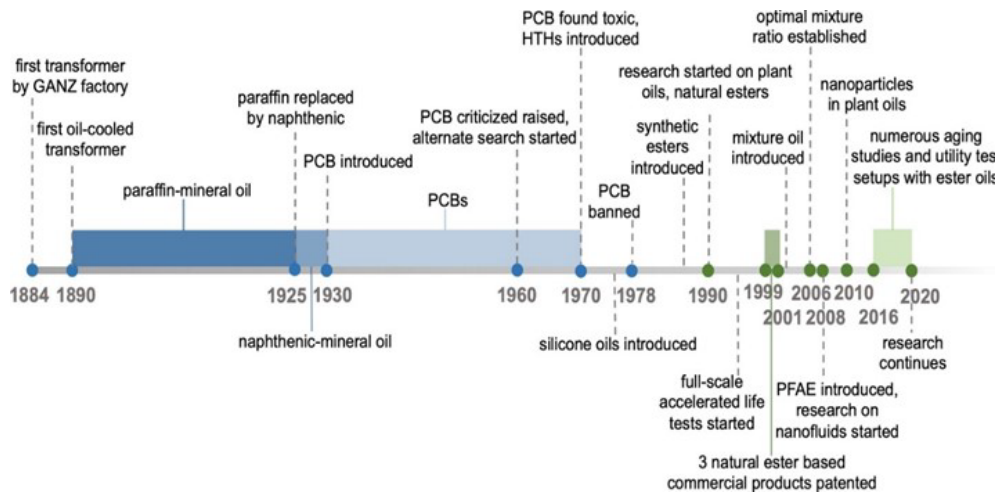


Figure 1.1: History of transformer insulating oil development (Shen et al., 2021)

Mineral-based oil has gained recognition for its cost-effectiveness and excellent electrical insulating characteristics, such as its low thermal conductivity (Beheshti et al., 2014; Nagendramma & Kaul, 2012). However, the utilisation of mineral oil as a liquid dielectric in power transformers is impeded by several drawbacks, largely stemming from its nonbiodegradability (Azli et al., 2019). The usage and disposal of this particular material have detrimental effects on the ecosystem, along with its non-renewable characteristics, indicating that it cannot be regenerated over a short period. Hence, in recent decades, researchers have begun to concentrate on exploring alternative insulating oils as potential solutions to address the limitations of traditional mineral oil, while working towards the utilisation of biodegradable resources and ensuring the long-term sustainability of insulating oil. The investigation of natural esters, especially palm oil, by researchers in this field has

led to positive outcomes, thereby contributing to current study efforts on the advancement of nanofluids based natural esters focusing on substantiating the viability and sustainability of employing biodegradable and renewable resources as promising insulating oil. One notable instance of a prevalent natural ester derived from palm oil, which can be categorised as a variant of biodiesel, is palm oil methyl ester (POME). It has been shown that POME exhibits traits that closely resemble those of petroleum diesel fuel (Pioch & Vaitilingom, 2005). Azli et al. (2019) also claimed that palm-based insulating oil is an ideal option as the basis fluid in research due to its high breakdown voltage and low dissipation factor.

The implementation of nanotechnology into palm-based natural ester has yielded promising results, as indicated by the study conducted in this field. The studies of POME-based NFs can be ascribed to their advantageous dielectric strength, lower thermal conductivity, and greater stability. Though there is an extensive number of investigations on NFs, there is a noticeable dearth of studies investigating the application of hybrid nanofluids (HNFs) as insulating oil in power transformers, a topic of remarkable significance and essentiality. HNFs can be defined as a novel class of nanofluids wherein several kinds of nanoparticles are suspended in a base fluid, resulting in the formation of composite materials (Sarkar et al., 2015). Senthilkumar et al. (2014) have reported that HNFs exhibit superior and stable dielectric properties compared to MNFs. The number of published papers on MNF and HNF from the year 2005 onwards is displayed in Figure 1.2 (Yasmin et al., 2023). The investigation of HNFs was solely carried out in the year 2015. The primary goal of this research is to investigate POME-based HNFs to contribute to existing knowledge, as well as the effects of thermal ageing on performance in real transformer applications. Besides, it is important to understand the potential of HNFs to perform as well as MNFs by focusing on the mixing of two distinct nanoparticle compositions, in this case, TiO₂ and ZnO NPs.

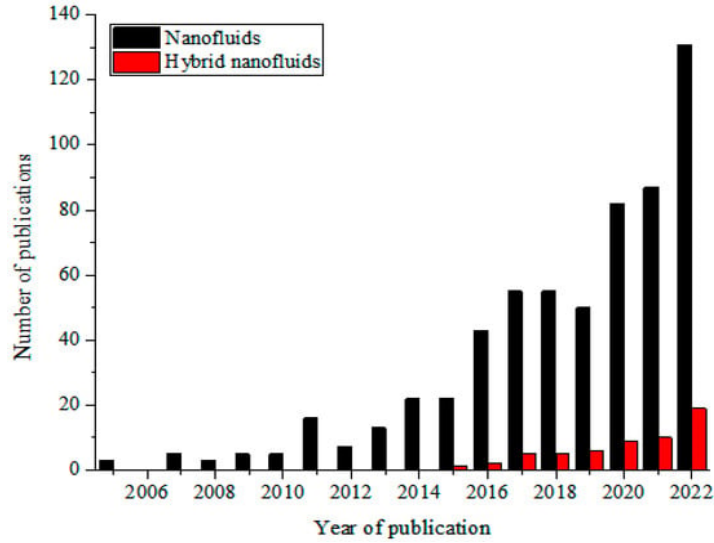


Figure 1.2: Paper publications on MNF and HNF (Yasmin et al., 2023)

1.2 Problem Statement

The continual use of mineral-based oil as an insulating liquid dielectric in transformers has prompted concerns not only about its rapid depletion and consequent future scarcity but also about its detrimental ecological impacts (Tiwari et al., 2024). Mineral-based oil is a prime example of a non-renewable and nonbiodegradable resource (Jacob, Preetha, & Krishnan, 2020; Lyutikova et al., 2022). It originated from a petroleum base, a natural resource that exists due to natural occurrences but cannot be produced in a shorter period, taking hundreds of thousands or even millions of years (Aluyor & Ori-jesu, 2009). Furthermore, nonbiodegradable material poses environmental threats because it cannot break down or degrade naturally. In the case of discarding used oil, it may leak from the transformer to the ground or be deliberately released into the river, thereby causing a threat of pollution (Fazzo et al., 2017; Karaman et al., 2023; United States Environmental Protection Agency & Office of Emergency and Remedial Response, 1993). Moreover, mineral-based oil characteristics are combustible and easily raise substation fire risk, producing excessive carbon dioxide and other greenhouse gases that indirectly contribute to

climate change (Fazzo et al., 2017; Treanor & Whitman, 1952). Therefore, this necessitates a shift towards more sustainable and renewable resourced-based alternatives from conventional mineral-based oil.

To address these issues, there has been growing interest in exploring alternative insulating materials such as natural esters-based oil over the last decades (Ab Ghani et al., 2018). The natural esters can be derived from vegetable oils which are a renewable resource (Lligadas et al., 2013). Palm oil, on the other hand, remains the primary driver of Malaysia's economic growth in early 2024 (Razak, 2024). Therefore, palm-based oil will be used in this research since its resource is abundant and readily accessible in Malaysia. Palm-based oil is a potential alternate replacement due to its biodegradability characteristic from vegetable oil, which normally has a higher flash point and lesser harmful effects on environmental impacts and safety problems than conventional mineral-based insulating oil (Abdelmalik, 2014; Srivastava & Sahai, 2013; Suhaimi et al., 2022). In recent years, the addition of nanoparticles into palm-based oil, the product called NF, to improve its poor dielectric properties has become an increasingly common practice among researchers (Hussain et al., 2022). These additions have been studied by Hasin et al. (2023) using graphene oxide (GO), Koutras et al. (2020) using silicon carbide (SiC), and Vasheghani et al. (2011) using aluminium oxide (Al₂O₃), which demonstrate an enhancement in heat transfer, dielectric strength, and thermal conductivity, respectively.

Large-scale investigations on MNFs have been extensively conducted up to this point, whereas HNF, especially utilising palm-based oil is less studied in transformer insulating oil application areas. In their review, Suneetha et al. (2022) reported that several investigations have not addressed specifically the vegetable oil used in hybrid insulation

applications. Furthermore, the application of HNF as insulation is common in the study of engine lubrication in the manufacturing sector. For example, Fadzli et al. (2020) explored Al₂O₃ and Silicon Oxide (SiO) added into water; Lim et al. (2022) investigated the hybrid nano mixture of graphene (G) and Al₂O₃ dispersed in an EG base fluid; as well as Ribeiro et al. (2023) assessed the addition between hexagonal boron nitride (h-BN) and carbon nanotubes (CNT) in a synthetic ester, FR3 oil. The exploration notion of HNF performance beyond application in manufacturing or similar to current MNF as a liquid dielectric may eventually lead to the groundbreaking discovery of this possibility for a new investigation. Prolonged heat exposure of HNFs is particularly crucial for the research investigation since this procedure allows them to reveal their properties accurately in conditions similar to those of a real transformer.

This highlights an experimental preparation that entails various concentrations between the two NPs used in this research of POME-based HNFs will lead to any improvement as such MNFs in terms of their electrical and physicochemical properties after undergoing accelerated thermal ageing. Thus, it also aims to ascertain whether the addition of several nanoparticles to POME can enhance their properties as the oil ages thermally.

1.3 Research Questions

The research questions of this research are:

- i. Does preparing several POME-Based HNF concentrations result in any form of obvious enhancement?
- ii. Does introducing hybrid nanoparticles in POME-based HNF enhance the transformer's dielectric properties as it ages thermally?

- iii. What is the optimal performance of HNF for its electrical and physicochemical properties as transformer oil application?

1.4 Objectives

The purposes of this research are:

- i. To investigate the electrical and physicochemical properties of POME-based TiO₂-ZnO HNFs at different concentrations and ageing times.
- ii. To formulate several concentrations of POME-based TiO₂-ZnO HNF for accelerated thermal ageing.
- iii. To analyse the performance of POME-based TiO₂-ZnO HNF at the optimal concentration following accelerated thermal ageing.

1.5 Hypotheses

The hypotheses of this research are:

- i. Palm oil can serve as a sustainable insulating oil, as the transesterification process is expected able to mitigate its inherent drawbacks, such as high viscosity and density making it more suitable for transformer applications.
- ii. The modification of palm oil, specifically POME, through the addition of NPs will enhance its dielectric properties, improving its insulating performance compared to unmodified palm-based oil.
- iii. HNF will undergo accelerated thermal ageing to evaluate their long-term stability and performance under actual transformer operating conditions.

1.6 Research Scope

The research focuses on the experimental preparation and investigation of POME-based HNFs as a potential alternate insulating oil in transformers. The first scope of this research is to prepare POME-based HNFs under transesterification and accelerated thermal ageing. A biodegradable-based oil, in this case, palm oil is transesterified into POME, with a combination of titanium dioxide (TiO₂) and zinc oxide (ZnO) NPs with a ratio of 80:20 dispersed in POME using a two-step NF method of preparation with concentrations of 0.05 g/L, 0.10 g/L, 0.15 g/L, and 0.20 g/L. The thermal exposure of POME-Based TiO₂-ZnO HNFs at 135 °C is conducted with non-accelerated thermal ageing of 0-hr and accelerated thermal ageing of 50-hrs, 500-hrs, and 1000-hrs. Then, the investigation of POME-Based TiO₂-ZnO HNFs encompasses the analysis of electrical and physicochemical properties such as AC BDV, kinematic viscosity, density, FTIR and UV-Vis spectroscopy that undergo accelerated thermal ageing. The results will be compared to the non-accelerated thermal ageing. Last but not least, the assessment of the outcomes to determine the optimum concentrations of POME-Based TiO₂-ZnO HNFs to attain mineral-based oil properties, along with the changes in the HNF's properties as they age thermally.

1.7 Significance of Research

The research investigation on transformer insulating oil has shown an incredible discovery for the past years in the transition from conventional insulating oil, such as mineral-based oil towards MNF. These MNFs especially are often developed using a biodegradable type of base fluid such as palm-based oil due to their abundance of resources especially in Malaysia. Therefore, this study pertains to the development of a biodegradable and sustainable insulating by using a modified palm-based oil which is a palm oil methyl ester that is produced through the transesterification process on the RBDPO. Not only that,

the formulation of the POME-based TiO₂-ZnO HNF also leads and adds to a new finding on the hybrid development of insulating oil in transformer insulation applications since it is still not commonly investigated. The benefits of extending the investigation on the TiO₂-ZnO HNF on prolonged heat exposure will allow the study to evaluate their dielectric properties after some time by imitating an actual transformer condition. Only then their potential as alternate insulating oil in power transformer applications can be determined.

1.8 Thesis Outlines

This thesis has been divided into five separate chapters. The following section offers a summary of the contents of each chapter.

1.8.1 Chapter 1: Introduction

Chapter 1 contains an introduction to the research background, problem statements, objectives, scopes, and thesis outlines for this research, which include the reasons for biodegradable oil exploration as a future alternate insulating oil in transformer applications over the decades.

1.8.2 Chapter 2: Literature Review

Chapter 2 reviews the transition from conventional mineral-based oil to palm-based NF as an alternative to the current insulating oil used in transformers, as well as the most recent trends discovered by researchers on this topic in real transformer conditions. A research gap has been addressed by comparing the type of NF, NPs, and base fluid used, methodology, outcomes, and limitations from the reviewed literature.

1.8.3 Chapter 3: Methodology

Chapter 3 discusses the methodology employed in this research to achieve the objectives that have been established. Designing a research plan before beginning the investigation simplified the experimental procedure and analysis methods. The experiment will be divided into three phases; POME preparation using transesterification, POME-based TiO₂-ZnO HNFs using a two-step method and accelerated thermal ageing. This chapter also comprised a detailed procedure for testing its electrical and physicochemical properties in the laboratory by following the established standard test for insulating oil.

1.8.4 Chapter 4: Results and Discussion

Chapter 4 explains the results and provides a discussion based on findings from the experimental analytical testing. The initial outcomes begin with the characterisation of TiO₂ and ZnO NPs by FESEM-EDS and POME percentage yield. Then, the main findings of the research entail the electrical properties such as AC BDV, and physicochemical properties such as kinematic viscosity, density, visual appearance, FTIR, and UV-Vis spectroscopy that will be compared in terms of POME-based TiO₂-ZnO HNF concentrations and ageing times. These results are significant for demonstrating that the addition of multiple NPs performs as well as the previously investigated MNF.

1.8.5 Chapter 5: Conclusion and Recommendations

Chapter 5 concludes the research and highlights the findings from the experimental investigation of POME-based TiO₂-ZnO HNFs that align with the objectives. Further, this thesis ends with recommendations for further HNF investigations on nanoparticle types and testing, as well as strategies for overcoming limitations.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

This chapter provides an in-depth review of the historical events that led to the growth of present-day investigations and developments in transformer insulating oil. Mineral-based oil has been widely commercialised as an insulating oil in transformers due to its unexceptional characteristics. Nevertheless, it bears two major drawbacks, such as its non-renewable nature and its non-biodegradability nature. Therefore, numerous researchers searched for different approaches to overcome the issues along with the transition to a greener technology while preserving and enhancing the characteristics of mineral-based oil. An example of this is the adoption of biodegradable oils with a specific focus on palm-based oil, which has demonstrated positive results through appropriate modifications. Additionally, multiple research studies have also found that the addition of nanoparticles to the base fluid of the proposed oil predominantly enhances its dielectric properties compared to the absence of nanoparticles. Not only that, but their prolonged heat exposure studies also play a crucial role in determining their stability in the long term. The following section will address the gap among studies conducted on HNFs, despite a significant breakthrough in MNFs as an up-and-coming alternative for insulating oils.

2.2 Insulating Oil

Insulating material in transformer is one crucial element sometimes disregarded when mentioning electrical substations. The claim that insulation plays a vital part in all electrical system devices because it keeps them safe to use has been investigated by several studies (Aljamali et al., 2021; S. Li et al., 2016). More industries have been using

insulating materials that correspond to the three states of insulating materials, such as solids, liquids, and gases, not just for transformer devices but also for spinning equipment and circuit breakers (Vishal et al., 2011). The insulation in electrical equipment can eventually reduce energy consumption with the proper development of new biodegradable and sustainable insulating materials (Shahee et al., 2024). Growing research on insulating oil occurs because of the continuous discoveries and knowledge of its significance in improving the reliability and efficiency of transformer performance. Muhamad & Razali (2016) state that insulating oil brings the same meaning as transformer oil and liquid dielectric. It acts as insulation to prevent electrical discharge and cools the transformer windings (Muhamad & Razali, 2016).

Many factors must be considered before injecting insulating oil into a transformer, regardless of how simple it might appear to choose the most suitable one. One of the most common insulating materials used in oil-immersed transformers is cellulose, a by-product of wood or fibre crops, because of its low dielectric constant and higher resistivity (M. et al., 2011). However, stabilisers containing amino groups, when added to cellulose materials that were initially non-toxic and non-corrosive, the transformer becomes poisonous and erodes (Jusner et al., 2021). Therefore, the insulating oil needs to be in safer operation in the transformer by fulfilling the established standard requirements such as ASTM and IEC for insulating oil. The influence on the transformer performance can be because of the insulating oil's electrical, thermal, and chemical, which include dielectric strength characteristics, power factor, thermal conductivity, heat capacity for cooling, oxidation stability, and moisture content (Z. Huang et al., 2019; Lu & Liu, 2016).

Lu & Liu (2016) discovered that silicone oil, mineral oil, and synthetic ester oil are the three types of insulating oils for appropriate usage in transformer insulation based on

their comparative analysis. Mineral-based oil has been the most widely used insulating oil for over a century due to its low cost and superior properties (Hao et al., 2017; Liao et al., 2011; Liu & Tornkvist, 2009). It is a liquid fluid derived from petroleum and has been significant in manufacturing sectors, such as pharmaceutical, cosmetic, and industrial (Hess et al., 2011; Sharma et al., 2023). However, there is still no clear definition for mineral-based oil. Mineral oil usage as the primary insulating oil in transformers has sparked many concerns due to its negative environmental impacts and resource scarcity (Adelman, 1990). In contrast, synthetic ester oils, unlike mineral oil, have not become more popular despite offering better fire protection and being better for the environment (Rozga et al., 2020). The reason is due to the cost of synthetic ester and its incompatibility with some transformer components, particularly those found in high-voltage equipment (Rozga et al., 2020).

Large-scale and high-voltage power transformers targeted for the utilisation of sustainable and renewable energy in future will unavoidably become necessary as the world struggles with the growing problems related to its energy supplies (S. Huang, 2022). To satisfy the growing demand for environmentally friendly technology, an alternative strategy that either fixes or replaces the root causes of the issues mentioned needs to be investigated before one insulating oil can be developed. When it comes to replacing mineral-based oil, current insulating oil development trends focus on improving synthetic ester and investigating vegetable oil properties, with prospects for the future that are consistent with sustainable and renewable values. Vegetable oils, or known as natural ester, with their superior thermal qualities, sustainability, and biodegradability for environment have consequently overtaken all of these insulating oils (Aluyor et al., 2009; Malik et al., 2023; Sarma & Vinu, 2022). The selection of the vegetable oil that will be

utilised in this research as a possible insulating oil will require additional study.

2.3 Vegetable Oil

As stated previously, concerns about the continuous usage of mineral-based oil in transformer insulation systems have raised the idea of innovation to comprehend the issues. One potential approach that meets the criteria of sustainability and renewable is through the utilisation of biodegradable-based material. According to Rahman & Bhoi (2021), they defined a biodegradable material as a material that will eventually undergo decomposition into simpler constituents such as carbon dioxide (CO₂), water (H₂O), and biomass when prolonged exposure to the presence of microbes. Although biodegradable nature is a noteworthy option while assessing its suitability, it should not be the sole criterion in choosing the base of innovation of a new insulating oil. This is discussed by Innocenti (2003) as his belief in the compostability concept in sustainable packaging material does not involve biodegradability only, but also disintegrability, illustrated in Figure 2.1. In addition, (Li et al., 2023) suggested the biodegradable materials should be utilised together with the principles of renewable resources, especially for insulating oil.

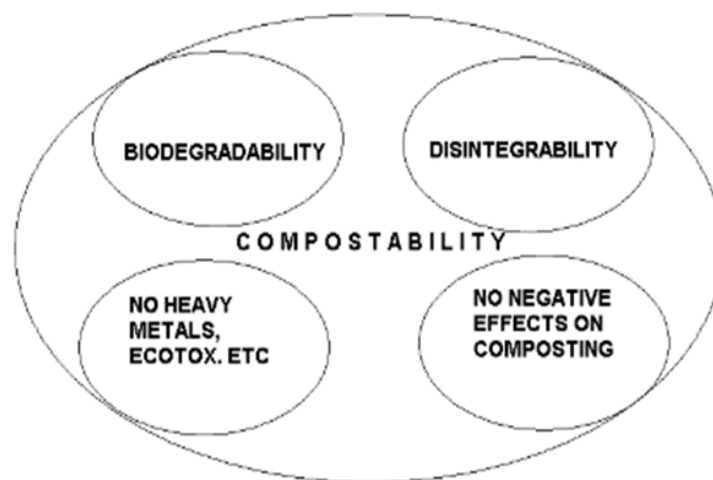


Figure 2.1: Compostability packaging characteristics (Innocenti, 2003)

Long-term efforts have been performed to innovate a new biodegradable insulating oil often founded from natural or synthetic esters commonly referred to as vegetable oils through chemical modifications (Nagendramma & Kaul, 2012). In between these two, vegetable oil has yielded more encouraging results based on numerous studies regarding the efficacy and longevity of biodegradable oil criteria. Because vegetable oil is derived from plants, and are believed to have the ability to break down spontaneously in the environment, lowering the risk of pollution (Aluyor et al., 2009). These features help in exhibiting greater prospects over petroleum-based materials in terms mitigating carbon emissions and addressing the issue of global warming. It is also necessary to note that the elastomeric seal in power transformers have the possibility to experience expansion and degradation when in contact with insulating oil (Avila et al., 2022).

