



Faculty of Engineering

**Accelerated Thermal Ageing on Electrical and Physicochemical
Performance of Palm Oil Methyl Ester-Based Nanofluids as Liquid
Insulation in Transformers**

Sharifah Masniah Wan Masra

**Doctor of Philosophy
2024**

Accelerated Thermal Ageing on Electrical and Physicochemical Performance
of Palm Oil Methyl Ester-Based Nanofluids as Liquid Insulation in
Transformers

Sharifah Masniah Wan Masra

A thesis submitted

In fulfillment of the requirements for the degree of Doctor of Philosophy

(Electrical Engineering)

Faculty of Engineering
UNIVERSITI MALAYSIA SARAWAK
2024

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.



.....

Signature

Name: Sharifah Masniah Wan Masra

Matric No.: 20010186

Faculty of Engineering

Universiti Malaysia Sarawak

Date : 28th June 2024

ACKNOWLEDGEMENT

First and foremost, all praises go to Allah for giving me the opportunity, determination, and strength to complete my studies. Thank you UNIMAS for the scholarship. I want to express my profound appreciation to my supervisor, Ir Ts Dr Yanuar Zulardiansyah Arief for his continuous support, encouragement, and invaluable expertise throughout this study. To my co-supervisors, Prof Ir Dr Andrew Ragai Henry Rigit, Assoc Prof Ir Ts Dr Siti Kudnie Sahari, and Assoc Prof Dr Md. Rezaur Rahman, I am highly grateful for their motivation, insightful feedback, constructive criticism, and encouragement during the various stages of my study. Their collective expertise and dedication have undoubtedly enriched the quality of my work.

I am indebted to all my colleagues, friends, and lab mates for their support and exciting discussions, which have fostered an environment of intellectual exchange and growth. I want to extend my gratitude to En Wahap Marni, En Shafri Senawi, and En Benedict Samling from the Faculty of Resource Science and Technology (FRTS) and En Airul Azhar Jitai from the Department of Chemical Engineering and Energy Sustainability for their valuable assistance in operating the equipment in the laboratory. I would like to thank the staff and technicians of the Faculty of Engineering for assisting me throughout my study.

My heartfelt thanks go to my husband, Mohd Saufee Muhammad; my daughters, Sofea Miyuki and Shaza Mizuki; and my parents and siblings for their love, understanding, belief, and moral support throughout this long journey. Thank you all for being an integral part of my pursuit of a PhD.

ABSTRACT

The paradigm shift from non-biodegradable and non-renewable mineral oil to acceptable biodegradable, renewable, and safer alternatives has prominently emerged in response to the United Nations Sustainable Development Goal (SDG)-7. However, their high viscosity, low dielectric strength, and poor oxidation stability prevent the broader usage as a replacement for dielectric liquid in transformers. Driven by the need for elevated properties of vegetable oils to align with the industry standards, the conversion process of the triglycerides, combined with modifications through the addition of nanoparticles and a surfactant, has been investigated. This thesis proposes enhancing the properties of refined, bleached, and deodorised palm oil olein (RBDPO_o) through chemical modification using a transesterification process to produce palm oil methyl ester (POME) as the base fluid. Additionally, semiconductive titanium dioxide (TiO₂) and conductive multi-walled carbon nanotube (MWCNT) nanoparticles, along with the hexadecyltrimethylammonium bromide (CTAB) surfactant, are added. Accelerated ageing experiments were conducted in a sealed condition at a temperature of 130 °C over a duration ranging from 0 to 1000 h for 0.01–0.10-g/L concentrations to understand their degradation behaviour. Overall, the findings demonstrated that all POME-based nanofluids showed an increase in AC BDV during the accelerated thermal ageing study, with the highest average value recorded at 57.08 kV, indicating a remarkable increment of 157.7% for POME-based TiO₂ nanofluid at a concentration of 0.05-g/L. After being subjected to accelerated thermal ageing, the BDV of POME-based TiO₂ nanofluids revealed a consistent trend across various doping concentrations. It was demonstrated that the proposed POME-based nanofluids performed comparably with other types of methyl ester oils in terms of breakdown voltage, kinematic viscosity, and flash point.

Keywords: Chemical modification, nanofluids, palm oil methyl ester, thermal ageing, transesterification

Penuaan Terma Dipercepat ke atas Sifat-sifat Elektrik dan Kimiafizik Nanobendalir Berasaskan Ester Metil Minyak Sawit sebagai Cecair Penebat di dalam Transformer

ABSTRAK

Anjakan paradigma dari minyak galian yang tidak terurai secara biologi dan tidak diperbaharui kepada alternatif yang boleh terurai secara biologi, boleh diperbaharui dan lebih selamat telah muncul sebagai tindak balas kepada Matlamat Pembangunan Mampan ke-7 (SDG-7) Pertubuhan Bangsa-bangsa Bersatu (PBB). Walaubagaimanapun, kelikatan tinggi, kekuatan elektrik rendah, dan kestabilan oksidasi yang lemah menghalang penggunaan meluas sebagai pengganti cecair dielektrik di dalam transformer. Dipacu oleh keperluan untuk meningkatkan sifat-sifat minyak sayuran agar selaras dengan piawaian industri, proses penukaran trigliserida, digabungkan dengan pengubahsuaian melalui penambahan nanopartikel dan surfaktan telah dikaji. Tesis ini mencadangkan peningkatan sifat-sifat olein minyak sawit ditapis, diluntur, dan dinyahbau (RBDPO_o), melalui pengubahsuaian kimia menggunakan proses transesterifikasi untuk menghasilkan ester metil minyak sawit (POME) sebagai cecair asas. Selain itu, nanopartikel semikonduktif titanium dioksida (TiO₂) dan nanopartikel konduktif nanotub karbon berbilang dinding (MWCNT), beserta surfaktan CTAB, ditambahkan. Eksperimen penuaan dipercepat telah dijalankan dalam keadaan tertutup pada suhu 130 °C selama tempoh antara 0 sehingga 1000 jam pada kelikatan 0.01–0.10-g/L untuk memahami tingkah-laku degradasi mereka. Secara keseluruhan, dapatan kajian menunjukkan bahawa semua nanobendalir berasaskan POME menunjukkan peningkatan dalam AC BDV semasa kajian penuaan terma dipercepat, dengan nilai purata tertinggi yang direkodkan pada 57.08 kV, menunjukkan peningkatan luar biasa sebanyak 157.7% bagi nanobendalir berasaskan POME-TiO₂ pada kelikatan 0.05-g/L. Setelah menjalani penuaan terma dipercepat, BDV nanobendalir berasaskan POME-

TiO₂ mendedahkan trend yang konsisten pada pelbagai kelikatan. Ini telah ditunjukkan bahawa nanofluid berasaskan POME yang dicadangkan menunjukkan prestasi yang standing dengan jenis minyak ester metal yang lain dari segi voltan keruntuhan, kelikatan kinematik, dan titik kilat.

Kata kunci: *Pengubahsuain kimia, nanobendalir, metil ester minyak sawit, penuaan terma, transesterifikasi*

TABLE OF CONTENTS

	Page
DECLARATION	i
ACKNOWLEDGEMENT	ii
ABSTRACT	iii
<i>ABSTRAK</i>	v
TABLE OF CONTENTS	vii
LIST OF TABLES	xii
LIST OF FIGURES	xiv
LIST OF ABBREVIATIONS	xvii
CHAPTER 1 INTRODUCTION	1
1.1 Study Background	1
1.2 Problem Statement	5
1.3 Research Questions	6
1.4 Research Hypothesis	7
1.5 Research Objectives	7
1.6 Scopes and Limitations of Study	8
1.7 Significance of Research	9
1.8 Thesis Outline	10
CHAPTER 2 LITERATURE REVIEW	12

2.1	Overview	12
2.2	Transformer Insulating Liquids	13
2.2.1	Mineral Oil	14
2.2.2	Synthetic Ester Oil	14
2.2.3	Natural Ester Oil	14
2.3	Methods to Improve Natural Ester Oils	16
2.3.1	Chemical Conversion of Triglycerides	16
2.3.2	Addition of Nanoparticles	19
2.3.3	Addition of Chemical Additives	26
2.4	Properties and Performance Characteristics for Transformer Oil	30
2.5	History of Palm Oil as Insulating Liquid	31
2.6	Properties of Palm Oil	32
2.7	Research Trends in Using Palm Oil as an Alternative to Mineral Oil	33
2.8	Properties of Palm Oil as Insulating Liquid	34
2.8.1	Physical Properties	35
2.8.2	Chemical Properties	37
2.8.3	Electrical Properties	44
2.9	Palm Oil-based Nanofluids	46
2.9.1	Development and Progress of Palm Oil-based Nanofluids	47
2.10	Preparation of Nanofluids	51

2.10.1	One-step Method	51
2.10.2	Two-step Method	51
2.11	Synthesis of Methyl Ester as the Base Fluid	53
2.12	Chapter Summary	56
CHAPTER 3 METHODOLOGY		59
3.1	Overview	59
3.2	Materials and Chemicals	61
3.2.1	Synthesis of the Understudy POME as the Base Fluids	61
3.2.2	Preparation of Nanofluids	62
3.3	Synthesis of POME by Transesterification Process	63
3.4	Characterisation of POME	66
3.4.1	FTIR Spectra Analysis for Functional Group Characterisation	66
3.4.2	GC–MS Analysis for Fatty Acid Composition	68
3.5	Characterisation of Nanoparticles	69
3.6	Preparation of POME-based Nanofluids	71
3.7	Methodology of Accelerated Thermal Ageing	74
3.8	Characterisation of Aged POME-based Nanofluids	75
3.8.1	FTIR Spectra Analysis	75
3.8.2	Ultraviolet–Visible Analysis	76
3.9	Experimental Setup	76

3.9.1	AC Breakdown Voltage Test	77
3.9.2	Characterising AC Breakdown Voltage Data Using a Probability Function	79
3.9.3	Resistivity	80
3.10	Measurement of Physicochemical Properties of Thermally Aged Nanofluids	81
3.10.1	Flash Point	82
3.10.2	Density	82
3.10.3	Kinematic Viscosity	83
3.10.4	Water Content	83
3.10.5	Acidity	84
3.10.6	Dissolved Gas Analysis (DGA)	84
3.11	Chapter Summary	84
	CHAPTER 4 RESULTS AND DISCUSSION	85
4.1	Overview	85
4.2	Characterisation of Laboratory-Synthesised POME	85
4.2.1	FTIR Spectra Analysis	85
4.2.2	Gas Chromatography–Mass Spectra Analysis	86
4.3	Characterisation of Used Nanoparticles	88
4.4	Characterisation of Aged Nanofluids	90
4.4.1	FTIR Spectra Analysis of Aged Nanofluids	91
4.4.2	Ultraviolet–Visible Analysis	93

4.5	Effects of Accelerated Thermal Ageing on Electrical Properties of POME-based Nanofluids	95
4.5.1	Analysis of Average AC Breakdown Voltage	95
4.5.2	Statistical Analysis of AC Breakdown Voltage	99
4.5.3	Resistivity	110
4.6	Effects of Accelerated Thermal Ageing on Physicochemical Properties of POME-based Nanofluids	111
4.6.1	Flash Point	111
4.6.2	Density	112
4.6.3	Kinematic Viscosity	112
4.6.4	Water Content	114
4.6.5	Total Acid Number (TAN)	116
4.6.6	Change of Gases Concentration Over Ageing Durations	118
4.7	Chapter Summary	126
	CHAPTER 5 CONCLUSION AND RECOMMENDATIONS	128
5.1	Conclusion	128
5.2	Recommendations for Future Work	131
	REFERENCES	132
	APPENDICES	156

LIST OF TABLES

	Page
Table 2.1	Natural ester oil-based nanofluids research works and their findings 21
Table 2.2	Summary of surfactants investigated as reported in the literature 28
Table 2.3	Basic characteristics of mineral oil vs natural ester oil [adapted from (Jacob et al., 2020; Raof, 2021; Rozga, 2013)] 30
Table 2.4	Fatty acids composition of CPO (Mohd et al., 2021) and RBDPO _o (Mohamad et al., 2018) 33
Table 2.5	International standards and specification of natural ester oils and mineral oil properties [adapted from (IEEE Std C57.147-2018, 2018; Judendorfer et al., 2014; Reffas et al., 2016; Sitorus et al., 2016)] 35
Table 2.6	Comparison of flash and fire points of palm oil with mineral oil and other types of natural ester oils (Abdullahi et al., 2004; Kanoh et al., 2008; Lewand, 2004; Suwarno et al., 2003) 37
Table 2.7	Fault types in the Rogers Ratio Method (RRM) (Golarz, 2016) 40
Table 2.8	Fault types in the gas ratio method as per IEC 60599 (Golarz, 2016; Piotrowski et al., 2019) 40
Table 2.9	Previous research on palm oil-based nanofluids and their findings 48
Table 2.10	Previous studies on methyl ester synthesis 54
Table 3.1	Properties of POME 62
Table 3.2	Selected nanoparticles used and their properties 63
Table 3.3	Nanofluids samples with different concentrations 72
Table 3.4	Test standards and equipment 82
Table 4.1	Major compositions of POME 87
Table 4.2	Average size of used nanoparticles 89
Table 4.3	Descriptive statistics of 36 measurements of 1000-h-aged nanofluids 99
Table 4.4	Hypothesis test of conformity to a normal distribution of 1000-h-aged nanofluids 102
Table 4.5	Skewness and kurtosis of AC BDV of 1000-h-aged nanofluids 103

Table 4.6	Hypothesis test of conformity to Weibull distribution of aged nanofluid samples	106
Table 4.7	Weibull parameters of 1000-h-aged oil samples	107
Table 4.8	AC BDV at 1%, 10%, and 50% probability levels	109
Table 4.9	Resistivity of oil 1000-h-aged nanofluid samples	110
Table 4.10	Comparison of flash points of different insulating oils	111
Table 4.11	Kinematic viscosity of nanofluid samples	114
Table 4.12	Gas concentration ratios based on RRM and IEC	120
Table 4.13	Results of fault prediction using various methods	124
Table 4.14	Comparative analysis of dielectric strength, kinematic viscosity, and flash point of POME with other methyl ester oils	125

LIST OF FIGURES

	Page
Figure 1.1 UN's SDG-7 (https://www.globalgoals.org/goals/7-affordable-and-clean-energy)	2
Figure 1.2 Number of publications related to vegetables- and natural ester oils-based transformer oil [adapted from (Suhaimi et al., 2022a)]	3
Figure 2.1 Bodies of literature and focus in this study	12
Figure 2.2 Transformer insulation system: (a) Solid insulation [adapted from (Montero et al., 2022)], and (b) Liquid insulation [adapted from https://www.electricalindia.in/transformer-oil-trends/]	13
Figure 2.3 Triglycerides molecular structure (where R ₁ , R ₂ , and R ₃ is hydrocarbon chain containing saturated and unsaturated carbon) (Mohd et al., 2021)	15
Figure 2.4 Research progress on developing methyl ester from natural ester oils	17
Figure 2.5 Production process of palm oil products [adapted from (Masra et al., 2022; Suhaimi et al., 2022a)]	32
Figure 2.6 Hydrolysis reaction of natural ester oil	38
Figure 2.7 Duval Triangle Method (Duval, 2002)	41
Figure 2.8 (a) Duval pentagon 1, and (b) Duval pentagon 2 (Duval and Lamarre, 2014)	43
Figure 2.9 Process of the preparation of nanofluids based on the two-step method	52
Figure 2.10 Transesterification reaction	55
Figure 2.11 Esterification reaction (Decarpigny et al., 2022)	55
Figure 2.12 Epoxidation reaction of MTCKO ^t to obtain EMTCKO ^o (Agu et al., 2019)	56
Figure 2.13 Chronology of palm oil-based insulating liquid development	58
Figure 3.1 Flowchart of the methodology	60
Figure 3.2 Separating methyl ester from glycerol in transesterification process	65
Figure 3.3 POME production process in the laboratory	65

Figure 3.4	Thermo Scientific Nicolet iS10 FTIR spectrometer	66
Figure 3.5	Flowchart of FTIR spectra analysis	67
Figure 3.6	QP2010 Plus GC spectrometer	68
Figure 3.7	Flowchart of GC–MS analysis	69
Figure 3.8	A JEOL JSM-IT5000HR FESEM equipment	70
Figure 3.9	A JEOL JEC-3000FC auto-fine coater	70
Figure 3.10	Flowchart of POME-based nanofluids preparation	72
Figure 3.11	Ultrasonication of POME-based NFs	73
Figure 3.12	Illustration of POME-based nanofluids preparation in the laboratory	73
Figure 3.13	Accelerated thermal ageing experiment using a convection oven	74
Figure 3.14	Agilent Cary 60 UV–Vis spectrophotometer	76
Figure 3.15	HZJQ-1B transformer oil BDV tester	78
Figure 3.16	Flowchart of AC BDV measurement	80
Figure 3.17	ADTR-2K PLUS test apparatus (Suhaimi et al. 2021)	81
Figure 3.18	DMA 35 Anton Paar handheld density meter	83
Figure 4.1	FTIR spectra of RBDPO _o and POME	86
Figure 4.2	GC–MS chromatogram of laboratory-synthesised POME	87
Figure 4.3	FESEM images of (a) TiO ₂ and (b) MWCNT nanoparticles showing their morphology at different magnifications	89
Figure 4.4	Visual observation of (a) TiO ₂ and (b) MWCNT nanofluid samples before (left) and after (right) the 1000-h accelerated ageing	90
Figure 4.5	FTIR spectra of (a) TiO ₂ and (b) MWCNT nanoparticles at varied concentrations and ageing durations	92
Figure 4.6	UV–Vis spectra of (a) TiO ₂ and (b) MWCNT nanofluids at varied concentrations and ageing durations	95
Figure 4.7	Scatter plots of AC BDV of pure POME, fresh, and 1000-h-aged POME-based (a) TiO ₂ and (b) MWCNT nanofluids at varied concentrations	96
Figure 4.8	Average AC BDV of 1000-h-aged POME-based nanofluids	98

Figure 4.9	Histograms of 1000-hour-aged POME-based TiO ₂ at (i) 0.01-g/L, (ii) 0.02-g/L, (iii) 0.05-g/L, and (iv) 0.10-g/L concentrations	100
Figure 4.10	Histograms of 1000-h-aged POME-based MWCNT at (i) 0.01-g/L, (ii) 0.02-g/L, (iii) 0.05-g/L, and (iv) 0.10-g/L concentrations	101
Figure 4.11	Weibull probability plots of AC BDV for aged POME-based (a) TiO ₂ and (b) MWCNT nanofluids	105
Figure 4.12	Change of kinematic viscosity at different ageing durations for POME-based (a) TiO ₂ and (b) MWCNT nanofluids	113
Figure 4.13	Change of water content at different ageing durations for POME-based (a) TiO ₂ and (b) MWCNT nanofluids	116
Figure 4.14	Change in TAN at different ageing durations for POME-based (a) TiO ₂ and (b) MWCNT nanofluids	117
Figure 4.15	Variation of gas content in 1000-h-aged POME-based nanofluids	119
Figure 4.16	Result analysis using DTM for POME-based TiO ₂ nanofluids	121
Figure 4.17	Result analysis using DTM for POME-based MWCNT nanofluids	122
Figure 4.18	Result analysis using DPM for 1000-h-aged POME-based (a) TiO ₂ , and (b) MWCNT nanofluids	123

LIST OF ABBREVIATIONS

AC BDV	Alternating Current Breakdown Voltage
Al ₂ O ₃	Aluminum Oxide
ASTM	American Society for Testing and Materials
BaTiO ₃	Barium Titanate
BDS	Breakdown Strength
BDV	Breakdown Voltage
GC–MS	Gas Chromatography–Mass Spectrometry
CH ₃ OH	Methanol
CJCO	Crude Jatropha Curcas Oil
CKO	Crude Karanji Oil
CO	Castor Oil
COME	Castor Oil Methyl Ester
CPKO	Crude Palm Kernel Oil
CPO	Crude Palm Oil
CP8	Crude Palm Oil Category 8
CP10	Crude Palm Oil Category 10
CSO	Cottonseed Oil
CTAB	Hexadecyltrimethylammonium Bromide
CuO	Copper (II) Oxide
DC BDV	Direct Current Breakdown Voltage
DGA	Dissolved Gas Analysis
DTM	Duval Triangle Method
Eh-BN	Exfoliated Hexagonal Boron Nitride

EMTCKO ^o	Epoxyl Methyl Ester of <i>Terminalia Catappa</i> Kernel Oil
FAME	Fatty Acid Methyl Ester
FESEM	Finite Element Scanning Electron Microscopy
Fe ₂ O ₃	Iron (III) Oxide
Fe ₃ O ₄	Iron (II, III) Oxide
FTIR	Fourier Transform Infrared
HMWH	High Molecular Weight Hydrocarbons
H ₂ SO ₄	Sulfuric Acid
IEC	International Electrotechnical Commission
IFT	Interfacial Tension
JCO	Jatropha Curcas Oil
JMEO	Jatropha Curcas Methyl Ester
KO	Karanja Oil
KOH	Potassium Hydroxide
KOME	Karanja Oil Methyl Ester
LI BDV	Lightning Impulse Breakdown Voltage
MEPKO	Methyl Ester of Palm Kernel Oil
MESSO	Methyl Ester of Sesbania Seed Oil
MPO	Modified Punga oil
MTCKO	Modified <i>Terminalia Catappa</i> Kernel Oil
MTCKO ^t	Transesterified Modified <i>Terminalia Catappa</i> Kernel Oil
MWCNT	Multi-walled Carbon Nanotube
NaOH	Sodium Hydroxide
PCB	Polychlorinated Biphenyl
PDEV	Partial Discharge Extinction Voltage

PDIV	Partial Discharge Inception Voltage
PFAE	Palm Fatty Acid Ester
PKO	Palm Kernel Oil
PKOAE	Palm Kernel Oil Alkyl Ester
PKOME	Palm Kernel Oil Methyl Ester
POME	Palm Oil Methyl Ester
R&D	Research and Development
RBDPO _o	Refined, Bleached, and Deodorised Palm Oil Olein
RRM	Rogers Ratios Method
SDBS	Sodium dodecylbenzene sulfonate
SDG	Sustainable Development Goal
SDS	Sodium Lauryl Sulphate
SEO	Synthetic Ester Oil
SiO ₂	Silicon Dioxide
TAN	Total Acid Number
Tan δ	Dielectric Dissipation Factor
TBHQ	Tert Butyl Hydroxyquinone
TCKO	<i>Terminalia Catappa</i> Kernel Oil
TiO ₂	Titanium Dioxide
UN	United Nations
UV–Vis	Ultraviolet–Visible
ZnO	Zinc Oxide
ZrO ₂	Zirconium Dioxide

CHAPTER 1

INTRODUCTION

1.1 Study Background

Petroleum-based oil, so-called mineral oil, has been utilised conventionally as a transformer liquid for decades and is widely established (Asano and Page, 2014; Fofana, 2013; Lv et al., 2017). It has emerged as the predominant insulating liquid of transformers due to its availability, good performance, low viscosity, and low cost. It is excellent as both a cooling and insulating liquid. However, the usage of mineral oil derived from non-renewable energy sources is affecting the environment because of its non-biodegradability properties, inflammable, and toxicity owing to its polyaromatic hydrocarbons content, which are potentially carcinogenic (Fofana, 2013; Jacob et al, 2020; Srivastava et al., 2021). In pursuit of a safer, non-flammable, and environmentally friendly insulating liquid for transformer applications, scholars and researchers have explored innumerable alternatives to mineral oil and have demonstrated significant results (Abdelmalik, 2014; Boss and Oommen, 1999; Mentlik et al., 2018; Stockton et al., 2007; Tokunaga et al., 2019; Totzauer and Trnka, 2019).

The shift of researchers' interest from conventionally nonbiodegradable mineral oil to biodegradable and renewable alternatives has greatly stood out as the most promising and directly relevant initiative in supporting the United Nations (UN) Sustainable Development Goal (SDG)-7, which aims to provide access to affordable, clean, and modern energy services, as illustrated in Figure 1.1.



Figure 1.1: UN's SDG-7 (<https://www.globalgoals.org/goals/7-affordable-and-clean-energy>)

Researchers have recently made notable attempts to develop high-performance, eco-friendly, or fully biodegradable insulating liquids that can substitute conventional mineral oil. A paradigm shift is sufficiently evident with tremendous attention, and many researchers redirecting their interests to biodegradable and renewable alternatives. Most biodegradable oil research for transformer applications has focused on vegetable oil or natural ester oil derived from seeds or other parts of plants. According to Suhaimi et al. (2022a), an aggregate of 900 articles was obtained between 2013 and 2022 from the IEEE Xplore database related to transformer oil based on vegetable or natural ester oils, as shown in Figure 1.2.

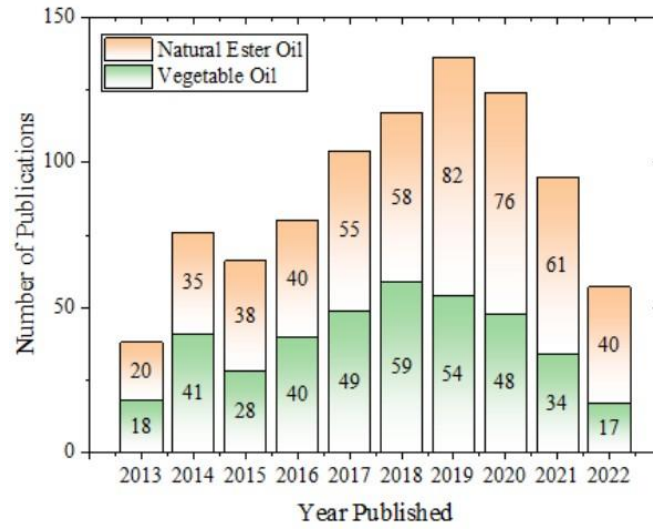


Figure 1.2: Number of publications related to vegetables- and natural ester oils-based transformer oil [adapted from (Suhaimi et al., 2022a)]

Around the early 1900s, experimental exploration of vegetable oils as dielectric coolants began (Fofana, 2013; McShane, 2002). Meanwhile, in the mid-1990s, Research and Development (R&D) laboratories commenced investigations to develop a fully biodegradable liquid (Oommen, 2002). Vegetable oil was considered the leading candidate and most promising option for such an insulating liquid (Fofana, 2013; Rafiq et al., 2015). As a non-fossil liquid alternative, vegetable oil offers a renewable, cost-saving, environment-friendly, fully biodegradable, sustainable, and safer alternative for transformers as an insulating and cooling medium (Amin et al., 2019; Fofana, 2013; Mahanta and Laskar, 2017; Rafiq et al., 2015; Shen et al., 2021).

The first commercial ester-based insulating liquid was BIOTEMP[®], which was derived from high oleic sunflower or rapeseed oil and patented in 1999 by ABB in the USA (Boss and Oommen, 1999; Oommen et al., 1997). It was followed by Envirotemp FR3[®] from soybean oil by Cooper Industries Inc. a year later (McShane et al., 2000). BIOTEMP[®],

Envirotemp FR3, and MIDELEn (M&I Materials) are today's leading brands of ester-based transformer fluids (Shen et al., 2021). The extensive literature on the aforementioned natural ester oils has ignited the interest of researchers from other countries, prompting them to initiate studies on their plant-based insulating liquids commercially available in local regions. For instance, palm oil by Abdullahi et al. (2004) and Azis et al. (2014) in Malaysia, also by Suwarno et al. (2003) in Indonesia, and by Saengkhumwong and Suksri (2017) in Thailand; palm kernel oil (PKO) by Oparanti et al. (2020), *Terminalia catappa* kernel oil (TCKO) by Menkiti et al. (2017), and olive oil by Jamo et al. (2019) in Nigeria; jatropha curcas oil (JCO) by Sitorus et al. (2016) in Indonesia; karanja oil (KO) by Maharana et al. (2018) in India; and castor oil (CO) by Nkouetcha et al. (2019) in Cameroon.

There has been extensive research into palm oil as an alternative transformer oil in Southeast Asia countries, especially Indonesia, Malaysia, and Thailand. Palm oil was chosen for several reasons. First, the trio is the world's leading palm oil producer. Palm oil is a potential source of biodegradable oil, making it possible to develop palm oil-based transformer oil to replace mineral oil. Secondly, previous empirical data has demonstrated that palm oil should be given preference as one of the options for transformer insulation. Pioneering works by Abdullahi et al. (2004) and Azis et al. (2014) have provided insights into the potential and suitability of palm oil as an oil insulation replacement. Since then, the study has evolved tremendously. As Suhaimi et al. (2022a) reported, most researchers prefer investigating refined, bleached, and deodorised palm oil olein (RBDPO_o) over other palm oil forms in their studies. Numerous published studies showcased the remarkable and unexpected outcomes of RBDPO_o (Jamail et al., 2018; Katim et al., 2018; Mohamad et al., 2016, 2018, 2019; Sinan et al., 2014; Suleiman and Muhamad, 2014; Yaacob and Alsaedi, 2015).

1.2 Problem Statement

The literature indicates that most studies on palm oil as a dielectric insulating liquid in transformers have focused on RBDPO_o. Although considered a potential alternative, RBDPO_o, like other vegetable oils, faces significant limitations for high-voltage applications. The primary challenges include high viscosity (McShane, 2000) and low dielectric strength (Bakruthen et al., 2018; Koutras et al., 2022; Peppas et al., 2016; Shen et al., 2021), and poor thermal and oxidative stability (Fofana, 2013; McShane, 2002).

To address these issues, research must focus on new material alternatives that can overcome these limitations. Chemical modification of natural ester oils represents an attractive alternative route for producing potentially usable oils to replace oil insulation. Several studies have explored chemically modifying natural ester oils for potential use as transformer liquids, but the specific modifications to palm oil remain underexplored. This study aims to fill this gap by investigating palm oil in its ester form, specifically converting RBDPO_o to palm oil methyl ester (POME) to achieve the desired properties for transformer liquids.

An extension of this chemical modification approach involves adding nanoparticles (1 to 100 nm in size) to create a mixture called nanofluid. Nanotechnology has shown promise in enhancing the thermal conductivity and electrical properties of insulating liquids (Ab Ghani et al., 2018; Kadim et al., 2021; Suhaimi et al., 2020). However, natural ester oil-based nanofluids stability challenges (Fofana, 2013; Jacob et al., 2020; Mariappan and Rengaraj, 2021). Adding small amounts of surfactants and antioxidants can improve stability, increase dielectric strength and enhance oxidation resistance (Ali and Salam, 2020;

Dey et al., 2017; Fofana, 2013; Khan et al., 2018). Until now, no findings have been found on the effect of surfactants on the electrical properties of POME-based nanofluids.

There is limited literature on the electrical, physical, and chemical properties of palm oil-based nanofluids subjected to thermal stress. An ageing study of nanofluids is essential to assess their performance under the transformer's actual and prolonged operating conditions (Jacob et al., 2020), but few studies have examined their thermal ageing evolution (Fernández et al., 2020; Maharana et al., 2019). A previous ageing study on RBDPO_o (Mohamad et al., 2018) under sealed conditions for 28 days showed no significant impact on alternating current breakdown voltage (AC BDV), possibly due to the short ageing duration.

This study focuses on the performance of POME-based nanofluids over a longer ageing duration. It aims to conduct laboratory experiments to uncover the behaviour of POME-based nanofluids subjected to an accelerated thermal ageing tests, an aspect that has not been comprehensively explored.

1.3 Research Questions

Based on the problems addressed, the present study attempted to answer the following questions:

- (i) Does the laboratory-synthesised POME meet the specifications of established standards as an insulating liquid?
- (ii) How do different types and concentrations of nanoparticles and accelerated thermal ageing collectively influence the electrical and physicochemical properties of POME-based nanofluids?

- (iii) How does the performance of POME-based nanofluids compare with that of other types of methyl ester oils?

1.4 Research Hypothesis

The utilisation of methyl ester oil as an alternative insulating liquid has been increasingly investigated in recent years to explore the potential of alternative materials in replacing conventional mineral oil. While many studies have examined different types of methyl ester oils as base fluids for nanofluid preparation, exploring POME remains limited. It is poorly understood whether chemically modified POME can optimise its performance as an insulating liquid by adding nanoparticles and surfactants. It appears that the use of titanium dioxide (TiO_2) nanoparticles and hexadecyltrimethylammonium bromide (CTAB) surfactant with RBDPO₆ showed AC BDV enhancement; however, their impact on POME has yet to be thoroughly investigated. Using multi-walled carbon nanotube (MWCNT) nanoparticles with high thermal conductivity is anticipated to enhance the insulating properties of POME. The endurance of POME-based nanofluid as a substitute for insulation depends on understanding of its compatibility in terms of chemical structure, and electrical and physicochemical characteristics, particularly under thermal ageing conditions. It is hypothesised that the utilisation of POME-based nanofluid will showcase improved insulating properties and greater stability during thermal ageing, making it a promising alternative to mineral oil in transformer insulation applications.

1.5 Research Objectives

To assess the suitability of POME-based nanofluids as transformer oil, the study aims to achieve the following objectives:

- i. To determine the compatibility of the laboratory-synthesised POME as the base fluid for nanofluids preparation in accordance with established standards.
- ii. To investigate the electrical and physicochemical properties of the prepared POME-based nanofluids with different types and various concentrations of nanoparticles, along with the surfactant, subjected to accelerated thermal ageing.
- iii. To evaluate the performance of POME-based nanofluids with other types of methyl ester oils in terms of electrical and physicochemical properties.

1.6 Scopes and Limitations of Study

The scopes of this research work are as follows:

- i. This research only considers two types of nanoparticles, namely semiconductive TiO_2 and conductive MWCNT, in assessing their influences on the POME-based nanofluids.
- ii. The concentrations of the prepared POME-based nanofluids used in this study are 0.01-g/L, 0.02-g/L, 0.05-g/L, and 0.10-g/L, with CTAB surfactant whose amount under investigation is equal to half the weight percentage of the nanoparticle's concentration.
- iii. The used nanoparticles are characterised using field emission scanning electron microscopy (FESEM). The characterisation of the laboratory-synthesised POME is performed using Fourier transform infrared (FTIR) and gas chromatography–mass spectrometry (GC–MS) analyses. The aged

POME-based nanofluids are characterised using FTIR and ultraviolet–visible (UV–Vis) spectrophotometer analyses.