Nevertheless, some studies have demonstrated through several investigations that transformer components made of copper, steel, and paper type of insulating material are compatible with biodegradable oil derived from vegetable oils (Martins, 2010; Oparanti et al., 2024; Siddique et al., 2024) In India, vegetable type of oilseed crops, such as rapeseed, soybean, and sunflower have been frequently used due to their affordability and accessibility (Morya et al., 2022). Another study has documented that by certain mixing between vegetable oils have lower flash and fire points than the others. For instance, the soybean almond oil blend had flash and fire points of 286 °C and 330 °C, respectively, when compared to other blended vegetable oils, which (Talkit et al., 2015). Insulating oil that has a low flash and fire point may cause a greater chance ignition to occur easier in a transformer at higher temperature. These values meet the standard requirement of IEC 62770 for an natural ester-based insulating oil's flash point and fire point 275 °C and 300 °C, respectively (Mehta et al., 2016). Therefore, the fact that some vegetable oils.

In reality, the majority of them show a rather good result. Because it contains unsaturated fatty acids, vegetable oil is a type of natural ester with a greater rate of oxidation. Conversely, mineral oil has a higher concentration of saturated fatty acids and a lower quantity of unsaturated fatty acids. Three of the four varieties of indigenous vegetable oils studied have greater levels of unsaturated fatty acids; more than 65% of which are composed of the two compounds called polyunsaturated (PUFA) and monounsaturated (MUFA) fatty acids (Muhammad et al., 2023). Thus, in an effort to address present problems with vegetable oils, researchers have been proposing methods for modification, as well as development of NF throughout the past few decades.

2.4 Palm-based Oil

Insulating oil is a part of insulating materials palm-based oil is acknowledged to be among the most important vegetable oil on a global scale and serves as the primary contributor to Malaysia's agricultural economic growth. In the year 2019, Malaysia had recorded around 33.7% of the global palm oil production, positioning the nation as the second largest producer and exporter after Indonesia, as depicted in Figure 2.2 (Zulqarnain et al., 2020). Besides, the government initiatives to promote agricultural development, the provision of land for production, and global demand for vegetable oils were some of the factors that pushed the development of the palm oil turnover. Over the years, palm oil has been produced for various purposes, including its use as a cooking oil, biofuel source, and even cosmetic products. Figure 2.3 shows an illustration created by Sulaiman et al. (2022), that highlights on the utilisation of Refined Palm Oil (RPO), which can be broken down into two different applications, namely Palm Olein (POI) and Palm Stearin (PS).

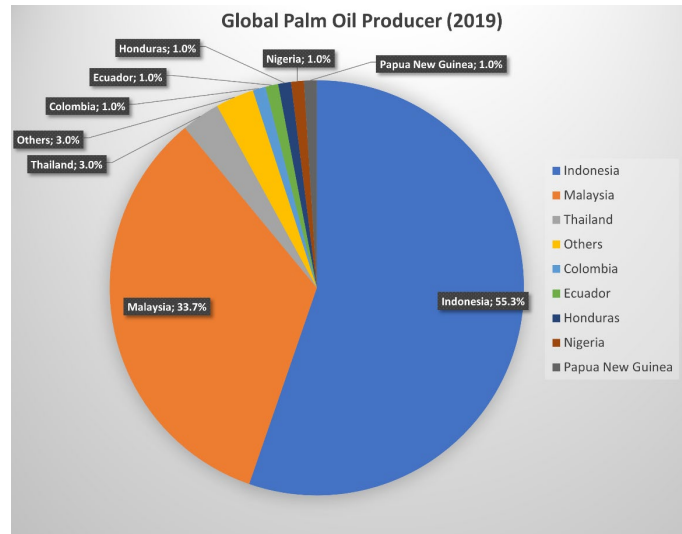


Figure 2.2: Global palm oil producer in 2019 (Zulqarnain et al., 2020)

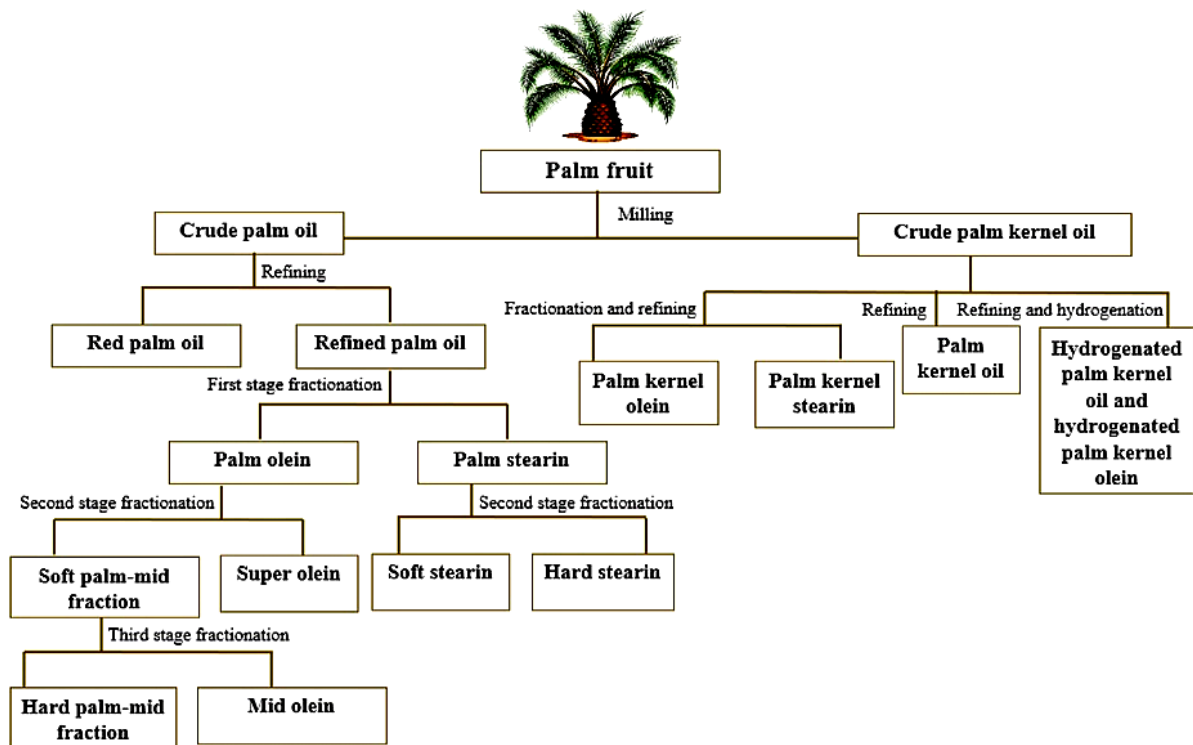


Figure 2.3: Fractionated palm fruit (Sulaiman et al., 2022)

These byproducts have undergone a thorough investigation in the healthcare industries. In accordance with Ranasinghe et al. (2022), their investigation area indicates tocotrienols and tocopherols, two types of vitamin E, exclusively derived from plants, such as palm-based oil, possess the ability to potentially aid the development pharmaceutical

interventions for cancer and diabetes prevention. Also, red POI contains higher carotene level compared to in PS, a form of vitamin A that serves as an antioxidant agent (Nabu et al., 2021). Presently, palm oil maintains its position as the largest produced vegetable oils worldwide, with Malaysia and Indonesia generating more than one-third of the world's vegetable oil over the past ten years ("Oilseeds and Oilseed Products," 2018). Malaysia, as among the largest producers of palm oil has a competitive advantage when it comes of environmentally advantageous palm oil supplies (Jamail & Muhamad, 2016). The ample and easily accessible resources contribute to the maximisation of the utilisation of palm-based oil, particularly in the development of new innovative technologies.

This development led to the study of possible palm-based oil applications for power transformer insulating systems. Multiple studies have demonstrated that palm oil offers favourable characteristics as an alternative liquid dielectric, providing proper procedures are adhered to and satisfies the established standards, such as those set by reliable organisations like IEEE and ASTM (Abdullahi et al., 2004; Roslan et al., 2021). Based on Saenkhumwong & Suksri (2017) research, palm ester-based oil exhibits superior properties, including its dielectric breakdown strength, flash and fire points, in comparison with mineral oil. This would thereby decrease the probability of transformer breakdowns in the event of incompatibility. Despite the fact that palm oil has biodegradable nature and favourable fire point and breakdown voltage, its high viscosity remains one of the main issues. Yet, it can be resolved by employing a procedure known as a transesterification process, which produces Palm Oil Methyl Ester (POME) as an end-product (Masra, Arief, Rigit, et al., 2023; Wajanasoonthorn & Suksri, 2021). This proves to be beneficial compared to the utilisation of raw cooking oil, known as Refined, Bleached, and Deodorized Palm Oil (RBDPO).

Aside from that, second-generation biofuels utilise non-edible feedstocks, such as algae, crop residue, and refuse vegetable oils, necessitating a more complex production method that promotes land conservation (Andrée et al., 2017; Malode et al., 2021). Algal biomass is the feedstock for third-generation biofuels, despite the fact that microalgal fatty acid composition making it not recommended for industrial use (Behera et al., 2015). For the last generation, also known as the advanced generation which is still in the development phase and favourable outcomes are anticipated because it is based on a genetically modified plant that will intake significantly more carbon dioxide (CO₂) than a common plant (Lee & Lavoie, 2013; Pandey & Kumar, 2017). Also, the production of biodiesel can be developed through a disparate of processes, including the blending of vegetables oils with petroleum-based fuel, transesterification, micro-emulsion, and pyrolysis (Abbaszaadeh et al., 2012; Ma & Hanna, 1999). It is possible to mitigate the fundamental issue involving vegetable oils, specifically its viscosity, through blending vegetable oils with solvent and undergoing microemulsions technique.

However, issues with engine performance may still arise. Thus, transesterification process is frequently used to further overcome the high viscosity issue. This method able to reduce viscosity by modifying the molar ratio of alcohol to glycerides between 6:1 to 30:1, as well as the reaction temperature and pressure, reaction intervals, and the total amount of free fatty acids (Demirbaş, 2003). Other factors such as the mode of reaction condition, the molar ratio of alcohol to oil, the types of alcohol, the type and quantity of catalysts, and the levels of reacting agents' purity all influence the transesterification results according to a study by Meher et al. (2006). Ishola et al. (2020) reported that the transesterification process utilising RBD palm olein mixing with methanol in the presence of KOH, producing a biodiesel yield of 62.5%. The selection of the molar ratio is led by research conducted by

Baroutian et al. (2009), who discovered that producing methyl or ethyl esters derived from RBDPO at molar ratio of 1:6 yielded the maximum biodiesel at 98.10%. The stoichiometric molar ratio of oil to alcohol of 1:3 is not recommended, as it make the process reversible, therefore it must be larger for the reaction to complete (J. Lee & Saka, 2010; Musa, 2016).

The base oil selected influences the performance of a biodiesel feedstock that complies with sustainable and biodegradable criteria, particularly its thermal and electrical characteristics as a potential transformer insulation use. Examples of modified palm-based oils investigated for possible use in transformer applications include Crude Palm Oil (CPO), POME, RBDPO, and Trimethylolpropane esters (TMPE). These oils have varied efficiency and oil stability characteristics. Compared to POME and TMPE, which undergo some modifications, CPO is an unprocessed oil extracted directly from the fruit of the palm tree (Alhaji et al., 2024). It has poorer oxidative stability due to its high molecular weight and free fatty acid content, which causes it to be more viscous and include more impurities (Alkabbashi et al., 2009; Emebu et al., 2022; Siti Nurul Akmal Yusof et al., 2021). Besides, RBDPO is a product that is made from CPO by chemically degumming, deodorizing, and bleaching it to eliminate impurities, phospholipids, and free fatty acids (Gharby, 2022). RBDPO is commonly manufactured and utilised as cooking oil in the food sector (Purnama et al., 2020). Thus, both have high viscosities (48.5 to 50 cSt, at 40 °C), which makes them undesirable and less suitable for direct industrial insulating oil applications (Abdullahi et al., 2004).

TMPE are the extended synthesis process of POME with TMP, whereas POME can be derived directly from CPO and RBDPO (Gheewala et al., 2022; Hamid et al., 2012; Nkem & Nwakaudu, 2019). Studies have shown that POME between 4.36 to 5.36 cSt offer better

dielectric qualities at extremely elevated temperatures and a higher enhancement than TMPE with viscosities between 49.7 and 57.47 cSt at temperature of 40 °C (Masra, Arief, Rigit, et al., 2023; Mohd Nor & Salimon, 2023; Oo et al., 2024; Yunus et al., 2003). Therefore, POME can lower the high viscosities of CPO and RBDPO compared to TMPE. The low viscosity of POME ensures enhanced heat transfer of the oils and enhances stability performance as transformer insulation. Selecting transformer insulating oil requires consideration of oxidation stability besides viscosity and dielectric qualities. Because CPO and RBDPO consist of high levels of triglycerides, which are often vulnerable to oxidation and accelerate degradation when heat is present, they break down faster than POME and TMPE (Niasar et al., 2015). POME undergoes a transesterification process in which the triglyceride of CPO or RBDPO reacts with methanol to remove the glycerol, which is the primary problem with contaminants in the palm-based oil (Miyuranga et al., 2022). TMP undergoes an additional esterification process after POME by adding TMP, which reduces oxidation and improves the oil's overall electrical stability performance (Sornvoratop et al., 2024).

POME is a better candidate for further study among these palm-based oils because it has been examined from an environmental and practical perspective as insulating oil in transformers. Despite TMPE's huge potential for insulating oil, its high saturation and high molecular branching may make them less compatible with transformer insulation materials, which could ultimately lead to material degradation (Raof et al., 2022; Samidin et al., 2021). TMPE is also more complex to produce and requires more purifying and chemical procedures that influence the environment and result in unwanted byproducts. On the other hand, POME's more straightforward molecular structure makes it possible to improve its fluidity, efficient heat dissipation, and easy integration into transformer systems without

requiring significant changes (Golam, 2024). Thus, regardless of the method, modifications of palm-based oil to natural esters must be biodegradable and sustainable. Table 2.1 tabulates the investigations on the palm-based oil modification to natural ester insulating oil in transformer, which will be referred in the methodology section.

Table 2.1: Palm-based Oil Modification as Natural Ester Insulating Oil

Author(s)	Modification Method	Descriptions	Outcomes of Research
Sitorus et al. (2024)	Addition of Additives	The addition of butylated hydroxytoluene to CPO and heated until temperature of 100 °C.	The addition of butylated hydroxytoluene to CPO is not suitable since the minimum requirement of BDV is 30kV/2.5mm, in this case is only 11.23kV
Hamid et al. (2022)	Filtering pre-processed	RBDPO is filtered using Thermo Fisher Nalgene membrane filter and degassed and dehydrated for 48 hrs in vacuum oven at 85 °C below 500 Pa.	Relative permittivity of modified PO is 2.176 closer to solid insulation (4.0), making the stress distribution among liquid and solid in transformers. AC BDV of PO is lowered than RBO but higher than MO.
Masra, Arief, Rigit, et al. (2023)	Transesterification	RBDPO is modified by adding methanol to it and KOH as catalyst at 60 °C for 60 minutes and leave overnight for separation. Then, the mixture will be washed using warm distilled water until pH 7 is achieved	The FAME will be used as the base fluid for NF preparation. The average POME yield production is 98.43%.
Egbuna et al. (2021)	Transesterification	PKO is dispersed with methanol and KOH at 60 °C for 120 minutes. Separation for 24 hrs and lower layer is drained with washing distilled	PKME with TMP: Yield percentage production is 49.82%. Viscosities recorded 42.35 cSt at 40 °C, and 10.65 cSt at 100 °C. Flash point of PKME is 210 °C.

Table 2.1: continued

		water. PKME with TMP as well as with epoxidation-esterification reaction.	
Deraman et al. (2020)	Transesterification	WCO is heated at 120 °C for 10 minutes and filtered the solid waste at 50 °C. Then, the methanol and NaOH is added to the filtered and dispersed with WCO at 55-60 °C then separated for 48 hrs. the washing with hot distilled water. Then the oil was heated at 120 °C for 5 mins. The solution was separated for 15 mins and repeated 6 times. The WCOME was heated for another 10 mins at above 120 °C.	The WCOME electrical as such BDV reached 33 kV and chemical properties as such water content have been reduced as much as 92% has improved and almost fulfilled the requirement insulating oil in transformer.

2.5 Trends on Palm-based Nanofluid

The approach of mixing base fluid, traditionally an insulating oil, with the tiny particles known as nanoparticles because they range in size from 1 to 100 nanometre has reignited enthusiasm. This is how the term "nanofluid" came to be. Several research have investigated that by adding only a few nanoparticles able to show a significantly improved heat transfer and even the thermal conductivity of a transformer performance (Begdouri & Fadar, 2023; Fal et al., 2018; Kong & Lee, 2020). A study demonstrates that when a nanoparticle, specifically CuO, is added to RBDPO, the AC breakdown voltage increases as contrasted with when nanoparticles are not present (Suhaimi et al., 2022). In addition

to improving the efficiency and sustainability of the transformer industry, the use of natural ester-based insulating oils and HNF as insulating materials can also help to reduce the environmental impact of transformer operation. By reducing the risk of transformer fires and improving the efficiency of transformer operation, these materials can contribute to reducing greenhouse gas emissions, which are a significant contributor to climate change.

On top of that, in relation to the palm oil's dielectric properties, it has been reported that palm-based oil may hold the ability to resemble mineral oil based on some studies that have been performed. Nevertheless, palm oil initially does not possess the same properties as mineral oil, unless certain further procedures have to be performed to obtain the end-product. Besides, the addition of 1.0% Titanium Dioxide (TiO₂) into palm oil engine lubricant shows a rise of 4.1% in its Viscosity Index (VI) with ethylene glycol as a surfactant that serves to mitigate the formation of aggression and sedimentation in the oil samples (Roselina et al., 2020). However, the elevated concentration of TiO₂ also leads to an increase, so it will affect the stability of the palm oil lubricant when operating inside the engine. Recent trends in insulating oil technology include the development of new synthetic ester oils with improved properties and the use of nanotechnology to enhance the performance of insulating oils. Future directions in insulating oil technology include the use of renewable and sustainable feedstocks for the production of vegetable oils and the development of new insulating oil testing methods to improve the reliability of power transformers. Exploring palm oil based insulating oil as a nanofluid has shown a potential discovery in offering sustainable and ecologically friendly alternatives. Since there has not been a significant amount of research on HNF based palm-based oil, future recommendations should emphasise reduce environmental impact, cost-effectiveness, and

enhance performance in coolant and insulator applications.

2.6 Ageing of Palm-based Oil and Nanofluid

The degradation of the transformer insulating oil is due to an inherent mechanism that occurs when the oil is exposed continuously to extreme temperatures and electrical stress throughout the operational life of the transformer (Chakraborty et al., 2021). Extended exposure can result in a gradual loss in the dielectric strength of an insulating oil, which may eventually lead to a deterioration of performance and efficiency and a total breakdown of the transformer (Pioch & Vaitilingom, 2005). This occurrence has been defined as insulating ageing by Elele et al. (2022). (Abdi et al. (2011) discovered that thermal ageing significantly promotes acidic product formation and water formation in the oil, which degrades its electrical and physicochemical properties. While numerous investigations have been performed to explore and dissect the palm-based NF dielectric properties as an alternate insulating oil, the landscape of publications on their effects as they age under actual transformer conditions has remained sparse on NF applications even in recent years, as listed in Table 2.2.

Table 2.2: Ageing of Palm-based Oil and NFs

Author(s)	NP(s)	BF(s)	Ageing Conditions	Outcomes of Research
Gutierrez et al. (2023)	N/a	SuO, RO, SoO, PO, SE, MO	Temperature: 150 °C Ageing time: 672 hrs NF conditions: N/a	PO has a comparable result to MO as they age.
Masra, Arief, Sahari, et al. (2023)	TiO ₂ , MWCNT	POME	Temperature: 130 °C Ageing time: 1000 hrs NF conditions: Sealed	0.05 g/L POME-based TiO ₂ NF exhibited highest AC BDV after 1000 hrs of ageing.
Mustam et al. (2023)	N/a	RBDPO, MO	Temperature: 140 °C Ageing time: 7 days NF conditions: Sealed	Impregnated paper-oil of RBDPO has an AC BDV comparable to

Table 2.2: continued

				impregnated paper-oil of MO.
Wajanasoonthon & Suksri (2022)	G	POME	Temperature: 100 °C Ageing time: 168, 336, and 504 hrs NF conditions: Sealed	BDV of NPME is higher than pure POME after 504 hrs of ageing.
Boyekong et al. (2021)	N/a	MEPKO	Temperatures: 110 °C Ageing time: 48, 72, and 96 hrs NF conditions: Sealed	Impregnated paper-oil of MEPKO has a higher BDV and improved stability which is comparable to impregnated MO.
Mohamad et al. (2018a)	N/a	RBDPO, CO	Temperatures: 90, 110, and 130 °C Ageing time: 7, 14, 21, and 28 days NF conditions: Open	Both AC BDV remain unchanged, whereas CO resistivity decreases with ageing.
Mohamad et al. (2018b)	N/a	RBDPO	Temperature: 170 °C Ageing time: 7, 14, 21, and 28 days NF conditions: Open	AC BDV remains unchanged, whereas viscosities and moisture content increase with ageing.
Mohamad et al. (2016)	N/a	RBDPO, CO	Temperature: 85 and 115 °C Ageing time: 1, 3, 5, 7, and 14 days NF conditions: Open	AC BDV of RBDPO remains unchanged at open condition, while its resistivity show a decrease trend at 115 °C.
(Shahee et al., 2024)	N/a	CoO, PO, MO	Temperature: 150 °C Ageing time: 15, 30, and 45 days NF conditions: Sealed	The BDV of CoO and PO decreased with ageing, but higher than MO.

This research should be expanded to include investigations on the effects of the temperature and ageing times under actual transformer conditions, with the NPs used in

palm-based oil as these factors should be further examined, especially regarding the prospective uses of HNFs as insulating oil in the future.

2.7 Research Gap

The production of insulating oil utilising biodegradable resource, such vegetable oils, will move in the direction towards the values of sustainable and renewable. Moreover, the growth of natural ester-based oil and the application of nanotechnology to elevate the performance of existing alternate insulating oil has shown a steadily rise over the year. A noteworthy finding that offers the earlier mentioned values has been shown through the exploration of palm-based MNF, however the potential application of HNF as insulating oil in transformer has yet to be extensively explored. Numerous researchers have documented the methods involved in developing the preparation of their proposed insulating oil and adhered to the established guidelines for testing.

Reducing the environmental impact of transformer performance inefficiency may be possible with the use of natural ester-based insulating as a biodegradable insulating oil and the creation of hybrid nanofluid. By reducing the danger due to their performance, the recommended oil also indirectly aids in mitigating greenhouse gas emissions, which are a significant global contributor to climate change. With palm oil's superior qualities over other vegetable oils making, it one of the most popular insulating oils according to recent research investigations, its use and application in conjunction with nanotechnology should be further investigated as it has a promising future as insulation materials in power transformers. For this research, a palm-based oil will be utilised in the investigation of HNF using the two-step method as well as the prolonged exposure ageing to evaluate their properties under actual transformer conditions.

According to several studies, further investigation into the characteristics of ZnO NP and TiO₂ NP has improved transformer oils' attributes, strengthening the foundation for the application of insulating oil. An example of HNF utilisation was carried out by dispersing it into various vegetable oils using NPs like Al₂O₃ and TiO₂. Among the hybrid vegetable-based oils, the study found that palm-based oil had superior thermophysical qualities compared to soybean and coconut-based oils (Wanatasanappan et al., 2022). This supports the study on palm-based oil quality benefits, as well as the addition of NPs. A similar investigation into the hybridisation but using TiO₂-ZnO, however in a new and used naphthenic oil was conducted by Paul and Goswami (2022) to observe the significance of HNFs concerning their thermal conductivity and dielectric properties. The investigation on HNF was primarily focused on radiator applications, besides transformer applications.

The research on the radiator was carried out using TiO₂ and ZnO NPs in pure water as a base fluid to increase pressure loss and enhance heat transfer performance for flat tubes (Elibol et al., 2024). The effect of an HNF on TiO₂-ZnO in ethylene glycol on the intensification of heat transfer across microchannels under laminar circumstances was also investigated (Nesakumar & Baskar, 2019). The utilisation of this HNF were able to improve the heat transfer of one oil in one application. These investigations show how TiO₂-ZnO HNFs can enhance transformer insulating oil performance, indicating its applicability to research goals focused on improving transformer oil formulations. The recent trends in insulating oil moving towards HNF applications are tabulated in Table 2.3, covering the methods, outcomes of research, and limitations of the study.

Table 2.3: Research Gap

Author(s)	Type	NP(s)	BF(s)	Methods	Outcomes of Research	Limitations
Afzal et al., 2022)	MNF	MgO, TiO ₂ , FeO ₃	MO	Three phases of dummy transformer and test cell model Simulation by using Quickfield software.	The addition of nanoparticles enhanced both the electrical and thermal properties of the nanofluid. TiO ₂ with the concentration n of 10% has the reduced voltage at center of the electrode by 46%.	The investigation only limited to simulation.
Ghislain et al. (2022)	MNF	FeO ₃	KPOME	The production of KPOME using : i) .transesterification, washing, filtered, dehumidification, and degassed. The production of NF using two-step method: i) NPs were dispersed with blends oil (0.10 wt% to 0.20 wt%). ii) Sonication at room temperature for 2 hrs.	The thermal conductivity of KPOME in the absence of FeO ₃ demonstrates optimal properties at a temperature of 65°C, followed by with a concentration of 0.20wt% FeO ₃ . The kinematic viscosity and fire point of KPOME show a reduction as the amount of FeO ₃ is reduced. The flash point, on the other hand, exhibits an upward trend with increasing concentration.	The analysis on the dielectric strength were limited.
Walvekar et al. (2021)	HNF	SiO ₂ -G	MO, POME,	The production of NF using two-step method: i) NPs were dispersed with blends	SiO ₂ -Graphene concentration improved thermal conductivity. Low-temperature POME-based	The analysis on the dielectric strength were limited.

Table 2.3: continued

			MO-POME	oil (0.01 wt% to 0.10 wt% for 10 minutes. ii) Sonication at room temperature for 5 hrs.	NF has low viscosity. The optimal MO, POME, and SiO ₂ -Graphene concentrations are 81.51 wt%, 18.48 wt%, and 0.10 wt%, respectively	
Benedict et al. (2020)	MNF, HNF	CNC, Al ₂ O ₃ -TiO ₂ , Al ₂ O ₃ -CNC	W-EG	The production of NF using two-step method: i) NPs were dispersed with W-EG (40:60) for 30 minutes. The NFs have volume concentrations of 0.1%, 0.5%, and 0.9%. ii) Sonication at 50 °C for 2 hours.	The study found that Al ₂ O ₃ -CNC HNF has the best thermal conductivity at 0.9% volume concentrations: but it had the highest viscosity and lowest specific heat capacity among others. Overall, CNC MNF demonstrated better specific heat capacity.	The exact concentration and ratio of NPs added into the W- EG were not clearly stated, and a lack of analysis on dielectric testing as it concentrated on radiator insulation.
Wanatasanapan et al. (2020)	HNF	TiO ₂ -Al ₂ O ₃	DI water	The production of NF using two-step method: i) TiO ₂ -Al ₂ O ₃ (20:80, 40:60, 50:50, 60:40, 80:20) were dispersed in DI water for 2 hours at 900rpm with a concentration of 1.0%. PVP was then added in a 1:10 ratio to NPs' mass. ii) Ultrasonication using ultrasonic bath for 2 hours.	TiO ₂ -Al ₂ O ₃ (80:20) sedimentation in DI water raised thermal conductivity by up to 71% and dynamic viscosity by up to 232%, while 50:50 ratio resulted led to the least sedimentation. The experimental were then used to construct a high-accuracy estimation correlation model for HNF	Further investigation will be required as higher viscosity did not simply imply it had the ideal viscosity values and there is a lack of analysis on dielectric testing.

Table 2.3: continued

					thermal conductivity and relative dynamic viscosity.	
Yunus et al. (2020)	MNF, HNF	NG powder	PO-TMP, PO.TMP-SSEO	The production of TMP ester using transesterification method: i) The synthesising of TMP were prepared by transesterified POME and trimethylolpropane alcohol. The production of NF using two-step method: i) TMP (0%, 2%, 4%, 5%, 6%, 7%) to RBDPO were dispersed for 30 minutes at 100 °C, then the best blends were dispersed further with SSEO (0%, 10%, 30%, 50%) with the same condition. Lastly, it was dispersed with NG NP with concentration of 0.25wt%, 0.50wt%, 0.75wt%) ii) Ultrasonication using ultrasonic bath for 8 hours	The blend of 30% PO-TMP and 70% SSEO improved all friction and wear properties of original SSEO. PO-TMP is a superior anti-wear element for engine oil than partially SSEO.	This study is only concentrated on engine insulation.

2.8 Chapter Summary

This chapter has delivered a summary of earlier studies conducted on the transition of alternative, sustainable, and biodegradable insulating oil from conventional mineral-based oil into transformers. Mineral-based oil is causing detrimental effects on the environment and harming living organisms. Vegetable oil, specifically palm oil, has been investigated as an alternate solution these days, as it is aligned with the mentioned values and exhibits similar characteristics to mineral-based oil when modified properly. Numerous studies have discovered that the thermal conductivity and dielectric properties of a nanofluid are greatly enhanced by adding nanoparticles to palm-based oil. While an enormous amount of research on monotype nanofluid has been issued and discussed, relatively limited investigation has been explored on the potential applications of HNF as an insulating oil. Further research has focused on exploring the extent to which a nanofluid can withstand being exposed to continuous heat for prolonged durations in a transformer. The last section of this chapter concluded with an overview of the research gap, covering methods employed by prior studies, the materials utilised, the findings obtained, and the limitations discovered.

CHAPTER 3

METHODOLOGY

3.1 Overview

This chapter gives an in-depth description of the methodological procedures used for the preparation and execution of the experiment. The initial step begins with designing a research plan that acts as a guideline for each phase of this research process. The three primary phases used to prepare the HNF sample utilising POME involve; i) the preparation of POME by the transesterification process, ii) the preparation of HNF using a two-step method, and iii) the accelerated thermal ageing of POME-based TiO₂-ZnO HNFs. In addition, the electrical and physicochemical properties of these HNF samples will be assessed using the ASTM and IEC standard testing methods for insulating oil at different ages. This step is known as the data analysis method, and it will be discussed sequentially.

3.2 Research Plan

The increasing need for sustainable, renewable, and reliable insulation for electrical equipment has spurred the exploration of novel materials suitable for these purposes. As mentioned in the previous chapter, palm-based HNFs have not yet gained attention, even though the potential of palm-based MNFs as future insulating oils has been studied and proven by numerous researchers to possess comparable dielectric properties to mineral-based oils. Given these facts, this research focused on using more than one nanoparticle in the proposed HNF, particularly a combination of TiO₂-ZnO. In addition, the method of accelerated thermal ageing in the actual transformer insulation application. Figure 3.1 is the flowchart that illustrates the overall research plan for POME-based TiO₂-ZnO HNFs.

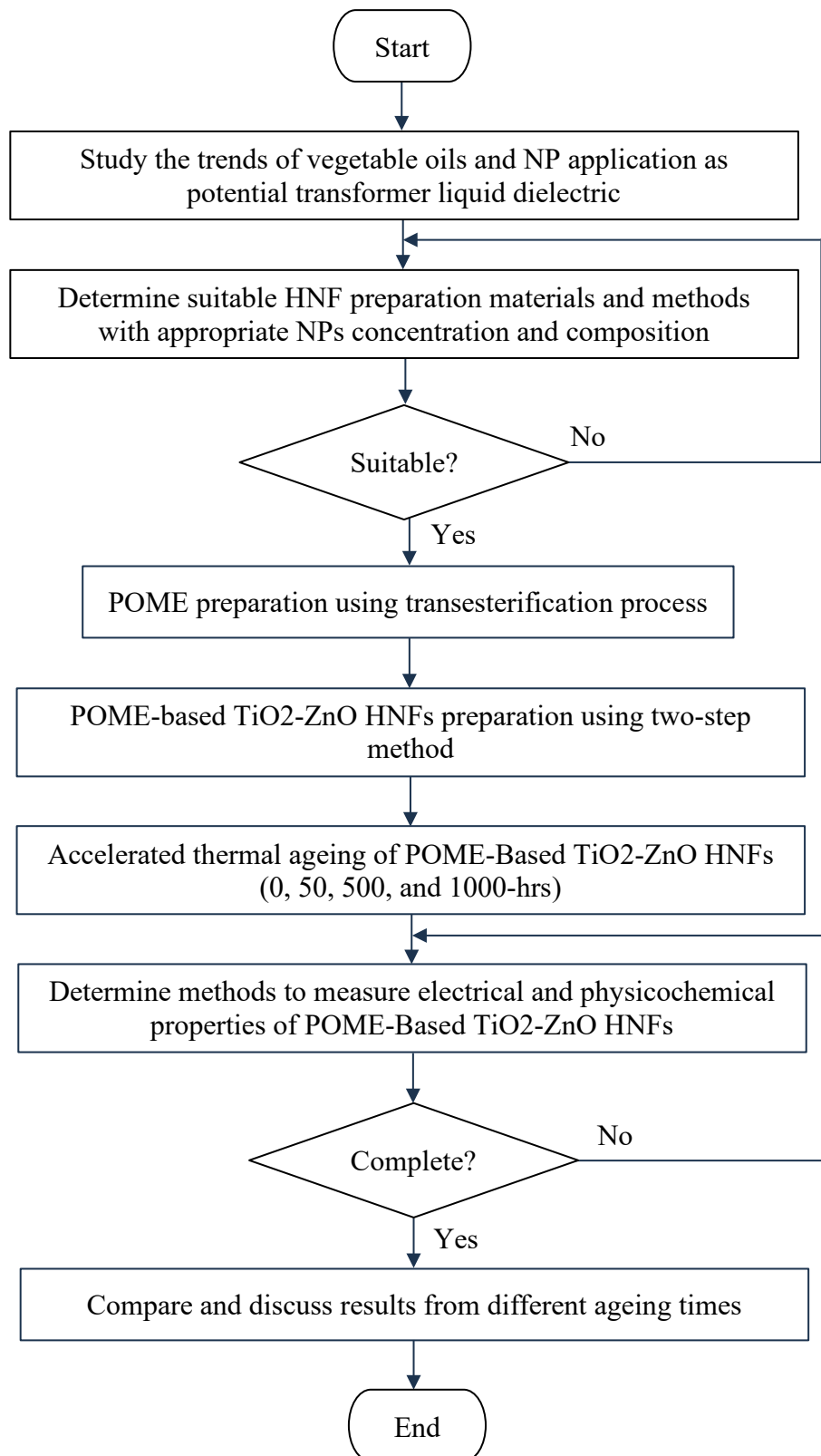


Figure 3.1: Overall Research Plan Flowchart of POME-based TiO₂-ZnO HNFs

Since liquid insulation is one of the important factors in improving the efficiency of a transformer, as well as the whole electrical power system, the topic of insulating oil will be addressed in this research. The utilisation of vegetable oils as a greener solution to promote sustainable technology has drawn interest, especially alongside the adoption of NPs to enhance their dielectric properties. So, it is essential to study the suitability of both these ideas as a potential liquid dielectric for transformer insulation. As shown in the flowchart in Figure 3.1, the determining process for necessary materials and equipment in HNFs preparation with specific composition and concentration involves narrowing down the list of ideal methods based on conducted studies. The transesterification process is utilised in POME preparation (dispersion between the methanol and KOH with RBDPO), signifying the beginning of the experiment once the appropriate materials and method have been determined.

The next phase of the experiment is to prepare the POME-based TiO₂-ZnO HNFs using the two-step methods, which involves dispersing POME with TiO₂ and ZnO NPs and then sonicating the HNF mixture for 3 hrs. The POME-based TiO₂-ZnO HNFs will undergo accelerated thermal ageing during the final phase. This process is performed at temperatures of 135 °C for four different ageing times; nonaccelerated (0-hr), and accelerated (50-hrs, 500-hrs, and 1000-hrs). The data analysis method for measuring the electrical and physicochemical properties based on the ASTM and IEC standard testing for insulating oil is then determined by considering the previous research investigations and the availability of analysis equipment in the laboratory. After the completion of the required data at different concentrations (0.05g/L, 0.10g/L, 0.15g/L, and 0.20g/L), it will be organised in an easily comprehensible manner. Last but not least, the outcomes are compared and analysed primarily regarding their ageing times and concentrations of HNFs.

3.3 Experimental Procedure

The following section comprises the materials, equipment, and detailed laboratory procedures required for the three primary phases involved of preparing POME-based TiO₂-ZnO HNFs in ensuing investigations.

3.3.1 Materials and Equipment

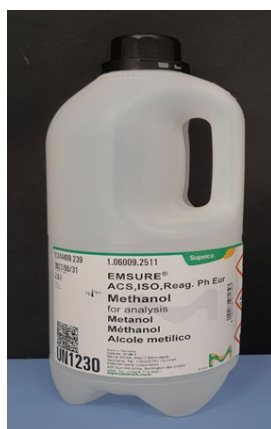
RBDPO will be modified into POME through a transesterification process with the utilisation of methanol (CH₃OH) and a catalyst of potassium hydroxide (KOH) in pellet form in this research (Munawar et al., 2020; Talebian-Kiakalaieh & Amin, 2015). The refined, bleached, or deodorised palm oil, or simplification for RBDPO based on Noor et al. (2017), is manufactured by Lam Soon and is widely accessible in local supermarkets. Next, TiO₂ NP and ZnO NP, manufactured by Sigma Aldrich, are the NPs utilised in the HNF preparations. These TiO₂ and ZnO NPs can be used as a hybrid due to their similar semiconductive qualities, which Nesakumar and Baskar (2019) and Sahid et al. (2017) have investigated. Because the band gap width and scattered energy band position of the TiO₂ NP are identical to those of ZnO, an end-product of HNF will be stable (Zhang et al., 2021). Cationic-type surfactants, named Cetyl Trimethyl Ammonium Bromide (CTAB), also manufactured by Sigma Aldrich, can be added to the HNF to increase its stability and prevent NPs from sedimentation (Haq et al., 2017).

Aside from this, the equipment used for material dispersion and heating are digital magnetic stirrers with temperature probes manufactured by Joan Lab meanwhile, the water bath ultrasonic cleaner is used for the sonication process. Duran bottles made of borosilicate glass are also used to store HNFs, particularly for accelerated thermal ageing because of their remarkable ability to withstand high temperatures for prolonged periods (Bouras et al.,

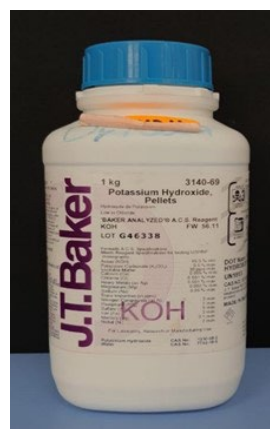
2009). Both the materials and equipment employed in this study are commonly utilised in small-scale biodiesel production and have no adverse effects on the environment. The materials and equipment required to prepare the POME-based TiO₂-ZnO HNFs are depicted in Figure 3.2 and Figure 3.3, respectively.



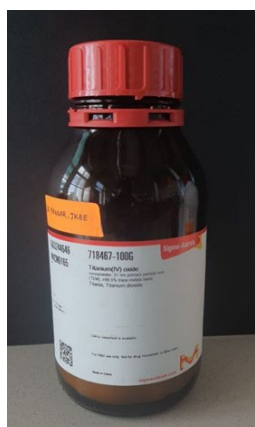
(a) RBDPO



(b) Methanol



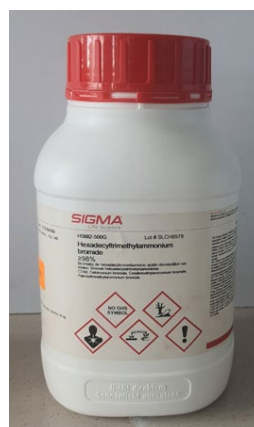
(c) Potassium Hydroxide



(d) TiO₂ NP



(e) ZnO NP



(f) CTAB

Figure 3.2: Materials Used in the Preparation of POME-based TiO₂-ZnO HNFs

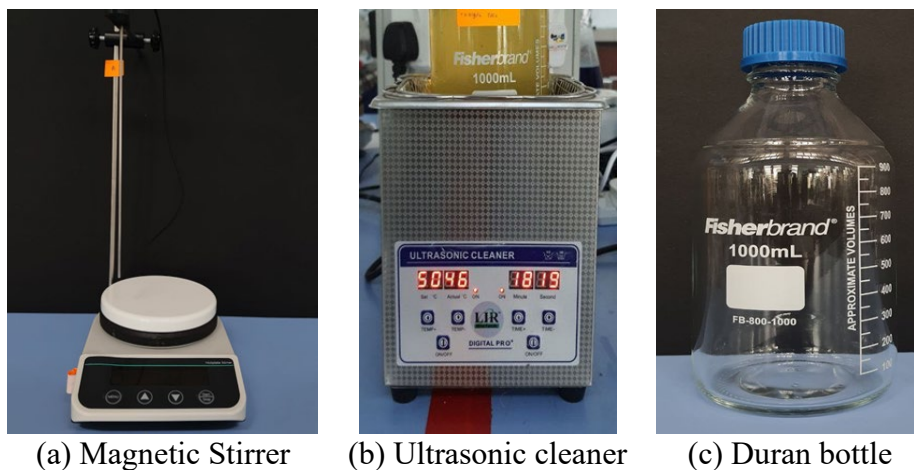


Figure 3.3: Equipment Used in the Preparation of POME-based TiO₂-ZnO HNFs

3.3.2 Preparation of POME

Using Equation 3.1 with the alcohol to oil ratio of molar mass at 1:6, one can determine the needed mass of methanol when 1000g of RBDPO is used in POME preparation procedure as indicated in Figure 3.4. In the first phase of the experiment, Figure 3.4 shows the step-by-step process of making Methyl Ester from RBDPO, to replicate the characteristics of biodiesel feedstock commonly derived from mineral-based oil.

$$\frac{mass_{(RBDPO)}}{molar\ mass_{(RBDPO)}, g/mol} = \frac{mass_{(Methanol)}}{6(molar\ mass_{(Methanol)}, g/mol)} \quad \text{Equation 3.1}$$

Table 3.1: Molar Mass and Mass Required for RDBPO and Methanol

Material	Molar Mass (g/mol)	Required Mass (g)
RBDPO	846.00	1000.00
Methanol	32.04	227.23

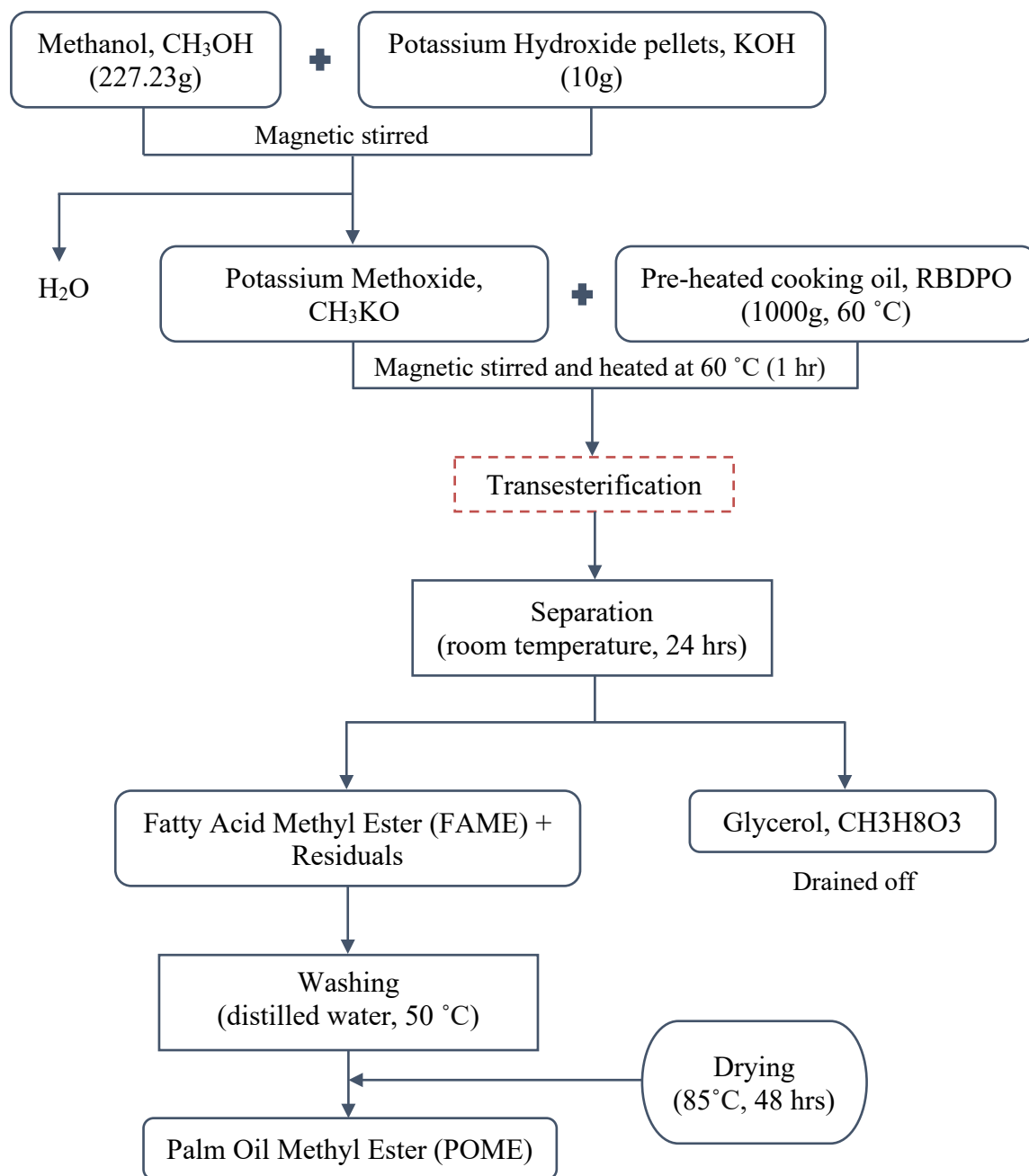


Figure 3.4: Preparation of POME

10g of KOH in the form of pellets will be added into 227.23g, 7.094 mol liquid methanol until it is completely dissolved using the magnetic stirrer at 900rpm. The product is identified as potassium methoxide (KOCH₃). The KOCH₃ will then be added into a preheated 1000g, 1.182 mol of RBDPO once 60 °C is reached and dispersed using the

magnetic stirrer at 900rpm for 1 hr. Then, the mixture will be naturally separated at room temperature for 24 hrs in a separatory funnel. Two layers, indicating fatty acid methyl ester (FAME) and glycerol ($\text{CH}_3\text{H}_8\text{O}_3$) are produced in the funnel after 24 hrs. The bottom layer, glycerol will be drained, while the top layer, FAME will be washed with distilled water heated to $50\text{ }^\circ\text{C}$ to remove the remaining KOH. The washing of FAME is repeated four times, each with a 20 minute interval until the pH value reaches 7 (neutral). The FAME mixtures are placed in a 1L Duran bottle and dried in the conventional oven at $85\text{ }^\circ\text{C}$ for 48 hrs to remove excess water molecules or moisture. The end product is called POME. The laboratory experiment procedures are illustrated in Figure 3.5.