- iv. Similar concentrations and durations at a specific operating temperature were considered in the ageing study.
- v. Since the nanoparticles are dispersed in the liquids, the study on the subject of ageing highlights the liquid's behaviour. This study only focused on assessing electrical, physical, and chemical properties such as AC BDV, resistivity, flash point, density, kinematic viscosity, water content, acidity, and dissolved gas analysis (DGA).
- vi. This study utilised ImageJ software to analyse the size of nanoparticles in a FESEM image. The particle analysis involved manually obtaining the diameter of 50 nanoparticles and calculating the average size using MS Excel.
- vii. The absence of in-house equipment necessitates the collaboration with Tenaga Nasional Berhad (TNB) Labs Sdn Bhd to conduct certain essential test and measurement activities on behalf of the research project.

1.7 Significance of Research

This study aims to obtain an alternative insulating ester-based derived from RBDPO_o through a chemical modification method that includes a transesterification process, nanoparticle dispersion, and the addition of a chemical surfactant additive. It is necessary to study the chemical modification-properties relationship of the nanofluids to properly prepare and develop a new methyl ester-based insulating liquid. The research outcomes have the

potential to yield new insights into the electrical and physicochemical properties of POME-based nanofluids.

The ageing study used accelerated thermal ageing to examine the degradation of ester-based nanofluids. To the authors' knowledge, the dielectric strength and physicochemical properties of POME-based nanofluids have not been investigated under the accelerated ageing study. The knowledge obtained from the ageing study can be used to understand the aged behaviours of POME-based nanofluids and consequently should provide a significant direction for future research work.

1.8 Thesis Outline

After the introduction and research background in Chapter 1, Chapter 2 presents the literature review of transformer insulating liquids, emphasising natural ester and palm oil, particularly as viable alternatives to conventional mineral oil. The chapter explores natural ester oil modification methods covering chemical conversion, nanoparticle incorporation, and the use of chemical additives. It comprehensively analyses the properties of palm oil as an insulating liquid, encompassing its physical, chemical, and electrical properties. Emerging research trends highlighting palm oil's option as a mineral oil replacement are discussed, along with palm oil-based nanofluids and their preparation methods. The synthesis of methyl ester as a base fluid is also detailed.

Chapter 3 outlines the materials and methods employed in this study, including the transesterification process for synthesising the methyl ester and preparing POME-based nanofluid samples. This section also provides a detailed description of the experimental setup, covering the characterisation of the nanoparticles and nanofluids, the accelerated

thermal ageing mechanism, and the procedures conducted to assess the performance of the prepared samples.

Chapter 4 presents the results following each of the objectives in the study. The results obtained from the characterisation of laboratory-synthesised methyl ester are discussed. It also includes comparing the ageing behaviour of POME-based nanofluids with different types of nanoparticles, doping concentrations, and ageing durations. The effects of ageing durations at a specific operating temperature on the electrical (AC BDV and resistivity) and physicochemical (flash point, density, kinematic viscosity, water content, acidity, and DGA) properties are studied.

Chapter 5 concludes the study by summarising the main findings and providing concluding remarks. Furthermore, this chapter offers recommendations for future research, following the list of references.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

This chapter will provide an overview of research development on the issues covered in this thesis. It establishes the framework that serves as the primary focus of the research presented herein. First, it begins with the background of mineral-based transformer oil, followed by a review of ester-based oils: synthetic ester oils and natural ester oils as potential alternative insulating liquids. Second, it discusses an overview of nanofluids using different base fluids with particular emphasis on mineral and natural ester oils through a brief review of some significant milestones in nanofluids. Finally, it ends with reviewing palm oil and palm oil-based nanofluids to discover their promising development as one of the options for transformer insulation. Figure 2.1 illustrates the bodies of the literature and research focus in this thesis.

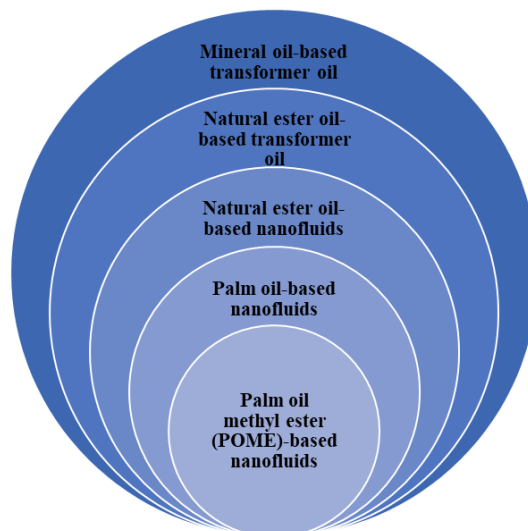


Figure 2.1: Bodies of literature and focus in this study

2.2 Transformer Insulating Liquids

The transformer insulation system comprises two necessary insulating materials: (a) solid insulation and (b) liquid insulation, as shown in Figure 2.2. The solid insulation is commonly composed of Kraft paper and pressboard. Insulating liquid is referred to by various terms such as transformer oil, insulating liquid, dielectric fluid, etc. As an essential part of the transformer, the insulating liquids are important in providing electrical insulation and cooling (Oommen, 2002), ensuring the transformer functions effectively and safely. Insulating liquids additionally provide analytical information on the operational condition of the transformer (Oommen, 2002; Raof, 2021). Generally, there are three categories of transformer-insulating liquids: mineral oil, synthetic ester oil, and natural ester oil (Mahanta and Laskar, 2017). Given that natural ester oils are the main focus of this study, the thesis primarily concentrates on more discussion restricted to them.



(a)



(b)

Figure 2.2: Transformer insulation system: (a) Solid insulation [adapted from (Montero et al., 2022)], and (b) Liquid insulation [adapted from <https://www.electricalindia.in/transformer-oil-trends/>]

2.2.1 Mineral Oil

The development of mineral oil as liquid insulation originated from the 1890s to the 1990s when paraffinic-mineral oil was used as a dielectric liquid in transformers due to its low viscosity, which provides superior insulation (Sheppard, 1986). However, after 1925, due to its high paraffin wax content, which is attributed to an elevated pour point, naphthenic-mineral oil was introduced as an alternative (Amin et al., 2019; Mahanta and Laskar, 2017), but this oil has the disadvantage of high flammability (Mahanta and Laskar, 2017). While the dominant worldwide usage of mineral oil is primarily attributed to its electrical and cooling properties, the main concern is its safety issue and poor biodegradability (Shen et al., 2021).

2.2.2 Synthetic Ester Oil

Synthetic ester oils are artificial esters created through the reaction of alcohol with short-chain fatty acids. These liquids have advantages over mineral oil due to their high pour point, high flash point, biodegradability, and lower water content (Khan et al., 2021). MIDEL 7131, produced by M & I Materials, is a well-known product in the synthetic-ester group (Jacob et al., 2020).

2.2.3 Natural Ester Oil

Natural ester oils are produced from vegetable oils, also known as green or biodegradable oils, derived from plants and seeds. They consist mainly of triglycerides, in which the molecular structure has three fatty chains chemically linked to a glycerol backbone, as shown in Figure 2.3, where R_1 , R_2 , and R_3 is hydrocarbon chain containing saturated and unsaturated carbon (Mohd et al., 2021). Fatty acids are the primary component of all-natural ester oils. The number of carbon atoms in the chain is usually between 12 and

24. Individual fatty acids are typically associated with a shorthand notation of two numbers separated by a colon. For example, palmitic acid, a saturated fatty acid with a chain of sixteen carbon atoms, is written as C_{16:0}, where the first number (16) indicates the number of carbon atoms, and the second number (0) indicates the number of double bonds is present in the chain (Hamid, 2011).

The fatty acid compositions act as unique characteristics among the various vegetable oils and esters; commonly present are stearic, palmitic, oleic, linoleic, and linolenic (Hamid, 2011). The composition of saturated, monounsaturated, and polyunsaturated influenced the physical and chemical properties of vegetable oils (Dupain et al., 2007; Totzauer and Trnka, 2019) and therefore linked with some drawbacks to the specific parameters: viscosity, oxidation stability, and pour point (Oommen, 2002; Oparanti et al., 2022; Rafiq et al., 2015).

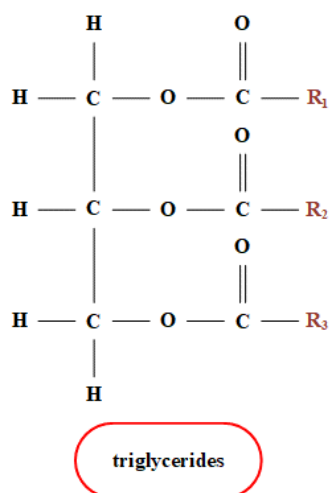


Figure 2.3: Triglycerides molecular structure (where R₁, R₂, and R₃ is hydrocarbon chain containing saturated and unsaturated carbon) (Mohd et al., 2021)

2.3 Methods to Improve Natural Ester Oils

From the perspective of the various properties to be considered in selecting natural ester oil as a new environmentally friendly insulating liquid, researchers eventually realised that the natural ester oil-based insulating liquid needed various modifications to overcome its shortcomings before being utilised in transformers. Researchers have investigated and proposed a few solutions to address these issues, which are believed to significantly enhance their potency for insulating liquids in transformers. The following subsection explains the solutions in detail.

2.3.1 Chemical Conversion of Triglycerides

Currently, chemical modification of natural ester oils represents an attractive route for producing potentially usable oils to replace oil insulation. Many studies reported and recognised the modification of natural ester oils as a key contributor to successfully exploring the possibility of employing the modified ones as transformer liquids. The literature in the last five years has been occupied with research investigating various methods with different types of natural ester oils as the base fluids. Figure 2.4 illustrates some of these remarkable attempts. Such studies include *Terminalia catappa* kernel oil (TCKO), jatropha curcas oil (JCO), karanji oil (KO), castor oil (CO), sesbania seeds oil (SSO), etc., showcasing a variety of sourcing options. Different proven modification methods for converting triglycerides into methyl esters, including direct purification, transesterification, esterification, and epoxidation-esterification, are prime candidates utilised and discussed in the literature.

Sitorus et al. (2016) presented a new natural ester oil called jatropha curcas methyl ester oil (JMEO), which is obtained by an alkali-base-catalysed esterification method of JCO

extracted from jatropha fruit seeds by mechanical pressing and filtering. The authors analysed the electrical and physicochemical properties of the JMEO, and the results indicated that the BDV of the JMEO is comparable to mineral oil. The average BDV of JMEO at 87.0 kV passes the average BDV of mineral oil (78.5 kV). It was discovered that the esterification reaction decreased the kinematic viscosity and acid number of JCO but not its water content. It is noted that the esterification reaction decreases the kinematic viscosity of JMEO from 30–55 cSt to 10.45 cSt. Except for the flash and pour points, the physicochemical properties of JMEO were compliant with the IEEE C57.147 standard (IEEE Std C57.147-2018, 2018).

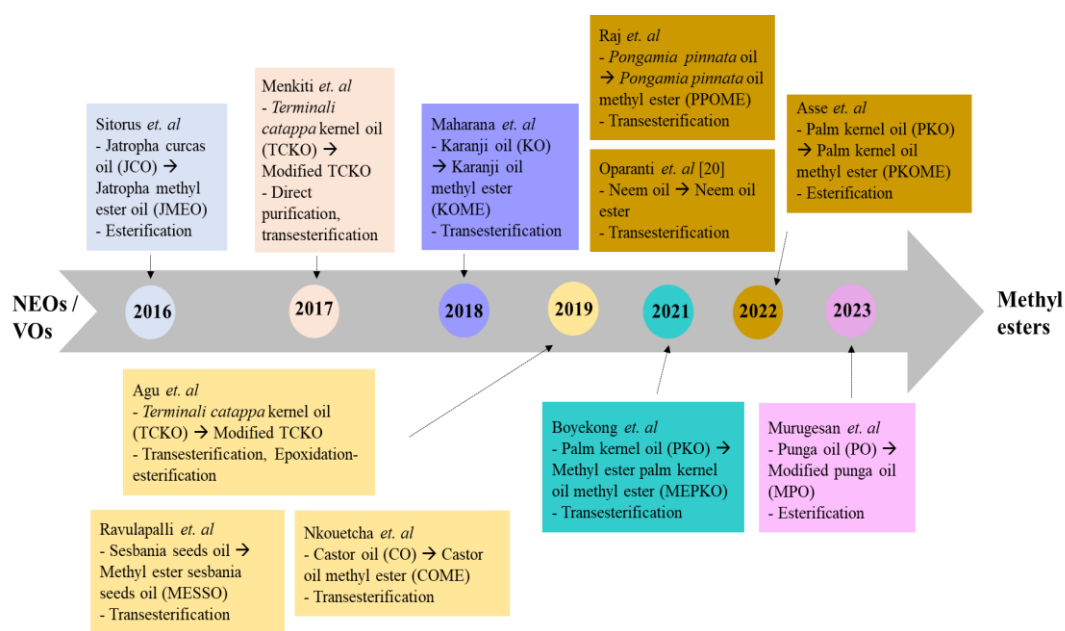


Figure 2.4: Research progress on developing methyl ester from natural ester oils

The TCKO that had been chemically modified using the direct purification-transesterification method carried out by Menkiti et al. (2017) and later using the sequential transesterification-epoxidation-esterification method by Agu et al. (2022) produced

modified TCKO with better physicochemical properties and dielectric strength compared to unmodified TCKO.

Some other important sources of triglycerides and edible methyl ester oils were recently studied by some researchers, such as karanji oil methyl ester (KOME), castor oil methyl ester (COME), methyl ester of sesbania seed oil (MESSO), and methyl ester of palm kernel oil (MEPKO). KOME is extracted from crude karanji oil (CKO) using a two-step transesterification process: acid and base transesterification. The findings suggested that KOME exhibits favourable physicochemical, thermal, and electric properties. Their electrical and thermal properties demonstrated superior performance to conventional mineral oil upon exposure to oxidative ageing (Maharana et al., 2018). Meanwhile, the studies conducted by Nkouetcha et al. (2019) and Ravulapalli et al. (2019) explored the viability of utilising castor oil (CO) and sesbania seed oil (SSO), respectively, as source materials for developing alternative insulating liquids. Their research findings also suggested promising results and merits for bio-based insulating liquids.

Modified punga oil (MPO) is an example of a non-edible methyl ester-based oil type that has recently attracted notice for its potential application as an insulating oil (Murugesan et al., 2023). The MPO was synthesised from punga oil using two sequential esterification phases: acid and base. The finding of this study revealed that the modification of punga oil leads to significant improvement in its electrical and rheological properties. The electrical properties demonstrated enhanced performance, likely attributed to the oil's molecular structure modification. Additionally, the rheological behaviour of the MPO displayed notable changes, indicating improved flow and viscosity attributes. These findings

emphasised the potential benefits of the chemical modification process in expanding the utility of punga oil.

Converting triglycerides into their methyl esters is a prerequisite step before their use as alternative insulating liquids. However, some parameters remain that need to be enhanced, such as dielectric strength, pour point, and oxidative stability (Murugesan et al., 2023; Okikiola et al., 2022), when using methyl ester-based oils as the base fluids for the transformers.

2.3.2 Addition of Nanoparticles

Introducing nanoparticles to enhance the performance of mineral oil and natural ester oils has been considered in many studies recently. Choi and Eastman (1995) proposed the term *nanofluid* to describe a mixture of a base fluid with good dispersion of nanoparticles with sizes ranging from 1 to 100 nm. Since then, a significant amount of research work on nano dielectrics has been conducted. According to the literature, the research on nanofluids is studied from various angles, from its preparation, stability, properties, and characteristics to challenges, progress, and applications (Ali and Salam, 2020; Amin et al., 2019; Babita et al., 2016; Bakthavatchalam et al., 2020; Kadim et al., 2021; Rafiq et al., 2021). The superior performance of mineral oil-based nanofluids by enhancing their insulation properties motivates the development of natural ester oil-based nanofluids.

Considerable work has been studied in the literature to demonstrate the natural ester oil-based nanofluids performance by dispersing nanoparticles based on their conductivity as conductive (Fe_2O_3 , Fe_3O_4 , CNT), semiconductive (TiO_2 , ZnO, CuO), and insulative (SiO_2 , Al_2O_3). Table 2.1 presents some studies on developing natural ester oil-based nanofluids, examining various properties such as AC BDV, viscosity, flash point, fire point, etc. In Table

2.1, the addition of nanoparticles shows its potential to enhance the dielectric strengths of the base fluids. Almost all studies agreed that the addition of nanoparticles, when prepared well and having optimum doping concentrations, could have the ability to address the persistent demand to develop a new generation of insulation systems with improved dielectric strength and thermal conductivity (Kadim et al., 2021), along with the requirement of being fully biodegradable owing to environmental concerns.

Table 2.1: Natural ester oil-based nanofluids research works and their findings

References	Base fluids	Nanoparticles	Findings
(Jariyanurat et al., 2017)	Natural ester oil	TiO ₂ , BaTiO ₃ , ZnO	The study found that adding nanoparticles to natural ester oil changed its AC BDV by 22%, 42%, and 69% with TiO ₂ , BaTiO ₃ , and ZnO, respectively. The impulse BDV for natural ester oil-based ZnO showed the highest improvement.
(Mohamad et al., 2017)	PFAE	Fe ₃ O ₄ , TiO ₂ , Al ₂ O ₃	The study compared how conductive Fe ₃ O ₄ , semiconductive TiO ₂ , and insulative Al ₂ O ₃ changed the AC BDV and viscosity of the PFAE oil. The results demonstrated that the PFAE-based Fe ₃ O ₄ showed the best dielectric performance. The authors concluded that adding the nanoparticles had an insignificant effect on the PFAE's viscosity.
(Madavan et al., 2018)	Honge oil, Neem oil, Mustard oil and Punna oil	Fe ₃ O ₄ , BN, Al ₂ O ₃	The authors found that when comparing the natural ester oils with Fe ₃ O ₄ , they showed the highest AC BDV among other combinations. The viscosity increased with the increased concentration of nanoparticles.

Table 2.1: continued

(Khan et al., 2018)	Rapeseed oil	Fe ₃ P	The results revealed a significant improvement in BDV at a particular concentration of nanoparticles. The optimisation of the nanoparticle's concentration needs to be further investigated to achieve maximum dielectric strength of the prepared nanofluids.
(Peppas et al., 2018)	FR3	Colloidal magnetic iron oxides (MIONs), SiO ₂	The results showed that adding MIONs increased the AC BDV, while adding SiO ₂ decreased the AC BDV. The reason for the SiO ₂ effect is these nanoparticles' low conductivity and permittivity, consequently reducing their capability to capture free electrons.
(Méndez et al., 2020)	Natural ester oil	Fe ₂ O ₃ , TiO ₂	The authors reported that the concentration of the nanoparticles influenced the AC BDV of natural ester oil. The best concentrations for Fe ₂ O ₃ and TiO ₂ have been identified to be 0.2 kg/m ³ and 0.5 kg/m ³ , respectively). The results showed that the thermal conductivity of the natural ester oil was improved between 2–38% with the addition of nanoparticles.

Table 2.1: continued

(Pyrgioti et al., 2020)	FR3	TiO ₂ with sorbitan monolaurate as surfactant	The authors revealed that the sorbitan monolaurate enhanced the dispersion of TiO ₂ in FR3 by showing no signs of sedimentation after one month of the nanofluid preparation. Consequently, the mean BDV of the surface-modified and TiO ₂ -based nanofluids improved compared to the base fluid.
(Khaled and Beroual, 2020)	MIDEL eN 1204	Fe ₃ O ₄ , SiO ₂ , Al ₂ O ₃	The study demonstrated that the DC BDV of the nanofluids depends on the types of added nanoparticles and their concentrations. The best increments were obtained with 0.2-g/L of Fe ₃ O ₄ and 0.3-g/L Al ₂ O ₃ . However, the DC BDV decreased regardless of SiO ₂ concentrations.
(Koutras et al., 2020a)	FR3	TiO ₂	This study showed that the increment of AC BDV in FR3-based TiO ₂ nanofluids was optimal at 0.020 vol% concentration. The TEM image of the nanofluid with the highest concentration (0.040 vol%) showed the largest aggregates, indicating an unstable nanofluid. The authors suggested investigating appropriate surface modifications to optimise the nanofluid's stability.

Table 2.1: continued

(Baruah et al., 2020)	Environtemp FR3	TiO ₂	The investigation demonstrated that natural ester oil-nanofluids performed better than the base fluids in their BDV before and after ageing. However, the BDV decreased after ageing due to sludge agglomeration and increased moisture content.
(Duzkaya and Beroual, 2021)	MIDEL eN 1204	ZnO	The most significant improvement in AC BDV was observed at a 0.1-g/L concentration. The authors hypothesised the presence of a critical threshold for ZnO concentration beyond 0.4-g/L.
(Raj et al., 2021)	Marula oil (MRO)	Al ₂ O ₃	The statistical analysis showed that adding Al ₂ O ₃ to MRO enhanced its electrical, physical, and thermal characteristics. The modified oil was subjected to thermal ageing for its stability assessment, revealing its behaviour following accelerated ageing. The treated MRO with Al ₂ O ₃ was found to be thermally stable and can withstand high temperatures for an extended period without significant degradation.

Table 2.1: continued

(Emara et al., 2022)	FR3	Pentyl-graphene nanosheets	The investigation demonstrated that 0.008% w/w was the best concentration for preparing nanofluids that worked well in terms of thermal and dielectric properties. Pentyl-graphene nanosheets exhibited excellent dispersibility without agglomeration at the optimum concentration.
(Pérez-Rosa et al., 2022)	NE Bioelectra	Fe ₃ O ₄	The study demonstrated that the presence of Fe ₃ O ₄ in the insulation system can significantly affect the ageing process of the oil. The findings indicate that this nanoparticle affects the oil's ageing by reducing its water content, which can help slow down its ageing.

2.3.3 Addition of Chemical Additives

Even though many studies have revealed that the dielectric nanofluids potentially possess superior dielectric properties to their base fluids, the stability of these liquids remains an important issue that needs to be resolved. Many factors have been studied with the expectation that they can significantly enhance the long-term stability of the prepared nanofluids, which include the addition of chemical additives such as surfactants (Amizhtan et al., 2022; Fofana, 2013; Huang et al., 2019). According to Das (2023), surfactants must be carefully chosen to allow their combined effect to achieve good dispersion of nanoparticles and overall performance.

In the literature, surfactants are added during the preparation of the nanofluids to modify the surface of the nanoparticles. Introducing a surfactant to the base fluid reduces the liquid's surface tension, which helps the nanoparticle dispersion and enhances the stability of the nanofluids (Gayathri et al., 2019; Yang and Hu, 2017). The presence of an appropriate surfactant with an optimal amount, preferably in low concentration, can improve the dispersion stability without causing adverse effects on the prepared nanofluids, such as a decrease in thermal conductivity and an increase in viscosity (Yang and Hu, 2017). The optimal amount of the surfactant remains a prime subject of ongoing investigation, as it varies depending on the specific base fluids, types of nanoparticles, and nanofluid preparation methods employed. For instance, in a recent study (Jacob et al., 2020), oleic acid was used as a surfactant to coat Al_2O_3 in soybean natural ester as the base fluid, reducing Van der Waals attractive force. Ethanol was used as a dispersant to ensure homogeneous dispersion of the nanoparticles in the base fluid, resulting in higher stability.

On the other hand, Pyrgioti et al. (2020) used sorbitan monolaurate surfactant, pairing it with FR3 as the base fluid and incorporating TiO₂ nanoparticles. A concentration of 3% by weight of the sorbitan monolaurate surfactant was used in the study. The authors observed no sedimentation even after a month from the preparation of the nanofluids. This contrasts with their earlier work without the surfactant, where sedimentation became noticeable two weeks after preparation.

Various surfactants have been investigated to establish which are compatible with the added nanoparticles and the base fluids. Surfactants are classified into three types (Babita et al., 2016): (i) anionic (negatively charged), (ii) cationic (positively charged), and nonionic (neutral). The studies in Table 2.2 reported some surfactants that were added to various natural ester oils as the base fluids to facilitate the mixing of the nanoparticles with the base fluids. Most of the studies demonstrated that surfactants help to enhance the stability of the prepared nanofluids by improving the dispersion of nanoparticles in the base fluids, which reduces the level of agglomeration or sedimentation of the particles.

Table 2.2: Summary of surfactants investigated as reported in the literature

Types of surfactant	Surfactant	Nanoparticles	Base Fluid	Critical Findings	References
Anionic	Sodium dodecyl sulphate (SDS)	Al ₂ O ₃	RBDPO	Introducing the SDS with Al ₂ O ₃ showed a promising impact on the partial discharge (PD) properties of RBDPO. With SDS, its PD amplitude and repetition rate improved by 38% and 80%, respectively.	(Mohamad et al., 2021)
	Sodium dodecylbenzene sulfonate (SDBS)	Graphene	Cottonseed oil (CSO)	The stability of the nanofluids is related to the addition of SDBS, synthetic antioxidant tert butyl hydroxyquinone (TBHQ), and sonication time. The nanofluids' stability increases as the sonication increases.	(Farade et al., 2020)
Nonionic	Sorbitan monolaurate	TiO ₂	FR3	The results showed that the mean BDV was improved by adding sorbitan monolaurate. The surface modification did not affect the performance of the dielectric strength of nanofluids.	(Pyrgioti et al., 2020)

Table 2.2: continued

Nonionic	Oleic acid	Al ₂ O ₃	FR3	The study showed that the AC BDV of the modified nanofluid with the addition of oleic acid was enhanced by 26.9% compared to the unmodified nanofluid. There were no signs of sedimentation in a range of eight months.	(Koutras et al., 2020b)
Cationic	Cethyl trimethyl ammonium bromide (CTAB)	TiO ₂	MIDEL 1215	The study indicated that the nanofluids with CTAB surfactant showed a higher corona inception voltage.	(Amalanathan et al., 2020)
		SiO ₂	FR3	A nanofluid with SiO ₂ exhibited an increment in corona inception voltage. A further increment can be seen when CTAB is added. However, dielectric properties decreased beyond a certain proportion of SiO ₂ and CTAB.	(Thakur and Sarathi, 2018)
		Al ₂ O ₃	RBDPO	The introduction of CTAB promotes lower agglomeration of Al ₂ O ₃ compared to SDS and oleic acid. The use of CTAB also caused an improvement in lightning BDV.	(Mohamad et al., 2020)

2.4 Properties and Performance Characteristics for Transformer Oil

Some basic parameters of natural ester oil should be considered to determine its suitability as an alternative to conventional mineral oil. Table 2.3 summarises the typical values for some physicochemical and electrical properties of mineral oil and natural ester oil.

Table 2.3: Basic characteristics of mineral oil vs natural ester oil [adapted from (Jacob et al., 2020; Raof, 2021; Rozga, 2013)]

Physicochemical properties			
	Units	Mineral oil	Natural ester oil
Density at 20°C	g/cm ³	0.83–0.89	0.9–0.919
Kinematic viscosity at 40°C	mm ² /s	3–16	29–45
Flash point	°C	110–150	225–330
Pour point	°C	-30–63	-5–-25
Dielectric properties			
	Units	Mineral oil	Natural ester oil
Breakdown voltage (IEC 60156 for 2.5 mm gap)	kV	≥30	≥35
Dielectric dissipation factor		<0.002	<0.003

Ideally, the desirable characteristics of insulating liquid used in transformers should have high flash and fire points (McShane, 2002), low viscosity (Kittikhuntharadol et al., 2023), excellent dielectric strength (Jacob et al., 2020), fully biodegradable (Fofana, 2013), and non-toxic. According to Fofana (2013) and McShane (2002), ester-based

liquids can mitigate the drawbacks associated with conventional mineral oil, particularly its adverse environmental effects. Moreover, ester-based liquids are eco-friendly, exhibiting a lower tendency for fire and explosion.

2.5 History of Palm Oil as Insulating Liquid

Palm oil is one of the vegetable oils being extensively studied for its feasibility and reality as a biodegradable transformer oil. Following these extensive studies and investigations, it has been demonstrated that palm-based oils have excellent potential as insulating liquids in transformers. Therefore, in the context of this study, it attempts to exploit palm oil for further investigation.

Palm oil is obtained from the mesocarp of the fruit of the oil palm tree, known as *Elaeis guineensis*. The British introduced it to Peninsular Malaysia (previously known as Malaya) in the 1870s as an ornamental plant from West Africa (“Fact Sheets Malaysian Palm Oil,” 2014). It can be harvested around the year during its long productive life (usually 25-30 years), ensuring a constant palm oil supply (“Fact Sheets Malaysian Palm Oil,” 2014). Two main products extracted from palm fruit: crude palm oil (CPO) from the mesocarp and crude palm kernel oil (CPKO) from the seeds. CPO is further refined to obtain RBDPO, which then undergoes the fractionation process to get RBDPO olein (RBDPO_o, edible oil) and RBDPO stearin (margarine fats, shortenings, etc.). On the other hand, CPKO can be further processed to obtain PKO products. Figure 2.5 briefly illustrates the production process of palm oil products.

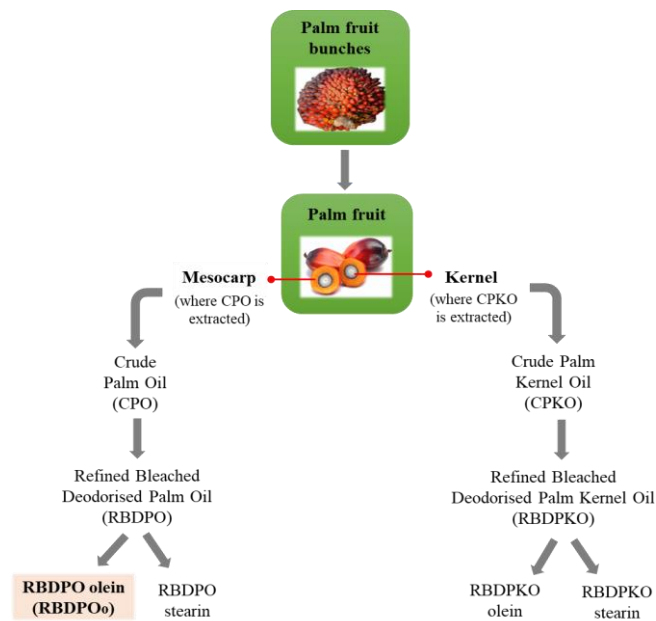


Figure 2.5: Production process of palm oil products [adapted from (Masra et al., 2022; Suhaimi et al., 2022a)]

2.6 Properties of Palm Oil

Different types of oils have different physical and chemical properties depending on the proportions of saturated, monounsaturated, and polyunsaturated fatty acids present (Ab Ghani et al., 2018). Table 2.4 shows the comparative composition of fatty acids in CPO and RBDPO_o.

Table 2.4: Fatty acids composition of CPO (Mohd et al., 2021) and RBDPO_o (Mohamad et al., 2018)

Types of fats	Types of fatty acids	CPO	RBDPO _o
Saturated	Caproic acid (C _{6:0})	—	—
	Caprylic acid (C _{8:0})	—	—
	Capric acid (C _{10:0})	—	—
	Lauric acid (C _{12:0})	0.0–0.5	43.0–45.4
	Myristic acid (C _{14:0})	0.9–1.5	0.9–1.1
	Palmitic acid (C _{16:0})	39.2–45.8	37.7–39.3
	Stearic acid (C _{18:0})	3.7–5.1	3.9–4.2
Monounsaturated	Oleic acid (C _{18:1})	37.4–44.1	41.1–43.0
Polyunsaturated	Linoleic acid (C _{18:2})	8.7–12.5	10.4–12.4
	Linolenic acid (C _{16:3})	0.0–0.6	0.2–0.3

2.7 Research Trends in Using Palm Oil as an Alternative to Mineral Oil

This section highlights the trends of research using palm oil as an insulating liquid for transformer applications in terms of article publications over the last 20 years, including the types of palm oil used, the areas studied, and the tests and assessments performed. Based on the literature survey, many palm oil products have been used as test subjects in research experiments. These include CPO, RBDPO, RBDPO_o, CPKO, PKO, and palm kernel oil alkyl ester (PKOAE). The key features discovered in these studies are summarised, compared, and discussed relative to commercialised natural ester oils and mineral oil, as reviewed by Suhaimi et al. (2022a).

Section 2.8 outlines the investigation of the basic properties of palm oil as an insulating liquid from studies which have been thoroughly conducted in Malaysia,

Indonesia, Thailand, Japan, Nigeria, and India. Most of the studies aimed to investigate palm oil's electrical and physicochemical properties and demonstrate its potential as an alternative to transformer oil.

2.8 Properties of Palm Oil as Insulating Liquid

Several key parameters of palm oil, including its physical, chemical, and electrical properties, must be rigorously analysed to determine its suitability and potential as an alternative insulating liquid to conventional mineral oil. Experiments carried out in the laboratory are based on standard testing methods according to the American Society for Testing Materials (ASTM), the International Electrotechnical Commission (IEC), and the Institute of Electrical and Electronics Engineers (IEEE). The properties of new unused natural ester oils for use in electrical equipment such as transformers are detailed in IEEE C57.147 (IEEE Std C57.147-2018, 2018) and IEC 62770 (IEC 62770, 2013). The properties of natural ester oils and the standard specifications for new unused natural ester oils and mineral oil are presented in Table 2.5 for comparison.