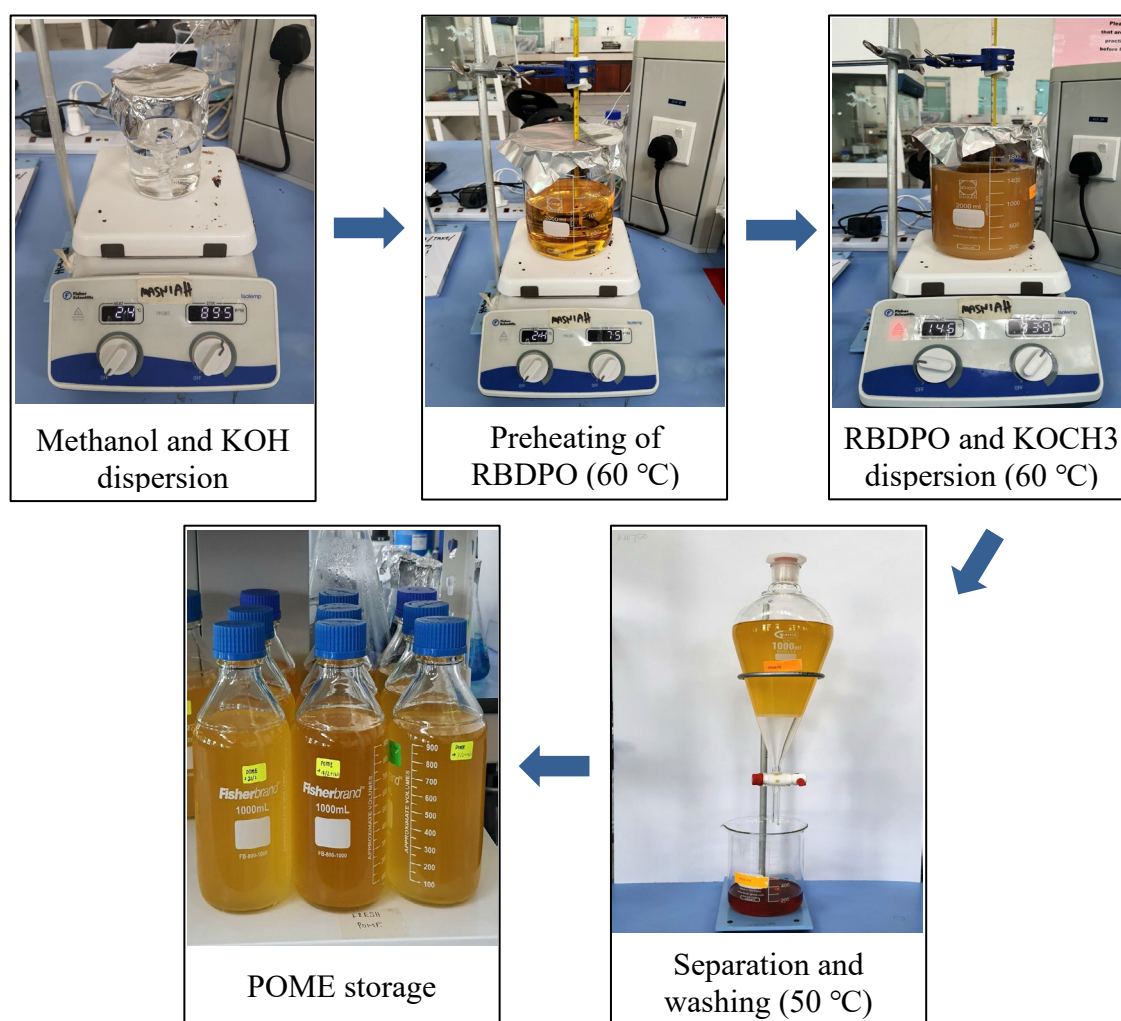


Figure 3.5: Experimental procedure of POME preparation

3.3.3 Preparation of POME-based TiO₂-ZnO HNFs

Table 3.2 includes the mass required based on the addition of surfactant to the NP ratio at 1:2 and the ratio of TiO₂ NP to ZnO NP at 80:20. The two-step method of NF preparation used following the POME preparation is shown in Figure 3.6, which details the step-by-step process of preparing HNFs in the second phase of the experimental procedures.

Table 3.2: Mass Required for CTAB, TiO₂ NP, and ZnO NP

CTAB	ZnO NP	TiO ₂ NP	Concentration of HNF (g/L)
0.025	0.01	0.04	0.05
0.500	0.02	0.08	0.10
0.075	0.03	0.12	0.15
0.100	0.04	0.16	0.20

Step 1: Direct Dispersion

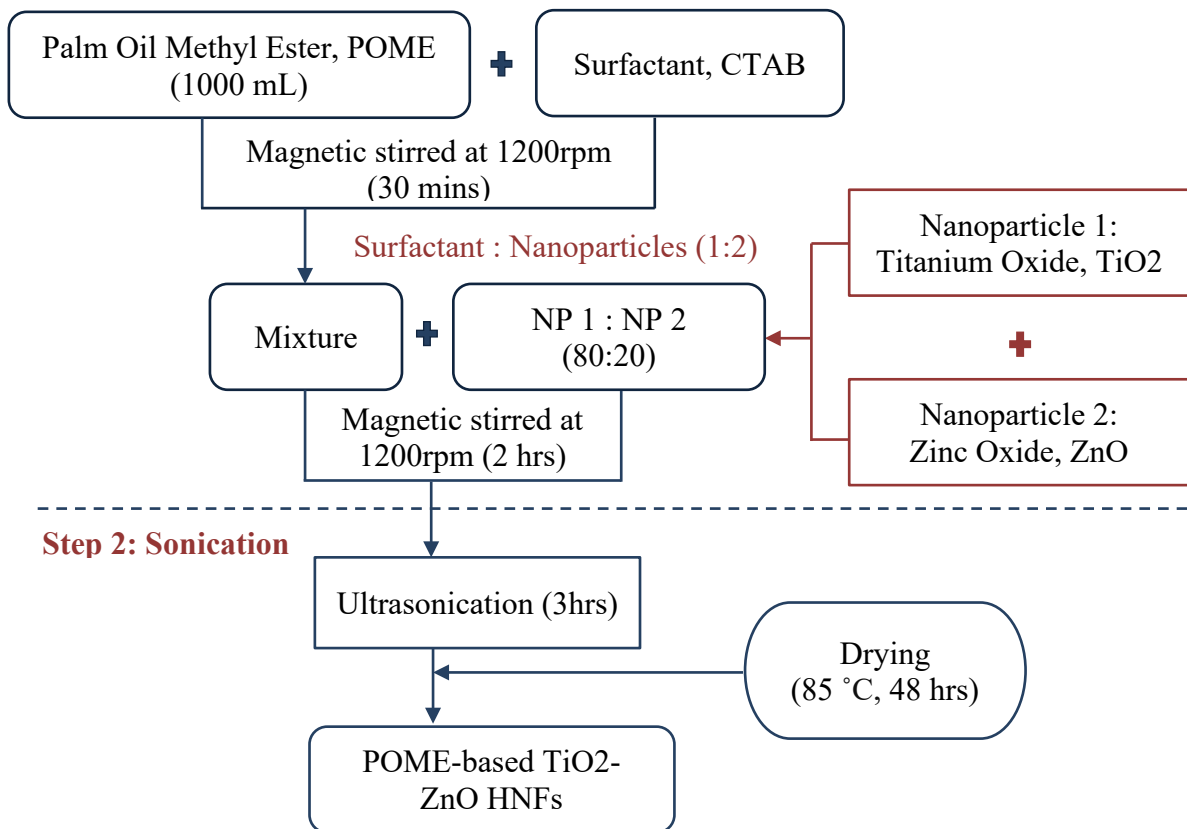


Figure 3.6: Preparation of POME-based TiO₂-ZnO HNF

Before commencing the two-step method for HNF preparation, it is necessary to subject the TiO₂ and ZnO NP to a drying procedure in a conventional oven. It is to eliminate the presence of moisture since the NPs tend to absorb water molecules, potentially affecting their overall properties. The dried POME from the previous preparation is the base fluid in this experiment and is measured to 1000 mL or approximately 857g. The surfactant, CTAB, is weighed and added into POME according to Table 3.2. The mixture is then subjected to dispersion for 30 minutes at a rotational speed of 1200 rpm using a magnetic stirrer without applying heat. TiO₂ and ZnO NPs are added directly into the mixture and another dispersion for 2 hrs at a speed of 1200rpm. The HNF is then kept in a Duran bottle once the dispersion procedure is completed. The sonication is carried out using ultrasonic water bath for 3 hrs, with the agitation taking place at regular intervals of half an hour. The HNFs will then be dried further for 48 hrs at 85 °C when it is completed. The laboratory experiment procedures are illustrated in Figure 3.7.

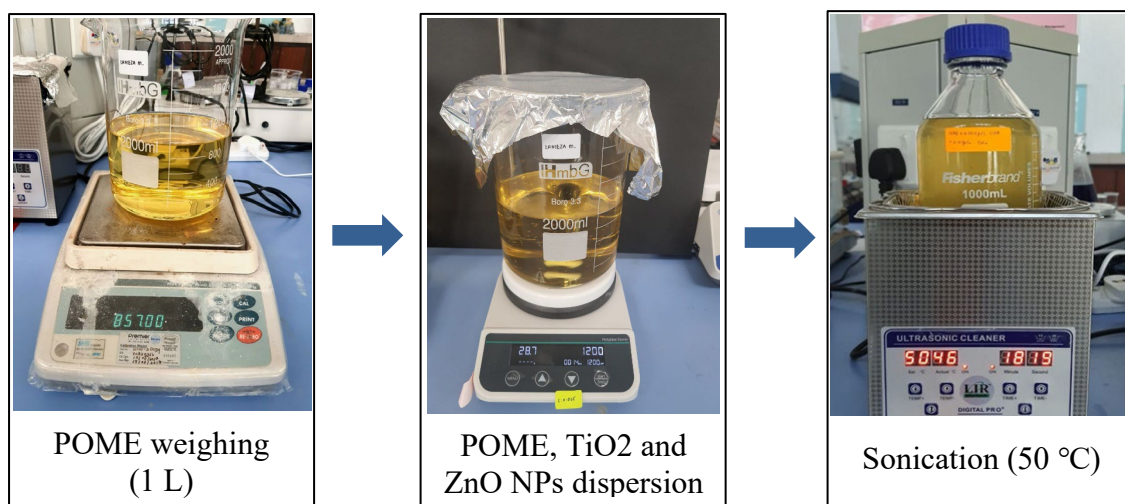


Figure 3.7: Experimental procedure of POME-based TiO₂-ZnO HNF preparation

3.3.4 Accelerated Thermal Ageing of POME-based TiO₂-ZnO HNFs

An accelerated thermal ageing experiment is conducted to investigate the stability and degrading behaviour of POME-based TiO₂-ZnO HNFs concerning their longevity in transformers under similar conditions. Besides, this ageing process is crucial in ensuring the potential of utilising these HNFs as alternate insulating oil. The pure POME and POME-based TiO₂-ZnO HNFs are placed in a thermal oven at 135 °C with a sealed condition and performed a thermal ageing for 50-hrs, 500-hrs, and 1000-hrs, as shown in Figure 3.8. After allowing the aged HNFs to cool at room temperature for a day, they will only be measured. Also, a comparison will be performed between the accelerated and nonaccelerated thermal ageing of the 0-hr data to observe changes in the properties under extended thermal exposure.



Figure 3.8: Thermal ageing using Thermal Oven

3.4 Experimental Analysis Method

The following section comprises the equipment, and detailed laboratory procedures required for the data analysis in Chapter 4, including TiO₂ and ZnO NPs characterisation, breakdown voltage test, measurement of kinematic viscosity and density, and POME-based TiO₂-ZnO HNFs characterisations according to IEC and ASTM standards for insulating oil testing measurements.

3.4.1 Characterisation of TiO₂ and ZnO NPs using FESEM-EDS

The Field Emission Scanning Emission Electron Microscope (FESEM) with energy-dispersive X-ray spectroscopy (EDS) allows visualising and characterising the surface topology and elemental compositions of the TiO₂ and ZnO NPs before the preparation of the TiO₂-ZnO HNFs. Figure 3.9 shows that the JEOL JSM-IT500HR equipment, which runs at 20 kV, can be used to conduct FESEM-EDS.



Figure 3.9: JEOL JSM-IT500HR FESEM Equipment

3.4.2 Breakdown Voltage Test

The Huazheng® HZJQ-1B Transformer Oil BDV Tester machine, shown in Figure 3.10 (a), is used to measure the breakdown voltage test for this research. International Electrotechnical Commission (2018) will be the standard for measuring the breakdown voltage (BDV) of the POME-based TiO₂-ZnO HNFs. Following the specifications shown in Figure 3.11, as specified in IEC 60156, the mushroom electrodes sized 12.5 mm in diameter must have a gap distance of 2.5 mm between them. 250 mL of HNF is injected into the test cell oil cup as shown in Figure 3.10 (b), until the electrode is almost immersed fully with oil. The voltage is continuously applied into the electrodes with a steady rise rate of 2 kV/s. An initial standing time of 5 minutes is set before the first BDV, including the recommended interval time between each BDV is 2 minutes long to let the HNF stabilise

after higher voltage is applied toward the electrodes (Khaled & Beroual, 2020). For this test, 18 total measurements are obtained, with each series comprised of 6 consecutive measurements of AC BDV that will be repeated another two times (Khelifa et al., 2022). These data are used to investigate the conformity of AC BDV data to normal and Weibull distributions using the AD-goodness-of-fit test.

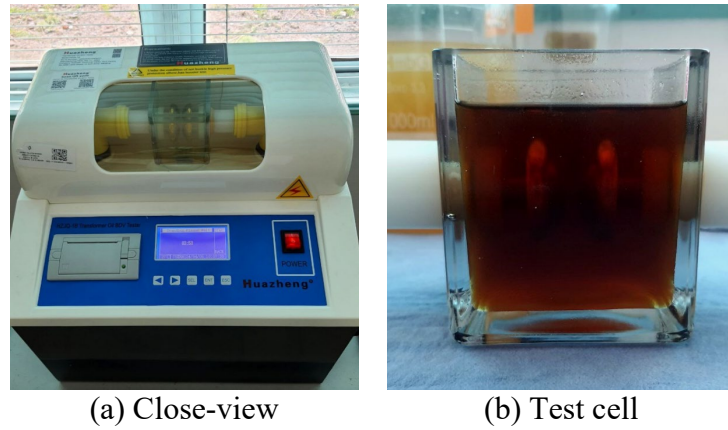


Figure 3.10: HZJQ-1B Transformer Oil BDV Tester Machine

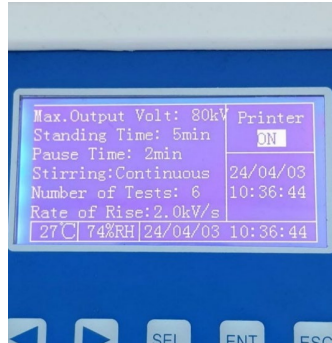


Figure 3.11: AC BDV testing specifications

3.4.3 Measurement of Kinematic Viscosity

The Rotavisc IKA viscometer depicted in Figure 3.12 is accessible in the laboratory and used to measure kinematic viscosity at standard room temperature, 27 °C and 40 °C. The viscosity obtained is obtained by following ASTM Standard D445-17a (2008). POME-based TiO₂-ZnO HNFs are prepared and poured into an empty beaker to a volume of about 250

mL. In this testing, spindle type 1, SP-1 is utilised since it is able to measure a low viscosity, or more precisely, the oil type of liquid. The oil will be immersed until it reaches the spindle's indentation line before starting the viscometer. The viscometer's speed is adjusted to 160 rpm. Then, these steps are repeated for each concentration for nonaccelerated and accelerated thermal ageing after heating the sample at 40 °C using a magnetic stirrer in Figure 3.13. The kinematic viscosity measurements are repeated three times for each sample and mean values are calculated.

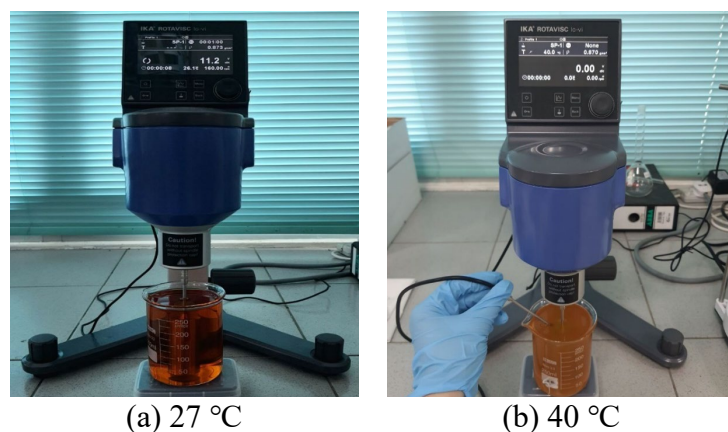


Figure 3.12: Rotavisc IKA Viscosity measurement at: (a) 27 °C and (b) 40 °C



Figure 3.13: Heating of POME-based TiO₂-ZnO HNF using magnetic stirrer at 40 °C

3.4.4 Measurement of Density

The density of the POME-based TiO₂-ZnO HNFs measured using an Aton Paar DMA 35 is shown in Figure 3.14 (a) and (b), according to ASTM Standard D7777-13 (2018)

for density determination with a portable digital density meter. Only 4 to 5 mL of oil samples will be needed for this digital DMA 35 density meter to measure. The density measurements are repeated three times for each sample and mean values are calculated for a more precise result.

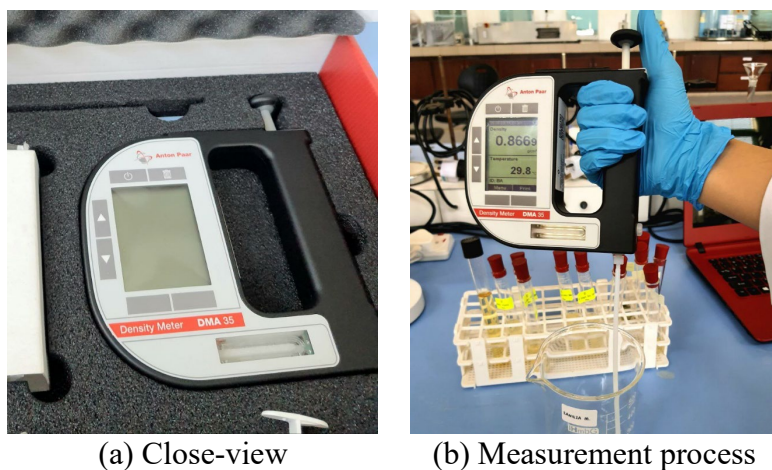


Figure 3.14: Aton Paar DMA 35 Density Meter

3.4.5 Characterisation of POME-based TiO₂-ZnO using FTIR Spectra

The functional groups of POME-based TiO₂-ZnO HNFs can be characterised through FTIR spectroscopy via the Shimadzu IRAffinity-1 spectrophotometer, as displayed in Figure 3.15, as this effectively detects changes in the functional groups (Younis et al., 2021). One drop of each sample of pure POME and POME-based TiO₂-ZnO HNFs is placed on the surface of the machine, while the lid will be closed afterwards to let the IR radiation pass through the oil sample. The absorbance rate (a.u.) ranges between 500 and 4000 cm⁻¹ wavenumbers with 42 scannings. The data is collected and adjusted to get the desired results by using the IRRolution Software by following these steps: Measure > File Name > Start > Manipulation > Smoothing > Manipulation > Peak Table > Add Peak > File > Save. Each graph will be merged from one ageing time into a single graph to study the variations and

parallels in the functional group and chemical bonding that occur with rising HNF concentrations and the effects of accelerated thermal ageing on the HNFs.



Figure 3.15: Shimadzu IRAffinity-1 Spectrophotometer

3.4.6 Stability of POME-based TiO₂-ZnO using UV-Vis

In addition to visual inspection using NP, which is through the formation of sediments, the stability of POME-based TiO₂-ZnO HNFs can also be assessed via UV-Vis spectroscopy on a Shimadzu UV-1800 UV/Visible Spectrophotometer as shown in Figure 3.16 (Arifutzzaman et al., 2019; Fikri et al., 2021). 4mL from each sample is poured into a 10-mm Quartz Cuvette glass and inserted within the UV-Vis spectroscopy machine once pure POME and POME-based TiO₂-ZnO HNFs for nonaccelerated and accelerated thermal ageing are prepared and completed. The absorbance (a.u.) of the light used in this research ranges from 336 to 700 nm at standard room temperature. Following this, using the UV-Probe Software, the data is collected and documented by following these steps: Start > Peak Pick > File > Save. The absorbance peak of pure POME and four concentrations of POME-based TiO₂-ZnO HNFs (0.05 g/L, 0.10 g/L, 0.15 g/L, and 0.20 g/L) were merged into one

graph to examine the variations in concentrations and the effects of accelerated thermal ageing towards the stability of HNFs.

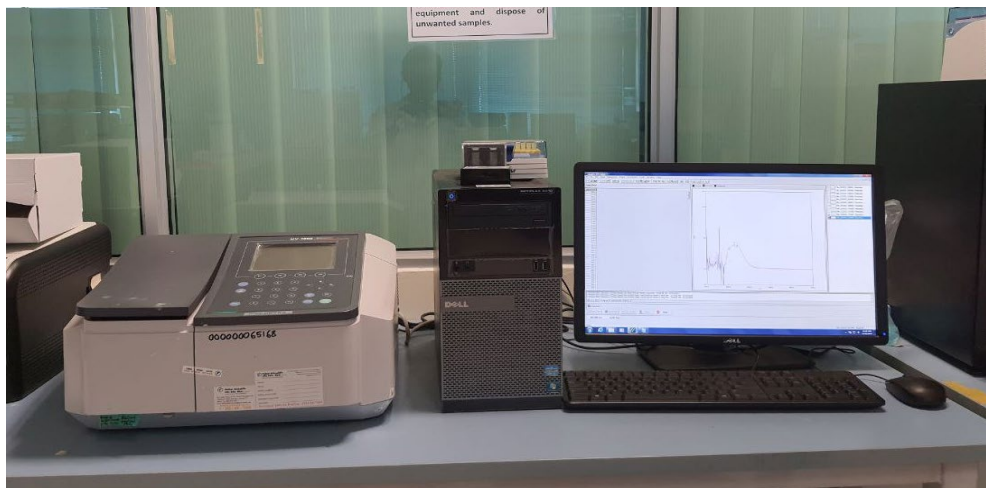


Figure 3.16: Shimadzu UV-1800 UV/Visible Spectrophotometer

3.5 Chapter Summary

In this chapter, the methodologies used in this study were categorised and discussed following the research plan. Afterwards, the experimental procedure required three phases: preparing POME using the transesterification process, preparing POME-based TiO₂-ZnO HNFs with concentrations of 0.05 g/L, 0.10 g/L, 0.15 g/L, and 0.20 g/L using the two-step method, and preparing for nonaccelerated (0-hr) and accelerated (50-hrs, 500-hrs, and 1000-hrs) thermal ageing at 135 °C. Besides, measuring the electrical and physicochemical properties of the POME-based TiO₂-ZnO HNFs method was determined. These include the data measurement method for AC BDV, kinematic viscosity, density, FTIR and UV-Vis spectroscopy. The findings will be analysed in more detail in the following chapter.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview

This chapter presents the experimental findings of an investigation on the POME-based TiO₂-ZnO HNFs that experienced transesterification, two-step HNF production, and accelerated thermal ageing. The POME percentage yield of the POME is assessed to validate that it surpasses 95% after the transesterification process before producing the TiO₂-ZnO HNFs. Besides, the FESEM-EDS analysis is carried out earlier to explore the elemental composition, size, and surface topology of the TiO₂ and ZnO NPs to analyse the HNFs later, respectively. The key discoveries of this research centre on analysing the results for POME-Based TiO₂-ZnO HNFs under nonaccelerated and accelerated thermal ageing. These results are divided into two parts; i) electrical property, such as AC BDV; and ii) physicochemical properties, including visual appearance, density and kinematic viscosity measurement, spectroscopy analysis of FTIR and UV-Vis. The AC BDV test is performed to evaluate the sample's dielectric strength by adhering to the standard value for insulating oil in transformers. Then, the POME-Based TiO₂-ZnO HNFs are examined on their changes in colour and NPs sedimentation due to ageing effects. The density and viscosity of the hybrid nanofluid are measured to ensure that the proposed oil will maintain its properties and effectiveness, by comparing it to an established value. FTIR spectroscopy is a method to determine the functional groups and molecular structure by analysing the chemical bonds, while UV-Vis spectroscopy is employed to observe the stability and detect the presence of conjugated double bonds in the HNFs.

4.2 Preliminary Analysis

This section will provide the preliminary analysis (before conducting main analysis test) of this research, which includes the percentage yield calculation of POME and the FESEM analysis before proceeding with the production of the TiO₂-ZnO HNFs.

4.2.1 Percentage Yield of POME

One noteworthy example of a biodiesel feedstock resource is palm-based oil, which may be characterised as an oil that possesses the characteristics of biodegradability and commercial viability as an insulating oil, in comparison to conventional mineral-based oils. The development of this particular type of palm-based insulating oil can be obtained through a transesterification process involving RBDPO, which is commonly referred to as palm cooking oil. The triglyceride present in the RBDPO reacts chemically with methanol, that is, its short-chain alcohol component, in the presence of KOH acting as a catalyst. This reaction will result in the production of FAME, which is also referred to as POME. The effectiveness of this transesterification process for the conversion of RBDPO into POME can be determined by calculating the POME sample percentage yield as specified in Equation 4.1, with reference to the theoretical biodiesel production percentage defined by Trisupakitti et al. (2018). Additionally, it was stated the mass of the produced biodiesel and palm oil used are measured in grams (Trisupakitti et al., 2018).

$$POME \text{ Percentage Yield (\%)} = \frac{mass_{(POME)}}{mass_{(RBDPO)}} \times 100\% \quad \text{Equation 4.1}$$

As mentioned in the previous chapter, this research utilised 1000g of RBDPO for the transesterification process in the preparation of POME. Table 4.1 presents the calculated percentage yield of the produced POME sample, which is used for the preparation of the

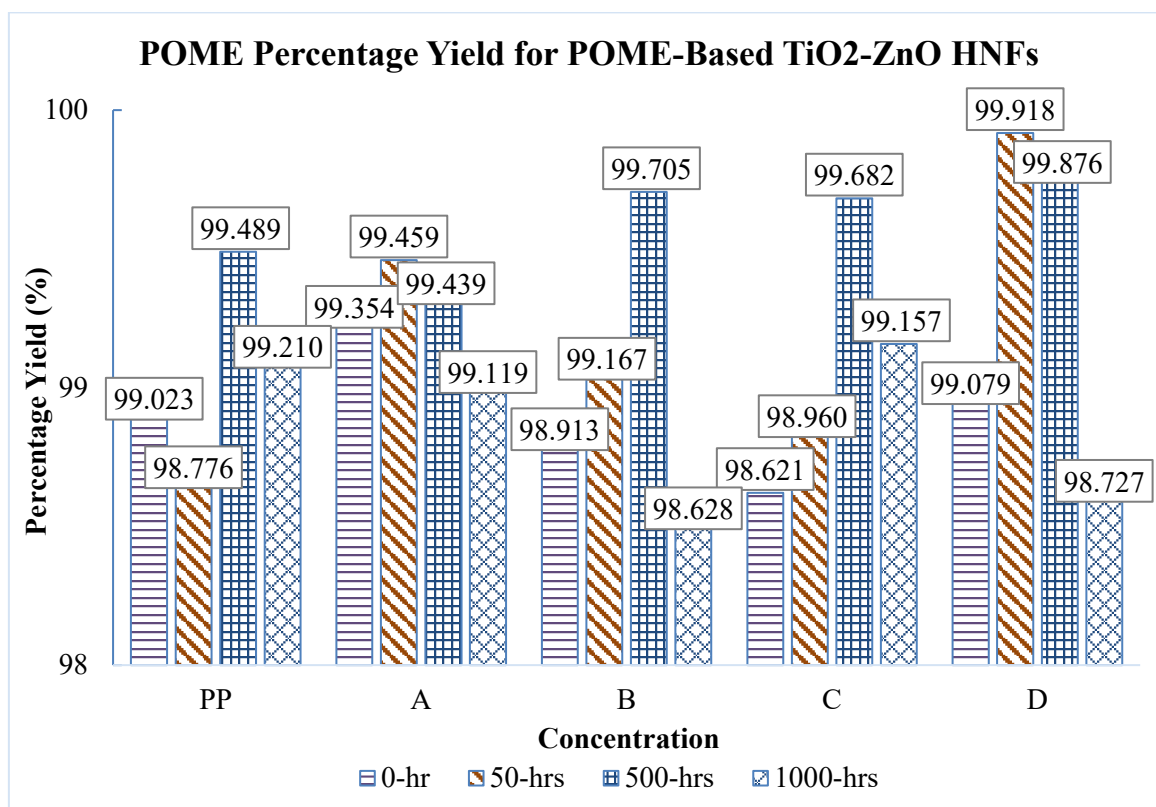
HNFs for different ageing times of 0-hr, 50-hrs, 500-hrs, and 1000-hrs. On the other hand, the data displayed on the graph in Figure 4.1 comes from the percentage yield of POME presented in the Table 4.1. The samples presented in Figure 4.1 have been classified into the following categories: Pure POME is referred to as PP, whereas POME samples with different amounts of TiO₂-ZnO HNFs are labelled as A, B, C, and D, corresponding to concentrations of 0.05g/L, 0.10g/L, 0.15g/L, and 0.20g/L, respectively.

Table 4.1: POME Percentage Yield for Ageing of 0-hr, 50-hrs, 500hrs, and 1000-hrs

Measurement	Mass of POME (g)	Percentage Yield (%)
Ageing Time: 0-hr		
Pure POME	990.23	99.023
0.05g/L TiO ₂ -ZnO + POME	993.54	99.354
0.10g/L TiO ₂ -ZnO + POME	989.13	98.913
0.15g/L TiO ₂ -ZnO + POME	986.21	98.621
0.20g/L TiO ₂ -ZnO + POME	990.79	99.079
Ageing Time: 50-hrs		
Pure POME	987.76	98.776
0.05g/L TiO ₂ -ZnO + POME	994.59	99.459
0.10g/L TiO ₂ -ZnO + POME	991.67	99.167
0.15g/L TiO ₂ -ZnO + POME	989.60	98.960
0.20g/L TiO ₂ -ZnO + POME	999.18	99.918
Ageing Time: 500-hrs		
Pure POME	994.89	99.489
0.05g/L TiO ₂ -ZnO + POME	994.39	99.439
0.10g/L TiO ₂ -ZnO + POME	997.05	99.705
0.15g/L TiO ₂ -ZnO + POME	996.82	99.682
0.20g/L TiO ₂ -ZnO + POME	998.76	99.876
Ageing Time: 1000-hrs		
Pure POME	992.1	99.210
0.05g/L TiO ₂ -ZnO + POME	991.19	99.119

Table 4.1: continued

0.10g/L TiO ₂ -ZnO + POME	986.28	98.628
0.15g/L TiO ₂ -ZnO + POME	991.57	99.157
0.20g/L TiO ₂ -ZnO + POME	987.27	98.727

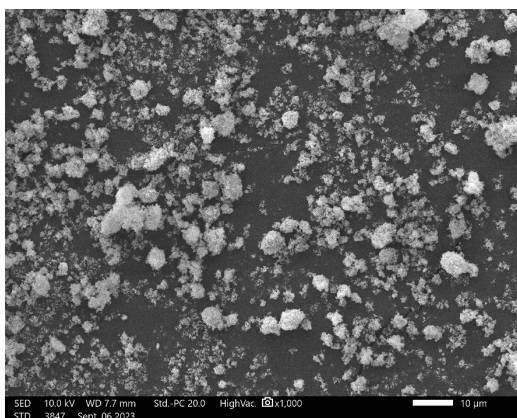
**Figure 4.1:** Graph of POME Percentage Yield for Pure POME and TiO₂-ZnO HNFs for Ageing of 0-hr, 50-hrs, 500-hrs, and 1000-hrs

Based on the data presented in Table 4.1 and Figure 4.1, it is apparent that all of the produced POME samples utilised in the preparation of TiO₂-ZnO HNFs have a percentage yield more than 98%. The average yield percentage of POME production (PP, A, B, C, and D) was calculated to be 98.998% for samples for 0-hr, 98.997% for samples aged for 50-hrs, 99.123% for samples aged for 500-hrs, and 98.9972% for samples aged for 1000-hrs. The overall mean of each sample combined has been determined to be 99.029%. Thus, all POME samples produced through this particular transesterification procedure have demonstrated a

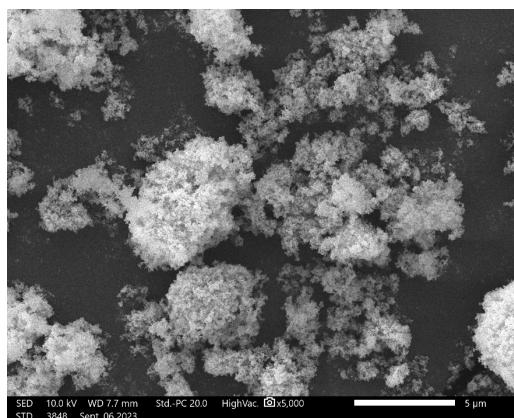
significant level of effectiveness, achieving a total average over 99%, as reflected by the maximum biodiesel output, which is reported to be achieved if it surpassed 95% (Kareem et al., 2017). A higher percentage yield signifies the effective production of the desired product, which is the FAME, in the reaction. This transesterification process offers consistent reaction outcomes for all POME samples, although slight variations in percentage yield may arise from intrinsic experimental drawbacks, such as practical losses during the procedure itself.

4.2.2 FESEM-EDS Analysis of TiO₂ and ZnO NPs

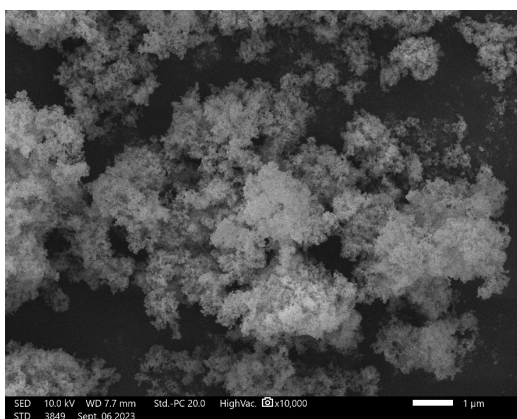
The addition of NPs into the production process of palm-based liquid dielectric is designed to augment both the dielectric and thermal properties of conventional mineral-based insulating oil (Hussain et al., 2022). This preliminary analysis research examines on the utilisation of TiO₂ and ZnO NPs in the production of POME-based TiO₂-ZnO HNFs. Therefore, it is crucial to investigate their characteristics since these factors could potentially influence the chemical and physical properties of the NF. The utilisation of FESEM-EDS equipment enables the precise imaging of each of the nanoparticles, within the nanometre range, with a better level of resolution. According to Hodoroaba et al. (2016), they stated that through SEM/FESEM-EDS analysis, it supports the purpose of assessing the surface morphology, internal structure, and elemental composition of nanoparticles. Figure 4.2 and Figure 4.3 shows the surface morphology and internal structure of the TiO₂ and ZnO NPs, respectively. The visualisations were obtained with different magnification levels.



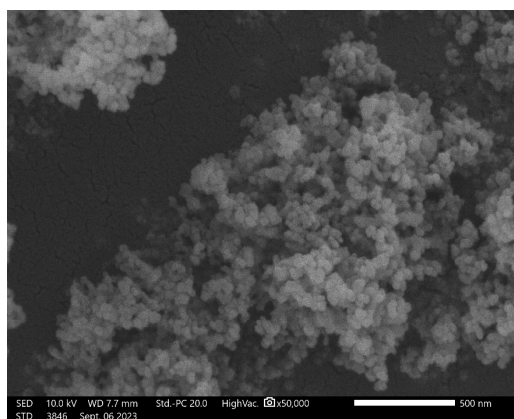
(a) x1,000



(b) x5,000

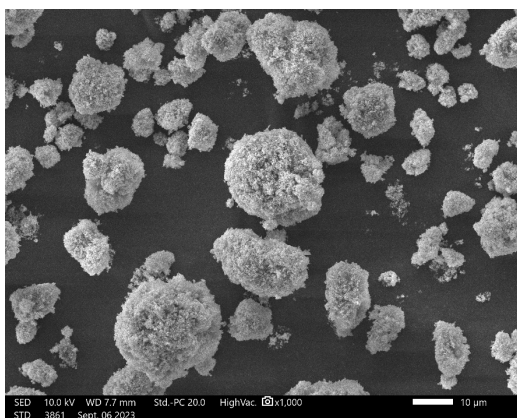


(c) x10,000

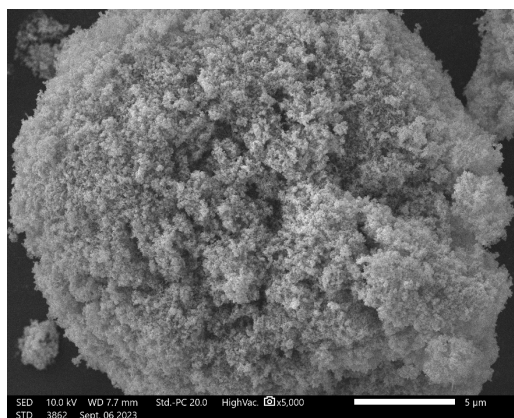


(d) x50,000

Figure 4.2: FESEM imaging of TiO₂ NP at 4 different magnifications: (a) x1,000, (b) x5,000, (c) x10,000, and (d) x50,000



(a) x1,000



(b) x5,000

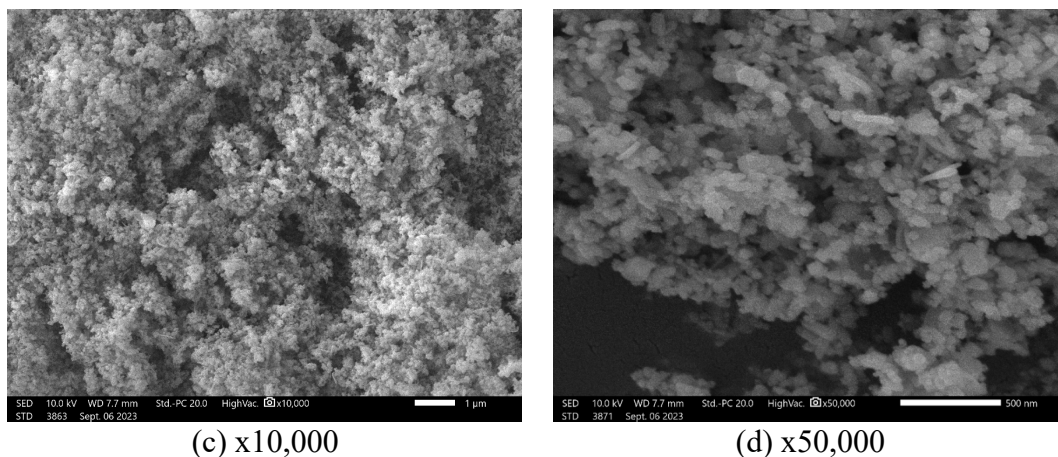


Figure 4.3: FESEM imaging of ZnO NP at 4 different magnifications: (a) x1,000, (b) x5,000, (c) x10,000, and (d) x50,000

Both TiO₂ and ZnO NPs were purchased in the form of powder from Sigma-Aldrich, a manufacturer based in St. Louis, Missouri, United State of America. Figure 4.2 reveals that TiO₂ has a predominantly a primarily spherical morphology, whereas Figure 4.3 illustrates that ZnO exhibits an essentially hexagonal morphology. According to Ilkhechi et al. (2020) and Kim et al. (2019), TiO₂ exhibits a spherical shape, while Taha et al. (2019) reported that ZnO nanoparticles have a hexagonal rod-like structure (Ilkhechi et al., 2020; Kim et al., 2019; Taha et al., 2019). The morphology of the TiO₂ and ZnO seen in the analysis is comparable, as pointed out in the previously reviewed paper. Along with this, the particle size of TiO₂ may range in between 1 to 150nm, as outlined in the *Safety Data Sheet: Titanium(IV) Oxide (2024)*, while ZnO is estimated to have a particle size smaller than 50nm.

Table 4.2: Average Particle Size of TiO₂ and ZnO NPs

Nanoparticle	Average particle size (nm)
TiO ₂	47.16 ± 17.44
ZnO	50.74 ± 19.49

The average particle size for TiO₂ and ZnO NPs were obtained by measuring 50 nanoparticles using the ImageJ software. The results are tabulated in Table 4.2. The TiO₂

and ZnO NPs used in this research possessed an average particle size of $47.16 \pm 17.44\text{nm}$ and $50.74 \pm 19.49\text{nm}$, respectively. From the results, the ZnO NP is slightly larger compared to TiO₂ NP. However, the functionality of both types of NPs remains the same, independent of any differences in their shapes and sizes. On the other hand, the predominant elemental composition of TiO₂ and ZnO NPs are measured through the EDS analysis, as depicted in Figure 4.4. Furuvik et al. (2022) and Kokla et al. (2023) carried out several investigations adopting imaging spectroscopy to accurately determine elemental composition of a small quantity of a certain material, which can be achieved through performing EDS analysis.