Table 2.5:International standards and specification of natural ester oils and mineral oil properties [adapted from (IEEE Std C57.147-2018, 2018; Judendorfer et al., 2014; Reffas et al., 2016; Sitorus et al., 2016)]

Properties	Standard Test Methods		Specification for natural ester		Specification for mineral oil
			IEEE C57.147	IEC 62770	IEEE C57.106
	ASTM	ISO/IEC			
Physical					
Flash point (°C)	D 92	ISO 2719	≥275	≥250	≥145
Fire point (°C)	D 92	ISO 2592		>300	
Density at 20 °C (g/cm ³)		ISO 12185		≤1.0	
Viscosity at 40 °C (mm ² /s)	D 445	ISO 3104	≤50	≤50	≤12
Electrical					
Breakdown voltage (kV)	D 877		≥30		
Breakdown voltage (kV), 2.5 mm gap		IEC 60156		≥35	≥30

2.8.1 Physical Properties

2.8.1.1 Viscosity

A liquid's viscosity measures its resistance to flow. In general, the viscosity of palm oil is much higher than that of typical mineral oil (12 mm²/s) at 40 °C, which has a wide range from 21 to 50 mm²/s depending on the types of palm oil forms (Azis et al.,

2014; Suwarno et al., 2003). Researchers have made considerable efforts to improve the undesirable viscosity of palm oil. Abdelmalik (2014) prepared a newly synthesised ester with lower viscosity (range between 4.6 to 7.0 mm²/s at 20 °C) by reducing the oil's fat content through transesterification and epoxidation processes.

Another study by Yaacob and Alsaedi (2015) investigated the kinematic viscosity of samples of CPO, RBDPO, and its derivatives (CP8 and CP10) with reduced fatty acid content. The viscosity of palm oil derivatives was reduced (6.3 mm²/s at 40 °C), which agrees with findings by (Abdelmalik, 2014; Aditama, 2005; Kanoh et al., 2008). Those findings suggested that reducing fat content leads to better performance of palm oil in terms of viscosity when chemical modification is used as a solution. This physical attribute is very crucial and cannot be discounted. A lower-viscosity liquid allows for a more efficient dissipation of heat (Lewand, 2004).

2.8.1.2 Flash point and fire point

Palm oil has a comparatively low flash point compared to other vegetable oils but is still substantially higher than mineral oil, as shown in Table 2.6. The flash point of RBDPO ranges between 220–330 °C; CPO ranges between 195–285 °C, and PFAE ranges between 176–178 °C. From the table, the minimum requirement for the flash point is 275 °C, and 300 °C for the fire point in accordance with ASTM D92 (ASTM D92, 2018). The elevated flash point and fire point are essential for ensuring higher safety standards during the in-service operation of the transformers.

Table 2.6: Comparison of flash and fire points of palm oil with mineral oil and other types of natural ester oils (Abdullahi et al., 2004; Kanoh et al., 2008; Lewand, 2004; Suwarno et al., 2003)

Parameter	ASTM D92	MO	BIOTEMP	FR3	RBDPO	CPO	PFAE
Flash point (°C)	≥275	145	328	326	220–330	195–285	176–178
Fire point (°C)	300	185	358	362	>220	206	n/a

n/a – not available

2.8.2 Chemical Properties

2.8.2.1 Water content

The presence of even a tiny droplet of water or moisture significantly impacts the BDV and dielectric loss (Wang et al., 2018). The effects of moisture on the performance of palm oil were studied by (Ab Ghani et al., 2016; Maneerot and Pattanadech, 2020; Murad et al., 2013; Shukri et al., 2021; Suleiman et al., 2014). The moisture measurement uses the Karl Fisher method. Murad et al. (2013) compared the BDV of red palm oil (carotina oil), RBDPO, and PFAE and identified which reacts better with moisture. PFAE had the highest BDV even when the moisture content was increased by 2500 ppm. Suleiman et al. (2014) and Ab Ghani et al. (2016) investigated the effects of moisture content on the BDV of palm oil-based insulating liquids and mineral oil. In their studies, PFAE exhibited excellent BDV. This was connected to its ability to absorb moisture from the oil (Ab Ghani et al., 2016).

Shukri et al. (2021) also investigated the effects of different moisture content resulting from the drying process at 0–48 h on AC BDV on RBDPO. The investigation

disclosed two key correlations: (i) the moisture decreases exponentially with drying time, and (ii) the AC BDV decreases exponentially with moisture levels. Such observations suggested that the drying process significantly reduces the moisture content of RBDPO_o.

2.8.2.2 Total Acid Number

Acidity, or the total acid number (TAN), refers to the presence of acids in the insulating liquid. The insulating liquid undergoes oxidation or degradation processes aided by oxygen availability, leading to the formation of more acids over time. The significant increase in TAN of natural ester oil with ageing times resulted from the hydrolysis reaction of esters, which produces more fatty acids (Bandara et al., 2016; Tokunaga et al., 2016). The hydrolysis reaction of natural ester oils takes place in three stages, as shown Figure 2.6, which are reversible processes (Bandara et al., 2016; Rooney and Weatherley, 2001). Consequently, the water content in the liquid becomes more soluble, leading to the formation of sludge that settles on the winding, adversely reducing heat dissipation (Raof, 2021).

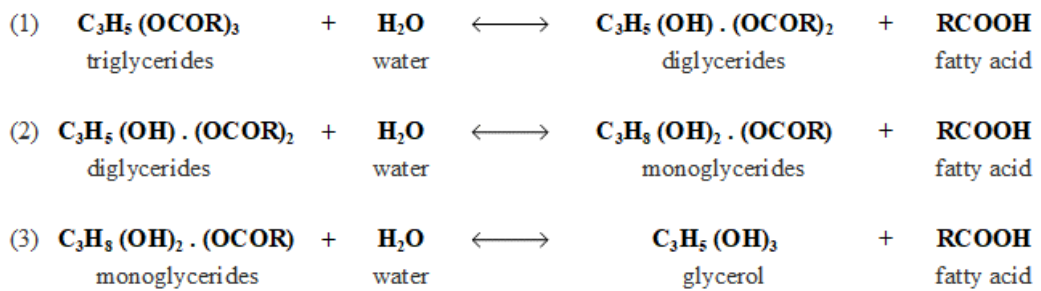


Figure 2.6: Hydrolysis reaction of natural ester oil

2.8.2.3 Dissolved Gas Analysis

Dissolved gas analysis (DGA) is an analysis technique that focuses on analysing gases dissolved in insulating liquids to establish a diagnosis in transformer predictive maintenance (Gomez et al., 2014; Perrier and Beroual, 2009). This technique monitors and analyses the dissolved gas content measured in parts per million (ppm) in the oil, by correlating it with a specific fault. There are three sets of essential gases monitored that are associated with incipient fault types: (i) hydrocarbon gases include hydrogen (H_2), methane (CH_4), ethane (C_2H_6), acetylene (C_2H_2), and ethylene (C_2H_4), (ii) carbon monoxide (CO), and carbon dioxide (CO_2), and (iii) oxygen (O_2) and nitrogen (N_2) are non-fault gases which are analysed to provide complementary information (Gouda et al., 2018; Perrier et al., 2012).

There are several interpretation diagnoses of DGA, such as gas ratio methods (Deornenburg, Rogers, and IEC), classical Duval triangle (Gouda et al., 2018; Perrier et al., 2012), and Duval pentagon. The details of the methods are as follows:

- (i) Rogers Ratio Method (RRM) and IEC Gas Ratio Method use the concentration ratios of the key gases H_2 , CH_4 , C_2H_6 , C_2H_2 , and C_2H_4 to identify the incipient fault types. The following ratios are calculated: C_2H_2/C_2H_4 , CH_4/H_2 , and C_2H_4/C_2H_6 , and the nature of the fault can be identified as shown in Tables 2.7 and 2.8.

Table 2.7: Fault types in the Rogers Ratio Method (RRM) (Golarz, 2016)

Fault	C_2H_2/C_2H_4	CH_4/H_2	C_2H_4/C_2H_6
Unit Normal	<0.01	<0.1	<1.0
Discharge low energy	≥ 1.0	$\geq 0.1, <0.5$	≥ 1.0
Discharge high energy	$\geq 0.6, <3.0$	$\geq 0.1, <1.0$	≥ 2.0
Thermal: $T < 300\text{ }^{\circ}\text{C}$	<0.01	≥ 1.0	<1.0
Thermal: $300\text{ }^{\circ}\text{C} < T < 700\text{ }^{\circ}\text{C}$	<0.1	≥ 1.0	$\geq 1.0 - <4.0$
Thermal: $T > 700\text{ }^{\circ}\text{C}$	<0.2	≥ 1.0	≥ 4.0

Table 2.8: Fault types in the gas ratio method as per IEC 60599 (Golarz, 2016; Piotrowski et al., 2019)

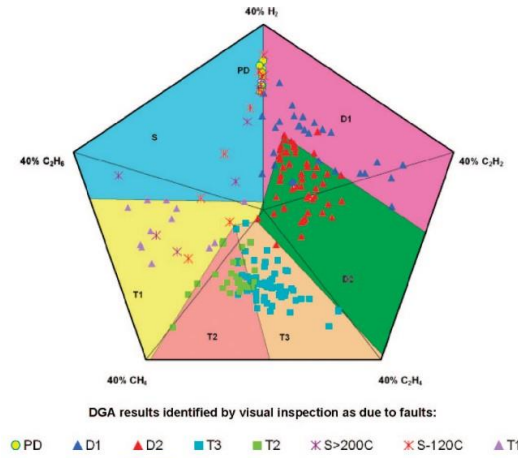
Fault	C_2H_2/C_2H_4	CH_4/H_2	C_2H_4/C_2H_6
Partial discharges	Not significant	<0.1	<0.2
Discharges of low energy	>1	$0.1-0.5$	>1
Discharges of high energy	$0.6-2.5$	$0.1-1$	>2
Thermal: $T < 300\text{ }^{\circ}\text{C}$	Not significant	>1	<1
Thermal: $300\text{ }^{\circ}\text{C} < T < 700\text{ }^{\circ}\text{C}$	<0.1	>1	$1-4$
Thermal: $T > 700\text{ }^{\circ}\text{C}$	<0.2	>1	>4

- (ii) Deornenburg Ratio Method (DRM) utilises the following ratios: CH_4/H_2 , C_2H_2/C_2H_4 , C_2H_4/C_2H_6 , and C_2H_2/CH_4 . To use DRM, the concentrations of gases must double the respective gases concentration limit (L1), as follows: $H_2 = 100\text{ ppm}$, $CH_4 = 120\text{ ppm}$, $CO = 350\text{ ppm}$, $C_2H_2 = 35$, $C_2H_4 = 50$, and $C_2H_6 = 65$) (Golarz, 2016).

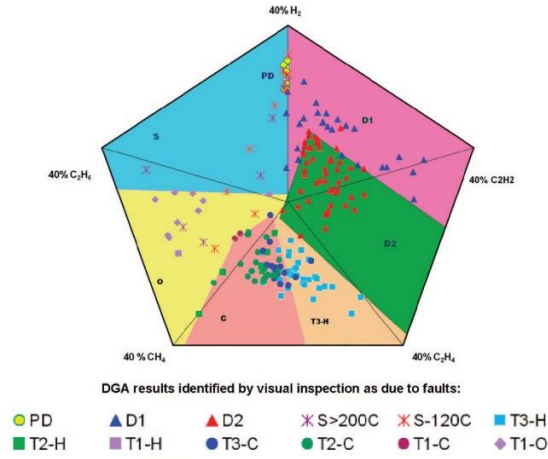
$$\% C_2H_4 = \frac{C_2H_4}{C_T} \times 100 \quad \text{Equation 2.3}$$

$$\% C_2H_2 = \frac{C_2H_2}{C_T} \times 100 \quad \text{Equation 2.4}$$

Duval and Lamarre (2014) introduced the Duval pentagon, which uses the relative percentages of five gases: H_2 , C_2H_6 , CH_4 , C_2H_4 , and C_2H_2 as five-digit coordinates to construct a pentagon. The Duval Pentagon (DPM) method utilises a regular pentagon with five fault zones. The zones are PD: corona partial discharge, D1: low energy discharges, D2: high energy discharges, T1: overheating (thermal faults < 300 °C), T2: thermal fault of 300 to 700 °C), and T3: arcing (thermal fault > 700 °C).



(a)



(b)

Figure 2.8: (a) Duval pentagon 1, and (b) Duval pentagon 2 (Duval and Lamarre, 2014)

The two versions of the DPM are Duval pentagon 1 (uses the regular pentagon with six zones) and Duval pentagon 2 (uses a modified pentagon with seven zones corresponding to PD, D1, D2, T3-H, C, O, and S), where T3-H: thermal faults in oil, C: carbonisation of paper, O: overheating, and S: stray gassing at temperature < 200 °C in the laboratory (Duval and Lamarre, 2014). Figure 2.8 compares the two pentagons.

The coordinates of each of the five points are calculated based on Equations 2.5–2.9 (Duval and Lamarre, 2014; Yue et al., 2022). The vertex coordinates of the pentagon with the relative content of five gases are according to Equations 2.5 and 2.6. The area of the pentagon with the relative content of the five gases as the vertex is calculated using Equation 2.7. The centre coordinates of the pentagon are calculated using Equations 2.8 and 2.9, where x_i and y_i are the coordinates of the five points, C_x and C_y are the coordinates of the centroid, and A is the surface of the polygon.

$$\begin{cases} x_0 = \%H_2 \cos \frac{\pi}{2} \\ x_1 = \%C_2H_6 \cos \left(\frac{\pi}{2} + \frac{2\pi}{5} \right) \\ x_2 = \%CH_4 \cos \left(\frac{\pi}{2} + \frac{4\pi}{5} \right) \\ x_3 = \%C_2H_4 \cos \left(\frac{\pi}{2} + \frac{6\pi}{5} \right) \\ x_4 = \%C_2H_2 \cos \left(\frac{\pi}{2} + \frac{8\pi}{5} \right) \end{cases} \quad \text{Equation 2.5}$$

$$\begin{cases} y_0 = \%H_2 \sin \frac{\pi}{2} \\ y_1 = \%C_2H_6 \sin \left(\frac{\pi}{2} + \frac{2\pi}{5} \right) \\ y_2 = \%CH_4 \sin \left(\frac{\pi}{2} + \frac{4\pi}{5} \right) \\ y_3 = \%C_2H_4 \sin \left(\frac{\pi}{2} + \frac{6\pi}{5} \right) \\ y_4 = \%C_2H_2 \sin \left(\frac{\pi}{2} + \frac{8\pi}{5} \right) \end{cases} \quad \text{Equation 2.6}$$

$$A = \frac{1}{2} \sum_{i=0}^{n-1} (x_i y_{i+1} - x_{i+1} y_i) \quad \text{Equation 2.7}$$

$$C_x = \frac{1}{6A} \sum_{i=0}^{n-1} (x_i + x_{i+1}) (x_i y_{i+1} - x_{i+1} y_i) \quad \text{Equation 2.8}$$

$$C_y = \frac{1}{6A} \sum_{i=0}^{n-1} (y_i + y_{i+1}) (x_i y_{i+1} - x_{i+1} y_i) \quad \text{Equation 2.9}$$

2.8.3 Electrical Properties

A number of characteristics are employed to determine the electrical strength of an insulating liquid. These include dielectric dissipation factor ($\tan \delta$), resistivity, relative permittivity, and BDV at different voltage responses such as AC, DC, or impulse. Literature shows that the most important and commonly investigated palm oil insulating

liquid parameters are the AC BDV and lightning impulse (LI BDV). There is, however, insufficient information for the DC BDV.

2.8.3.1 Tan Delta, Resistivity, and Relative Permittivity

Tan δ , or dissipation factor, gives information on the quality of the insulating liquid, which is directly linked to its resistivity. The lower the tan δ value, the better the insulating properties of the material (Yaacob and Alsaedi, 2015), and the better resistivity and permittivity have been attained (Aditama, 2005; Hamid et al., 2016; Suwarno et al., 2003; Yaacob and Alsaedi, 2015), indicating that palm oil has good insulator characteristics.

2.8.3.2 AC and DC Breakdown Voltages

For most of the literature, the BDV of the oils is measured using AC BDV in accordance with ASTM D1816 and IEC 60156 standards. Experimental findings demonstrated that the RBDPO and PFAE have excellent dielectric liquid potential in BDV, where they outperformed mineral oil (Abdullahi et al., 2004; Kanoh et al., 2008; Suwarno et al., 2003). The AC BDV of CPO measured by (Yaacob and Alsaedi, 2015) was reported at 16.57 kV, which was undoubtedly less than the standard limit; however, after chemical modification to reduce fat content, the BDV was increased to 41.92 kV (CP8) and 52.73 kV (CP10).

The breakdown strength (BDS) comparison between palm oil and other types of vegetable oils, such as sunflower oil, sesame oil, and honge oil, was performed by Bakrutheen et al. (2018). Their statistical analysis revealed that palm oil was one of the oils with a higher survival rate, implying that it was a reliable and potential alternative to conventional mineral oil in high-voltage transformers. Only Jamail et al. (2018) discussed how palm oil's breakdown characteristics with different moistures changed under DC

voltage in the literature. This study showed that the DC BDV for RBDPO is more significant than the AC BDV, but both values deteriorate when the moisture content increases.

2.8.3.3 Lightning Impulse Breakdown Voltage

Besides the BDV magnitude, LI BDV is also an important factor in real applications of liquid insulation in high-voltage transformers. The BDV of mineral, coconut, and palm oils was investigated under nonuniform and quasi-uniform field conditions. The experiment (Von Thien et al., 2016) used electrodes with a needle-sphere combination with 2.0-, 3.8-, and 6.0-mm gap distances with a normalised LI (1.2/50 μ s) according to the IEC 60897 standard. Meanwhile, the LI voltage test using a sphere-sphere electrode combination to produce a quasi-uniform electric field with IEC 60897 and ASTM D3300 standards was performed by (Katim et al., 2018). RBDPO_o and coconut oil were comparable to mineral oil under positive polarity. Their investigation also revealed that the LI BDVs of palm and coconut oils were consistent with mineral oil, and no polarity effect occurred in this electric field.

The above studies on palm oil's insulating properties suggest that there is a possibility of using it as a substitute for conventional mineral oil in transformers.

2.9 Palm Oil-based Nanofluids

Research studies have also been carried out to synthesise palm oil-based nanofluids. More research on palm oil-based nanofluids is needed; moreover, the existing research indicates a promising impact on dielectric strength and thermal conductivity. The following subsections discuss the development and progress, the preparation, the characterisation and stability, and the analysis of the electrical and physicochemical properties of palm oil-based nanofluids.

2.9.1 Development and Progress of Palm Oil-based Nanofluids

This section highlights the trends of researchers in terms of article publications regarding palm oil-based nanofluids over the last five years. Literature demonstrated the efforts of researchers to examine the effects of nanoparticles on the properties of palm oil. The following studies, as summarised in Table 2.9, are illustrations of those researching the effects of nanoparticles on the dielectric strength and other physicochemical properties of palm oil. Almost all studies revealed that the BDV of the nanofluids increases at a specific nanoparticle' concentration.

Table 2.9: Previous research on palm oil-based nanofluids and their findings

References	Base fluids / Nanoparticles / Surfactant	Objectives	Findings
(Nor et al., 2017)	RBDPO / TiO ₂ / CTAB	To investigate the electrical properties of RBDPO-based TiO ₂ with and without CTAB surfactant.	The AC BDV was slightly increased with the introduction of TiO ₂ . However, the presence of CTAB caused a reduction in its BDV at a specific TiO ₂ concentration.
(Saengkhumwong and Suksri, 2017)	Palm ester / ZnO, TiO ₂ / -	To develop a natural oil as a liquid insulator and improve heat exchange by adding nanomaterials.	Adding nanoparticles improved the dielectric breakdown of palm oil-based nanofluids but did not impact the flash point, fire point, and viscosity.
(Makmud et al., 2018)	RBDPO / Fe ₂ O ₃ , TiO ₂ / oleic acid	To investigate the influence of Fe ₂ O ₃ and TiO ₂ on the dielectric response of RBDPO-based nanofluids.	Stable nanofluids were developed with the introduction of oleic acid as a surfactant. The BDV of RBDPO-based nanofluids with conductive Fe ₂ O ₃ shows higher BDV at lower concentration (0.01-g/L).

Table 2.9: continued

(Suhaimi et al., 2018)	Palm oil / MWCNT / -	To investigate the BDV of palm oil-based MWCNT NFs.	The presence of MWCNTs decreased the AC BDV of palm oil-based MWCNT nanofluids. The agglomeration affected their performance.
(Mohamad et al., 2019)	RBDPO / Fe ₃ O ₄ , CuO, Al ₂ O ₃ / CTAB	To study the AC BDV of RBDPO-based nanofluids in the presence of CTAB as a surfactant.	The AC BDV was improved by introducing these nanoparticles at specific volume concentrations. However, CTAB had no apparent effect on the improvement of the AC BDV.
(Mohamad et al., 2021)	RBDPO / Al ₂ O ₃ / SDS	To examine the partial discharge property of RBDPO-based Al ₂ O ₃ nanofluids with or without SDS.	The introduction of Al ₂ O ₃ and SDS has a promising impact on partial discharge.
(Yahya and Amirrazli, 2018)	RBDPO / SiO ₂ / -	To investigate the AC BDV, partial discharge, and viscosity of RBDPO-based SiO ₂ nanofluids.	The addition of SiO ₂ did not improve the AC BDV and viscosity of the nanofluids at different temperatures.

Table 2.9: continued

(Maneerot et al., 2020)	Palm oil / ZnO, BaTiO ₃ / -	To study the partial discharge inception voltage (PDIV) and partial discharge extinction voltage (PDEV) of palm oil-based ZnO/ BaTiO ₃ nanofluids.	Adding BaTiO ₃ at 0.01% concentration demonstrated the highest PDIV and PDEV.
(Hussin et al., 2021)	Palm oil / ZnO, ZrO ₂ / -	To investigate the effect of low-concentration nanoparticles (0.0015-, 0.0025-, 0.005-g/L) on the dielectric properties of palm oil-based nanofluids.	Adding nanoparticles at a lower concentration has improved the nanofluids' dielectric properties and kinematic viscosity.
(Wajanasoonthon and Suksri, 2022; Wajanasoonthon and Suksri, 2021)	RBDPO, POME / Graphene / -	To investigate the BDV of RBDPO-based graphene and POME-based graphene nanofluids.	The BDV of RBDPO-based nanofluids was higher than that of POME-based nanofluids, but a higher concentration of graphene caused the decrement of their BDVs. The BDV of POME-based nanofluids was more significant than that of bare POME after 504 h of thermal ageing.
(Asse et al., 2022)	PKOME / FeO ₃ / -	To analyse the BDV and the acidity index of PKOME-based FeO ₃ nanofluids.	The addition of FeO ₃ improved the BDV of PKOME.

2.10 Preparation of Nanofluids

Two primary approaches commonly used for nanofluid preparation are the one-step and the two-step methods (Li et al., 2009; Yu and Xie, 2012).

2.10.1 One-step Method

The one-step method simultaneously synthesises and disperses nanoparticles in the base fluids. The nanoparticles' drying, storage, transportation, and dispersion processes are bypassed, leading to minimising nanoparticle agglomeration, thus producing more stable nanofluids. However, this approach has the disadvantage of being compatible only with low-vapor-pressure nanofluids (Li et al., 2009).

2.10.2 Two-step Method

The two-step method requires separate preparation of dried nanoparticles, then chemical and mechanical treatments to disperse the dried nanoparticles into the base fluids. Chemical treatment is performed with a surfactant (Atiya et al., 2015) or dispersant (Jacob et al., 2020). Stable nanofluids can only be produced with the help of surfactants and dispersants. On the other hand, mechanical treatment includes magnetic stirring and ultrasonication (Atiya et al., 2015). Magnetic stirring assists the nanoparticles in dispersing uniformly in the base fluids, but the energy is insufficient to avoid forming particle agglomeration. Ultrasonication is then performed to break up the agglomeration of nanoparticles. Researchers commonly use these two procedures to minimise particle agglomeration and maximise dispersion behaviour. Since nanoparticles are commercially available in the industry, many researchers have adopted the two-step method for economic reasons (Ali and Salam, 2020).

According to the literature, the synthesis of palm oil-based nanofluids follows a similar methodology to that used by researchers who prepare the nanofluids using mineral oil (Khaled and Beroual, 2019; Rafiq et al., 2020), as well as to the synthesis of other natural ester oils from soybean, sunflower, rapeseed, etc. (Baruah et al., 2019; Khaled and Beroual, 2020; Koutras et al., 2020b). Figure 2.9 illustrates the two procedures used in the preparation of nanofluids. The first is chemical treatment, which includes preparing the base fluid and adding nanoparticles and surfactant. The second is mechanical treatment, which includes magnetic stirring and ultrasonication.

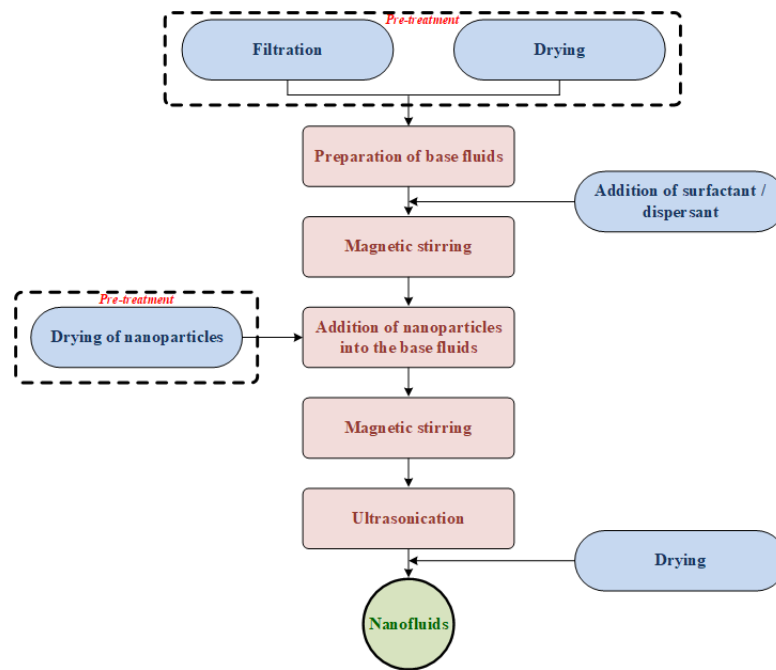


Figure 2.9: Process of the preparation of nanofluids based on the two-step method

Some researchers perform additional procedures to improve the stability of the prepared nanofluids. The procedures in the blue ovals represent these additional procedures. Before the preparation of the nanofluids, the base fluids undergo a pre-treatment process. This includes filtration of the base fluids, followed by drying the

nanoparticles and the base fluids separately at a specific time and temperature to remove unwanted impurities and water molecules. Subsequently, the nanofluids are dried to eliminate bubbles, moisture, and gases formed during the sonication process. The produced nanofluids are then ready for subsequent experimental testing and analysis.

2.11 Synthesis of Methyl Ester as the Base Fluid

Over recent years, researchers have successfully explored the chemical modification methods to modify vegetable oils to be utilised as transformer-insulating liquid, as explained briefly in subsection 2.3.1 on page 16. Different modification methods have been used to produce methyl esters from vegetable oils. Some important synthesis methods are noted and summarised in Table 2.10.

Based on Table 2.10, among all these alternatives, the most common method used to modify vegetable oils chemically is transesterification. During transesterification, triglycerides react with an alcohol in the presence of a catalyst to form esters and glycerol as the by-product (Anastopoulos et al., 2009; Musa, 2016), as seen in Figure 2.10. This reaction typically involves a molar alcohol-to-oil ratio of 6:1. Methanol (CH_3OH) is preferably used over ethanol due to its availability, cost, and physical and chemical advantages for quick reactions with triglycerides (Encinar et al., 2010; Musa, 2016). It is catalysed by a base, such as potassium hydroxide (KOH) or sodium hydroxide (NaOH), or an acid, such as sulfuric acid (H_2SO_4). These catalysts accelerate the conversion of the triglycerides into methyl esters and glycerol, releasing one glycerol molecule for each of the three fatty acid chains in the triglyceride molecule. The reaction is conducted at a moderate temperature, usually between 50–60 °C, and the duration can vary but is commonly around 1–2 h.

Table 2.10: Previous studies on methyl ester synthesis

Synthesis methods	Vegetable oils	Chemical used	Alcohol:oil molar ratio	Temperature (°C)	References
Esterification	JCO	CH ₃ OH, KOH, NaOH	6:1	70	(Sitorus et al., 2016)
	PKO	CH ₃ OH, KOH, NaOH	5:1	60	(Asse et al., 2021)
Transesterification	PKO	CH ₃ OH, KOH	n/a	n/a	(Boyekong et al., 2021)
	CO	CH ₃ OH, KOH	9:1	n/a	(Nkouetcha et al., 2019)
	SSO	CH ₃ OH, H ₂ SO ₄	6:1	50	(Ravulapalli et al., 2019)
	Neem oil	CH ₃ OH, NaOH	5:1	60	(Oparanti, et al., 2022)
Direct purification, Transesterification	KO	CH ₃ OH, KOH	6:1	60	(Menkiti et al., 2017)
Transesterification, Epoxidation- esterification	TCKO	CH ₃ OH, KOH, H ₂ SO ₄ , peracetic acid, acetic acid	6:1	30–60	(Agu et al., 2019)

n/a – not available

Transesterification significantly reduces the viscosity of vegetable oils to an adequate value for use as an alternative (Maharana et al., 2018; Okikiola et al., 2022). Methyl ester production could also be realised by the esterification reaction when a high level of free fatty acids is present (Decarpigny et al., 2022), as shown in Figure 2.11.

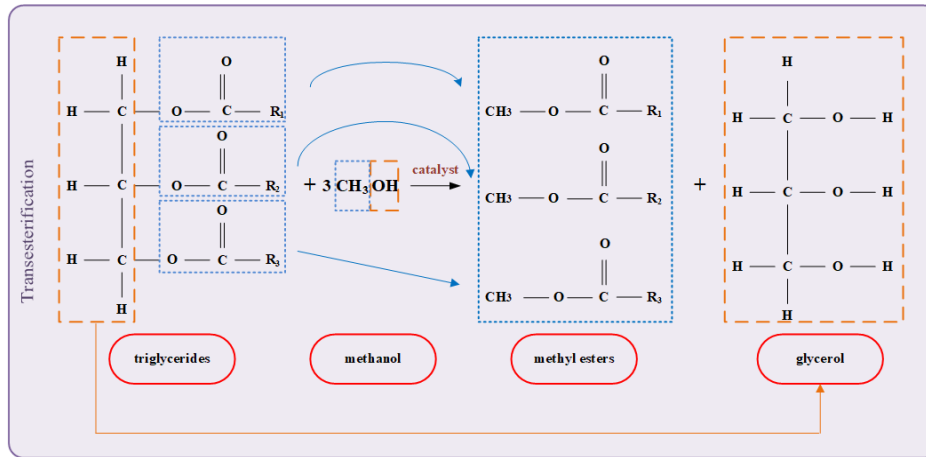


Figure 2.10: Transesterification reaction

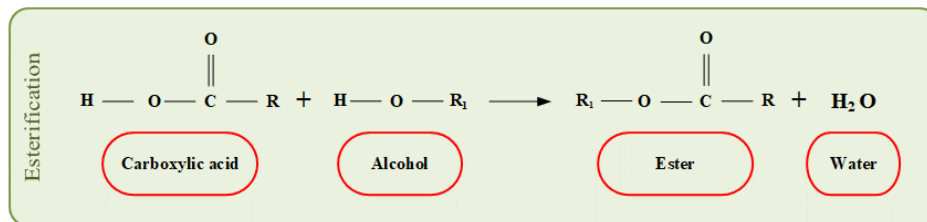


Figure 2.11: Esterification reaction (Decarpigny et al., 2022)

In addition, the study conducted by Agu et al. (2022) involves the modification of the TCKO using two different process routes: transesterification and epoxidation-esterification. The transesterification process involves the reaction of the TCKO with CH₃OH, and KOH as a catalyst. Epoxidation-esterification, on the other hand, adds oxygen to the C = C bonds of the transesterified fatty acid chain of the MTCKO

(MTCKO^t). The resulting epoxy methyl ester (oxirane) of TCKO is known as EMTCKO^o, which is then characterised and tested further. Figure 2.12 shows the epoxidation reaction, similar to the modification method presented in the work (Abdelmalik, 2014) for the modification of PKO.

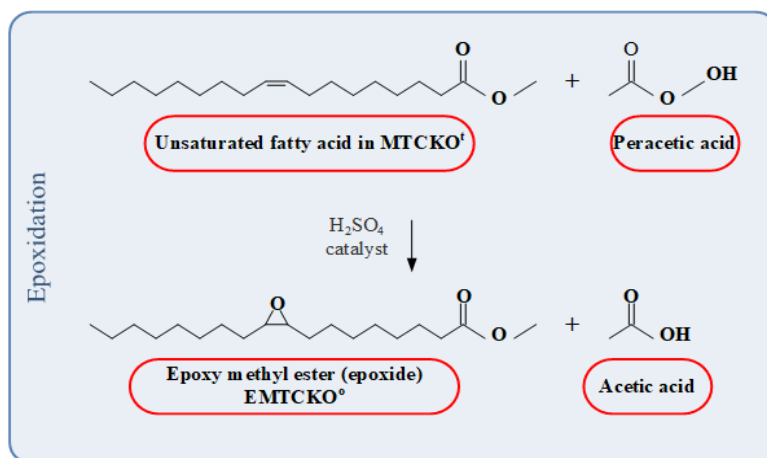


Figure 2.12: Epoxidation reaction of MTCKO^t to obtain EMTCKO^o (Agu et al., 2019)

2.12 Chapter Summary

In summary, the development of palm oil and its nanofluids as biodegradable oil insulation alternatives can be divided into three significant periods. In the early 2000s, some researchers started investigating the suitability and potential of palm oil to become one of the biodegradable transformer oils. Laboratory studies have conducted several experimental tests for property and characteristic determinations against standards. The main concern of the analysis was looking at the potency of palm oil as an alternative insulating liquid to mineral oil in transformer applications.

In the 2010s, comprehensive test measurement and analysis of palm oil-based insulating liquid in transformers have been conducted continuously by many researchers, covering different basic electrical, physical, and chemical properties, degradation,

oxidative, and ageing performance. According to the results, it is evident that palm oil complies with global standards, and most of its properties closely align with those of mineral oil. This indicates its potential value for further research and development towards becoming a world-class biodegradable insulating oil.