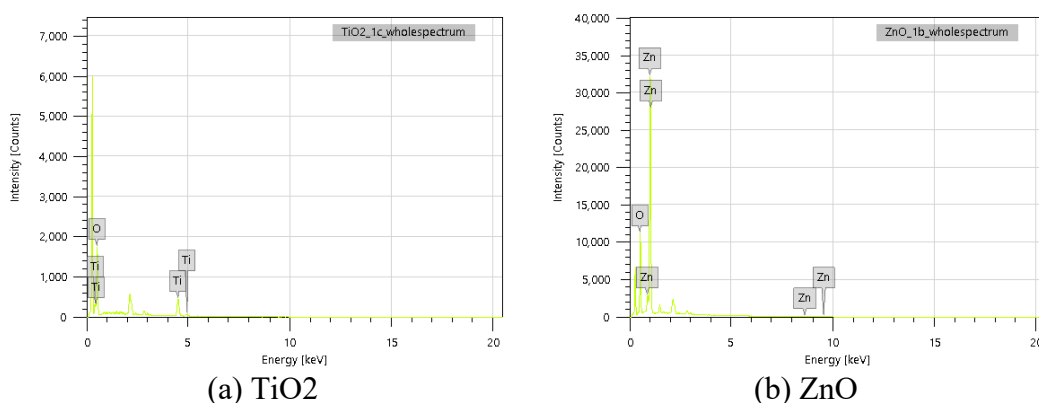


Figure 4.4: EDS analysis of (a) TiO₂ and (b) ZnO NPs

Figure 4.4 (a) and (b) show the elemental composition of TiO₂ and ZnO NPs obtained through the EDS spectra, respectively. Between these two EDS spectrums, the zinc (Zn) element exhibited the highest intensity. The detection of elements oxygen (O) and titanium (Ti) in the EDS Spectra seen in Figure 4.4 (a) suggests the formation of TiO₂. Meanwhile, Figure 4.4 (b) suggests that the detection of elements O and Zn leads to the formation of ZnO. This elemental composition of NPs utilised may have influenced the overall results of the data collected, especially when both NPs were introduced into the POME. The addition of NPs in the POME might change the POME structural groups as well

as increase in concentration will lead to agglomeration towards the HNFs that eventually lead to changes in behaviour of a good insulating oil.

4.3 Electrical Properties

The electrical property that are investigated and assessed for the POME-Based TiO₂-ZnO HNFs pertain to the measurement of dielectric strength. The AC BDV test was conducted to evaluate the dielectric strength possessed by the pure POME and POME-Based TiO₂-ZnO HNFs according to IEC 60156 standard for insulating oil in transformer. The aim of this test was to determine whether the improvement obtained by using pure POME in the production of POME-Based TiO₂-ZnO HNFs meets the desirable qualities specified in IEEE Std C57.147:2018 ($\geq 35\text{kV}$) for breakdown voltage, which would render them as a suitable and reliable alternative for insulating oil.

4.3.1 Average of AC BDV

The dielectric strength between pure POME and all POME-based TiO₂-ZnO HNFs, with varying concentrations and ageing times, was used to compare the 18 data obtained from the AC BDV measurements. In this section, Table 4.3 represents the average AC BDV data with the increment for the concentrations of 0.05 g/L, 0.10 g/L, 0.15 g/L, and 0.20 g/L of POME-based TiO₂-ZnO HNF enhancement rate based on the values of pure POME at each ageing time.

Table 4.3: Increment Percentage of AC BDV for pure POME and POME-Based TiO₂-ZnO HNFs

Measurement	Pure POME	0.05g/L TiO ₂ -ZnO + POME	0.10g/L TiO ₂ -ZnO + POME	0.15g/L TiO ₂ -ZnO + POME	0.20g/L TiO ₂ -ZnO + POME
Ageing Time: 0-hr					
AC BDV (kV)	19.6	23.3	24.3	29.0	29.1

Table 4.3: continued

Increment (%)	-	19.3	24.1	48.5	48.7
Ageing Time: 50-hrs					
AC BDV (kV)	19.7	26.8	28.7	51.2	54.3
Increment (%)	-	36.4	45.9	160.1	175.8
Ageing Time: 500-hrs					
AC BDV (kV)	18.1	52.0	42.3	49.4	49.5
Increment (%)	-	187.8	134.2	173.7	173.9
Ageing Time: 1000-hrs					
AC BDV (kV)	15.7	61.4	59.3	64.4	59.7
Increment (%)	-	290.8	277.0	309.3	279.9

As the ages went from 0-hr to 1000-hrs, the HNFs BDV increased compared to pure POME. These can be observed from the data tabulated in Table 4.3. For samples 0-hr and aged 50-hrs, the AC BDV and increment percentage continuously showed a positive trend as the concentration of the POME-based TiO₂-ZnO HNFs rose. The concentration of 0.20 g/L TiO₂-ZnO HNF achieved the highest AC BDV values at 29.1 kV with an increase of 49.7% at 0-hrs and 54.3 kV with 175.8% at 50-hrs. Meanwhile, the 500-hrs and 1000-hrs ageing times also showed an increase in percentage increment for all concentrations. However, the AC BDV values for TiO₂-ZnO HNFs at 500-hrs were lower than those at 50-hrs and then went up again at 1000-hrs. In contrast to the other concentrations, the concentration of 0.05g/L TiO₂-ZnO HNF demonstrated the most superior, continually stable increasing in AC BDV and increment percentage, and although it initially had the lowest BDV at 0-hr among the other HNFs. For instance, when it matures thermally, the increase in AC BDV was noted at 23.3 kV, 26.8 kV, 52.0 kV, and 61.4 kV.

In some cases, thermal ageing leads to degradation of the insulating characteristics, which are usually revealed through the physicochemical properties and the AC BDV analysis (Niasar et al., 2015). This investigation, however, revealed differently. According to study using TiO₂ NP and ZnO NP, which absorb free electrons and reduce their movement, including hybrid properties from both metal oxide NPs that can lessen the negative effects of ageing, thus the dielectric properties of these HNFs may be significantly improved as ageing time increased (Ilkhechi et al., 2020; Nawaz et al., 2020; Wanatasanapan et al., 2020; Wanatasanappan et al., 2022).

4.3.2 Normal distribution of AC BDV

The following section provides a more in-depth analysis normal distribution, and Weibull plotting of AC BDV data according to International Electrotechnical Commission (2007). In the statistical data utilised in the AC BDV analysis, the normality distribution was among the most commonly investigated. Khatun (2021) found that the Anderson Darling (AD)-goodness-of-fit test yielded the best statistical analysis when compared to the other Shapiro-Wilk (SW) and Shapiro Francia (SF) tests. Therefore, AD-goodness-of-fit test was utilised in this investigation to analyse these AC BDV data.

The AC BDV distributions of the pure POME and all POME-based TiO₂-ZnO HNFs' histograms for nonaccelerated (0-hr) and accelerated (50-hrs, 500-hrs, and 1000-hrs) thermal ageing was presented in Figure 4.5, Figure 4.6, Figure 4.7, and Figure 4.8 respectively. The histogram graph for colour yellow represented the samples of pure POME, magenta for 0.05 g/L TiO₂-ZnO HNF, red for 0.10 g/L TiO₂-ZnO HNF, green for 0.15 g/L TiO₂-ZnO HNF, and blue for 0.20 g/L TiO₂-ZnO HNF the same for all ageing times. On the other hand, Table 4.4 comprises the statistics, *p*-values, and conformity to the normal distribution of 0.05.

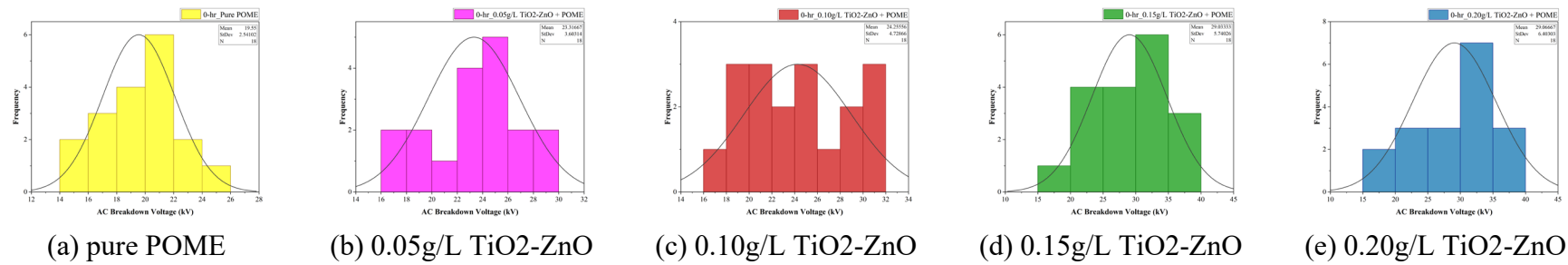


Figure 4.5: Histograms of 0-hr ageing for: (a) Pure POME, (b) 0.05g/L TiO₂-ZnO, (c) 0.10g/L TiO₂-ZnO, (d) 0.15g/L TiO₂-ZnO, and (e) 0.20g/L TiO₂-ZnO

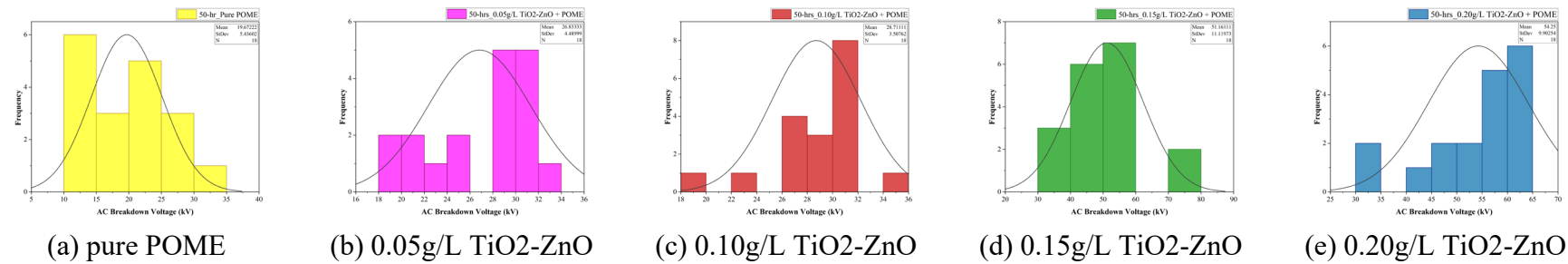


Figure 4.6: Histograms of 50-hrs ageing for: (a) Pure POME, (b) 0.05g/L TiO₂-ZnO, (c) 0.10g/L TiO₂-ZnO, (d) 0.15g/L TiO₂-ZnO, and (e) 0.20g/L TiO₂-ZnO

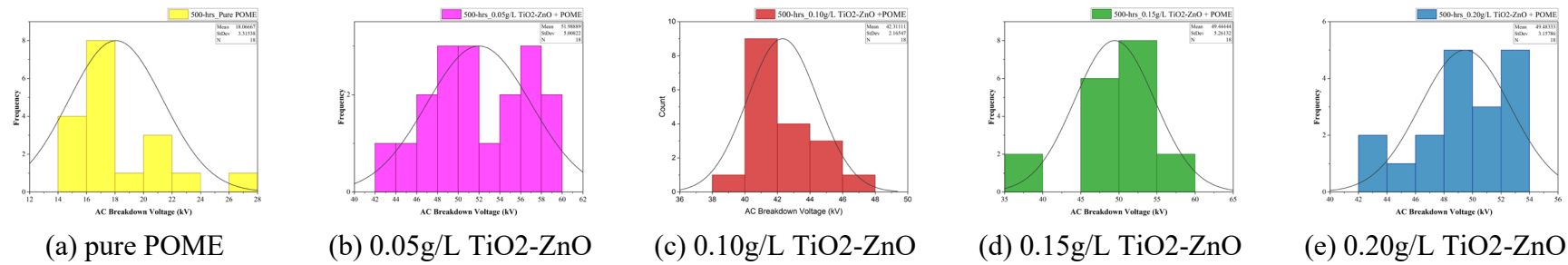


Figure 4.7: Histograms of 500-hrs ageing for: (a) Pure POME, (b) 0.05g/L TiO₂-ZnO, (c) 0.10g/L TiO₂-ZnO, (d) 0.15g/L TiO₂-ZnO, and (e) 0.20g/L TiO₂-ZnO

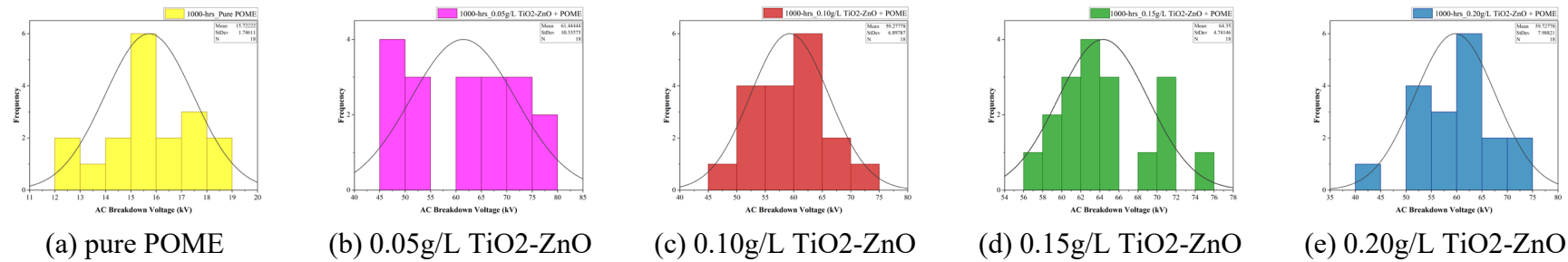


Figure 4.8: Histograms of 1000-hrs ageing for: (a) Pure POME, (b) 0.05g/L TiO₂-ZnO, (c) 0.10g/L TiO₂-ZnO, (d) 0.15g/L TiO₂-ZnO, and (e) 0.20g/L TiO₂-ZnO

Table 4.4: Hypothesis Test of Conformity to Normal Distribution of Pure POME and POME-based TiO₂-ZnO HNFs

Measurement	Statistic	<i>p</i> -value	Conformity to normal distribution (5%)
Ageing Time: 0-hr			
Pure POME	0.29102	0.56826	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.25248	0.69544	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.43949	0.25992	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.19077	0.88415	Accepted
0.20g/L TiO ₂ -ZnO + POME	0.3500	0.4323	Accepted
Ageing Time: 50-hrs			
Pure POME	0.42589	0.28125	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.64452	0.0776	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.65763	0.07177	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.5314	0.15029	Accepted
0.20g/L TiO ₂ -ZnO + POME	1.17532	0.0033	Rejected
Ageing Time: 500-hrs			
Pure POME	0.58545	0.11035	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.35662	0.41669	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.2337	0.76069	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.5195	0.16157	Accepted
0.20g/L TiO ₂ -ZnO + POME	0.43775	0.26256	Accepted
Ageing Time: 1000-hrs			
Pure POME	0.26694	0.64477	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.67822	0.06348	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.23153	0.76794	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.44222	0.25581	Accepted
0.20g/L TiO ₂ -ZnO + POME	0.2214	0.80071	Accepted

The histograms of pure POME and all four concentrations of POME-based TiO₂-ZnO HNFs for both nonaccelerated and accelerated thermal ageing were found to be mainly

symmetrical based on Figure 4.5, Figure 4.6, Figure 4.7, and Figure 4.8. The data collected, except for 0.20 g/L POME-based TiO₂-ZnO HNFs at 50-hour ageing, were all acceptable in conformance to normal distribution tests summarised in Table 4.4. Table 4.5 shows the kurtosis and skewness based on the normal distribution histogram to substantiate the aforementioned claims.

Table 4.5: Skewness and Kurtosis of Pure POME and POME-based TiO₂-ZnO HNFs

Measurement	Skewness	Kurtosis
Ageing Time: 0-hr		
Pure POME	0.31379	0.60504
0.05g/L TiO ₂ -ZnO + POME	-0.2367	-0.69888
0.10g/L TiO ₂ -ZnO + POME	0.14464	-1.33192
0.15g/L TiO ₂ -ZnO + POME	-0.01867	-0.73824
0.20g/L TiO ₂ -ZnO + POME	-0.56674	-0.60239
Ageing Time: 50-hrs		
Pure POME	0.25778	-1.0153
0.05g/L TiO ₂ -ZnO + POME	-0.46603	-1.19505
0.10g/L TiO ₂ -ZnO + POME	-1.19833	2.02425
0.15g/L TiO ₂ -ZnO + POME	0.96739	1.04186
0.20g/L TiO ₂ -ZnO + POME	-1.38125	1.13738
Ageing Time: 500-hrs		
Pure POME	1.30334	2.23929
0.05g/L TiO ₂ -ZnO + POME	-0.26331	-0.93427
0.10g/L TiO ₂ -ZnO + POME	0.3619	-0.08389
0.15g/L TiO ₂ -ZnO + POME	-0.99128	1.11791
0.20g/L TiO ₂ -ZnO + POME	-0.72561	-0.25341
Ageing Time: 1000-hrs		
Pure POME	-0.35727	-0.35335
0.05g/L TiO ₂ -ZnO + POME	-0.16026	-1.52688
0.10g/L TiO ₂ -ZnO + POME	0.4922	-0.07556

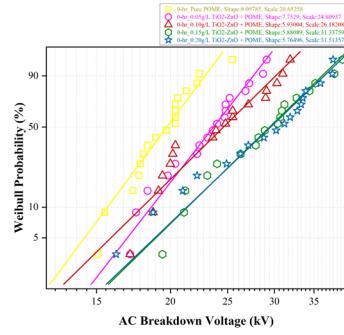
Table 4.5: continued

0.15g/L TiO ₂ -ZnO + POME	0.54831	-0.61693
0.20g/L TiO ₂ -ZnO + POME	-0.61693	1.07235

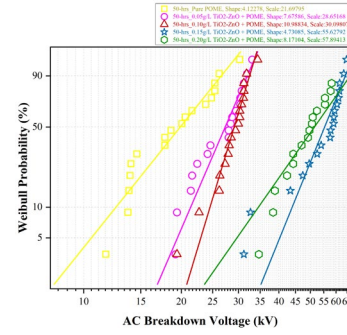
The skewness and kurtosis for pure POME were 0.31379 and 0.60504, respectively, at 0-hr ageing. Positive kurtosis suggested the distribution had a greater peak, whereas positive skewness values indicated the distribution's tail was on the right side. It showed the opposite of the positive values for the negative skewness and kurtosis values, which were -0.3572 and -0.35335, respectively, at ageing 1000-hrs for pure POME. When the skew values are close to zero, it indicates that the normal distribution is symmetric. If the kurtosis value is less than three, it indicates that the normal distribution is leptokurtic, or has a high peak for positive values and a platykurtic, or flat peak, for negative values (Shukri et al., 2021).

4.3.3 Weibull Distribution of AC BDV

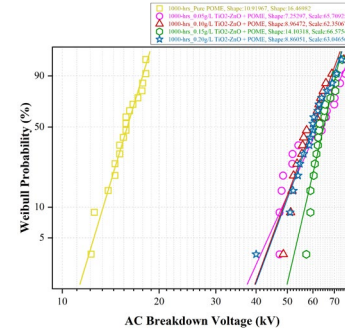
The AC BDV distributions of the pure POME and all POME-based TiO₂-ZnO HNFs for Weibull distribution were shown in Figure 4.9 (a), (b), (c), and (d) for nonaccelerated and accelerated thermal ageing, respectively, and were similar to normal distribution. The colour indications matched the normal distribution, but in the Weibull plotting case, the AC BDV plot values for pure POME, 0.05 g/L TiO₂-ZnO HNF, 0.10 g/L TiO₂-ZnO HNF, 0.15 g/L TiO₂-ZnO HNF, and 0.20 g/L TiO₂-ZnO HNF, respectively, were represented by the square, circle, triangle, hexagonal, and star, and they were the same for all ageing times. The data, *p*-values, and hypothesis test of conformance to Weibull distribution charting are also included in Table 4.6.



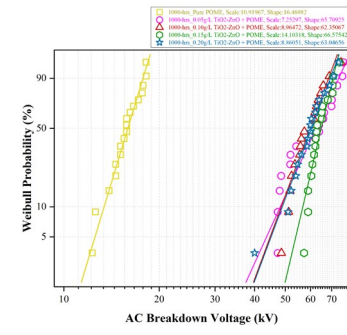
(a) 0-hr



(b) 50-hrs



(c) 500-hrs



(d) 1000-hrs

Figure 4.9: Weibull Probabilities Plot of Pure POME and POME-based TiO₂-ZnO HNFs for ageing at: (a) 0-hr, (b) 50-hrs, (c) 500-hrs, and (d) 1000-hrs

Table 4.6: Hypothesis Test of Conformity to Weibull Distribution of Pure POME and POME-Based TiO₂-ZnO HNFs

Measurement	Statistic	<i>p</i> -value	Conformity to Weibull distribution (5%)
Ageing Time: 0-hr			
Pure POME	0.29102	≥ 0.25	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.21605	≥ 0.25	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.47263	0.23076	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.19506	≥ 0.25	Accepted
0.20g/L TiO ₂ -ZnO + POME	0.29487	≥ 0.25	Accepted
Ageing Time: 50-hrs			
Pure POME	0.42734	≥ 0.25	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.58407	0.12337	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.34928	≥ 0.25	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.74237	0.04576	Rejected
0.20g/L TiO ₂ -ZnO + POME	0.94368	0.01464	Rejected
Ageing Time: 500-hrs			
Pure POME	0.85238	0.02355	Rejected
0.05g/L TiO ₂ -ZnO + POME	0.43676	≥ 0.25	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.45921	0.24369	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.24747	≥ 0.25	Accepted
0.20g/L TiO ₂ -ZnO + POME	0.30946	≥ 0.25	Accepted
Ageing Time: 1000-hrs			
Pure POME	0.30302	≥ 0.25	Accepted
0.05g/L TiO ₂ -ZnO + POME	0.63561	0.08809	Accepted
0.10g/L TiO ₂ -ZnO + POME	0.3843	≥ 0.25	Accepted
0.15g/L TiO ₂ -ZnO + POME	0.68201	0.06785	Accepted
0.20g/L TiO ₂ -ZnO + POME	0.80071	≥ 0.25	Accepted

Based on Table 4.6, it was evident that the AC BDV charting for the Weibull distribution rejected 0.15 g/L TiO₂-ZnO HNF and 0.20 g/L TiO₂-ZnO HNF at 50-hrs and

pure POME at 500-hrs, in contrast to normal distributions. While the normal distribution was acknowledged, the Weibull distribution charting did not match for 0.15 g/L TiO₂-ZnO HNF at 50-hrs and pure POME at 500-hrs. Weibull and normal distribution rejection occurred only for 0.20 g/L TiO₂-ZnO HNF at 50-hrs of ageing. The failure rate of 63.2% for the AD-goodness-of-fit test given in Table 4.6 will be predicted using the scale (α) and shape (β) displayed in Figure 4.9.