From the end of the 2010s until recently, efforts have been devoted to improving the properties of palm oil. In particular, different modifications have been introduced, such as performing chemical conversion of triglycerides, adding different types of nanoparticles with different concentrations, adding different types of surfactants, exploring different techniques for nanofluid preparation, investigating the interaction of the nanoparticles with the base fluids, etc. Several published studies provide a comprehensive analysis of the modified palm oil in terms of its chemical composition, oxidation stability, physicochemical properties, and dielectric properties such as AC BDV, lightning impulse BDV, resistivity, and dielectric dissipation factor in accordance with relevant standards. However, studies on developing POME as the base fluid of the nanofluid preparation as an alternative transformer oil have been relatively scanty. Moreover, studies on the effects of ageing mechanisms on the electrical and physicochemical properties of palm oil-based nanofluids have been limited. Therefore, conducting an ageing study becomes crucial to understanding the ageing behaviour of palm oil, particularly POME-based nanofluids. Figure 2.13 illustrates the chronology of palm oil's development as an insulating liquid based on the literature.

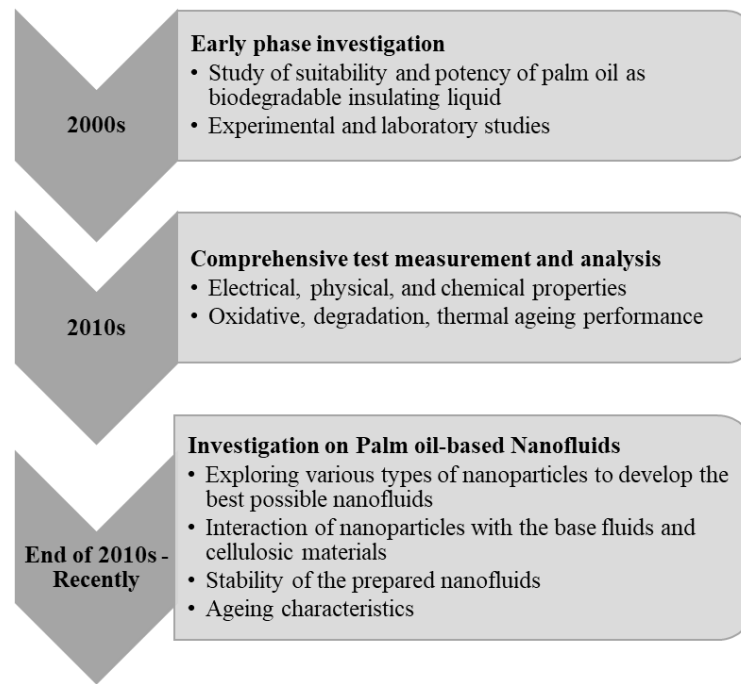


Figure 2.13: Chronology of palm oil-based insulating liquid development

CHAPTER 3

METHODOLOGY

3.1 Overview

This chapter presents the methodological procedures employed in the proposed work. The research methodology unfolds in several phases, commencing with the preparation of nanoparticles and the synthesis of methyl ester from RBDPO_o. Subsequently, the synthesised methyl ester undergoes characterisation to assess its compatibility as a transformer-insulating liquid. The ensuing phase involves the preparation of the POME-based nanofluids at various concentrations, which are then subjected to accelerated thermal ageing. Within this phase, the electrical and physicochemical properties of the aged nanofluids are thoroughly investigated and analysed. Finally, in the concluding phase, the ageing behaviour of these nanofluids is meticulously evaluated to establish their suitability for the intended purpose. The flowchart is illustrated in Figure 3.1.

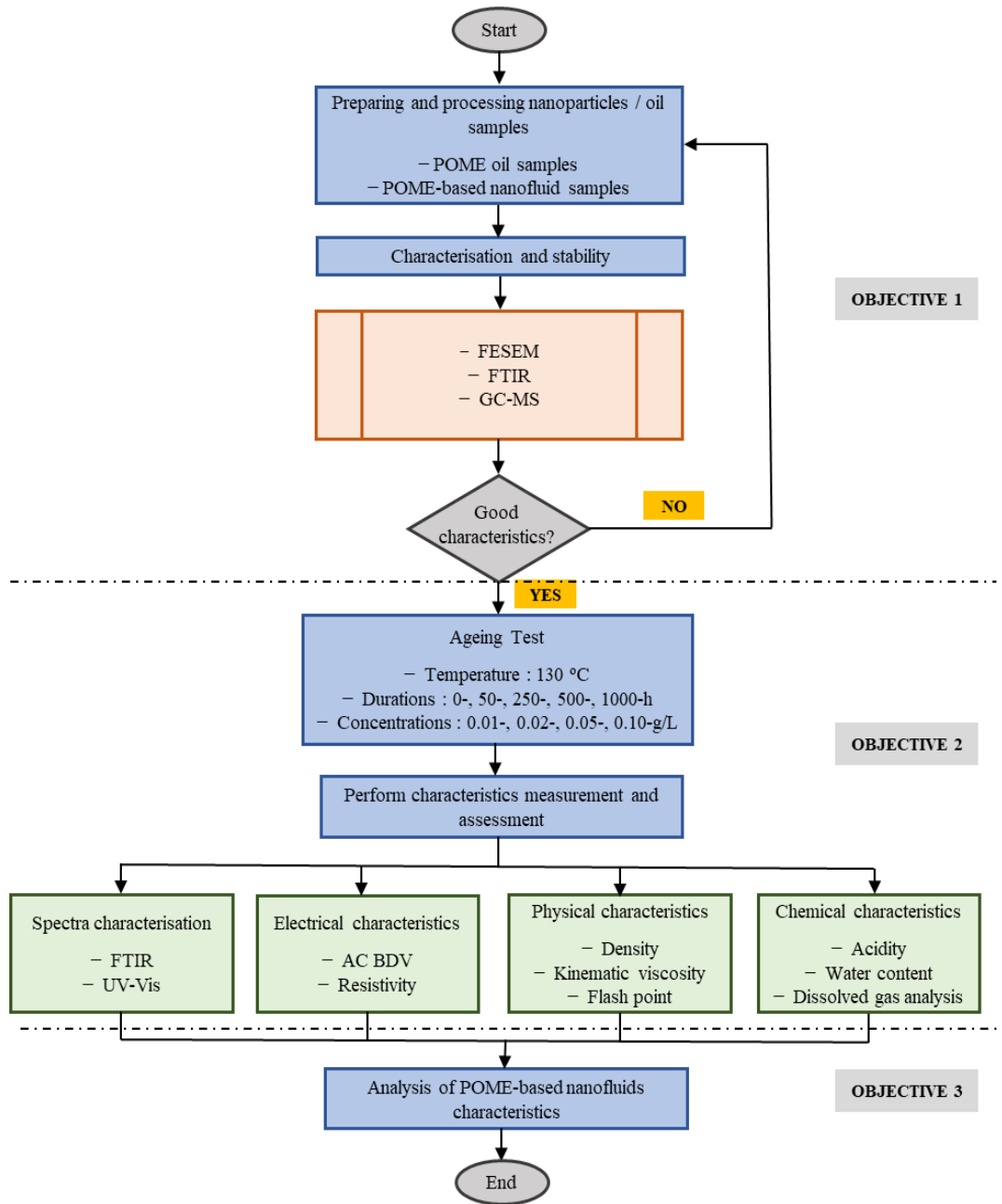


Figure 3.1: Flowchart of the methodology

3.2 Materials and Chemicals

The materials and chemicals employed in this study are categorised based on their functions: (i) synthesis of the understudy POME as the base fluids, and (ii) preparation of the nanofluids.

3.2.1 Synthesis of the Understudy POME as the Base Fluids

The RBDPO₆ was manufactured by Lam Soon Edible Sdn. Bhd. and purchased from a local grocery store. It was used as received without further filtration or purification. Within the framework of this thesis, the base dielectric fluid studied is POME, synthesised by the transesterification process of the RBDPO₆. Pure methanol CH₃OH (99.9%) and the catalyst potassium hydroxide (KOH) in the hemispherical pellets were obtained from commercial industries.

In this study, hexadecyltrimethylammonium bromide, often known as CTAB, was used as a surfactant to achieve homogeneous dispersion of the nanoparticles in the base fluids. The use of CTAB for improving stability in the nanofluids dispersed with MWCNT and TiO₂ nanoparticles has been reported by (Bakthavatchalam et al., 2022) and (Mohamad et al., 2020; Nor et al., 2017), respectively. The fatty acid methyl ester (FAME) standard mixture (comprised of 13 fatty acids) of >95% purity used was obtained from Sigma Aldrich. Table 3.1 presents the properties of POME. Nearly similar findings were reported by (Baroutian et al., 2009).

Table 3.1: Properties of POME

Property	Value
Density at 20°C (g/cm ³)	0.87
Kinematic Viscosity at 40°C (mm ² /s)	4.54
Water Content (ppm)	477
Acidity (mg KOH/g)	0.0913
Flash Point (°C)	175

3.2.2 Preparation of Nanofluids

Two different categories of nanoparticles explored for nanofluid preparation were conductive multi-walled carbon nanotube (MWCNT) and semiconductive titanium dioxide (TiO₂), with various sizes and properties manufactured by Sigma Aldrich. Table 3.2 contains the properties of the nanoparticles as obtained from the manufacturer's datasheet. The nanoparticles are subjected to a drying process in an oven for at least 24 h to remove the moisture and impurities. According to the literature, the BDV characteristics and statistical analysis of RBDPO-based nanofluids blending with metal oxides, particularly TiO₂, have been studied, for example, by Nor et al. (2017). However, the impact of TiO₂ nanoparticles on POME as the base fluid has yet to be thoroughly investigated. The study also looks at the nanofluids prepared by dispersing MWCNT into POME base fluid and how they could be used in different concentrations. This is because there has yet to be an exploratory data analysis for POME-based MWCNT nanofluids. The higher thermal conductivity of MWCNT, attributed to their strong atomic bonds (Alizadeh et al., 2022; Bakthavatchalam et al., 2022), prompted the selection of MWCNT in this study. The dispersion of MWCNT

in palm oil significantly enhanced its electrical properties but decreased with the increasing amount of MWCNT (Suhaimi et al., 2018).

Table 3.2: Selected nanoparticles used and their properties

Properties	Conductive	Semiconductive
	MWCNT	TiO ₂
Size (nm)	50–90	< 50
Appearance (Colour)	Black	White
Appearance (Form)	Powder	Powder
Density (g/cm ³)	1.7–1.9	4.26
Molecular Weight (g/mol)	n/a	79.90
Purity	>95%	≥99.5%

n/a – not available

3.3 Synthesis of POME by Transesterification Process

The production of the base fluid began with the synthesis of POME from RBDPO_o via the transesterification process. The RDBPO_o (1000 g, 1.182 mol) and methanol (227.3 g, 7.094 mol) were placed in separate beakers. In this work, methanol was particularly preferred to produce methyl ester because of its availability, cost, and physical and chemical advantages for quick reactions with triglycerides (Encinar et al., 2010; Musa, 2016). Then, KOH was added as a catalyst to the methanol beaker in the predetermined amounts for the experiment and stirred with the magnetic stirrer until the KOH pellets were fully dissolved. The second beaker containing RDBPO_o was heated to 60 °C. The methanol and KOH mixture was poured into the RDBPO_o, and both mixtures were heated to 60 °C and magnetically stirred at a speed of 1000 revolutions per min (r/min) with a reaction time of

60 min. The reaction used the recommended methanol:oil molar ratio of 6:1 (Baroutian et al., 2009; Musa, 2016). The ratio is universally accepted and often used to drive the reaction to the product side, as an incomplete reaction was observed for molar ratios below 6:1, and higher molar ratios increase the cost of recovering the excess alcohol after the completion of the reaction (Encinar et al., 2010).

The mixture was allowed to cool to room temperature before being transferred to a separatory funnel for separation. Glycerol and methyl ester were split into two layers when the transesterification process was completed, as shown in Figure 3.2. The mixture of crude glycerol and KOH, which had settled at the bottom of the funnel, was discarded. The top layer, the fatty acid methyl ester (FAME), was then washed with warm distilled water several times to remove excess KOH until it achieved a neutral pH level. It was then heated and stirred at 60 °C for 30 min to eliminate the remaining water molecules and methanol. After the transesterification process, the RDBPO₀ was converted to POME and used as the base fluid for preparing the nanofluids. Figure 3.3 shows the synthesis of POME in the laboratory using a transesterification process based on the previous study, where the production process and methodology were preferred accordingly.

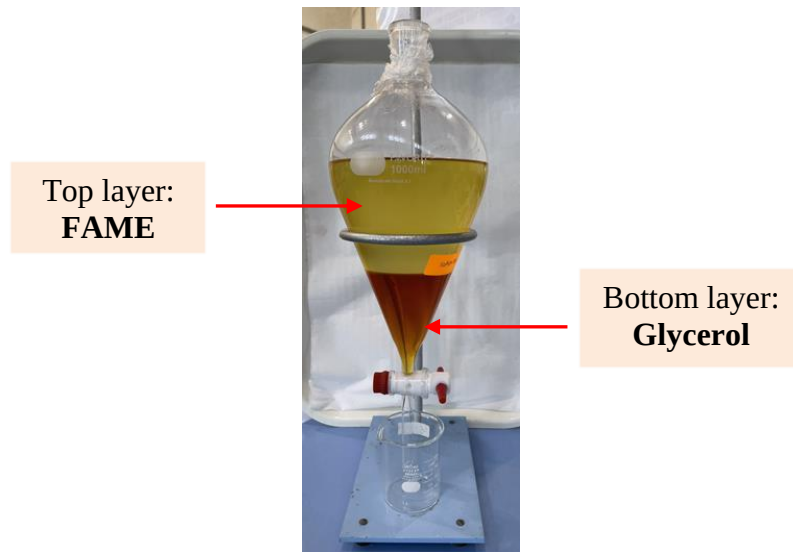


Figure 3.2: Separating methyl ester from glycerol in transesterification process

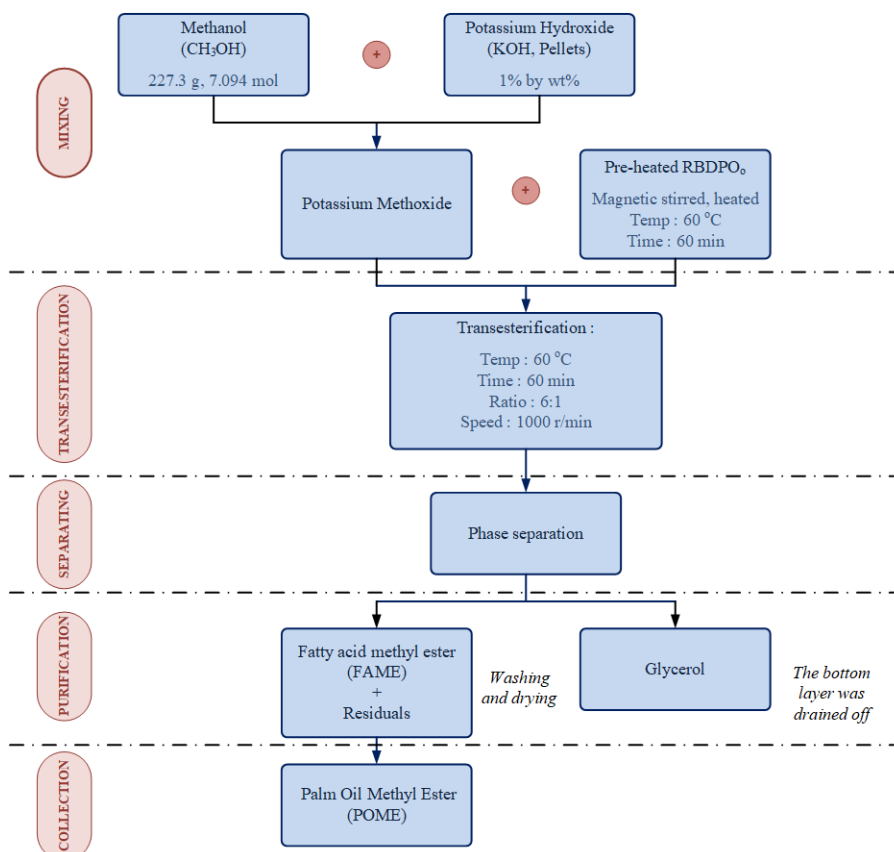


Figure 3.3: POME production process in the laboratory

3.4 Characterisation of POME

The conversion of RBDPO_o to its methyl ester is studied with the characterisation of POME by acquiring insights into its infrared spectroscopic and chemical composition. This involves characterisation by applying Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography–Mass Spectrometry (GC–MS) analyses.

3.4.1 FTIR Spectra Analysis for Functional Group Characterisation

The spectra of POME samples were recorded using a Thermo Scientific iS10 Nicolet spectrometer equipped with an attenuated total reflectance (ATR) accessory, as shown in Figure 3.4. A background spectrum was obtained before a sample could be acquired. The oil sample was placed on the ATR cell, and the measurement was taken by combining 32 interferograms at a resolution of 4. The data was collected using OMNIC spectra software (ver 8.2). The FTIR spectra displayed the sample's absorbance band at a wavenumber between 4000 and 600 cm^{-1} . After the spectrum was taken, the ATR plate was cleaned in-situ by wiping it with a solvent and drying it with a blower bulb before placing the following sample.



Figure 3.4: Thermo Scientific Nicolet iS10 FTIR spectrometer

Figure 3.5 shows the flowchart that outlines the steps involved in the scanning FTIR spectra analysis.

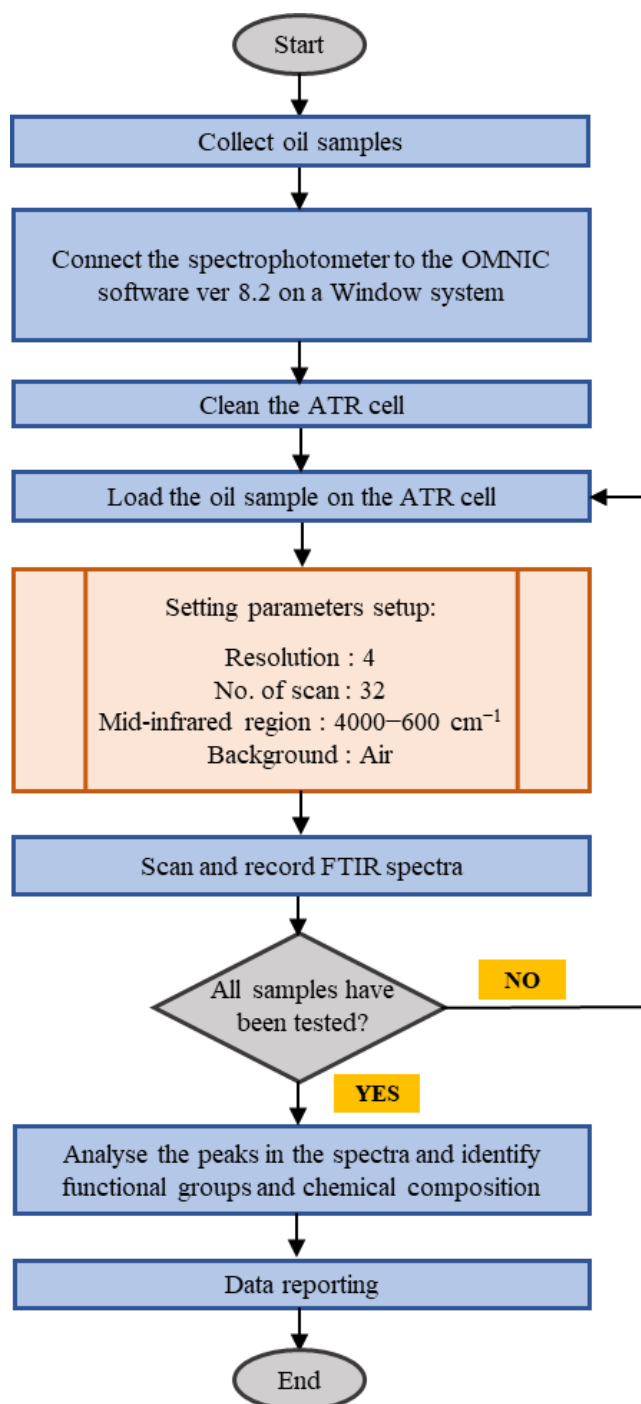


Figure 3.5: Flowchart of FTIR spectra analysis

3.4.2 GC–MS Analysis for Fatty Acid Composition

Gas chromatography was used to profile the composition of fatty acid methyl ester in POME using a QP2010 Plus GC (Shimadzu, Japan), as shown in Figure 3.6, equipped with an AOC-20i autosampler and a capillary BPX5 (30 m × 0.25 mm × 0.25 µm). Liquid sample injection was performed in the GC inlet port with the injector temperature kept at 280 °C, and the injection mode was set to the split mode (with a split ratio of 20:1). Helium was the carrier gas, with the rate set to 1.0 mL/min. The oven temperature programme was set at 50 °C. The detected fatty acids were identified by comparing their retention times with the authentic standard mixture from Sigma Aldrich. The total run time was 45 min. Figure 3.7 illustrates the flowchart of fatty acid composition in POME using GC–MS analysis.



Figure 3.6: QP2010 Plus GC spectrometer

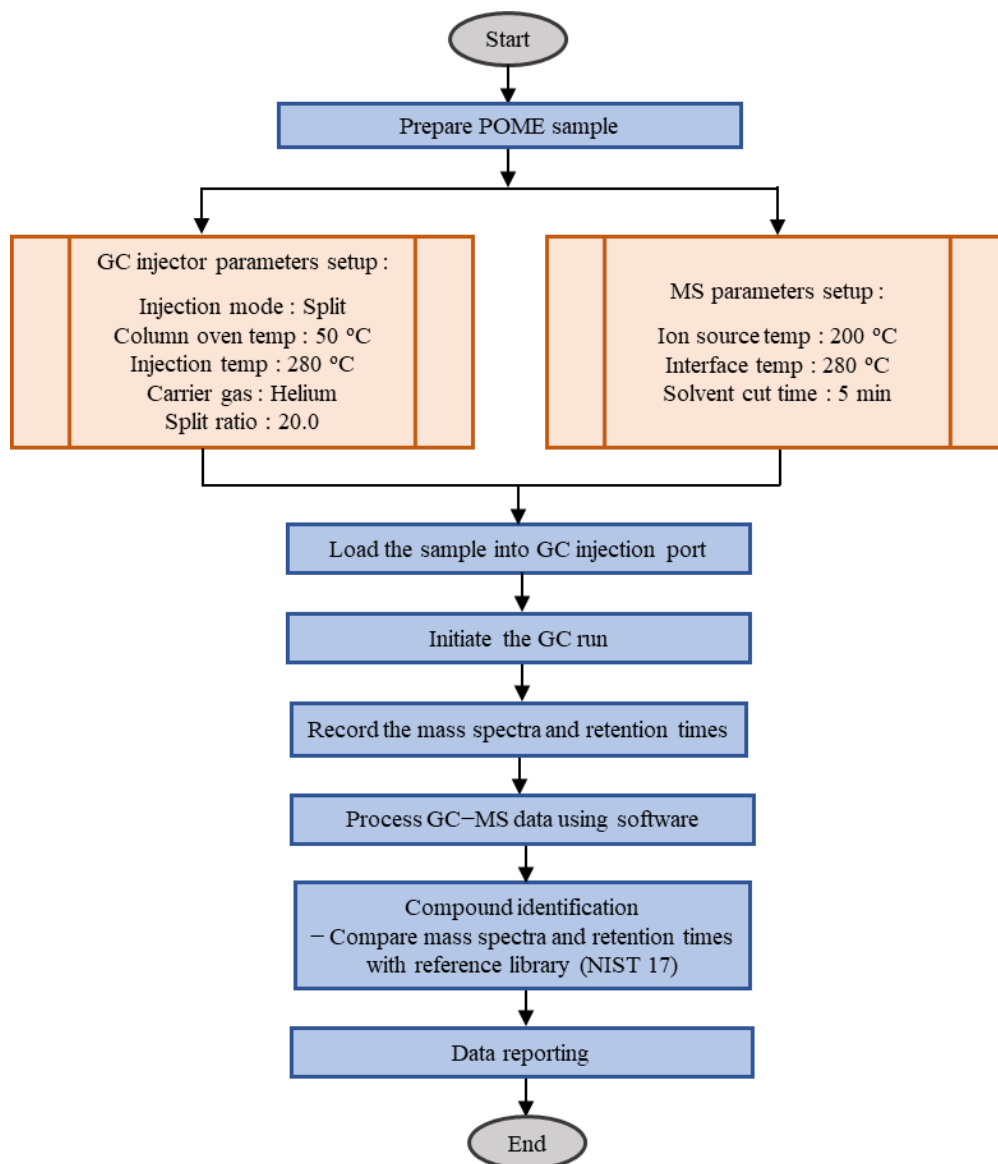


Figure 3.7: Flowchart of GC-MS analysis

3.5 Characterisation of Nanoparticles

Characterisation work on used nanoparticles was performed using a JEOL JSM-IT500HR field emission scanning electron microscopy (FESEM) device, as shown in Figure 3.8. It was used to determine the morphology, nanostructure, and size of the as-received nanoparticles. The FESEM analyses were conducted with an accelerating voltage of 20 kV

to reduce any possible damage to the investigated nanoparticles. The nanoparticles were deposited on carbon tapes before being coated with gold using the JEOL JEC-3000FC auto-fine coater, as shown in Figure 3.9, to prepare the specimens for FESEM observation and characterisation.

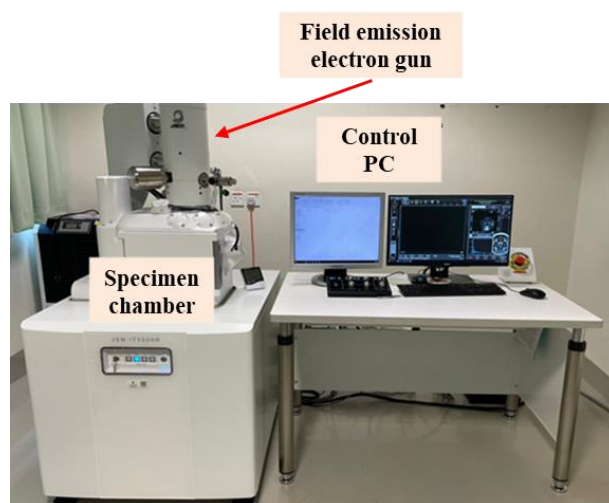


Figure 3.8: A JEOL JSM-IT5000HR FESEM equipment

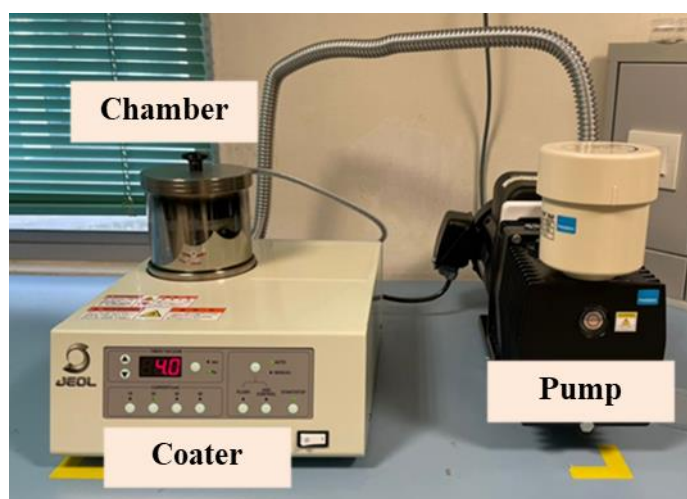


Figure 3.9: A JEOL JEC-3000FC auto-fine coater

3.6 Preparation of POME-based Nanofluids

The preparation of the nanofluids was performed using a two-step method. The two-step method involves the separate preparation of dried nanoparticles, followed by chemical and mechanical treatments of the dried nanoparticles in the POME base fluids.

Prior to the sample preparation, POME was preprocessed by being dried in a convection oven at 80 °C for 2 h to remove moisture and then allowed to cool to room temperature. A 1000-ml (1 L) volume of predried POME is determined to be the base fluid for the preparation of the nanofluids. The CTAB was then suspended in the POME base fluid and magnetically stirred at 1000 r/min for 30 min. The purpose was to disperse it in the base fluid evenly. According to the investigation by (Mohamad et al., 2019), the concentration of the CTAB understudy was equal to half the weight percentage of the nanoparticle's concentration. However, the optimal concentration is still being researched (Pyrgioti et al., 2020). The nanoparticles were accurately weighed for the nanofluid sample preparation and dispersed into POME to achieve the desired concentration. The amounts of TiO₂ and MWCNT nanoparticles considered in g/L are tabulated in Table 3.3. The concentration span provides a range to allow investigation of the optimal dielectric strength and physicochemical properties of the nanofluids. Each mixture must be exposed to energetic magnetic stirring and ultrasonication to ensure that the nanoparticles are well dispersed and that the end product of nanofluid samples is homogeneous to minimise possible agglomeration and sedimentation (Fasehullah et al., 2022). Figure 3.10 shows the flowchart to produce POME-based nanofluids.

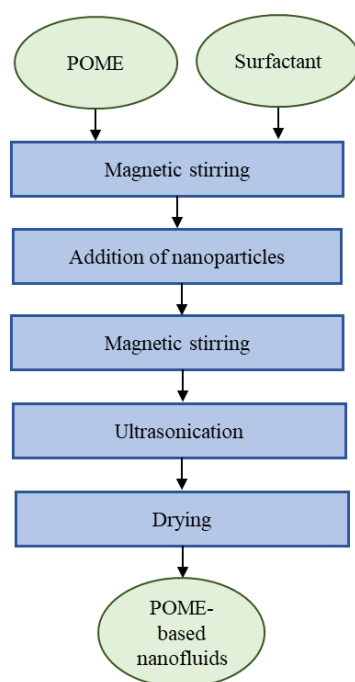


Figure 3.10: Flowchart of POME-based nanofluids preparation

Table 3.3: Nanofluids samples with different concentrations

NFs	POME-based TiO ₂				POME-based MWCNT			
Concentrations	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L

The ultrasonic procedure using an ultrasonic cleaner, as shown in Figure 3.11, with a heating power of 100 W and a frequency of 40 kHz, was applied for 2 h in intervals of 30 min of agitation with a 10-min break in between to prevent the nanofluids from becoming overheated. The energetic magnetic stirring and ultrasonication ensure that the nanoparticles are well dispersed and that the end product is homogenous to minimise the possibility of aggregation and sedimentation. After being dried in a convection oven for 48 h at 85 °C, all

nanofluid samples were ready for the ageing test experiment. Figure 3.12 illustrates the flowchart of the POME-based nanofluid preparation in the laboratory.



Figure 3.11: Ultrasonication of POME-based NFs

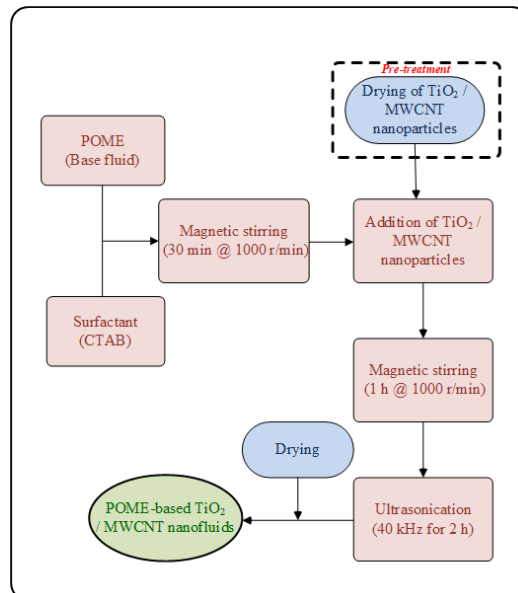


Figure 3.12: Illustration of POME-based nanofluids preparation in the laboratory

3.7 Methodology of Accelerated Thermal Ageing

An accelerated thermal ageing study was conducted to understand their degradation behaviour to validate the feasibility and potential of POME-based nanofluids as transformer-insulating liquids. Experiments were performed on two groups of samples: one with TiO_2 and the other with MWCNT. This was intended to understand the impact of different types of nanoparticles on the ageing behaviour of nanofluids based on POME. The prepared nanofluid samples were exposed to the accelerated thermal ageing process in the laboratory to simulate the actual ageing process of the transformer insulation in a shortened time frame. All POME-based nanofluid samples were stored in sealed 1-L Duran® bottles, referred to as *closed ageing* or *sealed ageing conditions* for the simulation. They were subjected to a temperature of 130 °C in a convection oven, as shown in Figure 3.13.



Figure 3.13: Accelerated thermal ageing experiment using a convection oven

A temperature of 130 °C was strategically chosen for the ageing process to mitigate the risk of exceeding the liquid's flash point (Münster et al., 2021). This decision is supported

by previous investigations that have been reported and published by Carcedo et al. (2015), Cong et al. (2022), Tenbohlen and Koch (2010), and Karambar and Tenbohlen (2021). Furthermore, in accordance with IEEE Std C57.147 (IEEE Std C57.147-2018, 2018), the thermal ageing temperature for natural ester oils is maintained at levels below 180 °C to avoid fluid scorching (Thakur et al., 2019). This aligns with the requisite accelerated ageing temperature of at least 130 °C for a meaningful comparison of the performance of the natural ester oil nanofluids with mineral oil.

To investigate the specific ageing process, the aged products were then characterised at 0, 50, 250, 500, and 1000 h of ageing time in terms of dielectric strength and physicochemical properties. The period was chosen to allow for the study of both short-term and long-term thermal ageing effects on the nanofluids. A total of 40 nanofluid samples were prepared for the ageing experiments. The nanofluid oil samples were allowed to cool down to ambient temperature before being measured.

3.8 Characterisation of Aged POME-based Nanofluids

The characterisation of aged POME-based nanofluids was assessed using the FTIR and UV–Vis analysis.

3.8.1 FTIR Spectra Analysis

The FTIR spectra were recorded using Thermo Scientific iS10 Nicolet FTIR spectrometer. As previously described in subsection 3.4.1, FTIR analysis was used to investigate the functional groups of the POME. Similarly, the FTIR analysis was utilised to delve further into the molecular composition of the aged nanofluid samples. Consistent measurement techniques and procedures were employed, enabling meaningful comparisons and thorough insights into the characterisation of the functional group.

3.8.2 Ultraviolet–Visible Analysis

The analysis of absorption spectra and peaks in the various concentrations and durations of aged POME-based nanofluids was done using an Agilent Cary 60 Ultraviolet–Visible (UV–Vis) spectrophotometer, as shown in Figure 3.14, with a standard 10-mm pathlength, 4-mL volume quartz cuvette.

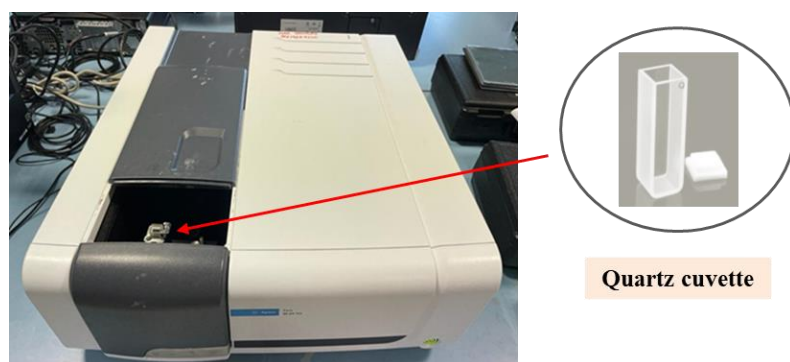


Figure 3.14: Agilent Cary 60 UV–Vis spectrophotometer

The spectrum reveals how the nanofluids absorb light across different wavelengths and provides information about their optical properties. Meanwhile, the intensity of the peaks indicates the presence of molecules or nanoparticles within the aged nanofluids. The measurement was performed by pipetting the aged nanofluid sample into a cuvette, placing it in the spectrophotometer, and initiating the measurement programme to record the absorbance spectrum.

3.9 Experimental Setup

This section explains the experimental study for analysing the effects of thermal ageing on electrical characteristics of aged POME-based nanofluids, considering various

doping concentrations and ageing durations. A detailed description of the process and setup of the AC BDV experiment is included.

3.9.1 AC Breakdown Voltage Test

The AC BDV measurement test of oil samples was guided by the IEC 60156 (IEC 60156, 2018) standard using an HZJQ-1B transformer oil BDV tester (Huazheng Electric Manufacturing Co. Ltd., China), as shown in Figure 3.15. The volume capacity of the oil cup was 250-mL. The brass hemispherical electrodes of 12.5-mm diameter with a distance of 2.5-mm were used for the testing. The input voltage of 240 V at a frequency of 60 Hz is steadily applied through the electrode terminals. The electrodes were subjected to a continuously increasing voltage at a uniform rise rate of 2 kV/s until the breakdown occurred, and the reading was shown on the LCD.

All measurements were conducted while the oil sample was constantly stirred, following the recommended approach as an advanced measurement (Baur et al., 2019; Calcara et al., 2019), which found that stirring the sample helped to reduce the scatter in the BDV results. Six sets of six consecutive breakdown voltage measurements were carried out, with a 2-min gap in between each measurement. The output BDV is up to 80 kV with ± 1 kV accuracy. In total, 36 breakdown values were collected for each sample, and the average value of those measurements was considered for analysis.

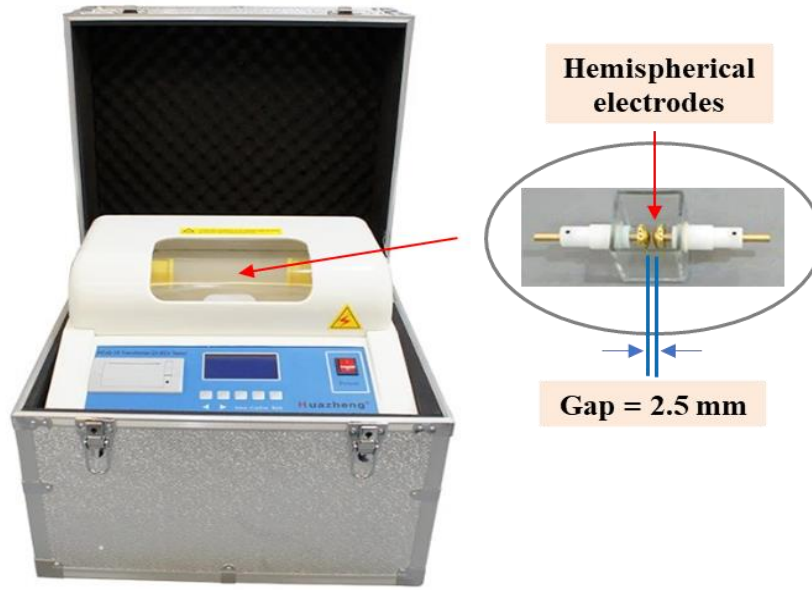


Figure 3.15: HZJQ-1B transformer oil BDV tester

The average BDV of each nanofluid sample was calculated as Equation 3.1:

$$V_{BDV_ave} = \sum_{n=1}^N \left(\frac{V_{BDV_n}}{n} \right) \quad \text{Equation 3.1}$$

where V_{BDV_ave} represents the average of the BDV, V_{BDV_n} represents the n th BDV measurement, and n represents the total number of BDV measurements. The acceptable range criteria based on the ASTM D877 (ASTM D877-02, 2002) is given by Equation 3.2:

$$\text{Acceptable Range} = \frac{R}{V_{BDV_ave}} \times 100 \quad \text{Equation 3.2}$$

where R represents the range of the BDV, which is the difference between the maximum and minimum of the collected measured BDV. The standard stated that the acceptable range of the BDVs is no greater than 92% of the average value. Figure 3.16 illustrates the flowchart of the overall process of AC BDV measurement using the HZJQ-1B transformer oil BDV tester.

3.9.2 Characterising AC Breakdown Voltage Data Using a Probability Function

An important step in analysing breakdown data is selecting an appropriate distribution. Common distribution for electrical breakdown includes the Weibull, Gumbel, and lognormal, with the Weibull distribution being the most commonly used (IEC 62539, 2007).

The expression for the cumulative distribution function (CDF) for the two-parameter Weibull distribution is shown in Equation 3.3 (IEC 62539, 2007):

$$F(x) = 1 - e^{-\left(\frac{x}{\alpha}\right)^\beta} \quad \text{Equation 3.3}$$

where α and β are the scale and shape parameters (both are positive), respectively, and x is the breakdown voltage. The scale represents the voltage for which the failure probability is at a 63.2% risk level, and the shape is a measure of the range of voltages (IEC 62539, 2007). The larger the value of β , the smaller the range of breakdown voltages.

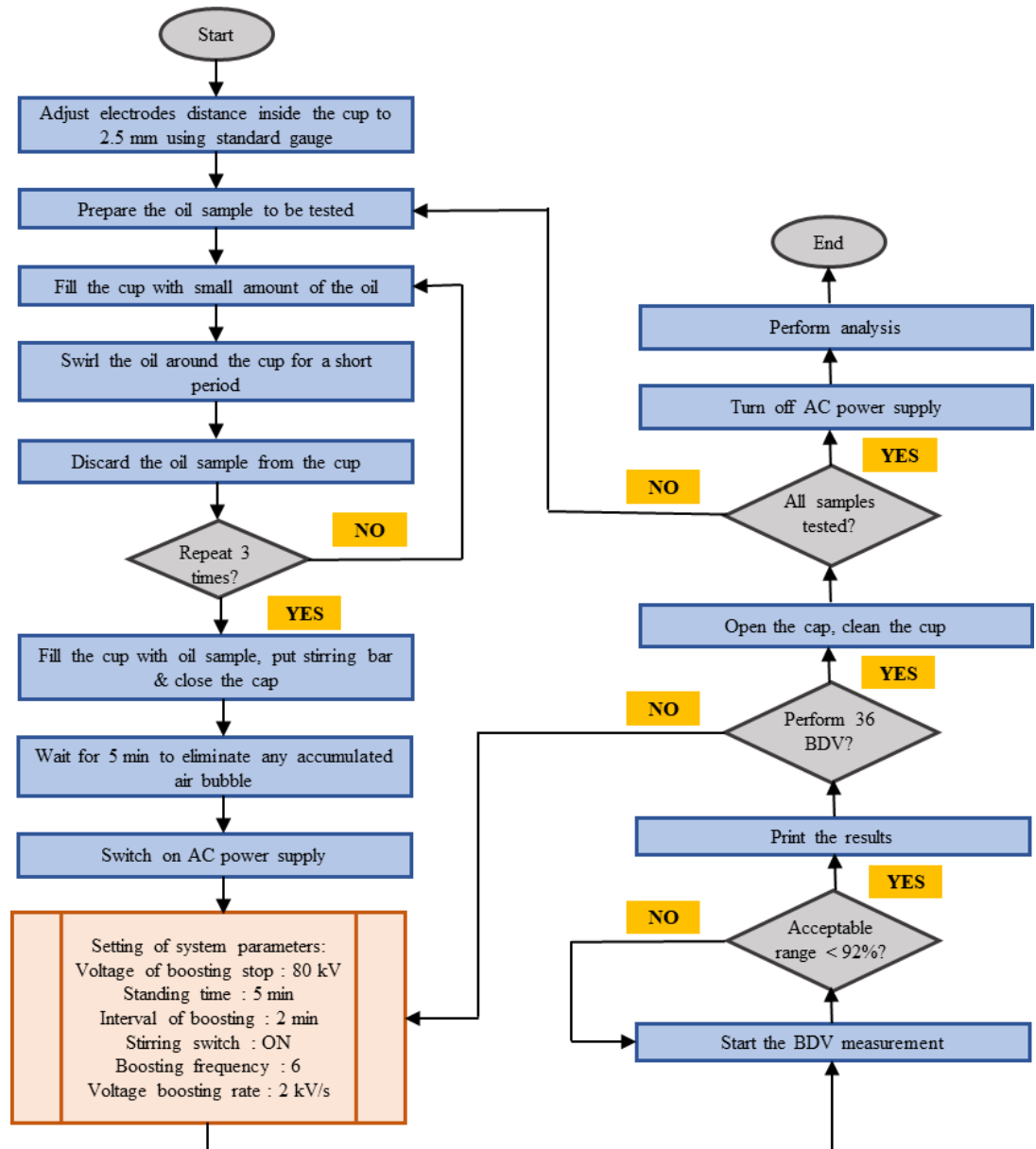


Figure 3.16: Flowchart of AC BDV measurement

3.9.3 Resistivity

The resistivity measurements of the oil samples were carried out in accordance with the IEC 60247 (IEC 60247, 2004) standard using an automatic ADTR-2K PLUS test apparatus of ELTEL Industries, as shown in Figure 3.17. The three-terminal test cell

apparatus was filled with oil samples, and the test was conducted at a setting temperature of 90 °C.

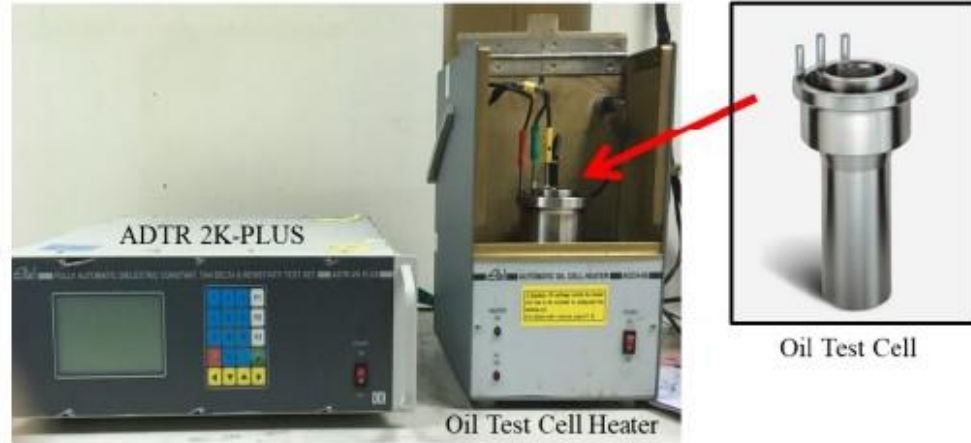


Figure 3.17: ADTR-2K PLUS test apparatus (Suhaimi et al. 2021)

3.10 Measurement of Physicochemical Properties of Thermally Aged Nanofluids

In the subsequent section, an exploration into the physicochemical properties of aged POME-based nanofluids, encompassing diverse doping concentrations and ageing durations, was undertaken. This investigation is essential as it provides valuable insights into the performance and suitability of these nanofluids subjected to thermal ageing. Detailed explanations of the measurement procedures employed for the investigations are provided. Additionally, Table 3.4 offers a summary of the standard methods and the equipment used for data acquisition.

Table 3.4: Test standards and equipment

Properties	Standards	Equipment
Flash point (°C)	ISO 2719	Stanhope-SETA
Density (g/cm ³)	ASTM D7777	Anton Paar DMA 35 Basic Density Meter
Kinematic viscosity at 40°C (mm ² /s)	ASTM D7042	Anton Paar SVM 3001 Viscometer
Water content (ppm)	IEC 60814	Metrohm 831 Karl Fischer
Acidity (mg KOH/g)	IEC 62021-1	Metrohm 888 Titrand
DGA	IEC 60567	Agilent 6890 Gas Chromatograph

3.10.1 Flash Point

The flash point of the nanofluid oil sample was measured using a Stanhope-SETA Cleveland Open Cup apparatus, following ASTM D92 (ASTM D92, 2018). The sample was heated at a controlled rate, and a test flame was periodically applied until a flash was observed. The temperature at which the flash occurred was recorded as the flash point.

3.10.2 Density

The density of the oil samples was determined using the DMA 35 basic handheld density meter, as shown in Figure 3.18, following the procedure outlined in ASTM D7777 (ASTM D7777-12, 2018). Each measurement involved introducing a 2-mL volume of the oil sample, which was filled into the measuring cell of DMA 35 by simply pushing the lever on the built-in pump, and the density measurement displayed was recorded. The

measurement process was repeated thrice for each sample, and the resulting values were averaged to obtain the representative density.

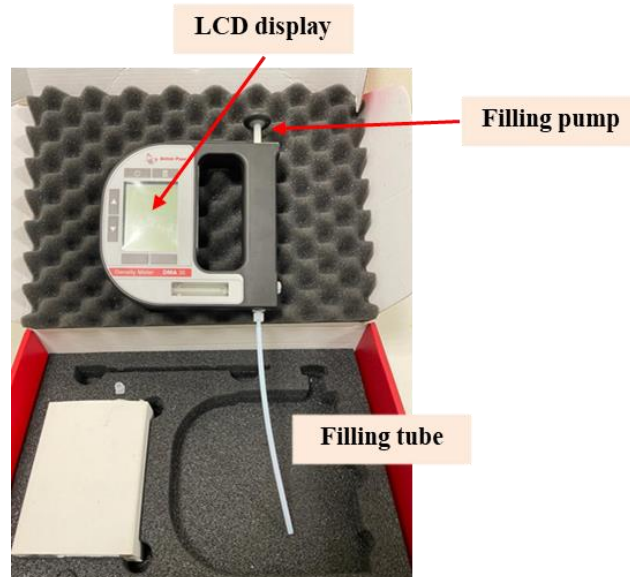


Figure 3.18: DMA 35 Anton Paar handheld density meter

3.10.3 Kinematic Viscosity

Kinematic viscosity measurements at 40 °C were determined based on standard compliance of ASTM D7042 (ASTM D7042, 2004) using an automatic viscometer SVM 3001 of Anton Paar with approximately 1.5-mL of nanofluid oil samples.

3.10.4 Water Content

The water content of the nanofluid samples was measured by using the coulometer model Methrohm 831 Karl Fischer (KF) following the IEC 60814 method (IEC 60814, 1997). The 1-mL oil sample was pipetted into an automatic coulometric KF titration cell. The coulometer will automatically detect the water content by measuring the amount of iodine consumed in the reaction with water.

3.10.5 Acidity

The measurement of acidity in nanofluid samples was performed using Metrohm 888 titrator under the IEC 62021-1 standard (IEC 62021-1, 2003). The acidity measurement for each nanofluid sample involved using 50-mL of oil.

3.10.6 Dissolved Gas Analysis (DGA)

DGA of the 1000-h thermally aged nanofluid samples was carried out using Agilent 6890 Gas Chromatograph as per IEC 60567 standard (IEC 60567, 2011).

3.11 Chapter Summary

This chapter detailed the research methodology employed in this study. The chemically modified palm oil, through a sequential transesterification process, addition of CTAB surfactant, and addition of nanoparticles, was investigated as a potential alternative transformer liquid. The process starts with the chemical conversion of POME from RDBPO_o using a transesterification process, followed by the preparation of POME-based nanofluid samples and the accelerated thermal ageing experiment of the nanofluid samples. Detailed explanations were provided for the characterisation of nanoparticles using FESEM, POME using FTIR and GC–MS, and aged nanofluids using FTIR and UV–Vis. The assessments of the electrical and physicochemical properties of thermally aged nanofluids were discussed. The evaluation of the modified and aged nanofluid samples is presented in Chapter 4.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview

This chapter represents the heart of the thesis, where the results, analyses, and findings obtained from the laboratory work, assessments, and measurements are presented and critically examined. Subsections 4.2 and 4.3 cover the characterisation of the laboratory-synthesised POME and the used nanoparticles, respectively. Subsection 4.4 continues with the characterisation of POME-based nanofluids. The final subsections, 4.5 and 4.6, deliberate on the effects of accelerated thermal ageing on the prepared nanofluids.

4.2 Characterisation of Laboratory-Synthesised POME

Characterisation using FTIR and GC–MS of the laboratory-synthesised POME is carried out to ascertain the formation of methyl ester oil.

4.2.1 FTIR Spectra Analysis

The FTIR spectra of a sample of RBDPO_o and the POME obtained from the transesterification process are presented in Figure 4.1. The transesterification yielded a product that is chemically similar to the precursor; only slight variations can be noticed between the two spectra. The wavenumbers at 3005, 2922, and 2852 cm⁻¹ represent both RBDPO_o and POME; its C–H alkyl stretching (Maharana, 2019). The stretching vibration of C=O at 1742 cm⁻¹, typical of ester and thus common in both RBDPO_o and POME spectra, is the most characteristic absorption band in the spectra of RBDPO_o and POME (Rabelo et al., 2015; Torres et al., 2020). The peak at wavenumber 1436 cm⁻¹ corresponds to the asymmetric stretching of –CH₃ present in the POME spectra and was absent in the RBDPO_o

spectra (Torres et al., 2020). Again, the peak at the wavenumber 1195 cm^{-1} represents the single bond stretching of C–O, confirming the formation of ester (Maharana, 2019).

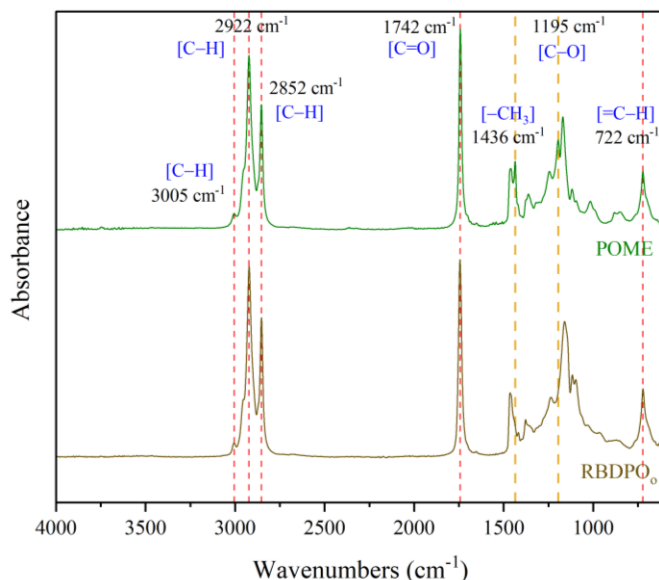


Figure 4.1: FTIR spectra of RBDPO₀ and POME

4.2.2 Gas Chromatography–Mass Spectra Analysis

Figure 4.2 illustrates the GC–MS chromatogram of laboratory-synthesised POME. The x- and y-axis represent the retention time in minutes and intensity in %, respectively. The results of methyl ester analysis using GC-MS prove that the product produced by the transesterification process in this study contains 100% fatty acid methyl ester. The chromatogram of POME identified the presence of six major methyl esters between C_{12:0} and C_{25:0}: methyl laurate, methyl myristate, methyl stearate, methyl arachidate, methyl behenate, and methyl tetracosanoic. Stearic acid (C_{18:0}) showed a prominent presence. Each peak corresponds to the POME's fatty acid methyl ester content and was identified from the library match software (NO. NIST 17). The result shows that a single run of FAME analysis

required 45 minutes. The major peaks of the GC–MS analysis, which are the methyl ester compositions, are tabulated in Table 4.1. The percentages of stearic acid and myristic acid are observed to be the highest, at 82.7% and 6.89%, respectively.

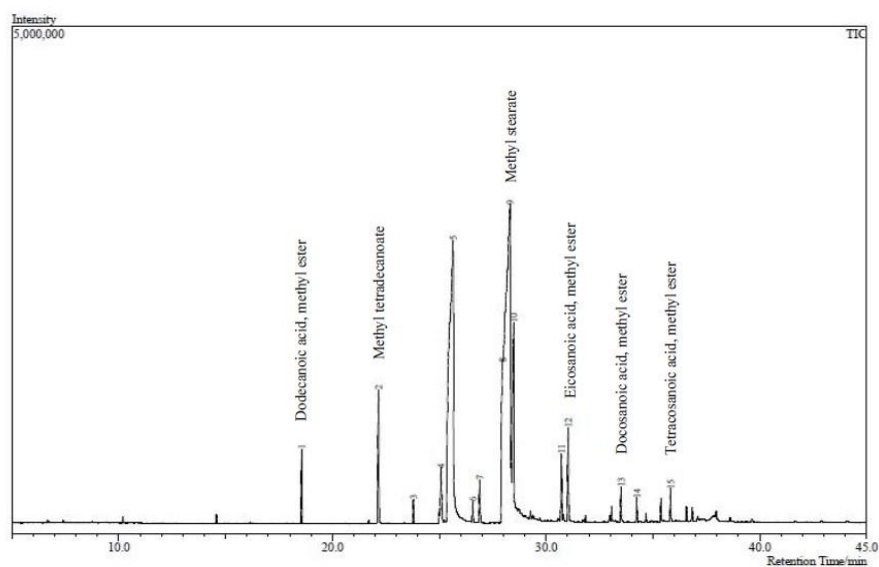


Figure 4.2: GC–MS chromatogram of laboratory-synthesised POME

Table 4.1: Major compositions of POME

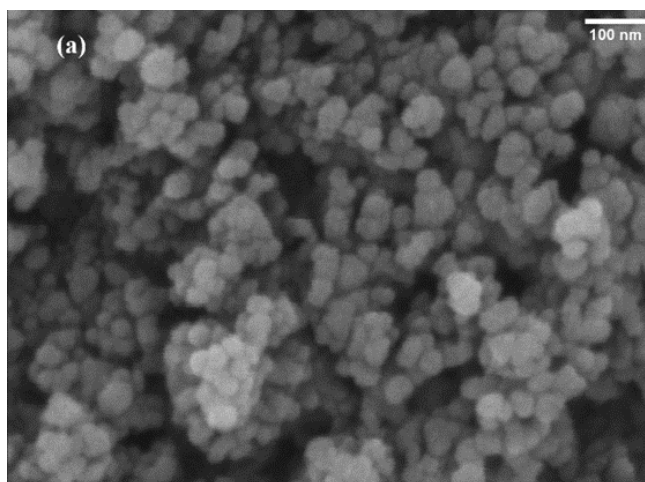
FAME composition	Compounds identified	Retention time (min)	Molecular Formula	Peak area	Area percentage (%)
Lauric acid (C _{12:0})	Dodecanoic acid, methyl ester	18.56	C ₁₂ H ₂₄ O ₂	1750485	2.92
Myristic acid (C _{14:0})	Methyl tetradecanoate	22.16	C ₁₄ H ₂₈ O ₂	4141184	6.89
Stearic acid (C _{18:0})	Methyl stearate	28.31	C ₁₈ H ₃₆ O ₂	49645887	82.70

Table 4.1: continued

Arachidic acid (C _{20:0})	Eicosanoic acid, methyl ester	31.04	C ₂₀ H ₄₀ O ₂	2827135	4.71
Behenic acid (C _{22:0})	Docosanoic acid, methyl ester	33.51	C ₂₂ H ₄₄ O ₂	810480	1.35
Tetracosanoic acid (C _{24:0})	Tetracosanoic acid, methyl ester	35.82	C ₂₄ H ₄₈ O ₂	873538	1.45

4.3 Characterisation of Used Nanoparticles

Figures 4.3(a) and (b) show the FESEM images of the applied TiO₂ and MWCNT nanoparticles at different magnifications, respectively. The TiO₂ particles were observed to be spherical, while the MWCNT appeared as cylindrically shaped tubes and had multiple concentric tubes in a single configuration.



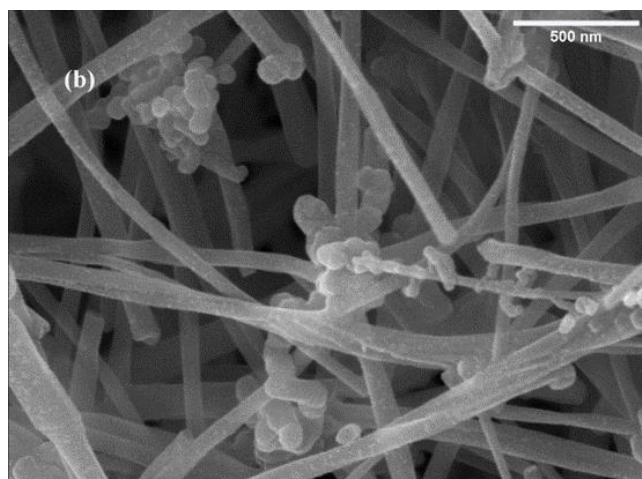


Figure 4.3: FESEM images of (a) TiO₂ and (b) MWCNT nanoparticles showing their morphology at different magnifications

ImageJ software was used to measure the average size of TiO₂ and MWCNT nanoparticles in FESEM images. Data of 50 particles were taken into consideration for measurement to ensure statistical significance, and the results were expressed as the mean $\pm$ standard deviation. Table 4.2 tabulates the average size of both nanoparticles. A higher standard deviation means more variability in the sizes of MWCNT nanoparticles.

Table 4.2: Average size of used nanoparticles

Nanoparticles	Average size (nm)
TiO ₂	37.0 $\pm$ 9.6
MWCNT	69.7 $\pm$ 27.1

4.4 Characterisation of Aged Nanofluids

Figure 4.4 shows the nanofluid samples that were prepared by dispersing the two types of nanoparticles before and after the ageing process of 1000 h. It is apparent to notice the colour difference when comparing fresh nanofluid samples to 1000-h aged samples, as well as the colour difference between the two types of nanofluids and the colour difference among the nanofluid samples with the lowest and highest nanoparticle doping concentrations. The aged nanofluids were darkened due to accelerated ageing. A comprehensive characterisation using FTIR and UV–Vis of the aged nanofluids is conducted and compared to demonstrate the significant impact of accelerated ageing on the nanofluid samples.

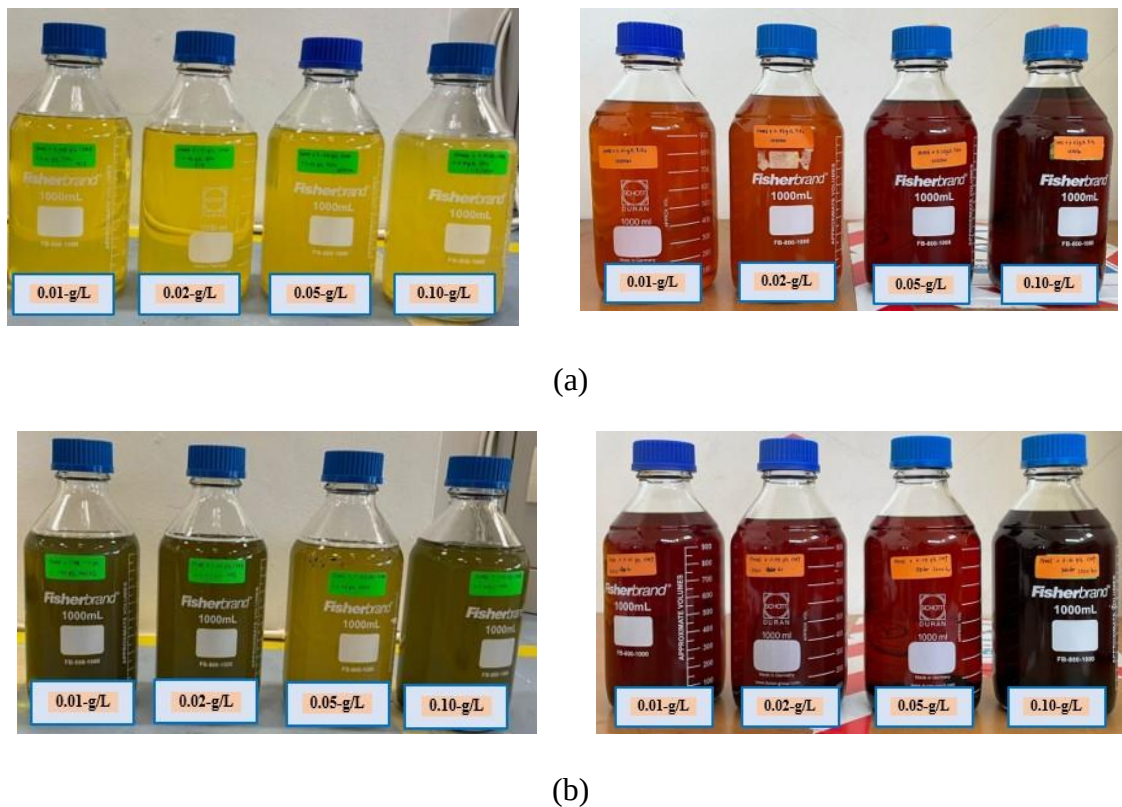
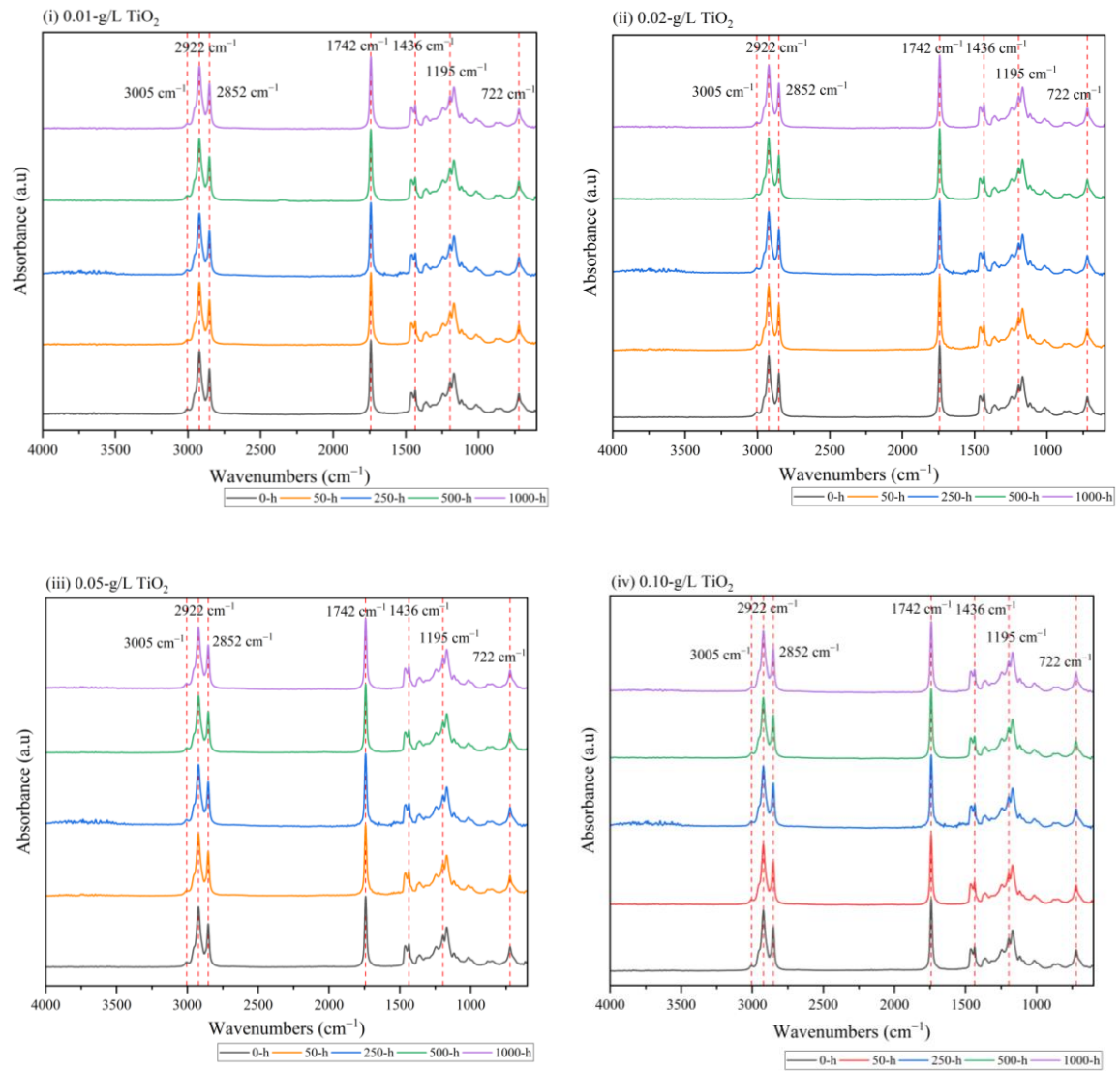


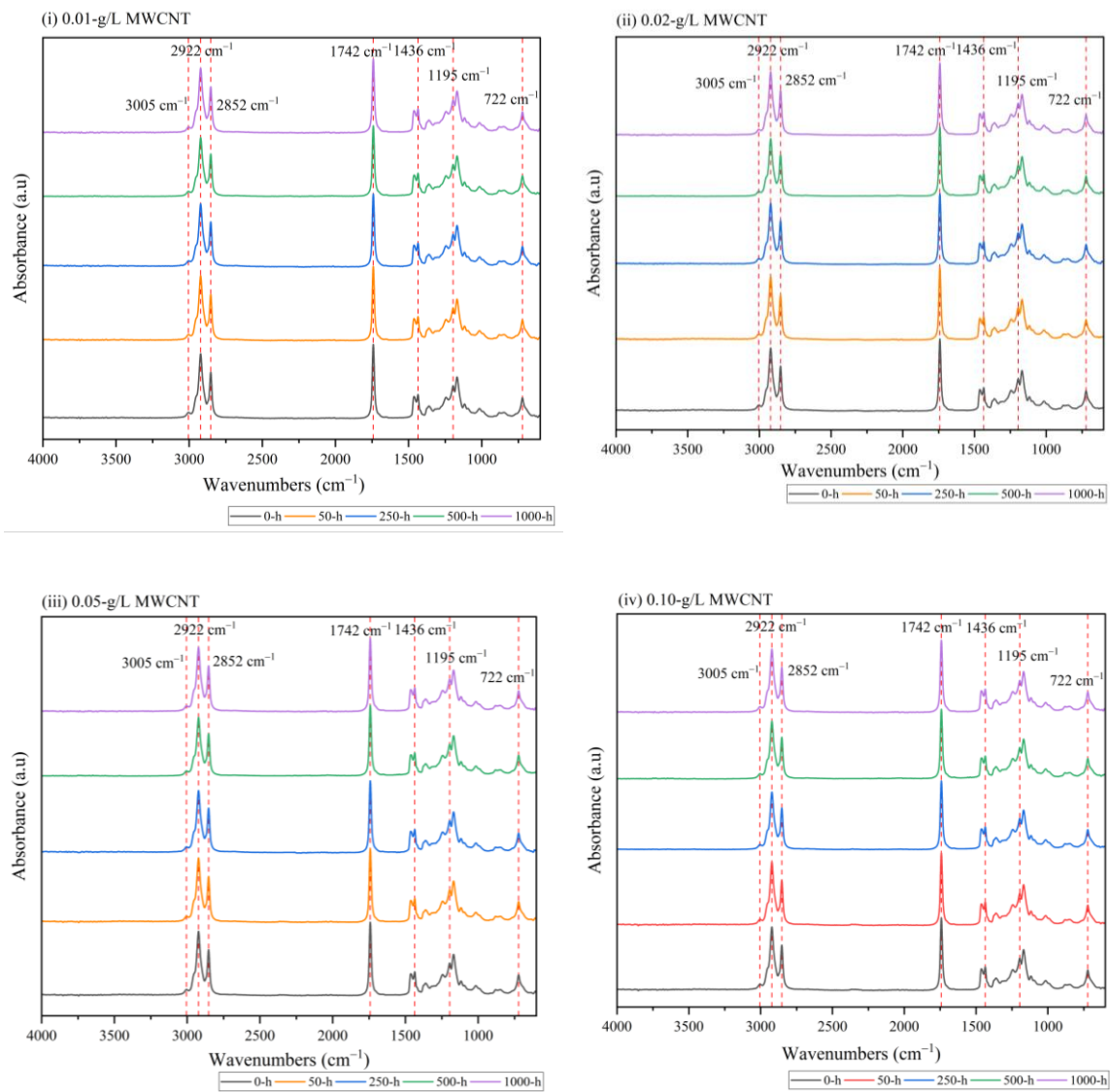
Figure 4.4: Visual observation of (a) TiO_2 and (b) MWCNT nanofluid samples before (left) and after (right) the 1000-h accelerated ageing

4.4.1 FTIR Spectra Analysis of Aged Nanofluids

The FTIR spectra of aged nanofluids were recorded and compared to examine the chemical composition changes with ageing. The comparative FTIR analysis in the POME-based TiO_2 and MWCNT nanofluids with ageing at 0-, 50-, 250-, 500-, and 1000-h are shown in Figures 4.5 (a) and (b), respectively.