Table 4.7: AC BDV Failure Probabilities of Pure POME and POME-Based TiO₂-ZnO HNFs

Measurement	AC BDV (kV)			
	U _{1%}	U _{10%}	U _{50%}	U _{63.2%}
Ageing Time: 0-hr				
Pure POME	11.70212	15.64175	19.73868	20.652
0.05g/L TiO ₂ -ZnO + POME	13.70658	18.5591	23.66381	24.808
0.10g/L TiO ₂ -ZnO + POME	12.05331	17.91412	24.61286	26.181
0.15g/L TiO ₂ -ZnO + POME	14.33348	21.37368	29.44416	31.336
0.20g/L TiO ₂ -ZnO + POME	14.18902	21.32896	29.57243	31.512
Ageing Time: 50-hrs				
Pure POME	7.10949	12.57085	19.85227	21.696
0.05g/L TiO ₂ -ZnO + POME	15.73537	21.37105	27.31574	28.650
0.10g/L TiO ₂ -ZnO + POME	19.80277	24.52432	29.11072	30.097
0.15g/L TiO ₂ -ZnO + POME	21.03762	34.57068	51.48097	55.624
0.20g/L TiO ₂ -ZnO + POME	32.97117	43.95684	55.35466	57.892
Ageing Time: 500-hrs				
Pure POME	8.1703	12.72816	18.16009	19.459
0.05g/L TiO ₂ -ZnO + POME	37.68633	45.36314	52.63282	54.176
0.10g/L TiO ₂ -ZnO + POME	34.48418	38.75418	42.55635	43.338
0.15g/L TiO ₂ -ZnO + POME	35.61841	43.03877	50.08977	51.589
0.20g/L TiO ₂ -ZnO + POME	40.75851	45.63168	49.95616	50.843
Ageing Time: 1000-hrs				

Table 4.7: continued

Pure POME	10.80769	13.40257	15.92620	16.469
0.05g/L TiO ₂ -ZnO + POME	34.84800	48.18130	62.47127	65.706
0.10g/L TiO ₂ -ZnO + POME	45.46830	48.50901	59.85293	62.348
0.15g/L TiO ₂ -ZnO + POME	48.04593	56.75655	64.86755	66.574
0.20g/L TiO ₂ -ZnO + POME	37.51343	48.90581	60.49185	63.044

Table 4.7 displays the data about four different failure probabilities of AC BDV at 1%, 10%, 50%, and 63.2%. These failure probabilities of POME-based TiO₂-ZnO HNFs were compared to the values from pure POME as the values of HNFs remain higher. Consistent with the average of AC BDV, the values for AC BDV for HNFs grew with increasing age at all probability levels. In this instance, the use of TiO₂ and ZnO NPs as HNFs produced an exceptional result, particularly for 0.05 g/L TiO₂-ZnO HNF, despite prolonged exposure to high heat temperatures.

4.4 Physicochemical Properties

The physicochemical properties of the research were covered in this section. These properties include the visual appearance through NP's sedimentation assessment of the POME-based TiO₂-ZnO HNFs, the measurement of density using ASTM D1298 with a limit of no more than 0.91 g/cm³, the kinematic viscosity of natural ester-based oil measured at 27°C and 40°C based on ASTM D455, and the functional groups' characterisation using FTIR spectra and UV-Vis.

4.4.1 Visual Appearance

This research explores the utilisation of POME-based TiO₂-ZnO HNF as a potential insulating oil in transformers. In general, the visual appearance of a nanofluid, including its

apparent colour and less suspended particulate, was among the visible indicators for assessing its suitability in insulating oil applications. The concentration and composition of the NPs in a nanofluid can affect how different colours seem to the eye, as N'cho et al. (2016) found in their study. The study conducted by N'cho et al. (2016) discussed the visual appearance of various colours within a nanofluid was influenced by the concentration and composition of the NPs. However, Choi & Eastman (1995) and Yu & Xie (2012) contended that the assessment on its appearance was inappropriate as it only provides visual information regarding dispersion and agglomeration tendencies. Its visual appearance also frequently corresponded with degradation.



(a) 0-hr



(b) 50-hrs



(c) 500-hrs



(d) 1000-hrs

Figure 4.10: Visual Appearance of Pure POME and POME-based TiO₂-ZnO HNFs for Ageing at: (a) 0-hr, (b) 50-hrs, (c) 500-hrs, and (d) 1000-hrs

Figure 4.10 (a), (b), (c), and (d) displayed the visual appearance of the pure POME and POME-based TiO₂-ZnO HNFs for nonaccelerated (0-hr) and accelerated thermal ageing (50-hrs, 500-hrs, and 1000-hrs), respectively. The TiO₂-ZnO HNFs showed a consistent turbid appearance when without undergoing a thermal ageing process, and over several days, there were less noticeable sedimentation of nanoparticles settling at the bottom of Duran bottle. In this case, transparency of fluid decreased as NP concentrations in HNFs increased because of the increased light scattering that these particles produced. Extended exposure to high temperatures resulted in the pure POME colour turning intensely dark after ageing 500-hrs and 1000-hrs, as seen in these figures. Similarly, the TiO₂-ZnO HNF at 0.20 g/L also

had the darkest brownish colour with the most sedimentation of all the concentrations as the ageing time increased.

The sedimentation of NPs in a sealed container was used to evaluate the stability of one NF by many researchers (Ettfaghi et al., 2017). Therefore, these layers of sedimented NPs suggested low stability and NP accumulation due to strong Van Der Waals forces in the NFs (Ilyas et al., 2017; Wanatasanappan et al., 2022). In contrast to MNF, an investigation by Kulandaivel et al. (2024) found that palm-based HNF exhibited little sedimentation. The employment of various NPs, TiO₂ and ZnO in this research showed a consistent finding of sedimentation that was equivalent to palm-based HNF, which is not a turbidly thick layer, even though the 0.20 g/L TiO₂-ZnO HNF exhibited the most sedimentation (Kulandaivel et al., 2024). The observed colour difference raised the possibility that oxidation had occurred and influenced the thermal conductivity and other thermal properties of these pure POME and TiO₂-ZnO HNFs. These visual appearances provided an insight into assessing only their physicochemical properties further, however, it did not validate the reason for the enhancement of the electrical properties which may be due to the hybridisation properties of the TiO₂ and ZnO NPs.

4.4.2 Density

POME-based TiO₂-ZnO HNF application as a potential insulating oil in power transformers may not solely rely on their visual appearance. It is crucial to consider measuring and analysing the density of the insulating oil because density and viscosity correlate with oil performance in transformers. Based on IEEE Standards Association (2018), the density of an insulating oil must not exceed 0.91 g/cm³, as specified by the ASTM D1298 standard, to prevent the ice formation in cold climates. Falatah & El-Naggar

(2018) mentioned that the transformer oil samples collected and analysed have the density range between 0.8803 and 0.8850 gml⁻¹, which falls within the required limit. Following IS 1866:2000, 0.89 gm/cm³ was the density for insulating oil of new power transformers at a high voltage of more than 72.5 kV (Mahanta & Laskar, 2017). Figure 4.11 and Table 4.8 depicts the pure POME and POME-based TiO₂-ZnO HNFs density at standard room temperature, 27 °C for nonaccelerated (0-hrs) and accelerated thermal ageing (50-hrs, 500-hrs, and 1000-hrs).

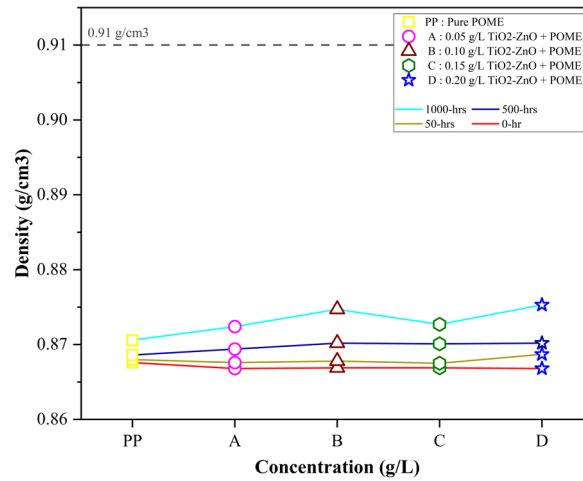


Figure 4.11: Density of Pure POME and POME-Based TiO₂-ZnO HNFs

Table 4.8: Density of Pure POME and POME-Based TiO₂-ZnO HNFs

Measurement	Density (g/cm ³)
Ageing Time: 0-hr	
Pure POME	0.8676
0.05g/L TiO ₂ -ZnO + POME	0.8668
0.10g/L TiO ₂ -ZnO + POME	0.8669
0.15g/L TiO ₂ -ZnO + POME	0.8669
0.20g/L TiO ₂ -ZnO + POME	0.8668
Ageing Time: 50-hrs	
Pure POME	0.8680

Table 4.8: continued

0.05g/L TiO ₂ -ZnO + POME	0.8676
0.10g/L TiO ₂ -ZnO + POME	0.8678
0.15g/L TiO ₂ -ZnO + POME	0.8675
0.20g/L TiO ₂ -ZnO + POME	0.8687
Ageing Time: 500-hrs	
Pure POME	0.8686
0.05g/L TiO ₂ -ZnO + POME	0.8694
0.10g/L TiO ₂ -ZnO + POME	0.8702
0.15g/L TiO ₂ -ZnO + POME	0.8701
0.20g/L TiO ₂ -ZnO + POME	0.8702
Ageing Time: 1000-hrs	
Pure POME	0.8706
0.05g/L TiO ₂ -ZnO + POME	0.8724
0.10g/L TiO ₂ -ZnO + POME	0.8747
0.15g/L TiO ₂ -ZnO + POME	0.8727
0.20g/L TiO ₂ -ZnO + POME	0.8753

At 27°C, the RBDPO density was measured to be 0.9057 g/cm³, nearly passing the minimum necessary limit for an insulating oil. It was successfully reduced to 0.8676 g/cm³ by modifying the RBDPO into POME. Table 4.8 showed that pure POME and all POME-based TiO₂-ZnO HNFs had densities ranging between 0.8676 and 0.8753 g/cm³. As the concentrations increased, the densities tended to increase as well. Similar findings by A. H. Ali et al. (2019) discovered that an increased NP addition influenced density to be higher. Long-term exposure to heat may also cause tiny molecules to evaporate, commonly known as oxidation (Engeländer et al., 2022). Although these densities met the standard density of transformer oil, the data does not definitively conclusive evidence that the HNF's density is

suitable for insulating oil. As Sai et al. (2020) discussed, temperature fluctuations may influence the oil's density, especially at lower temperatures.

4.4.3 Kinematic Viscosity

One crucial technique to assess an insulating oil's ability to perform as an insulating oil in transformer is kinematic viscosity. The transformer insulating oil required limit for usage must not exceed 50 cSt, while the kinematic viscosity of a standard insulating oil utilised was recorded to be 12.5 cSt at 40 °C (Hassan et al., 2013; Masra, Arief, Rigit, et al., 2023). The RBDPO was measured to be 56.97 cSt in a study by Nor et al. (2019). The palm oil tended to exhibit higher viscosities of 45.9 and 38.2 cSt; however, the transesterification procedure led the kinematic viscosity of POME to decrease to 4.5 cSt (Derawi & Salimon, 2014; Lamba et al., 2024) It indicated an influential factor in lowering the high viscosity of palm-based oil through the transesterification process. While this investigation examines the variations in kinematic viscosity at room temperature, 27 °C and 40 °C, as tabulated in Figure 4.12 and Table 4.9, between pure POME and TiO₂-ZnO HNFs.

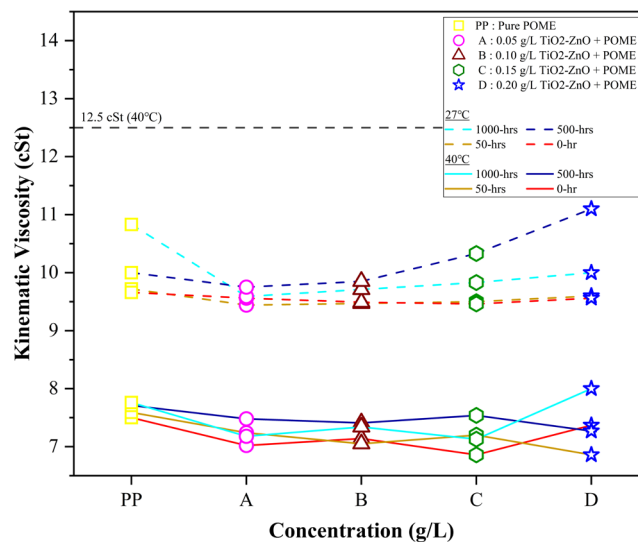


Figure 4.12: Kinematic Viscosities of Pure POME and POME-Based TiO₂-ZnO HNFs

Table 4.9: Kinematic Viscosity of Pure POME and POME-Based TiO₂-ZnO HNFs

Measurement	Kinematic Viscosity (cSt)	
	27 °C	40 °C
Ageing Time: 0-hr		
Pure POME	9.72	7.50
0.05g/L TiO ₂ -ZnO + POME	9.44	7.02
0.10g/L TiO ₂ -ZnO + POME	9.47	7.14
0.15g/L TiO ₂ -ZnO + POME	9.50	6.86
0.20g/L TiO ₂ -ZnO + POME	9.60	7.37
Ageing Time: 50-hrs		
Pure POME	9.66	7.59
0.05g/L TiO ₂ -ZnO + POME	9.56	7.24
0.10g/L TiO ₂ -ZnO + POME	9.49	7.05
0.15g/L TiO ₂ -ZnO + POME	9.46	7.20
0.20g/L TiO ₂ -ZnO + POME	9.56	6.86
Ageing Time: 500-hrs		
Pure POME	10.83	7.71
0.05g/L TiO ₂ -ZnO + POME	9.59	7.48
0.10g/L TiO ₂ -ZnO + POME	9.71	7.41
0.15g/L TiO ₂ -ZnO + POME	9.83	7.54
0.20g/L TiO ₂ -ZnO + POME	10.00	7.27
Ageing Time: 1000-hrs		
Pure POME	10.00	7.76
0.05g/L TiO ₂ -ZnO + POME	9.75	7.18
0.10g/L TiO ₂ -ZnO + POME	9.85	7.34
0.15g/L TiO ₂ -ZnO + POME	10.33	7.13
0.20g/L TiO ₂ -ZnO + POME	11.10	8.00

As tabulated in Table 4.9, the kinematic viscosities of thermal ageing for both nonaccelerated (0-hr) and accelerated (50-hrs, 500-hrs, and 1000-hrs) varied 9.44 to 11.10

cSt at 29 °C and 6.86 to 8.00 cSt at 40 °C. The investigated pure POME's kinematic viscosity was able to be lowered to 7.50 cSt at 40 °C by modifying the RBDPO into POME through a transesterification procedure while still meeting the necessary limit for insulating oil, in contrast to the viscosities of palm-based oil and RBDPO that stated previously. Based on these data, the viscosities decreased with increasing temperature. In a study by Ghislain et al. (2022), they discovered comparable patterns while examining the effects of FeO₃ NP on kernel POME at 40 °C, 60 °C, 80 °C, and 100 °C. It was caused by the NP and base fluid molecule's bond weakening with increasing temperature (Urmi et al., 2022). The kinematic viscosity of pure POME was slightly higher than the TiO₂-ZnO HNFs until 500-hrs, despite increased concentrations at both temperatures.

However, after 1000-hrs of ageing, the 0.20g/L of TiO₂-ZnO HNF had the highest viscosity at both temperatures. The possible explanation for this might involve the relationship between the viscosity of NF and the weight percentage of the NP used, as well as the possibility of NP clustering in base fluid (Karimi et al., 2024; Urmi et al., 2022). Furthermore, the marginally longer ageing time could be causing a rise in high molecular HNFs caused by the oil's oxygenated functional groups, which was a product of the oxidation process that occurred in the thermal oven (Oparanti, Fofana, Jafari, Zarrougui, et al., 2024). Nevertheless, the low viscosities of these POME-based TiO₂-ZnO HNFs continued to be lower than pure POME with the previously reported transformer oil utilised, indicating a good performance in terms of their cooling and heat transfer characteristics in the transformer.

4.4.4 Spectroscopy of FTIR Analysis

The infrared absorbance spectra of pure POME and the four concentrations of POME-based TiO₂-ZnO HNFs were measured using FTIR spectroscopy. These spectra were illustrated in Figure 4.13 (a), (b), (c), and (d), including a non-accelerated thermal ageing time of 0-hr and accelerated thermal ageing times of 50-hrs, 500-hrs, and 1000-hrs. The x-axis indicates the wavenumbers (cm⁻¹) while the y-axis indicates the measured sample's absorbance rate (a.u) in the figures. Similar to those investigated by Masra, Arief, Rigit, et al. (2023), the wavenumber region lies within the range of 500-4000 cm⁻¹.

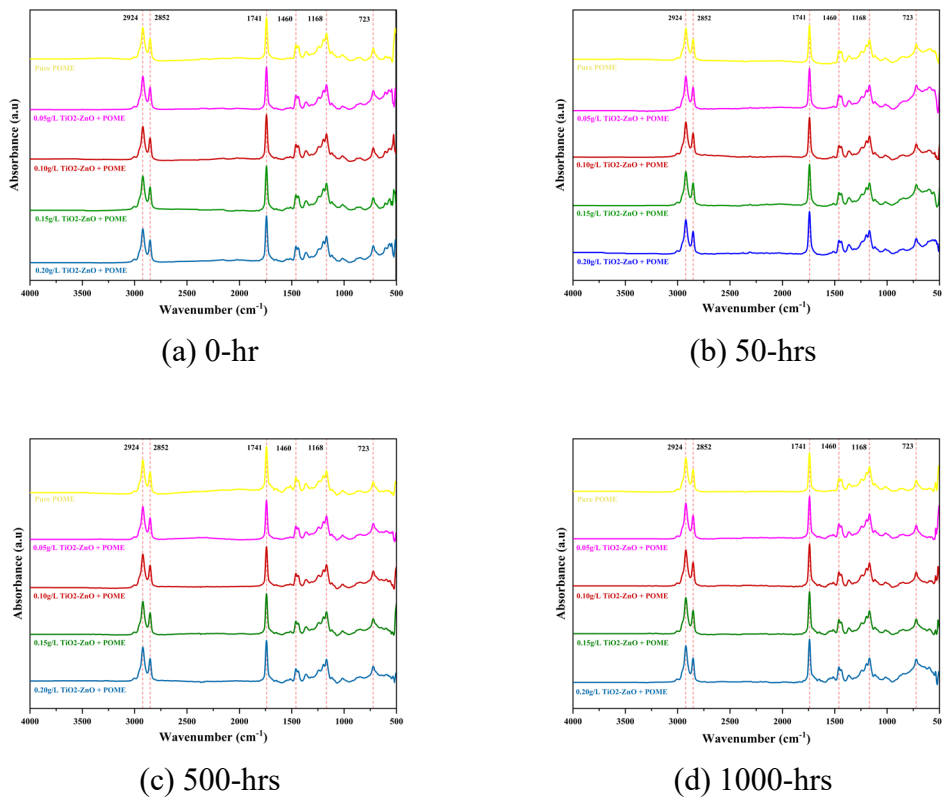


Figure 4.13: FTIR Spectra of Pure POME and POME-based TiO₂-ZnO HNFs for Ageing at: (a) 0-hr, (b) 50-hrs, (c) 500-hrs, and (d) 1000-hrs

Figure 4.13 illustrates several visibly identical peaks at 2924, 2852, 1460, 1168, and 723 cm⁻¹ for non-accelerated and accelerated ageing times. The asymmetric and symmetric

stretching vibrations of lipid compounds comprising -CH₃, -CH₂, and -CH correlate to the strong absorbance peak at 2924 and 2852 cm⁻¹, respectively (Foschi et al., 2022; Jacob, Preetha, & Sindhu, 2020; Wagh, 2020; Windarsih et al., 2022). The indication of C-H stretching of carboxylic acids and alkanes in POME, a type of fatty acid commonly present in natural ester-based oil or vegetable oil, was addressed by Masra, Arief, Sahari, et al. (2023) on these spectra ranges. Another extended, strong absorbance peak was noticed at 1741 cm⁻¹ wavenumber, signifying the ester functional group, carbonyl of -C=O stretching vibrational (Iwański et al., 2023; Masra, Arief, Rigit, et al., 2023; Yuliani et al., 2018).

At a wavenumber of 1460 cm⁻¹, a methylene functional group of -CH₂ bending was observed (Baruah et al., 2020; Irnawati et al., 2019; Yuliani et al., 2018). At 1168 cm⁻¹ wavenumber, a stretching vibration of C-O resulting from asymmetric vibrations of C-C(=O)-O bonds, indicative of a methyl ester group, was observed, while at 723 cm⁻¹, the C-H vibration of the methylene group found in alkanes was apparent (Mazlan et al., 2020; Mekonnen, 2023). These identical peaks indicated that they belong to the same functional group. Both TiO₂ and ZnO NPs added to the base fluid of pure POME did not significantly alter the structural compound of POME. Moreover, Masra, Arief, Rigit, et al. (2023) found that increasing the concentration of NPs did not entirely change the composition's overall structure. Similarly, the extended exposure to extreme temperature also demonstrated unnoticeable changes in POME-based TiO₂-ZnO HNFs molecular structure.

4.4.5 Spectroscopy of UV-Vis Analysis

The absorbance of pure POME and the four concentrations of POME-based TiO₂-ZnO HNFs were also measured using UV-Vis spectroscopy with wavelengths ranging from 300 nm to 700 nm. These absorption spectra were illustrated in Figure 4.14 (a), (b), (c), and

(d), including a non-accelerated thermal ageing time of 0-hr and accelerated thermal ageing times of 50-hrs, 500-hrs, and 1000-hrs. Based on (Hussein et al., 2020), UV-Vis was usually used to assess both agglomeration and the stability of the HNFs as they age over time due to the presence of nanoparticles when exposed to UV or visible light. In this study, UV-Vis was used to observe the interaction effects of the multiple NPs (TiO₂-ZnO) following accelerated thermal ageing on the HNF optical stability.