(a)



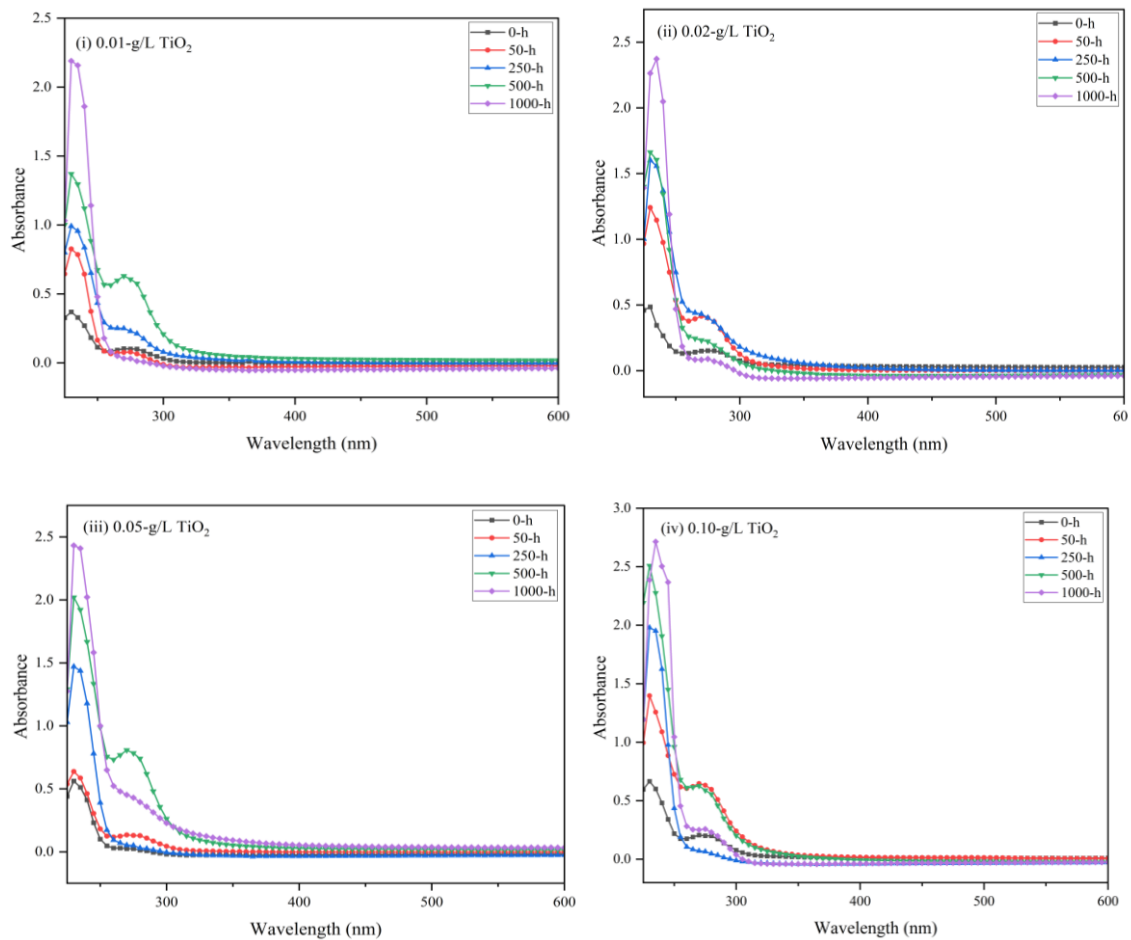
(b)

Figure 4.5: FTIR spectra of (a) TiO_2 and (b) MWCNT nanoparticles at varied concentrations and ageing durations

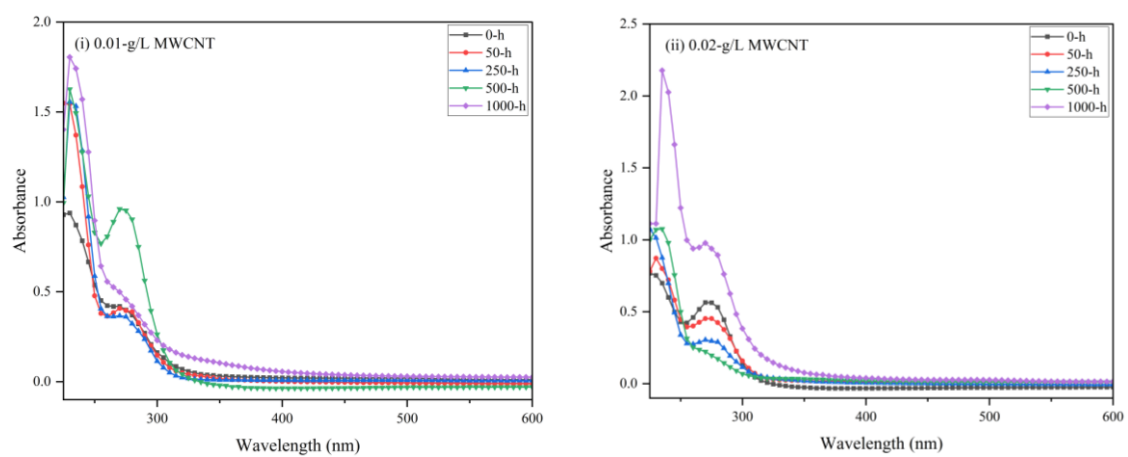
Both POME-based nanofluids share similarities in their spectra at 3005, 2922, 2852, and 1742 cm^{-1} wavenumbers, which is evidence that both of their compositions contain a similar chemical group. Visual examination of the spectra for both oil samples indicated that the introduction of TiO_2 and MWCNT nanoparticles, CTAB surfactant, and the accelerated ageing process did not significantly alter the spectra. These results suggest the absence of interactions between the POME as the base fluid and the additives. The outcomes are in line with prior studies conducted by (Dombek et al., 2018; Maharana et al., 2019), which examined thermal ageing effects on various types of esters dispersed with nanoparticles.

4.4.2 Ultraviolet–Visible Analysis

The UV–Vis spectra of the aged POME-based TiO_2 and MWCNT nanofluids are presented in Figures 4.6 (a) and (b) as a graph of absorbance on the y -axis and wavelength on the x -axis. Figures 4.6 (a) and (b) show that as the nanofluids undergo thermal ageing, the spectra have shifted to a higher absorbance value and longer wavelength for 1000-h-aged nanofluid samples. This shift indicates the presence of ageing products, probably resulting from the sedimentation of nanoparticles, and is correlated with an increase in oil concentration due to ageing (Amalanathan, 2020; Chakraborty et al., 2021). The highest increase is observed in 1000 h-aged 0.10-g/L MWCNT nanofluid, as shown in Figure 4.6 (b)(iv). This phenomenon is consistent with the visual observation of the darkening of nanofluid colour (Amalanathan, 2020).



(a)



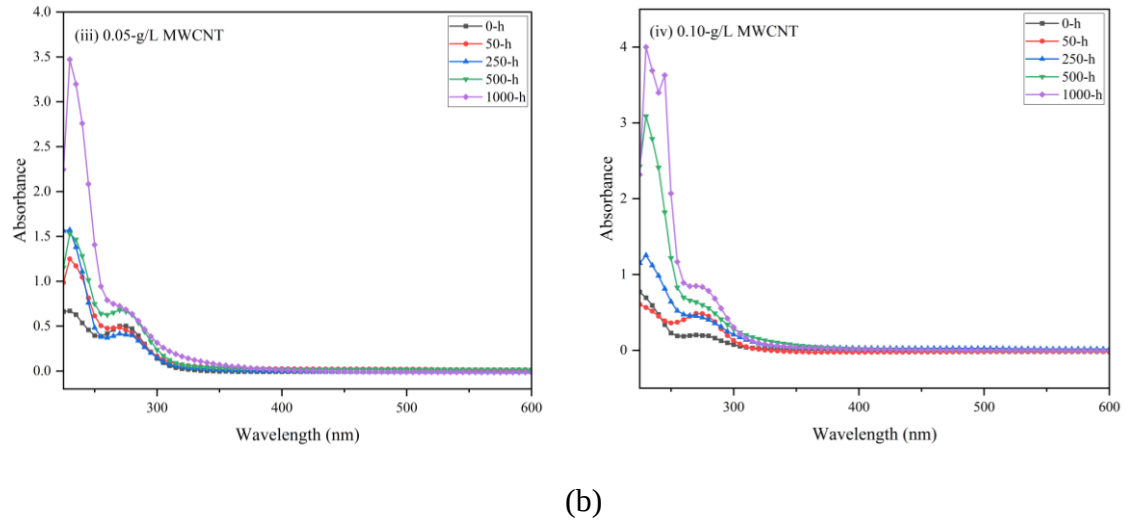


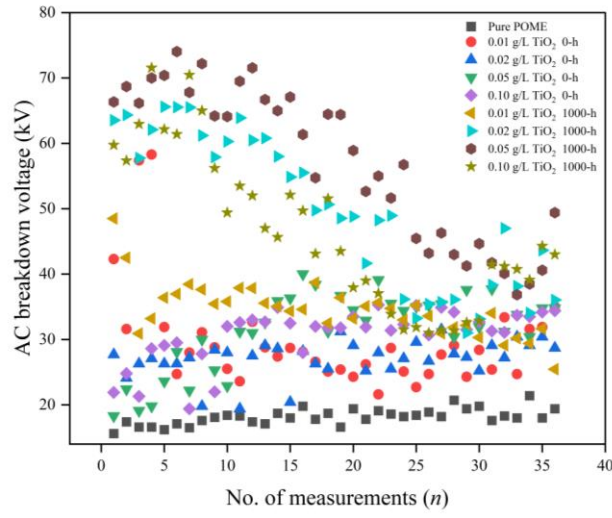
Figure 4.6: UV–Vis spectra of (a) TiO₂ and (b) MWCNT nanofluids at varied concentrations and ageing durations

4.5 Effects of Accelerated Thermal Ageing on Electrical Properties of POME-based Nanofluids

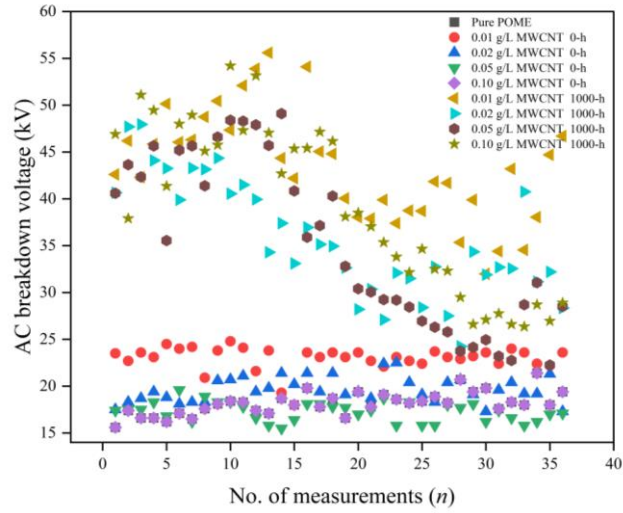
The aged nanofluids were further tested and assessed according to the IEC 60156 standard method.

4.5.1 Analysis of Average AC Breakdown Voltage

The changes in AC BDV for the two types of aged POME-based nanofluids are shown in Figures 4.7 (a) and (b), respectively.



(a)



(b)

Figure 4.7: Scatter plots of AC BDV of pure POME, fresh, and 1000-h-aged POME-based (a) TiO_2 and (b) MWCNT nanofluids at varied concentrations

The scatter plots show the variation in the distribution pattern of the 36 AC BDV readings for pure POME, POME-based TiO_2 , and POME-based MWCNT nanofluids before and after 1000 h of ageing at varied concentrations. It can be seen that the BDV of the pure

POME varied between 15–21 kV. After 1000 h of ageing, it is observed that the scattered data showed the AC BDV readings range between 25.45–74.05 kV. Interestingly, the trend demonstrated that adding nanoparticles at all doping concentrations had no negative effect on the AC BDV. Overall, it is shown that the aged POME-based TiO₂ nanofluids have greater dielectric strength compared to their counterparts of aged POME-based MWCNT nanofluids. The POME-based TiO₂ nanofluids exhibited a significant enhancement of 74.05 kV in BDV by up to 233% at a 0.05-g/L doping concentration.

On the other hand, the most significant enhancement in BDV for POME-based MWCNT nanofluids was at a 0.01-g/L concentration of as much as 95.8%, with 43.37 kV. It can be observed that as the doping concentrations of POME-based MWCNT nanofluids increase, there is a tendency for the BDV to decrease. The decrease could be attributed to the instability of the MWCNT nanofluids, resulting from the formation of aggregations and sedimentation of MWCNT nanoparticles. A similar result was reported by Suhaimi et al. (2022b) when mineral oil was used as the base fluid. The addition of CNTs to transformer oil has been shown to significantly impact the BDV. At lower concentrations, the adsorption of free charge by CNTs can slow down the movement of fast-moving free charges, which can improve the BDV of the nanofluids. However, at a higher concentration of the CNTs, the electric field distortion caused by the presence of the CNTs can enhance the breakdown propagation of the streamer, leading to earlier breakdown, as proposed by (Hwang et al., 2010). The average of AC BDV at varied concentrations for these two types of 1000-h-aged nanofluids is compared in Figure 4.8.

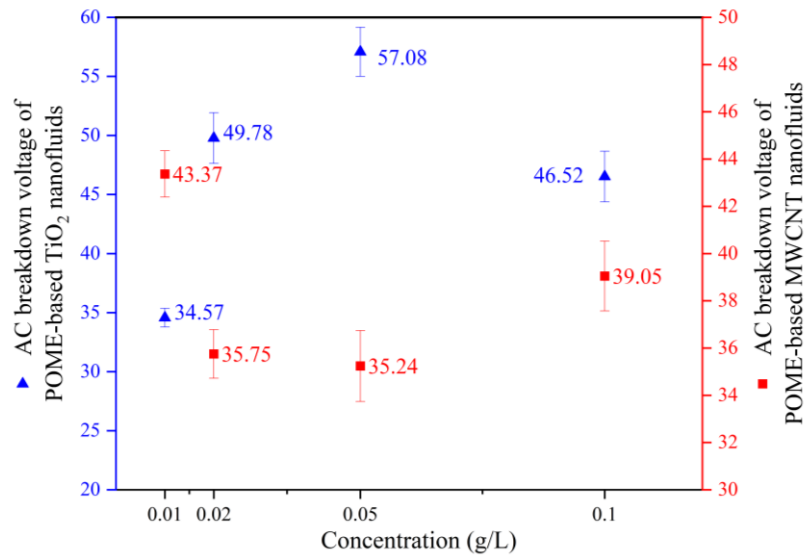


Figure 4.8: Average AC BDV of 1000-h-aged POME-based nanofluids

Overall, it is demonstrated that the aged POME-based TiO₂ nanofluids have greater dielectric strength compared to their POME-based MWCNT counterparts. The highest average BDV of 57.08 kV at a 0.05-g/L concentration of TiO₂.

Table 4.3 provides comprehensive descriptive statistics on the grouped AC BDV data. According to (ASTM D877-02, 2002), the acceptable criteria for the range or mean percentage of recorded BDV should be less than 92% of the average value; otherwise, the measurement test must be repeated. Observing that every sample was recorded below the allowable range criteria, the collected results in this study are considered credible.

Table 4.3: Descriptive statistics of 36 measurements of 1000-h-aged nanofluids

Samples	POME-based TiO ₂				POME-based MWCNT			
Concentrations(g/L)	0.01-	0.02-	0.05-	0.10-	0.01-	0.02-	0.05-	0.10-
Average (kV)	34.57	49.78	57.08	46.52	43.37	35.75	35.24	39.05
Standard Deviation (kV)	4.05	11.63	11.78	11.83	5.86	6.21	9.02	8.85
Minimum	25.45	31.00	36.85	31.00	32.00	24.25	22.25	26.35
Median	34.68	49.35	60.13	43.9	42.9	34.33	34.18	38.30
Maximum	48.50	65.60	74.10	71.6	55.60	47.95	49.10	54.20
Range	23.05	34.60	37.25	40.60	23.60	23.70	26.85	27.85
(Range/Average) ×100	66.68	69.51	65.26	87.27	54.42	66.29	76.19	71.31

4.5.2 Statistical Analysis of AC Breakdown Voltage

The AC BDV data were statistically analysed in accordance with IEC 62539 (IEC 62539, 2007). Normality testing is frequently an initial step in data analysis. The Anderson-Darling (AD) goodness-of-fit test determines whether the experimental data of AC BDV measurements meet the normality assumption. The p -value, determined by the AD statistic, serves as the guideline for conformity (Zhou et al., 2018). Based on the statistics, a greater p -value than the level of significance, alpha (α), means that the data is not statistically different from a normal distribution. The p -value of 0.05 was considered statistically significant, and the p -value for normal distribution was calculated and compared to the value of α using the AD statistic.

4.5.2.1 Normal Distribution

The frequency distribution of the AC BDV data set is graphically visualised using the histograms of all nanofluid samples, which are displayed in Figures 4.9 and 4.10.

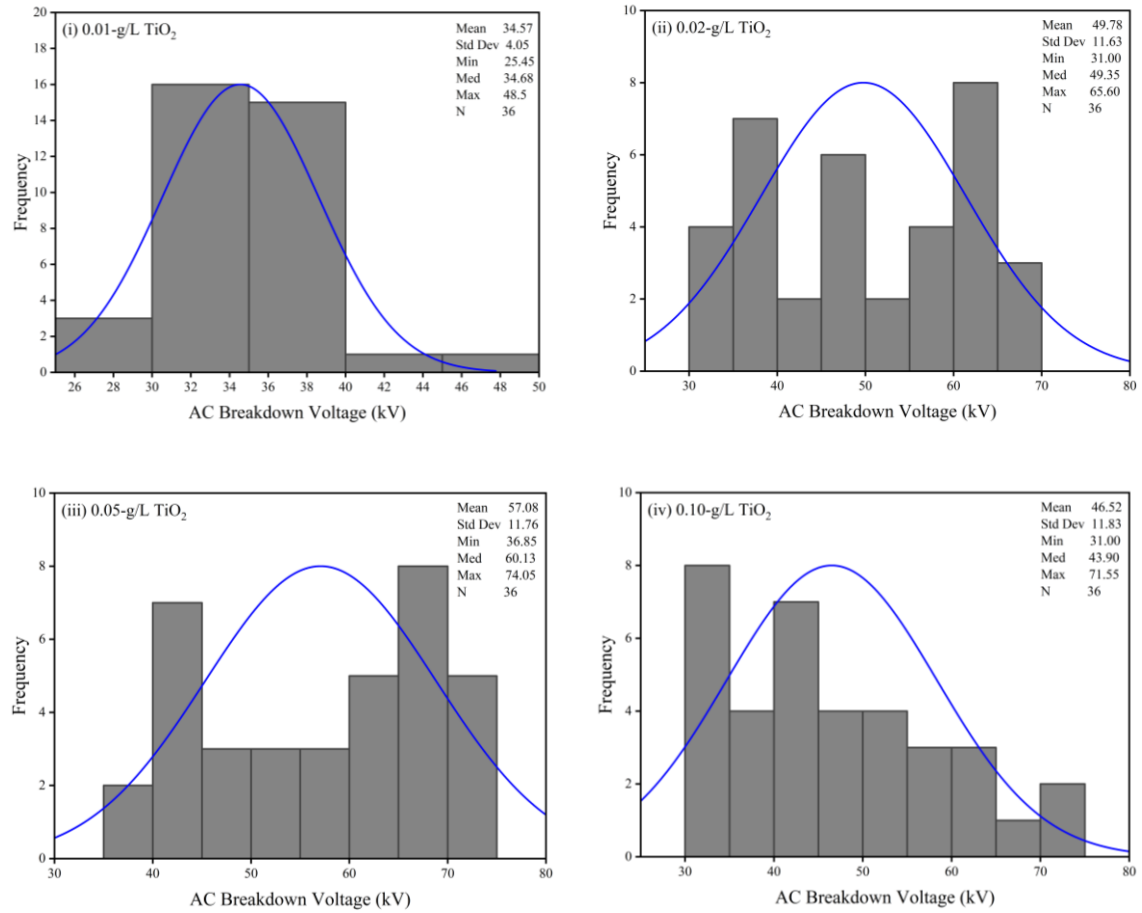


Figure 4.9: Histograms of 1000-hour-aged POME-based TiO_2 at (i) 0.01-g/L, (ii) 0.02-g/L, (iii) 0.05-g/L, and (iv) 0.10-g/L concentrations

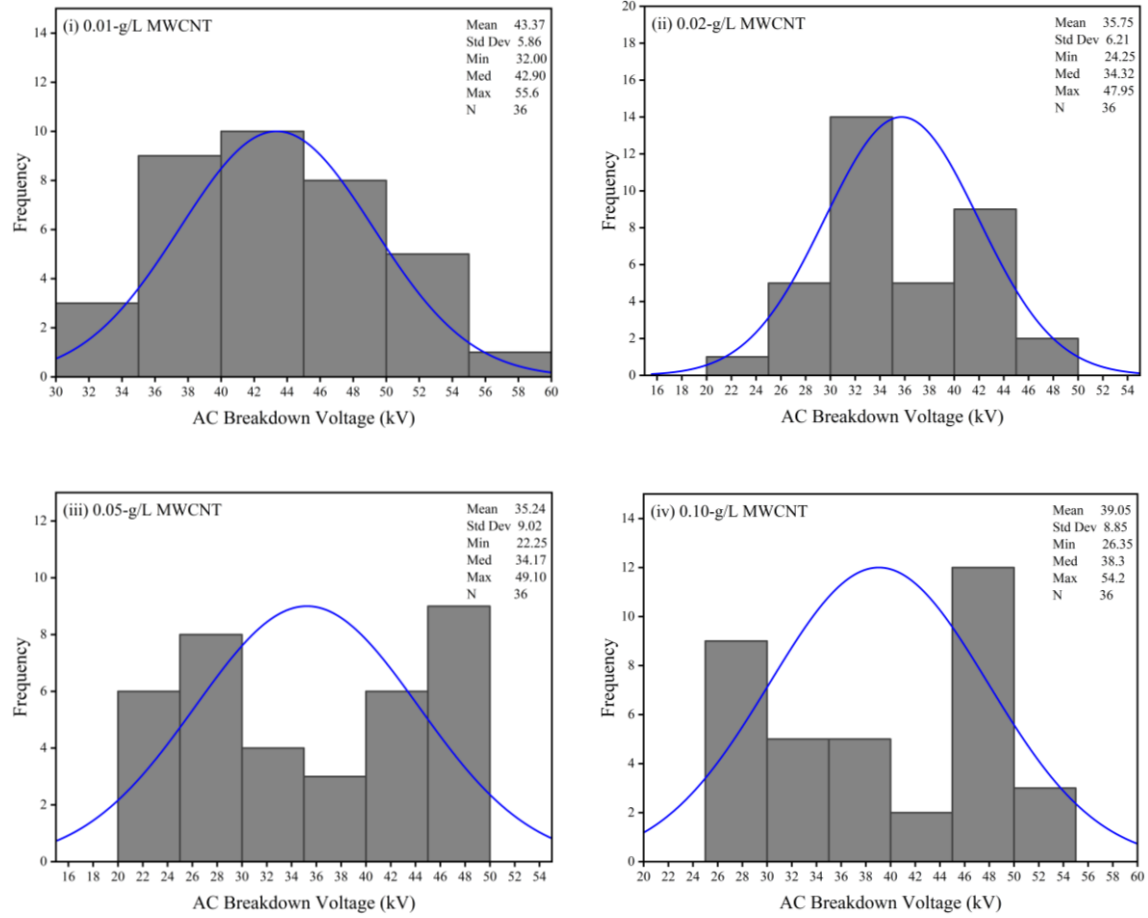


Figure 4.10: Histograms of 1000-h-aged POME-based MWCNT at (i) 0.01-g/L, (ii) 0.02-g/L, (iii) 0.05-g/L, and (iv) 0.10-g/L concentrations

The asymmetric histograms of the BDVs suggested that some of the experimental results did not follow the normal distribution. Table 4.4 summarises the *p*-values for the experimental data, as well as the normality or non-normality of the investigated aged nanofluids.

Table 4.4: Hypothesis test of conformity to a normal distribution of 1000-h-aged nanofluids

Nanofluid samples	<i>p</i> -values	Conformity to normal distribution
POME + 0.01-g/L TiO ₂	0.13655	Accepted
POME + 0.02-g/L TiO ₂	0.00589	Rejected
POME + 0.05-g/L TiO ₂	0.14599	Accepted
POME + 0.10-g/L TiO ₂	0.00238	Rejected
POME + 0.01-g/L MWCNT	0.86184	Accepted
POME + 0.02-g/L MWCNT	0.62982	Accepted
POME + 0.05-g/L MWCNT	0.00500	Rejected
POME + 0.10-g/L MWCNT	0.01209	Rejected

It is discovered that the datasets of POME + 0.01-g/L TiO₂, POME + 0.05-g/L TiO₂, POME + 0.01-g/L MWCNT, and POME + 0.02-g/L MWCNT obey the normal distribution, where the *p*-values are higher than 0.05. Skewness and kurtosis are used as evaluation metrics when assessing the experimental data for independence and conformity to a normal distribution. Table 4.5 tabulates the skewness and kurtosis of the 1000-h-aged nanofluid samples. The data strictly conforms to the normal distribution when skewness equals 0 and kurtosis equals 3 (Zhou et al., 2018). The aged nanofluid samples exhibited varying skewness and kurtosis, indicating a departure from normality. The negative skewness value indicates a negatively skewed distribution, indicating that most AC BDV data were concentrated on the right side of the distribution, with a longer tail extending to the left. Conversely, a positive skewness value represents a positively skewed distribution, indicating that AC BDV data were concentrated on the left side of the distribution, with a longer tail

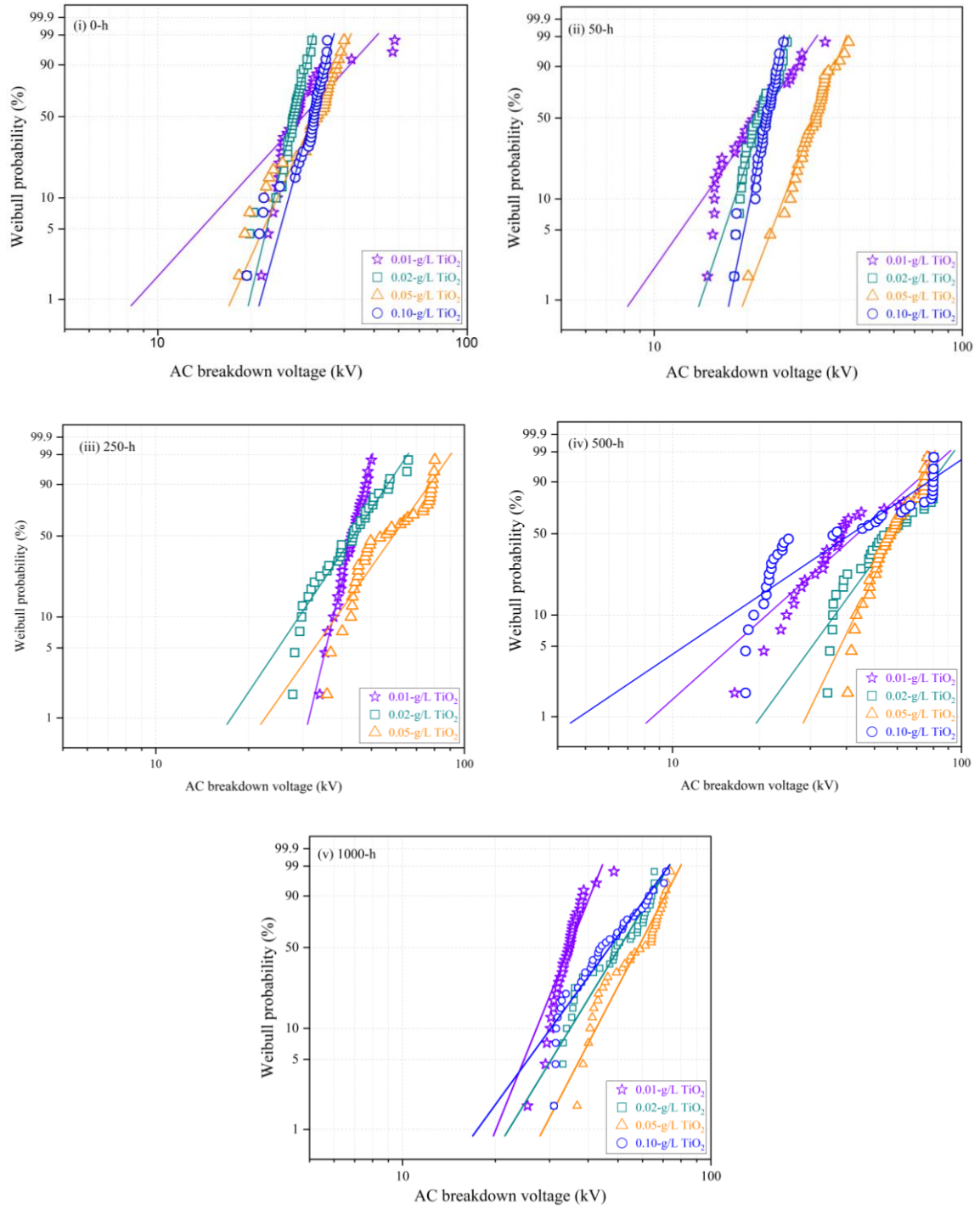
on the right, as seen in Figure 4.10 (i) – (iii). The kurtosis values of less than 3 suggest that the collected AC BDV data have lighter tails than a normal distribution, meaning that the data are less likely to have extreme values.

Table 4.5: Skewness and kurtosis of AC BDV of 1000-h-aged nanofluids

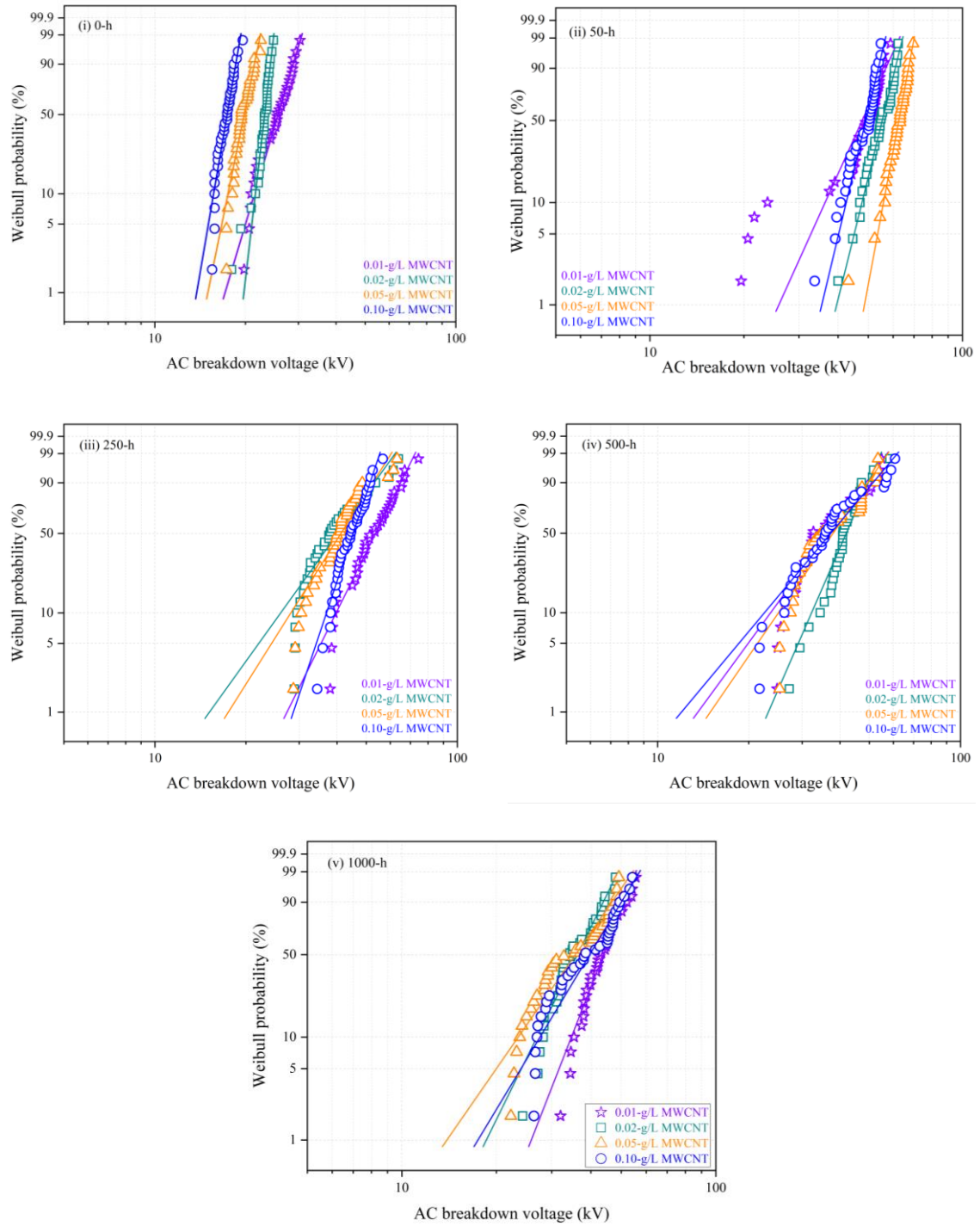
Nanofluid samples	Skewness	Kurtosis
POME + 0.01-g/L TiO ₂	0.9415	3.2911
POME + 0.02-g/L TiO ₂	−0.1461	−1.4824
POME + 0.05-g/L TiO ₂	−0.3046	−1.4332
POME + 0.10-g/L TiO ₂	−0.4618	−0.7491
POME + 0.01-g/L MWCNT	0.2281	−0.4668
POME + 0.01-g/L MWCNT	0.2673	−0.8682
POME + 0.01-g/L MWCNT	0.1246	−1.5244
POME + 0.01-g/L MWCNT	−0.0299	−1.4120

4.5.2.2 Weibull Distribution

According to the IEC 62539 standard, although the normal distribution is widely recognised and its mean and standard deviation parameters are easy to compute, it is often not usually appropriate for electrical breakdown data. The most common type of failure distribution analysis for electrical breakdown is Weibull (IEC 62539, 2007). The Weibull probability plots were made for the two groups of 1000-h-aged nanofluids, as shown in Figure 4.11, demonstrating how the BDV is mapped onto the corresponding fitting Weibull line. The star, square, triangle, and circle plots indicate the doping concentrations of 0.01-, 0.02-, 0.05-, and 0.10-g/L nanofluids, respectively. Table 4.6 summarises the *p*-values and conformity of the datasets for the Weibull distribution of the nanofluids.



(a)



(b)

Figure 4.11: Weibull probability plots of AC BDV for aged POME-based (a) TiO_2 and (b) MWCNT nanofluids

Table 4.6: Hypothesis test of conformity to Weibull distribution of aged nanofluid samples

Nanofluid samples	<i>p</i> -values	Conformity to Weibull distribution
POME + 0.01-g/L TiO ₂	0.136	Accepted
POME + 0.02-g/L TiO ₂	0.005	Rejected
POME + 0.05-g/L TiO ₂	0.051	Accepted
POME + 0.10-g/L TiO ₂	<0.01	Rejected
POME + 0.01-g/L MWCNT	0.862	Accepted
POME + 0.02-g/L MWCNT	0.629	Accepted
POME + 0.05-g/L MWCNT	0.054	Accepted
POME + 0.10-g/L MWCNT	0.012	Rejected

Noticeably, the experiment data from the low concentrations of nanofluids, particularly those with MWCNT dispersion, conforms to the Weibull distribution, except for the nanofluid sample at a 0.10-g/L concentration. From Figure 4.11 (b), the measured AC BDVs of 1000-h-aged POME-based nanofluids are well represented by fits to Weibull distributions compared to the normal distribution.

The details of the Weibull distribution parameters for 1000-h-aged nanofluids are tabulated in Table 4.7. It is observed that the 63.2% breakdown failure rates of POME-based TiO₂ and MWCNT nanofluids were relatively stable and were almost similar, indicating that adding nanoparticles significantly improved the dielectric strength of the nanofluids. Furthermore, the average AC BDV of the two types of nanofluids met the acceptance limit (>35 kV) of the IEC 60156.

Table 4.7: Weibull parameters of 1000-h-aged oil samples

Oil samples	α (Scale)	β (Shape)
Pure POME	24.2	15.8
POME + 0.01-g/L TiO ₂	36.5	3.4
POME + 0.02-g/L TiO ₂	55.0	13.0
POME + 0.05-g/L TiO ₂	62.1	6.9
POME + 0.10-g/L TiO ₂	51.1	11.3
POME + 0.01-g/L MWCNT	39.5	3.6
POME + 0.02-g/L MWCNT	39.2	3.9
POME + 0.05-g/L MWCNT	37.8	3.0
POME + 0.10-g/L MWCNT	41.1	3.4

The Weibull fit lines, as shown in Figure 4.11, were used to evaluate the AC BDV of the 1000-h-aged nanofluids at three failure probability failures of 1%, 10%, and 50% in comparison to pure POME, and the results are presented in Table 4.8. The assessment of the AC BDV at low probability failures (1% and 10%) probabilities serves as an evaluation of the voltage limit for safety and continuous operation (Fasehullah et al., 2022). Hence, this signifies the reliability of the insulating liquid. The 50% probability is an indication of the average BDV (Khaled and Beroual, 2019).

From the data, it is noted that at $U_{1\%}$, $U_{10\%}$, and $U_{50\%}$ breakdown probabilities, the AC BDVs of 1000-h-aged POME-based TiO₂ nanofluids at a 0.05-g/L concentration were increased by 58.6%, 101.9%, and 145.2%, respectively. In general, POME-based TiO₂ nanofluids consistently demonstrate an improvement in AC BDV across all doping concentrations, accompanied by higher increment percentages, indicating a positive impact.

Conversely, MWCNT nanofluids generally exhibit a decline in AC BDV, indicating a negative impact, aligning with findings from a previous study (Suhaimi et al., 2018). This study suggested that the primary reason for the decrement may be attributed to the high surface energy of MWCNT, leading to agglomeration that adversely affects the performance of palm oil. The findings underscore the potential of POME-based TiO₂ nanofluids as promising alternatives to conventional mineral oil, as they showed improvements in electrical properties.

Table 4.8: AC BDV at 1%, 10%, and 50% probability levels

Nanofluid samples	U _{1%}		U _{10%}		U _{50%}	
	AC BDV (kV)	Increment (%)	AC BDV (kV)	Increment (%)	AC BDV (kV)	Increment (%)
Pure POME	18.1	-	21.1	-	23.7	-
POME + 0.01-g/L TiO ₂	20.2	11.6	27.4	29.9	34.8	46.8
POME + 0.02-g/L TiO ₂	22.2	22.7	35.3	67.3	50.6	113.5
POME + 0.05-g/L TiO ₂	28.7	58.6	42.6	101.9	58.1	145.2
POME + 0.10-g/L TiO ₂	17.5	-3.31	30.4	44.1	47.0	98.3
POME + 0.01-g/L MWCNT	10.8	-40.3	21.0	-0.5	35.6	50.0
POME + 0.02-g/L MWCNT	12.3	-32.0	22.2	5.2	35.7	50.6
POME + 0.05-g/L MWCNT	8.3	-54.1	17.9	-15.2	33.5	41.4
POME + 0.10-g/L MWCNT	17.5	-3.31	27.7	31.3	39.8	67.9

4.5.3 Resistivity

Table 4.9 shows the resistivity of the oil samples. It was observed that the nanofluids always presented lower resistivity than pure POME during thermal ageing. Analysing the trend, it was noted that the resistivity values are lower for aged POME-based MWCNT as compared to POME-based TiO₂ nanofluids. In summary, the nanofluid samples exhibit a decreasing trend in resistivity compared to pure POME after the 1000-h ageing period. This trend indicates that the resistivity of the oil samples deteriorates due to the increasing levels of sludge, polar impurities, and unwanted byproducts following extended ageing periods, which is consistent with previous findings (Baruah et al., 2019; Gutierrez et al., 2023).

Table 4.9: Resistivity of oil 1000-h-aged nanofluid samples

Nanofluid samples	Resistivity ($\times 10^9 \Omega m$)
Pure POME	1.26
POME + 0.01-g/L TiO ₂	0.21
POME + 0.02-g/L TiO ₂	0.13
POME + 0.05-g/L TiO ₂	0.31
POME + 0.10-g/L TiO ₂	0.26
POME + 0.01-g/L MWCNT	0.04
POME + 0.02-g/L MWCNT	0.04
POME + 0.05-g/L MWCNT	0.04
POME + 0.10-g/L MWCNT	0.08

A published report (Raof, 2021) also demonstrated a similar trend of declining oil resistivity over ageing durations. It is also observed that while the resistivity varied with the

doping concentrations of the nanoparticles, these variations were not significant enough to warrant further discussion.

4.6 Effects of Accelerated Thermal Ageing on Physicochemical Properties of POME-based Nanofluids

The aged POME-based nanofluids were further evaluated based on the tests according to standard methods. The oil physicochemical parameters analysed as a part of the study were the flash point, density, kinematic viscosity, water content, total acid number, and DGA.

4.6.1 Flash Point

The measurements of the flash points for all aged nanofluids ranged from 171 to 182 °C. These values are not complying with the IEEE C57.147 (≥ 275 °C) and IEC 62770 (≥ 250 °C) standards. However, the flash point of aged POME-based nanofluids remains higher than that of mineral oil, comparable to other types of methyl ester-based oils investigated earlier and commercial natural ester oil PFAE, as compared in Table 4.10.

Table 4.10: Comparison of flash points of different insulating oils

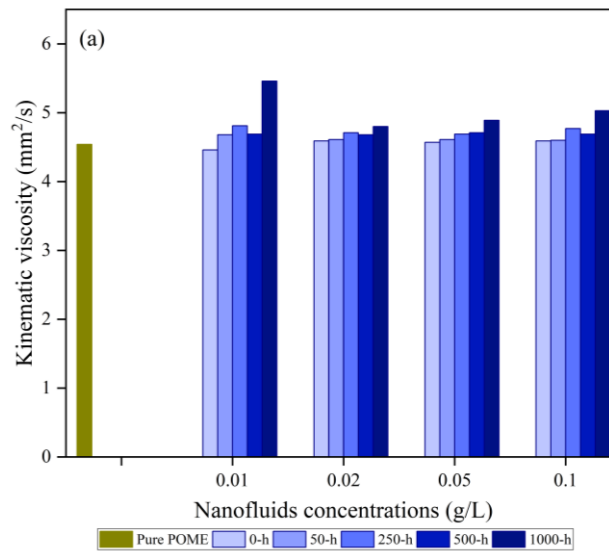
Oil samples	Flash point (°C)
Mineral oil	110–150
PFAE (Kano et al., 2008; Tokunaga et al., 2016)	176–178
JMEO (Sitorus et al., 2016)	191
Neem oil ester (Oparanti, et al., 2022)	175
PKOME (Boyekong et al., 2021)	167
POME-based nanofluids (current study)	171–182

4.6.2 Density

The measured density of both types of aged POME-based nanofluids at 0.01–0.10-g/L doping concentrations for 0–1000-h of ageing durations according to ASTM D7777 is 0.8733–0.8779 g/cm³. The density of the aged POME-based nanofluids is discovered to stay constant as the doping concentrations and the ageing durations increase. The findings align with earlier studies by (Gutierrez et al., 2023), where the investigation was conducted with mineral oil and different natural ester oils.

4.6.3 Kinematic Viscosity

The kinematic viscosities of the aged POME-based nanofluids measured at 40 °C ranged between 4.54 and 5.27 mm²/s, as presented in Figure 4.12. The bar graphs show that the kinematic viscosity barely increased over the ageing durations.



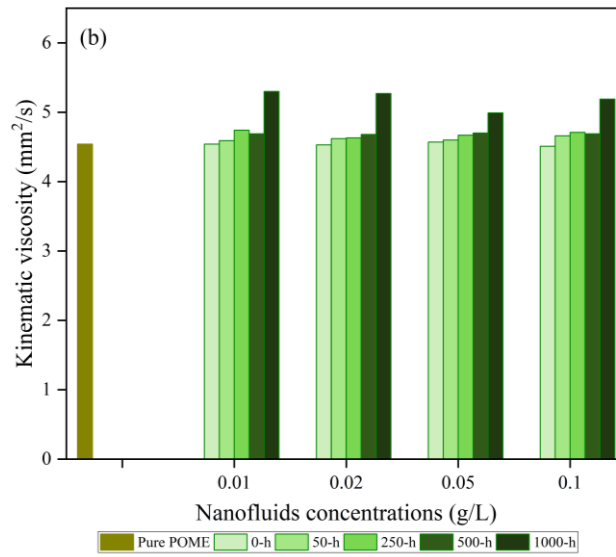


Figure 4.12: Change of kinematic viscosity at different ageing durations for POME-based (a) TiO₂ and (b) MWCNT nanofluids

The comparison of kinematic viscosity between the two types of nanofluids at 1000-h of ageing is tabulated in Table 4.11. The slight increase in the viscosity of the aged nanofluids may be due to an increase in the high molecular ageing product in the oil (Gutierrez et al., 2023; Okikiola et al., 2022), which is consistent with a previous study of different types of methyl esters. As anticipated, the low viscosity of POME-based nanofluids was favourable, indicating superior cooling performance and a more efficient heat dissipation (Lewand, 2004).

Table 4.11: Kinematic viscosity of nanofluid samples

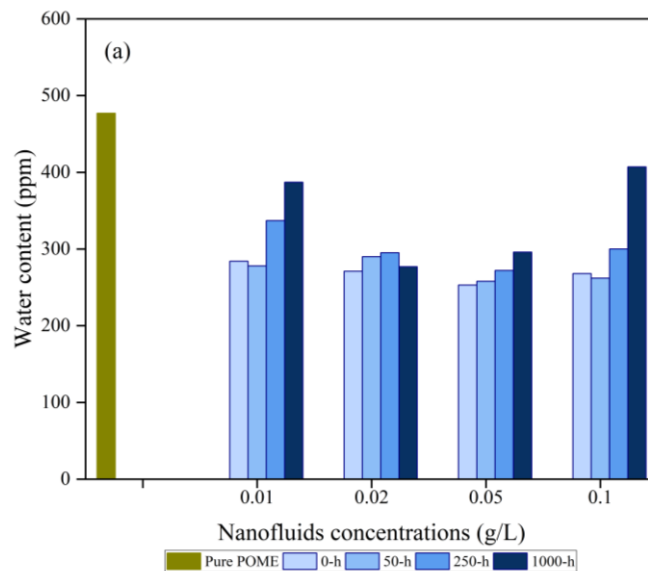
Nanofluid samples	Kinematic viscosity at 40 °C (mm ² /s)
Pure POME	4.54
POME + 0.01-g/L TiO ₂	5.46
POME + 0.02-g/L TiO ₂	4.80
POME + 0.05-g/L TiO ₂	4.89
POME + 0.10-g/L TiO ₂	5.03
POME + 0.01-g/L MWCNT	5.30
POME + 0.02-g/L MWCNT	5.27
POME + 0.05-g/L MWCNT	4.99
POME + 0.10-g/L MWCNT	5.19

4.6.4 Water Content

The test method according to IEC 60814, was used to determine the water content in the oil samples. The water content of the pure POME was 477 ppm. The variations of the water contents at two distinct ageing stages are represented visually as a bar graph, as shown in Figure 4.13.

Based on the results depicted in Figure 4.13, it can be seen that the water content of 1000-h aged POME-based nanofluids was relatively high, ranging between 478–557 ppm, reaching a peak of 557 ppm for POME-based MWCNT at a concentration of 0.02-g/L. These elevated water content levels were observed after extended ageing durations, consistent with findings in a previous study involving different insulating liquids and ageing durations (Singha et al., 2014). This phenomenon aligns with established prior knowledge and can be

attributed to the naturally hygroscopic nature of natural ester oils due to their chemical composition (Bandara et al., 2016; Gutierrez et al., 2023; Jeong et al., 2012; Lewand, 2004). The higher amount of water content than the levels normally found in mineral oil can be attributed to the ageing process (Jeong et al., 2012), during which the liquid generates water. However, when observing their respective AC BDV, it is essential to note that this increase in water content does not necessarily imply an adverse effect on their dielectric strength, as they remain relatively above the requirement limit of the standard.



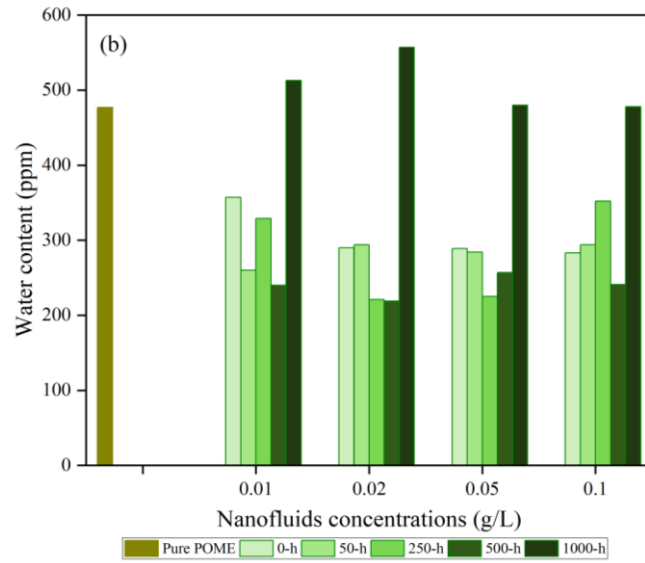


Figure 4.13: Change of water content at different ageing durations for POME-based (a) TiO_2 and (b) MWCNT nanofluids

4.6.5 Total Acid Number (TAN)

Figure 4.14 compares the total acid number (TAN) in nanofluid samples measured at 0-h–1000-h ageing periods. The initial TAN for pure POME was 0.0913 mg KOH/g of oil. Based on the bar graph, it is observed that the acidities of the 1000-h-aged nanofluids were significantly higher than those of fresh nanofluids (0-h) under thermal stress, which agrees with the findings reported by (Bandara et al., 2016; Gutierrez et al., 2023; Matharage et al., 2013; Tokunaga et al., 2016). According to those authors, the significant increase in TAN of natural ester oil with ageing times resulted from the hydrolysis reaction of esters, which produces more fatty acids (Bandara et al., 2016; Tokunaga et al., 2016).

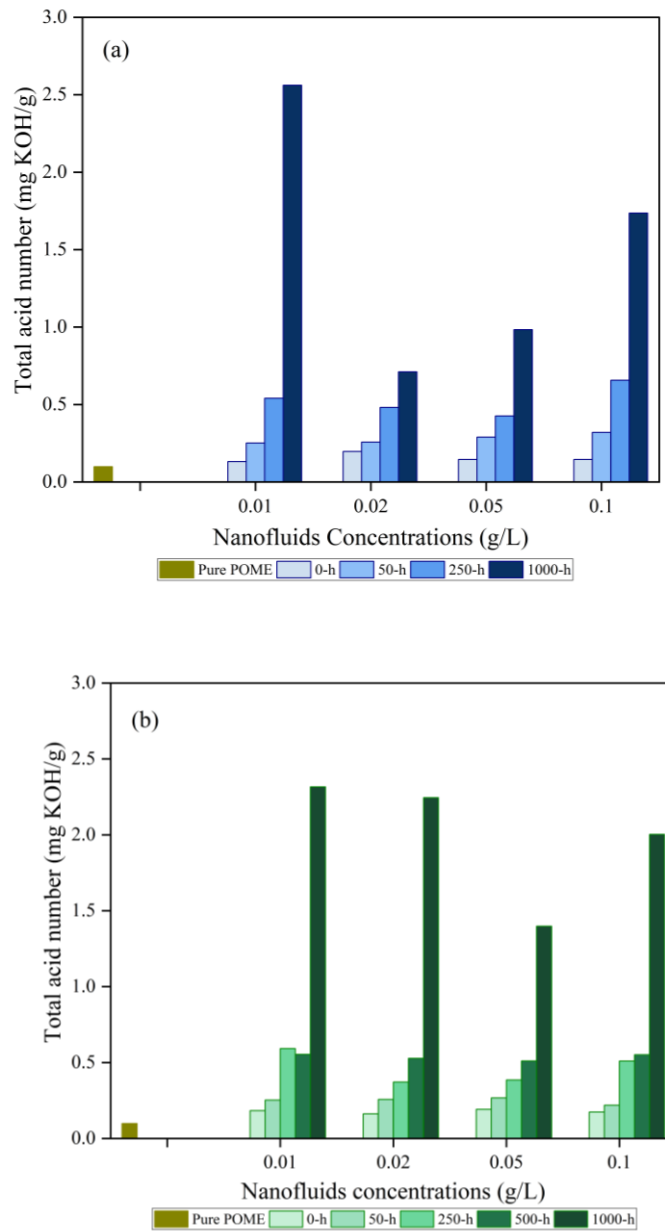


Figure 4.14: Change in TAN at different ageing durations for POME-based (a) TiO₂ and (b) MWCNT nanofluids

Conversely, despite having higher acidity and water content, the BDV of the 1000-h-aged nanofluid samples consistently surpasses the recommended limit, highlighting the favourable attributes of POME-based nanofluids.

4.6.6 Change of Gases Concentration Over Ageing Durations

Figure 4.15 depicts the presence of gases H_2 , CH_4 , C_2H_4 , C_2H_6 , CO , and CO_2 in the 1000-h-aged nanofluid samples. The gas generation of POME-based TiO_2 and POME-based MWCNT nanofluids is almost similar. It is found that H_2 and C_2H_6 are the dominant characteristic gas under the low-temperature thermal fault ($t < 300\text{ }^{\circ}C$), as reported by (Xing et al., 2023). CO is generated due to thermal degradation of the insulating oil itself (Zafeiris et al., 2011). Concentrations of CO and CO_2 in this study, which follow the guide in IEEE Std C57.155, correspond to normal gassing in transformers without faults. C_2H_4 and C_2H_2 were almost not generated in all nanofluid samples during the entire ageing experiment at $130\text{ }^{\circ}C$. The thermal fault in POME-based nanofluids dissipated insufficient energy for producing these particular gases.

In this study, the DGA methods used are the Rogers Ratio Method (RRM), IEC Ratio Method, Duval Triangle Method (DTM), and Duval Pentagon Method (DPM). The DRM is not being employed for analysis in this study due to the unfilled requisite gas concentrations, as mentioned earlier in subsection 2.8.2.3.

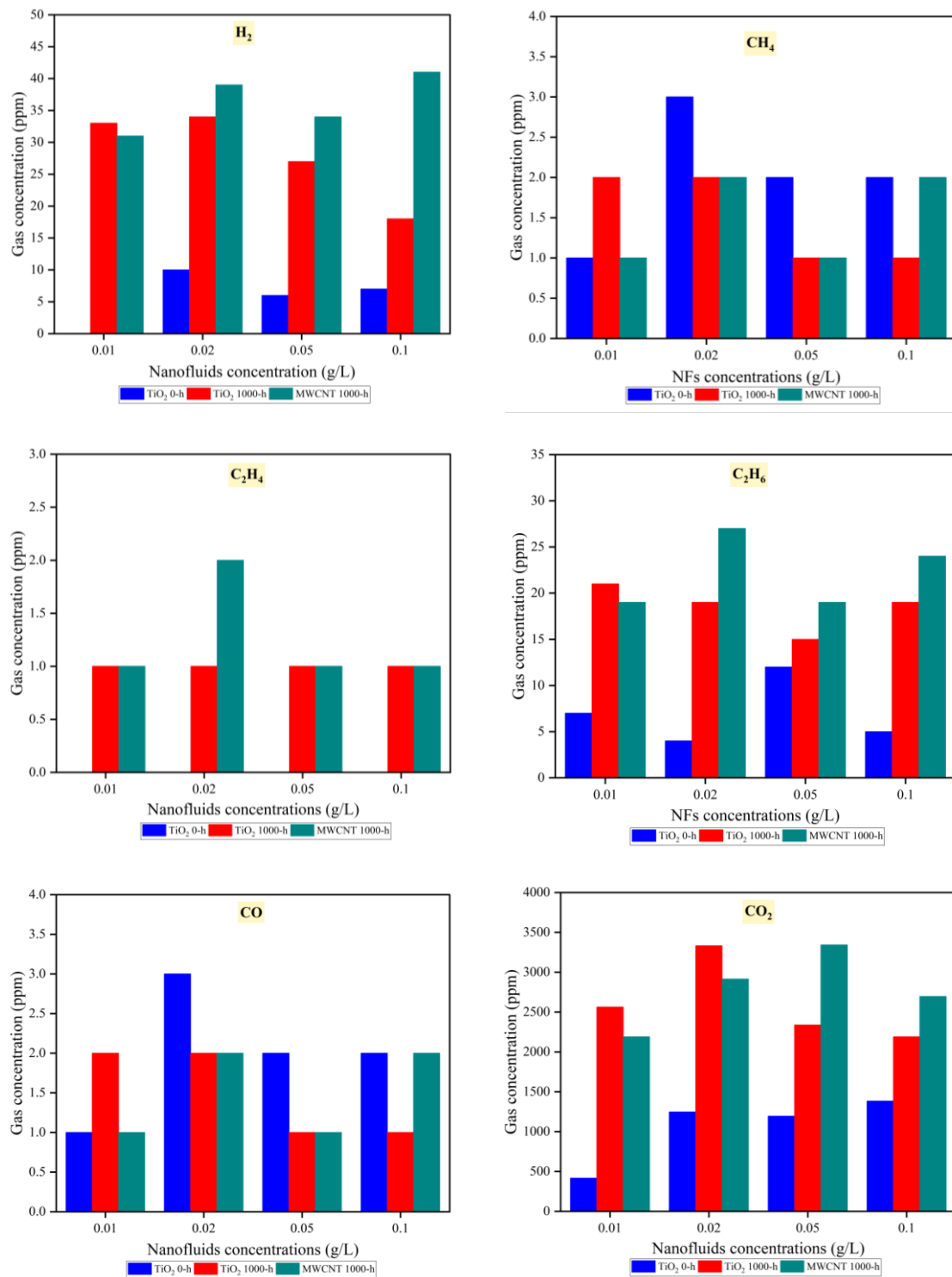


Figure 4.15: Variation of gas content in 1000-h-aged POME-based nanofluids

4.6.6.1 Rogers Ratio Method (RRM) and IEC Gas Ratio Method

Table 4.12 shows the ratios and the predicted incipient fault types based on the RRM and IEC Gas Ratio Method. According to IEEE standard C57.104 (IEEE Std C57.104, 2019), the RRM suggested that the nanofluid samples do not show any signs of fault or degradation due to thermal ageing. However, according to IEC 60599-2022 (IEC 60599, 2022), the IEC ratio method suggested that the nanofluid samples had a partial discharge fault. According to (Kaur et al., 2022), the partial discharge is a localised fault in the transformer, which is attributed to elevated voltage field stress.

Table 4.12: Gas concentration ratios based on RRM and IEC

POME-based NFs	Gas ratios			Fault diagnosis	
	C ₂ H ₂ /C ₂ H ₄	CH ₄ /H ₂	C ₂ H ₄ /C ₂ H ₆	Rogers	IEC
TiO ₂ 0.01-g/L	0	0.061	0.048	Normal	Partial discharges
TiO ₂ 0.02-g/L	0	0.059	0.053		
TiO ₂ 0.05-g/L	0	0.037	0.067		
TiO ₂ 0.10-g/L	0	0.056	0.053		
MWCNT 0.01-g/L	0	0.032	0.053		
MWCNT 0.02-g/L	0	0.051	0.074		
MWCNT 0.05-g/L	0	0.029	0.053		
MWCNT 0.10-g/L	0	0.049	0.042		

4.6.6.2 Duval Triangle Method (DTM)

The DTM is based on the relative percentages of three gases: CH₄, C₂H₄, and C₂H₂. The Duval triangle 3 (recommended for non-mineral oils) is applicable for fault types diagnosis in vegetable oils with some modifications on the boundaries of fault zones in

Duval triangle 1 (Baruah et al., 2020; Du et al., 2019). For instance, based on the calculated relative percentage of $\text{CH}_4 = 66.7\%$, $\text{C}_2\text{H}_4 = 33.3\%$, and $\text{C}_2\text{H}_2 = 0\%$, for POME-based TiO_2 at a concentration of 0.01-g/L, the point (yellow dot) falls in the T1 fault zone in the Duval triangle 3, which indicates possible overheating (thermal fault $< 300^\circ\text{C}$), as shown in Figure 4.16 (i). Overall, according to the interpretation using DTM of POME-based TiO_2 and MWCNT nanofluids as illustrated in Figures 4.16 and 4.17, all nanofluid samples were at the T1 fault zone in Duval triangle 3, indicating overheating of thermally aged samples $< 300^\circ\text{C}$.