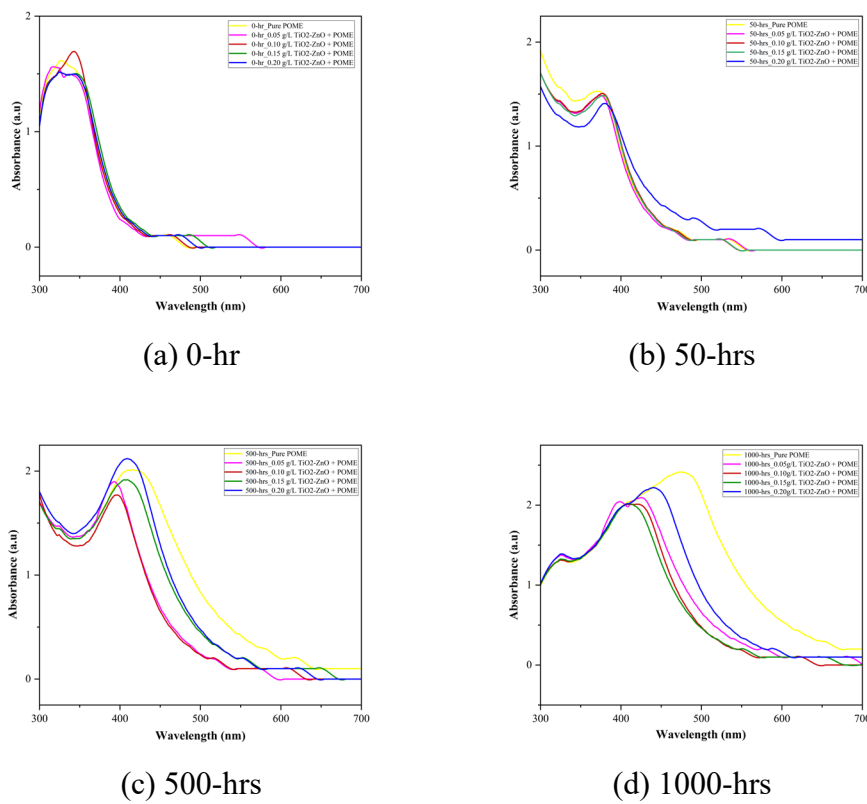


Figure 4.14: UV-Vis of Pure POME and POME-based TiO₂-ZnO HNFs for Ageing at: (a) 0-hr, (b) 50-hrs, (c) 500-hrs, and (d) 1000-hrs

Figure 4.14 provides an overview of the dispersion performance of TiO₂ NP and ZnO NP when dispersed into POME and exposed to thermal ageing under a prolonged time in addition to the visual appearance observation. At 0-hr, the POME-based TiO₂-ZnO HNFs and pure POME showed a similar absorbance peak within wavelength 315 nm to 360 nm,

indicating favourable dispersion in the base fluid. Vukoje et al. (2022) studied ZnO NP scattered at the visible spectral radiation region up to 374 nm, while TiO₂ NP scattering at up to 329 nm. It implies that the hybrid between TiO₂-ZnO HNFs was within the NP range at all concentrations. The absorbance peak moved progressively into a further wavelength range of 360 nm to 380 nm as the ageing time increased to 50-hrs.

Similarly, the absorbance peak shifted further wavelength towards a more visible region from 390 nm to 450 nm at 500-hrs. The absorbance trends between pure POME and the HNFs remained comparable despite the samples showing observable sedimentation as the ageing period rose, as indicated in visual appearance section. It indicates that with TiO₂ NP and ZnO NP hybridisation prevents the TiO₂-ZnO HNFs from further degrading and undergoing obvious accelerated fluid transitions (Ali et al., 2018; Elibol et al., 2024; Nawaz et al., 2020; Zainuri et al., 2018). At 1000-hrs, the absorbance peak extended at wavelengths between 400 and 500 nm, indicating that the fluid's concentration intensity had increased for pure POME and TiO₂-ZnO HNFs as ageing time accelerated (Ridzuan et al., 2019).

Comparing pure POME to POME-based TiO₂-ZnO HNFs reveals a discernible shift peak towards a longer wavelength. The absence of the TiO₂ and ZnO NPs suggests that the pure POME experienced further oxidative degradation (Azizie et al., 2023; Radu et al., 2020). Even though the absorbance peak changed with accelerated thermal ageing for both pure POME and the POME-based TiO₂-ZnO HNFs, the HNFs formulation remained stable and had better identical absorbance peak movement than pure POME. A study conducted by Nazir et al. (2024) showed a similar finding when a coolant was modified into an HNF, the absorbance spectra did not show significant changes when observed through UV-Vis, which

suggested that the addition of NPs into HNFs did not alter the optical properties of the HNFs due to strong van der Waals forces and electrostatic reactions.

The UV-Vis spectra also determined the concentration besides the dispersion rate of the TiO₂ NP and ZnO NP. Higher concentrations with a good dispersion method of HNFs should result in a higher light absorbance (Lim et al., 2022). For this study, the absorbance peak indicated otherwise, as pure POME without the addition of NPs showed an almost similar peak with the concentration of 0.5 g/L, 0.10 g/L, 0.15 g/L, and 0.20 g/L TiO₂-ZnO HNFs with a consistency, except at 1000-hrs. This suggests that the TiO₂-ZnO HNF might be suitable for long-term use in actual transformer settings.

4.5 Chapter Summary

In short, this chapter thoroughly studied and properly explained the development of the TiO₂-ZnO HNFs by using POME. These samples were compared to the required limit standard of the insulating oil that has been widely employed in transformer insulation systems. Based on the preliminary analysis, the yield percentage of the produced POME showed sufficient efficient efficiency of a successful transesterification process with an average ranging from 98.9972% to 99.123%, meanwhile, FESEM analysis revealed the composition of TiO₂ and ZnO NPs, respectively. For the investigation of their properties, 0.05 g/L exhibited superior AC BDV than the others as it aged, including its physicochemical properties, such as kinematic viscosity and density were within the standard values for insulating oil within the ranges, with the highest 8 cSt at 40 °C and 0.8668 to 0.8753 g/cm³, respectively. Both FTIR and UV-Vis spectroscopy were utilised to determine the characteristics of the functional group and stability performance as concentration increases as well as the ageing time of the HNFs after modifications.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The intent of this experimental investigation was to explore the potential use of nanoparticles and palm oil in adopting the notion of hybrid-type nanofluid as a sustainable and renewable insulating liquid dielectric for transformer insulation applications, compared to the often-studied monotype nanofluids. This research focused on producing nanofluids by adding multiple nanoparticles, in this case, TiO₂ and ZnO NPs, into a base fluid derived from RBDPO or a palm-based oil. The RBDPO was successfully modified into POME using the process of transesterification, which achieved an average yield of more than 98%. POME is obtained before the preparation of TiO₂-ZnO HNFs. After that, the HNFs were then prepared using the two-step method with four different concentrations of 0.05g/L, 0.10g/L, 0.15g/L, and 0.20g/L. The POME-Based TiO₂-ZnO HNFs experienced accelerated thermal ageing for 0-hr, 50-hrs, 500-hrs, and 1000-hrs at a high temperature of 135 °C. Following that, the electrical and physicochemical properties were obtained through several test analyses. For both accelerated (50-hrs, 500-hrs, and 1000-hrs) and non-accelerated (0-hr) thermal ageing times, the AC BDV results showed an elevated incremental percentage for all concentrations of POME-based TiO₂-ZnO HNFs. With the concentration of 0.10g/L TiO₂-ZnO HNF at 1000-hrs ageing, the highest AC BDV value was 64.4kV at $\pm 309.3\%$. 0.05g/L TiO₂-ZnO was the only concentration that consistently showed a stable increase in AC BDV values and increment percentages, although not every time to the highest. For all ageing times, the density of pure POME ranges from 0.8676-0.8706 g/cm³, while that of TiO₂-ZnO ranges from 0.8668-0.8753 g/cm³. As the duration of accelerated thermal ageing

becomes longer, the density of HNFs also increases with concentrations, while maintaining the IEC standard for density at below than 0.91 g/cm³. For both accelerated (50-hrs, 500-hrs) and non-accelerated (0-hr) thermal ageing of pure POME, the highest kinematic viscosity was recorded. The kinematic viscosity was seen to be highest under accelerated thermal ageing over 1000-hrs, with values of 11 cSt and 8 cSt at temperatures of 27 °C and 40 °C, respectively. Similar to their density, the high kinematic viscosity of the POME-Based TiO₂-ZnO HNFs was recorded as similar to being within the IEC standard for mineral-based oil that is used as insulating oil is 12.5 cSt at 40 °C. Besides, darker nanofluids, which tend to be used as an indication of higher densities and kinematic viscosities, sometimes offer an initial insight into the dispersion and stability of the nanoparticles inside the HNFs. In terms of their visual appearance, the lower concentration of HNFs exhibited more brownish colouration over the early accelerated thermal ageing (50-hrs), whereas higher concentration showed greater brownish colouration over the prolonged accelerated thermal ageing (500-hrs and 1000-hrs). The spectrograph of FTIR analysis indicates that all the POME-Based TiO₂-ZnO HNFs have the same functional group as the pure POME although the absorbance rate ranging from 500-400cm⁻¹ indicates the presence of TiO₂ and ZnO NPs. Last but not least, the spectrograph of UV-Vis also shows the stability of the POME-Based TiO₂-ZnO HNF as it ages through its absorbance rate. Although with ageing time increases, the HNFs did not vary much with POME. This research on HNFs will contribute to the development of sustainable and high-performance liquid dielectrics for transformers that can overcome the limitations of traditional mineral-based oil and improve the reliability and efficiency of the power grid.

5.2 Limitations and Recommendations

First, the findings relating to electrical properties indicated a declining trend in the AC BDV value on the concentration of 0.15g/L and 0.20g/L of the TiO₂-ZnO HNFs at ageing times of 500-hrs from 50-hrs and a rise back at 1000-hrs, thus further investigation is necessary on this problem. However, since the AC BDV results showed a favourable trend on incremental percentage as the HNFs aged, the surfactant, in this case CTAB, might have led to their stable performance. Concerning other issues, the nanoparticles gradually sunk at the bottom of the Duran bottle despite their tiny sizes and prolonged sonication time. Thus, future research could involve using a probe sonicator, as carried out in certain studies, to sonicate or break the particles of nanoparticles rather than the ultrasonic cleaner. Most importantly, other nanoparticle hybridisations should be investigated as well, as this work only examined hybrid of TiO₂ and ZnO NPs.

Besides, all tests involving its specifications as a suitable alternative insulating oil must be conducted to meet the established standards set forth by the mineral-based oil. These tests include water content, acidity, flash point, pour point, fire point, dissolved gas analysis, and resistivity. A tool based on Finite Element Analysis (FEA), which is included in software, such as Quickfield, can be used to visualise the heat transfer of the nanofluid as it cannot be seen with the human eye. This tool can be used to simulate the heat transfer of nanofluid and determine the heat localisation tendency in the actual transformer. However, advanced equipment is required to test the dielectric constant or permittivity of the produced nanofluid as its value might differ from the measured unmodified natural ester to correlate the experimental results with simulation.

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APPENDICES

Appendix A: Conferences Attended

(1)

Conference : 7th Technology and Innovation International Conference (TECHON)
2023

Date : 12 July 2023

Location : Waterfront Hotel, Kuching, Sarawak, Malaysia

Role : Presenter

Title : “A Comparative Review on Palm Oil-Based Hybrid Nanofluid as
Biodegradable Liquid Dielectric Material”

(2)

Conference : 15th International Engineering Conference (ENCON) 2024

Date : 15 February 2024

Location : Waterfront Hotel, Kuching, Sarawak, Malaysia

Role : Presenter

Title : “Simulation Investigation on Heat Transfer Characteristics of Palm-
Based Oil as Biodegradable Dielectric for Power Transformer Oil
Application Using Quickfield Software”

Appendix B: Journal Publications

1. **Musa, E.**, Arief, Y. Z., Saing, L. A. H., Mubarakah, N., Masra, S. M. W., & Rufus, S. A. (2024). Simulation Investigation on Heat Transfer Characteristics of Palm-Based Oil as Biodegradable Dielectric for Power Transformer Oil Application Using Quickfield Software. *Journal of Advanced Research in Applied Sciences and Engineering Technology*. [Scopus - Under Review]
2. **Musa, E.**, Arief, Y. Z., Masra, S. M. W., & Hashim, N. I. (2024). Ageing effects on electrical and physicochemical properties of modified palm oil methyl ester in TiO₂-ZnO Hybrid Nanofluid as Transformer Insulation. *IEEE Transactions on Dielectrics and Electrical Insulation (IEEE TDEI)*. [Web of Science, Q2 - Submitted for Review]
3. **Musa, E.**, Arief, Y. Z., Masra, S. M. W., & Hashim, N. I. (2024). A Review on Palm-Based Hybrid Nanofluid as a Potential Biodegradable Liquid Dielectric Material. *Journal of Palm Research (JOPR)*. [Web of Science, Q4 - Submitted for Review]

Appendix C: Aton Paar DMA 35 Density Meter Specification

Technical specifications

Measuring range	Density: 0 g/cm ³ to 3 g/cm ³ Temperature: 0 °C to 40 °C (32 °F to 104 °F) Viscosity: 0 mPa·s to 1000 mPa·s
Accuracy	Density*: 0.001 g/cm ³ Temperature: 0.2 °C (0.4 °F)
Repeatability	Density: 0.0005 g/cm ³ Temperature: 0.1 °C (0.2 °F)
Resolution	Density: 0.0001 g/cm ³ Temperature: 0.1 °C (0.1 °F)
Reproducibility, s.d.	Density: 0.0007 g/cm ³
Ambient temperature**	-10 °C to +50 °C (14 °F to 122 °F)
Supported measuring units	Density, density @ xx °C, Specific Gravity (SG), alcohol %v/v, alcohol %w/w, alcohol US (°Proof), API Gravity, API SG, API Density, °Baumé, H ₂ SO ₄ % w/w, H ₂ SO ₄ @ 20 °C, °Brix, <u>extract</u> (°Plato), ten programmable custom functions
Data memory	1024 measurement results
Power supply	Two 1.5 V LR06 AA alkaline batteries
Sample volume	2 mL
Dimensions	140 mm x 138 mm x 27 mm (5.5 in x 5.4 in x 1.0 in)
Weight	345 g (12.2 ounces)
Interfaces	IrOBEX/IrLPT
Protection class	IP54
Available options	Elongated filling tube, transportation suitcase, portable thermal printer with IrDA interface, IrDA USB adapter, wristband, rubber housing

Appendix D: HZJQ-1B Transformer Oil BDV Tester Machine Specification

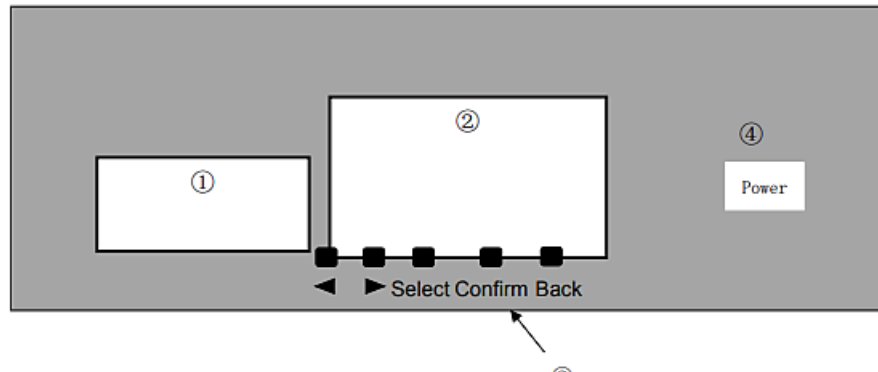
HUAZHENG ELECTRIC MANUFACTURING(BAODING)CO.,LTD.

Huazheng

III. Major Technical Indicators

- 1、Output voltage: 0~80KV (optional)
- 2、Voltage distortion rate: <3%
- 3、Voltage raising speed: 0.5~5KV/S (adjustable)
- 4、Standing time: 15min (adjustable)
- 5、Boosting interval: 5 min (adjustable)
- 6、Boosting frequency: 1~6 time(s) (optional)
- 7、Booster capacity: 1.5KVA
- 8、Measuring accuracy: $\pm 3\%$
- 9、Supply voltage: AC220V $\pm 10\%$ 50Hz ± 1 Hz
- 10、Power: 200W
- 11、Applicable temperature: 0℃~45℃
- 12、Applicable humidity: <75%RH
- 13、Overall dimension: 465×385×425

IV Panel Description



Appendix E: HZJQ-1B Transformer Oil BDV Tester Machine Specification

TECHNICAL DATA

Viscosity accuracy	1 %
Viscosity repeatability	0,2 %
Display	TFT
Motor rating output	4,8 W
Working temperature	min.: -100 °C max.: +300 °C
Speed	0,01 – 200 rpm
Setting accuracy speed	0,01 rpm
Temperature measurement resolution	0,1 K
Connection for ext. temperature sensor	PT 100
Graph function	Yes
Calibration option (temperature)	Yes
Overload protection	Yes
Operating mode	Timer and continuous operation
Attachment on stand	Extension arm
Interfaces	USB, RS 232, Analog output

GENERAL DATA

Weight	7,1 kg
Dimensions (W x H x D)	351 x 629 x 372 mm
Permissible ambient temperature	5 – 40 °C
Permissible relative humidity	50 %
Voltage	100 – 240 V
Frequency	50 / 60 Hz
Power input	24 W, Standby: 0,06 W
Protection class according to DIN EN 60529	IP 40

FOUR DEVICES. FOUR MEASURING RANGES.

ROTAVISC lo-vi

Viscosity measuring range	1 – 6 000 000 mPas
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ROTAVISC me-vi

Viscosity measuring range	100 – 40 000 000 mPas
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ROTAVISC hi-vi I

Viscosity measuring range	200 – 80 000 000 mPas
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ROTAVISC hi-vi II

Viscosity measuring range	800 – 320 000 000 mPas
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STAND FOR ROTAVISC

ROTASTAND

Stroke max.	200 mm
Diameter	16 mm
Dynamic load	5 kg
Dimensions (W x H x D)	351 x 318 x 246 mm
Weight	4,8 kg

HELISTAND

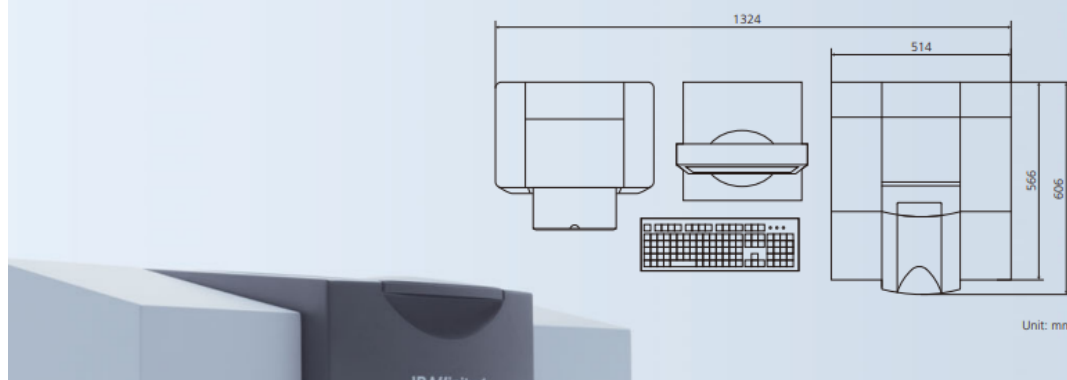
Stroke max.	142 mm
Diameter	16 mm
Dynamic load	3,2 kg
Overload protection	yes
Height adjustable safety switch	yes
Dimensions (W x H x D)	351 x 343 x 252 mm
Weight	5,5 kg
Permissible ambient temperature	0 - 40 °C
Permissible relative humidity	80 %
Protection class according to DIN EN 60529	IP 40
Voltage	220 - 230 / 100 - 115 V
Frequency	50/60 Hz
Power input max.	7 W

Appendix F: Fourier Transform Infrared Spectrophotometer IRAffinity-1 Specification

Hardware

Interferometer	Michelson interferometer (30° incident angle)
	Equipped with dynamic alignment system (JPN patent No. 2115670, No. 3613171)
	Sealed interferometer with auto dryer (JPN registration of utility model No. 3116465)
Optical system	Single-beam optics
Beam splitter	Germanium-coated KBr with 3 years guaranteed
Light source	High-energy ceramic light source
Detector	DLATGS detector equipped with temperature control mechanism
Wavenumber range	7,800 to 350 cm^{-1}
Resolution	0.5, 1, 2, 4, 8, or 16 cm^{-1}
S/N ratio	30,000:1 or higher (peak-to-peak, 4 cm^{-1} resolution, in a neighborhood of 2,100 cm^{-1} , 1-minute accumulation)
Mirror speed	4-step selection of 2.0, 2.8, 5, or 9 mm/sec
Data sampling	He-Ne laser
Gain	Automatic or manual setting ($\times 1$ to $\times 128$)
Sample compartment	Equipped with automatic accessory recognition mechanism
	200 (W) $\times$ 230 (D) $\times$ 170 (H) mm
	Center focus
Dimensions	514 (W) $\times$ 606 (D) $\times$ 273 (H) mm
Weight	35 kg

Dimensions



Appendix G: Gantt Chart

	Year 1						Year 2						Year 3					
	Semester 1			Semester 2			Semester 1			Semester 2			Semester 1					
Experiment Setup & Preparation																		
Research Methodology Webinar & Presentation																		
Writing: Background, Literature Review & Methodology																		
Experimental Work: POME & HNFs preparation																		
Journal Writing: Review Paper and Conference (1)																		
Proposal Defense Thesis & Presentation																		
Experimental Work: Thermal Ageing																		
Journal Writing: Research Article and Conference (2)																		
Data Collection & Analysis																		
Writing: Result and Discussion, and Conclusion																		
Thesis Writing & pre-ViVa Preparation																		
Journal Writing: Research Article (3)																		
Final Thesis Completion & ViVa Preparation																		