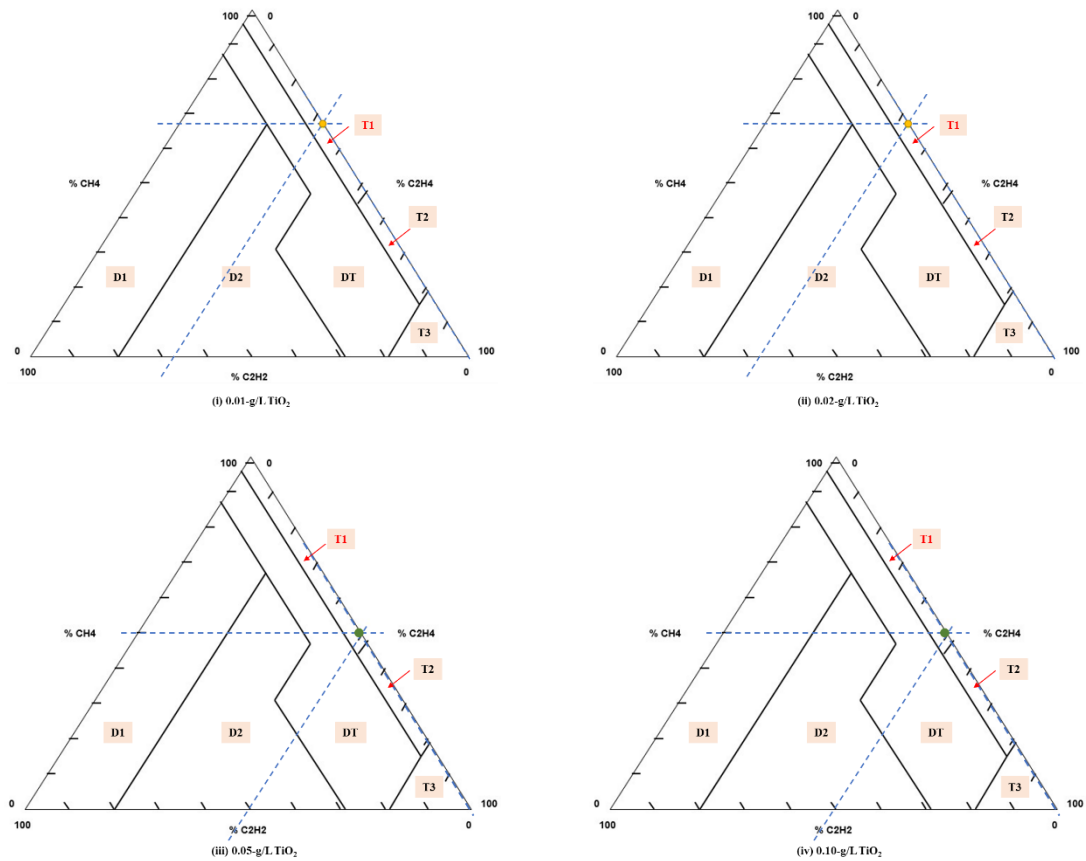


Figure 4.16: Result analysis using DTM for POME-based TiO_2 nanofluids

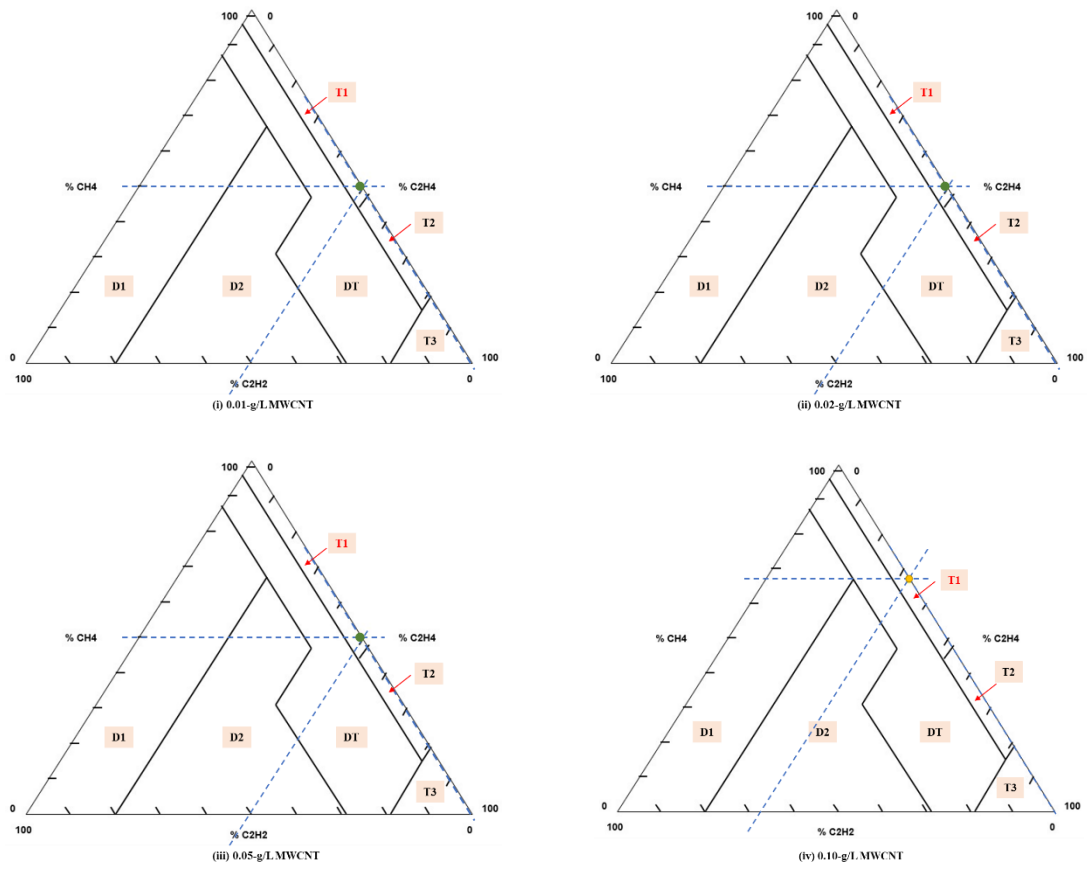
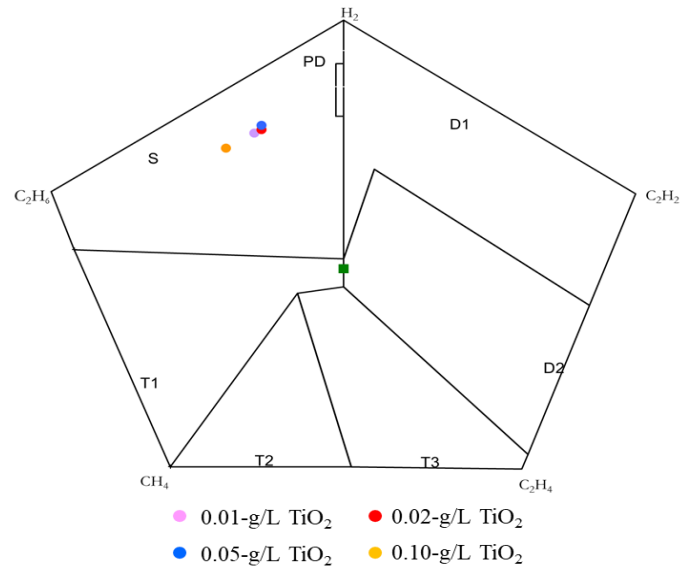
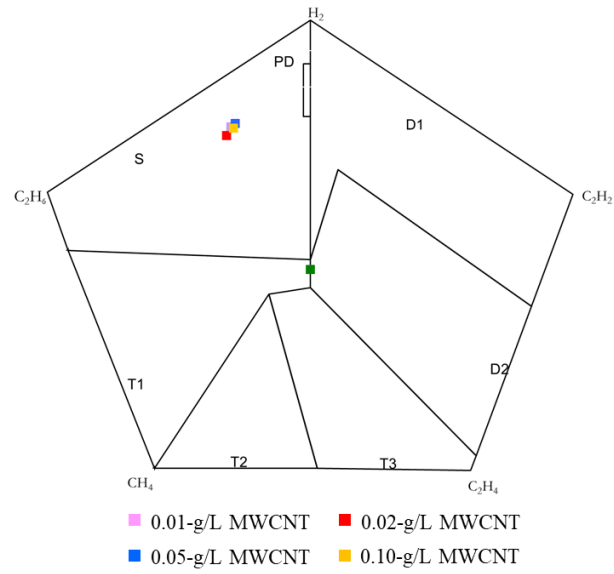


Figure 4.17: Result analysis using DTM for POME-based MWCNT nanofluids

The DGA diagnosis results of the 1000-h-aged POME-based NFs using the DPM are illustrated in Figure 4.18. These tasks were done easily with a small algorithm provided for free from duvalm@ireg.ca, established by Duval (2014). It can be seen that the DPM method identified ‘S’ status, signifying the presence of stray gassing of oil at a temperature < 200 °C for all nanofluid samples. This observation infers that natural ester oils might produce combustible gases under thermal stress (IEEE Std C57.155-2014, 2014).



(a)



(b)

Figure 4.18: Result analysis using DPM for 1000-h-aged POME-based (a) TiO_2 , and (b) MWCNT nanofluids

Table 4.13 summarises the fault prediction for 1000-h-aged POME-based nanofluids after thermal stress using various methods. The utilisation of different fault prediction

methods enabled the diagnosis of incipient faults under thermal stress in the insulation. According to the RRM, the transformer is considered to be in a ‘normal’ condition, indicating that the POME-based nanofluids did not show any signs of faults or degradation due to thermal ageing. In contrast, the IEC ratio gas methods predicted partial discharges, while the DTM and DPM predicted the signs of overheating (thermal fault < 300 °C) and stray gassing at temperatures < 200 °C for all aged POME-based nanofluids, respectively.

Table 4.13: Results of fault prediction using various methods

DGA diagnosis methods	Fault prediction
RRM	Normal
IEC Ratio Gas Method	Partial discharges
DTM	Overheating (thermal fault <300 °C)
DPM	Stray gassing at temperatures <200 °C

In conclusion, Table 4.14 shows the comparative performance in terms of dielectric strength, kinematic viscosity, and flash point in this study with various methyl esters that have been investigated in the literature. The results showed that POME performed comparably to other methyl ester oils.

Table 4.14: Comparative analysis of dielectric strength, kinematic viscosity, and flash point of POME with other methyl ester oils

Modification methods	References	Methyl ester oils	Performance		
			Dielectric strength (kV)	Kinematic viscosity at 40 °C (mm ² /s)	Flash point (°C)
Direct purification	(Menkiti et al., 2017)	MTCKO	32.88	15.22	256
Esterification	(Sitorus et al., 2016)	JMEO	87	10.45	191
Transesterification	(Maharana et al., 2018)	KOME	85.40	12.00	284
	(Agu et al., 2019)	MTCKO	48.55	10.29	280
	(Nkouetcha et al., 2019)	COME	74.67	18.42	n/a
	(Ravulapalli et al., 2019)	MESSO	24	0.98×10 ⁻¹¹	260
	(Asse et al., 2022)	PKOME	32.22	n/a	n/a
	(Okikiola et al., 2022)	Neem ester oil	n/a	5.17	175
	Current study	POME	57.08	4.54 – 5.27	171–182

4.7 Chapter Summary

In this chapter, the experimental results, analysis, and discussion were presented. Section 4.2 explained the characterisation of the laboratory-synthesised POME used in the experiment. The transesterification process led to a substantial reduction in kinematic viscosity from 33–50 mm²/s to 4.54–5.27 mm²/s, which was below the maximum value required of 50 mm²/s by the IEEE standard C57.147. The POME contained 100% fatty acid methyl ester, mainly methyl stearate (82.70%), methyl myristate (6.89%), methyl arachidate (4.71%), methyl laurate (2.92%), methyl tetracosanoic (1.45%), and methyl behenate (1.35%).

In the accelerated ageing study, two batches of nanofluids were prepared: POME-based TiO₂ and MWCNT. These nanofluids were prepared by dispersing 0.01–0.10-g/L of nanoparticles in the developed POME base fluid. Comparative measurements and analyses were carried out to assess the behaviour of POME-based nanofluids at various concentrations and times under a specific operating temperature. Several key parameters were thoroughly investigated, including AC BDV, resistivity, flash point, density, kinematic viscosity, water content, acidity, and DGA analysis. Many observations are made from this study, which will provide necessary information about the effectiveness of the nanofluids and their long-term performance with ageing.

However, there were certain limitations in the properties, notably in terms of resistivity, water content, and TAN, which did not meet the IEC standards. Thus, there is a necessity for further improvement to increase the feasibility of POME-based nanofluids as replacements for transformer oil.

The DGA was conducted on 1000-h-aged POME-based nanofluid samples using RRM, the IEC gas ratio method, DTM, and DPM. Employing fault prediction techniques using these DGA diagnostic methods enables the gaining of diagnoses related to partial discharges, overheating, and stray gassing in the nanofluids. These initial findings indicate the feasibility of obtaining incipient fault types for POME-based nanofluids based on the collected DGA data. Overall, the findings presented in this chapter contribute to our understanding of the performance of nanofluids during accelerated ageing.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In conclusion, this study has provided insights into the characteristics and behaviour of nanofluids utilising POME as the base fluid. These nanofluids were incorporated with conductive MWCNT and semiconductive TiO₂ nanoparticles at various concentrations, and enhanced with a CTAB surfactant, contributing a deeper understanding of how these nanofluids perform during accelerated thermal ageing at different time intervals at a specific operating temperature.

The findings of this study offer important insights into the accelerated thermal ageing of modified palm oil, laying the foundation for future investigations in the field of nanofluids for transformer liquid. This study has demonstrated that the prepared nanofluids exhibited commendable performance in particular physicochemical and electrical properties that influence their effectiveness. The significant findings can be summarised as follows:

- 1) The characteristics of the laboratory-synthesised POME as the base fluid for the preparation of nanofluids have been assessed. This study shows that the transesterification process significantly reduced kinematic viscosity, lowering it from 37–50 mm²/s to 4.54 mm²/s. This demonstrated compliance with the required limit of ≤ 50 mm²/s specified in the IEEE C57.147 standard (IEEE Std C57.147-2018, 2018). The density of POME was found to be 0.87 g/cm³, further aligning with the IEEE C57.147 standard, which sets a requirement limit of 0.96 g/cm³. The value is comparable to mineral oil, which has a density of

0.88 g/cm³ (Kano et al., 2008). The results of methyl ester analysis performed using GC–MS demonstrate that the POME produced by the transesterification process in this study contains 100% fatty acid methyl ester.

- 2) The influences of incorporating semiconductive TiO₂ and conductive MWCNT with a specific amount of CTAB surfactant into POME base fluids at doping concentrations ranging from 0.01 to 0.10-g/L on electrical properties have been investigated. The results reveal that adding these nanoparticles improved the BDV, regardless of the doping concentrations. Notably, POME-based TiO₂ nanofluid exhibited the highest elevation in breakdown strength at a concentration of 0.05-g/L. The AC BDV increased from 22.15 kV to 31.38 kV, marking an impressive 42% increment. An ageing duration of 1000 h was selected to observe long-term thermal ageing. All POME-based nanofluids showed an increase in AC BDV during the accelerated thermal ageing study, with the highest value recorded at 57.08 kV, indicating a remarkable increment of 157.7% for POME-based TiO₂ nanofluid at a concentration of 0.05-g/L. After being subjected to accelerated thermal ageing, the chemical modification effect of POME-based TiO₂ nanofluids on the BDV revealed a consistent trend across various doping concentrations. Particularly, the dielectric strength of POME-based TiO₂ nanofluids was less affected by ageing when compared to POME-based MWCNT under similar conditions of concentration, times, and temperature of thermal ageing. As the ageing period progressed, the resistivity was observed to decrease.

The investigation of the physicochemical properties of the aged POME-based nanofluids, including the flash point, density, water content, acidity, and DGA. The kinematic viscosity of aged nanofluids remained low at 4.54–5.27 mm²/s, while the density remained at 0.87 g/cm³. Furthermore, these findings suggest that the kinematic viscosity of the aged nanofluids could potentially serve as a low-viscosity natural ester oil-based dielectric, which would enhance the cooling capacity and extend the service life of the transformer. Notably, the water content of POME-based TiO₂ nanofluids was lower than their MWCNT counterparts. The DGA interpretation is employed using RRM, IEC gas ratios method, DTM, and DPM only. In this study, the IEC gas ratio method identified partial discharges as the incipient fault type in aged POME-based nanofluids. Meanwhile, the Duval triangle 3 detected overheating (thermal fault <300 °C), and stray gassing (primarily H₂, CH₄, C₂H₆) was observed within the Duval pentagon.

- 3) It has been demonstrated that the proposed POME-based nanofluids have outstanding properties and are comparable to other types of methyl ester oils. Incorporating POME with TiO₂ and MWCNT nanoparticles, along with the addition of CTAB surfactant, did not have an adverse effect on their breakdown strengths regardless of the doping concentrations. This combination offers elevated breakdown voltage, kinematic viscosity, and flash point than RBDPO_o. The results demonstrated that POME performed comparably to other methyl ester oils.

5.2 Recommendations for Future Work

- 1) The present study is exclusively concerned with fundamental electrical properties, including AC BDV and resistivity. It would be intriguing to delve into the impact of POME-based nanofluids on more advanced electrical properties, such as permittivity, lightning breakdown voltage, partial discharge, and so forth.
- 2) Although the ageing study on the insulating liquid alone has been completed, the forthcoming research aims to understand the ageing of cellulose insulation in POME-based nanofluids.
- 3) The study on accelerated thermal ageing has raised several inquiries, necessitating further investigation. The current study was limited to only a single operating temperature, i.e., 130 °C. Future research can be done at different temperatures to compare the variations of the ageing effect on the insulation liquid and paper in different test samples.
- 4) Future research in the experimental domain of the prepared nanofluids with the optimum performance should be undertaken to assess their performance in actual transformers. This practical approach will clearly explain their efficacy in real-world applications.

REFERENCES

- Ab Ghani, S., Muhamad, N. A., Chairul, I. S., & Jamri, N. (2016). A study of moisture effects on the breakdown voltage and spectral characteristics of mineral and palm oil-based insulation oils. *ARPJ. Eng. Appl. Sci.*, 11(8), 5012–5020.
- Ab Ghani, S., Muhamad, N. A., Noorden, Z. A., Zainuddin, H., Abu Bakar, N., & Talib, M. A. (2018). Methods for improving the workability of natural ester insulating oils in power transformer applications: a review. *Electric Power Systems Research*, 163, 655–667.
- Abdelmalik, A. A. (2014). Chemically modified palm kernel oil ester: A possible sustainable alternative insulating fluid. *Sustainable Materials and Technologies*, 1, 42–51.
- Abdullahi, U. U., Bashi, S. M., Yunus, R., & Nurdin, A. (2004). The potentials of palm oil as a dielectric fluid. In PECon 2004. Proceedings. *National Power and Energy Conference*, (pp. 224–228).
- Aditama, S. (2005). Dielectric properties of palm oils as liquid insulating materials: effects of fat content. In *Proceedings of 2005 International Symposium on Electrical Insulating Material (ISEIM)*. (Vol. 1, pp. 91–94).
- Agu, C. M., Nwosu-Obieogu, K., Ani, K. A., Agulanna, A. C., Iwu-Ejike, S. O., Oguegbu, O. O., & Menkiti, M. C. (2022). Chemical modification of *Terminalia catappa* L. kernel oil methyl ester using epoxidation-esterification approach, for application as bio-transformer fluid – Physicochemical characterization, kinetics and thermodynamics. *Cleaner Chemical Engineering*, 3, 100035.
- Agu, C. M., Menkiti, M. C., Nwabanne, J. T., & Onukwuli, O. D. (2019). Comparative assessment of chemically modified *Terminalia catappa* L. kernel oil samples – A

- promising ecofriendly transformer fluid. *Industrial Crops and Products*, 140, 111727.
- Ali, A. R. I., & Salam, B. (2020). A review on nanofluid: preparation, stability, thermophysical properties, heat transfer characteristics and application. *SN Applied Sciences*, 2(10), 1636.
- Alizadeh, H., Pourpasha, H., Heris, S. Z., & Estellé, P. (2022). Experimental investigation on thermal performance of covalently functionalized hydroxylated and non-covalently functionalized multi-walled carbon nanotubes/transformer oil nanofluid. *Case Studies in Thermal Engineering*, 31, 101713.
- Amalanathan, A. J., Sarathi, R., Harid, N., & Griffiths, H. (2020). Investigation on flow electrification of ester-based TiO₂nanofluids. *IEEE Transactions on Dielectrics and Electrical Insulation*, 27(5), 1492–1500.
- Amalanathan, A. J., Sarathi, R., Prakash, S., Mishra, A. K., Gautam, R., & Vinu, R. (2020). Investigation on thermally aged natural ester oil for real-time monitoring and analysis of transformer insulation. *High Voltage*, 5(2), 209–217.
- Amin, D., Walvekar, R., Khalid, M., Vaka, M., Mujawar, N. M., & Gupta, T. C. S. M. (2019). Recent progress and challenges in transformer oil nanofluid development: a review on thermal and electrical properties. *IEEE Access*, 7, 151422–151438.
- Amizhtan, S. K., Amalanathan, A. J., Sarathi, R., Srinivasan, B., Gardas, R. L., Edin, H., & Taylor, N. (2022). Impact of surfactants on the electrical and rheological aspects of silica based synthetic ester nanofluids. *IEEE Access*, 10, 18192–18200.
- Anastopoulos, G., Zannikou, Y., Stournas, S., & Kalligeros, S. (2009). Transesterification of vegetable oils with ethanol and characterization of the key fuel properties of ethyl esters. *Energies*, 2(2), 362–376.
- Asano, R., & Page, S. A. (2014). Reducing environmental impact and improving safety and

- performance of power transformers with natural ester dielectric insulating fluids. *IEEE Transactions on Industry Applications*, 50(1), 134–141.
- Asse, J. B., Mengounou, G. M., & Imano, A. M. (2021). Investigation of the effect of zinc nanoparticles (ZnO) on the ac breakdown voltage and acidity index of based nanofluid on methyl ester of palm kernel oil. *International Journal of Scientific Engineering and Technology*, 10(06), 262–267.
- Asse, J. B., Mengounou, G. M., & Imano, A. M. (2022). Impact of FeO₃ on the ac breakdown voltage and acidity index of a palm kernel oil methyl ester based nanofluid. *Energy Reports*, 8, 275–280.
- ASTM D7777-12. (2018). *Standard Test Method for Density, Relative Density, or API Gravity of Liquid Petroleum by Portable Digital Density Meter*, West Conshohocken, PA, 2018. ASTM International, 1–7. <https://doi.org/10.1520/D7777>
- ASTM D877-02. (2002). *Standard Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes*.
- ASTM D92. (2018). *ASTM D92 Standard Test Method for Flash and Fire Points by Cleveland Open Cup Tester*. <https://www.astm.org/astm-tpt-86.html>
- Atiya, E. G., Mansour, D. E. A., Khattab, R. M., & Azmy, A. M. (2015). Dispersion behavior and breakdown strength of transformer oil filled with TiO₂ nanoparticles. *IEEE Transactions on Dielectrics and Electrical Insulation*, 22(5), 2463–2472.
- Azis, N., Jasni, J., Ab Kadir, M. Z. A., & Mohtar, M. N. (2014). Suitability of palm based oil as dielectric insulating fluid in transformers. *Journal of Electrical Engineering and Technology*, 9(2), 662–669.
- Babita, Sharma, S. K., & Gupta, S. M. (2016). Preparation and evaluation of stable nanofluids for heat transfer application: A review. *Experimental Thermal and Fluid*

Science, 79, 202–212.

- Bakruthen, M., Iruthayaran, M. W., & Narayani, A. (2018). Statistical failure reliability analysis on edible and non edible natural esters based liquid insulation for the applications in high voltage transformers. *IEEE Transactions on Dielectrics and Electrical Insulation*, 25(5), 1579–1586.
- Bakthavatchalam, B., Habib, K., Saidur, R., Saha, B. B., & Irshad, K. (2020). Comprehensive study on nanofluid and ionanofluid for heat transfer enhancement: A review on current and future perspective. *Journal of Molecular Liquids*, 305, 112787.
- Bakthavatchalam, B., Habib, K., Wilfred, C.D., Saidur, R., & Saha, B. B. (2022). Comparative evaluation on the thermal properties and stability of MWCNT nanofluid with conventional surfactants and ionic liquid. *Journal of Thermal Analysis and Calorimetry*, 147, 393–408.
- Bandara, K., Ekanayake, C., Saha, T. K., & Annamalai, P. K. (2016). Understanding the ageing aspects of natural ester based insulation liquid in power transformer. *IEEE Transactions on Dielectrics and Electrical Insulation*, 23(1), 246–257.
- Baroutian, S., Aroua, M. K., Raman, A. A., & Sulaiman, N. M. (2009). Rbd palm olein-based methyl/ethyl esters. *Journal of Oil Palm Research*, 21, 659–666.
- Baruah, N., Maharana, M., Dey, S. S., & Nayak, S. K. (2019). Behavioural assessment of aged natural ester based nanofluid using statistical technique. In *2019 IEEE Conference on Electrical Insulation and Dielectric Phenomena (CEIDP)*, (pp. 653–656).
- Baruah, N., Maharana, M., & Nayak, S. K. (2019). Performance analysis of vegetable oil-based nanofluids used in transformers. *IET Science, Measurement and Technology*, 13(7), 995–1002.
- Baruah, N., Dey, S. S., & Nayak, S. K. (2020). Evaluation of dissolved gas analysis and

- long-Term performance of non-edible natural ester. *IEEE Transactions on Dielectrics and Electrical Insulation*, 27(5), 1561–1569.
- Baruah, N., Sangineni, R., Chakraborty, M., & Nayak, S. K. (2020). Statistical analysis of natural ester based insulating liquid using hypothesis testing. In *2020 International Symposium on Electrical Insulating Materials (ISEIM)*, (pp. 347–350).
- Baur, M., Knauel, J., Calcara, L., Pompili, M., Sangiovanni, S., & Muzi, F. (2019). Stirring effect in dielectric liquids breakdown voltage determination. In *2019 IEEE 20th International Conference on Dielectric Liquids (ICDL)*, (pp. 1–4).
- Boss, P., & Oommen, T. V. (1999). New insulating fluids for transformers based on biodegradable high oleic vegetable oil and ester fluid. In *Proceedings IEE Colloquium Insulating Liquids*, 119, 39–48.
- Boyekong, G. O., Mengounou, G. M., & Imano, A. M. (2021). Comparative evaluation of the thermal aging of solid insulation in mineral oil and methyl ester of palm kernel oil. *Journal of Power and Energy Engineering*, 09(05), 166–183.
- Calcara, L., Sangiovanni, S., & Pompili, M. (2019). Standardized methods for the determination of breakdown voltages of liquid dielectrics. *IEEE Transactions on Dielectrics and Electrical Insulation*, 26(1), 101–106.
- Carcedo, J., Fernández, I., Ortiz, A., Delgado, F., Renedo, C. J. & Pesquera, C. (2015). Aging assessment of dielectric vegetable oils. *IEEE Electrical Insulation Magazine*, 31(6), 13–21.
- Chakraborty, M., Baruah, N., Kumar Nayak, S., & Maiti, P. K. (2021). Correlating uv visible spectral response and thermal ageing of blended transformer oil. In *2021 IEEE International Conference on Properties and Applications of Dielectric Materials (ICPADM)*, (pp. 127–130).

- Choi, S. U., & Eastman, J. A. (1995). Enhancing thermal conductivity of fluids with nanoparticles. (No. ANL/MSD/CP-84938; CONF-951135-29) Argonne National Lab. (ANL), Argonne, IL (United States).
- Cong, H., Shao, H., Du, Y., Hu, X., Zhao, W., & Li, Q. (2022). Influence of nanoparticles on long-term thermal stability of vegetable insulating oil. *IEEE Transactions on Dielectrics and Electrical Insulation*, 29(5), 1642–1650.
- Das, A. K. (2023). Investigation of electrical breakdown and heat transfer properties of coconut oil-based nanofluids. *Industrial Crops and Products*, 197, 116545.
- Decarpigny, C., Aljawish, A., His, C., Fertin, B., Bigan, M., Dhulster, P., Millares, M., & Froidevaux, R. (2022). Bioprocesses for the biodiesel production from waste oils and valorization of glycerol. *Energies*, 15(9), 3381.
- Dey, D., Kumar, P., & Samantaray, S. (2017). A review of nanofluid preparation, stability, and thermo-physical properties. *Heat Transfer - Asian Research*, 46(8), 1413–1442.
- Dombek, G., Nadolny, Z., & Marcinkowska, A. (2018). Effects of nanoparticles materials on heat transfer in electro-insulating liquids. *Applied Sciences*, 8(12), 2538.
- Du, S., Cong, H., & Li, Q. (2018). Fault diagnosis methods for vegetable oil transformers. In *2018 IEEE International Conference on High Voltage Engineering and Application (ICHVE)*, (pp. 1–4).
- Dupain, X., Costa, D. J., Schaverien, C. J., Makkee, M., & Moulijn, J. A. (2007). Cracking of a rapeseed vegetable oil under realistic FCC conditions. *Applied Catalysis B: Environmental*, 72(1–2), 44–61.
- Duval, M. (2002). A review of faults detectable by gas-in-oil analysis in transformers. *IEEE Electrical Insulation Magazine*, 18(3), 8–17.
- Duval, M., & Lamarre, L. (2014). The duval pentagon-a new complementary tool for the

- interpretation of dissolved gas analysis in transformers. *IEEE Electrical Insulation Magazine*, 30(6), 9–12.
- Duzkaya, H., & Beroual, A. (2021). Statistical analysis of ac dielectric strength of natural ester-based ZnO nanofluids. *Energies*, 14(1), 99.
- Emara, M. M., Peppas, G. D., Pyrgioti, E. C., Chronopoulos, D. D., Bakandritsos, A., Tegopoulos, S. N., Kyritsis, A., Tsovilis, T. E., Polykrati, A. D., & Gonos, I. F. (2022). Thermal and dielectric performance of ester oil based pentyl-graphene nanofluids. *IEEE Transactions on Dielectrics and Electrical Insulation*, 29(2), 510–518.
- Encinar, J. M., González, J. F., Pardal, A., & Martínez, G. (2010). Transesterification of rapeseed oil with methanol in the presence of various co-solvents. In *Third International Symposium on Energy from Biomass and Waste*, (pp. 1–17).
- Fact Sheets Malaysian Palm Oil. (2014). In *Malaysian Palm Oil Council*. [https://doi.org/10.1016/s1351-4210\(05\)70698-3](https://doi.org/10.1016/s1351-4210(05)70698-3)
- Farade, R. A., Wahab, N. I. B. A., Mansour, Di. E. A., Azis, N. B., Jasni, J., Banapurmath, N. R., & Soudagar, M. E. M. (2020). Investigation of the dielectric and thermal properties of non-edible cottonseed oil by infusing h-BN nanoparticles. *IEEE Access*, 8, 76204–76217.
- Fasehullah, M., Wang, F., & Jamil, S. (2022). Significantly elevated ac dielectric strength of synthetic ester oil-based nanofluids by varying morphology of CdS nano-additives. *Journal of Molecular Liquids*, 353, 118817.
- Fernández, I., Valiente, R., Ortiz, F., Renedo, C. J., & Ortiz, A. (2020). Effect of TiO₂ and ZnO nanoparticles on the performance of dielectric nanofluids based on vegetable esters during their aging. *Nanomaterials*, 10(4).
- Fofana, I. (2013). 50 years in the development of insulating liquids. *IEEE Electrical*

Insulation Magazine, 29(5), 13–25.

- Gayathri, R., Deiva Sundari, P., Thakur, S., & Sarathi, R. (2019). Investigation on the dielectric performance of titania nanoparticles and surfactant added mixture of mineral and natural ester oil. *Materials Research Express*, 6(12), 125020.
- Golarz, J. (2016). Understanding dissolved gas analysis (DGA) techniques and interpretations. In 2016 *IEEE/PES Transmission and Distribution Conference and Exposition (T&D)*, (pp. 1–5).
- Gomez, N. A., Wilhelm, H. M., Santos, C. C., & Stocco, G. B. (2014). Dissolved gas analysis (DGA) of natural ester insulating fluids with different chemical compositions. *IEEE Transactions on Dielectrics and Electrical Insulation*, 21(3), 1071–1078.
- Gouda, O. E., El-Hoshy, S. H., & El-Tamaly, H. H. (2018). Proposed three ratios technique for the interpretation of mineral oil transformers based dissolved gas analysis. *IET Generation, Transmission and Distribution*, 12(11), 2650–2661.
- Gutierrez, C. M., Fernandez, A. O., Estebanez, C. J. R., Salas, C. O., & Maina, R. (2023). Understanding the ageing performance of alternative dielectric fluids. *IEEE Access*, 11, 9656–9671).
- Hamid, M. H. A., Ishak, M. T., Din, M. F., Suhaimi, N. S., & Katim, N. I. A. (2016). Dielectric properties of natural ester oils for transformer application under temperature variation. In 2016 *IEEE International Conference on Power and Energy (PECon)*, (pp. 54–57).
- Hamid, S. (2011). Production and purification of fatty acid methyl esters from plant oils of different origin. In *PhD's Thesis, University of Greenwich*. <http://gala.gre.ac.uk/8056/>
- Huang, Z., Li, J., Yao, W., Wang, F., Wan, F., Tan, Y., & Mehmood, M. A. (2019). Electrical and thermal properties of insulating oil-based nanofluids: a comprehensive overview.

IET Nanodielectrics, 2(1), 27–40.

- Hussin, N., Subri, N. A., Azizie, N. A., Khalil, A. N. M., Jamil, M. K. M., Abd-Rahman, R., & Arshad, S. N. M. (2021). Low concentration vegetable oil based nanofluid : dielectric properties, ac breakdown voltage and kinematic viscosity. *Journal of Physics: Conference Series*, 1878(1).
- Hwang, J. G., Zahn, M., O’Sullivan, F. M., Pettersson, L. A. A., Hjortstam, O., & Liu, R. (2010). Effects of nanoparticle charging on streamer development in transformer oil-based nanofluids. *Journal of Applied Physics*, 107(1).
- IEC 60156. (2018). *Insulating Liquids - Determination of the breakdown voltage at power frequency - Test method. Ed. 3.0 20*.
- IEC 60247. (2004). *Insulating liquids - Measurement of relative permittivity , dielectric dissipation factor and d . c . resistivity*.
- IEC 60567. (2011). *IEC 60567: Oil-filled electrical equipment-sampling of gases and analysis of free and dissolved gases - Guidance*.
- IEC 60599. (2022). *IEC 60599: Mineral oil-filled electrical equipment in service - Guidance on the interpretation of dissolved and free gases analysis*.
- IEC 60814. (1997). *IEC 60814 Karl fischer titration*.
- IEC 62021-1. (2003). *IEC 62021-1 - Insulating liquids - Determination of acidity - Part 1: Automatic potentiometric titration*.
- IEC 62539. (2007). *IEEE Guide for the statistical analysis of electrical insulation breakdown data*.
- IEC 62770. (2013). *IEC 62770 Fluids for electrotechnical applications - Unused natural esters for transformers and similar electrical equipment*. In 61010-1 © Iec:2001 (Vol. 2003).

- IEEE Std C57.104. (2019). *IEEE Std C57.104* (Vol. 2019).
- IEEE Std C57.147-2018. (2018). *IEEE Guide for Acceptance and Maintenance of Natural Ester Fluids in Transformers*. IEEE Power & Energy Society.
- IEEE Std C57.155-2014. (2014). *IEEE Std C57.155. IEEE Guide for Interpretation of Gases Generated in Natural Ester and Synthetic Ester-Immersed Transformers*. IEEE Power and Energy Society.
- Jacob, J., Preetha, P., & Sindhu, T. K. (2020). Stability analysis and characterization of natural ester nanofluids for transformers. *IEEE Transactions on Dielectrics and Electrical Insulation*, 27(5), 1715–1723.
- Jacob, J., Preetha, P., & Thiruthi Krishnan, S. (2020). Review on natural ester and nanofluids as an environmental friendly alternative to transformer mineral oil. *IET Nanodielectrics*, 3(2), 33–43.
- Jamail, N. A. M., Azali, N. A., Sulaiman, E., Kamarudin, Q. E., Othman, S. M. N. S., & Kamarudin, M. S. (2018). Breakdown characteristic of palm and coconut oil with different moisture. *Indonesian Journal of Electrical Engineering and Computer Science*, 12(1), 363–369.
- Jamo, H. U., Umar, I. D., Yusuf, B., & Auwalu, I. A. (2019). Enhancement of physical properties of biodiesel extracted from palm oil by the addition MgO nanoparticles. *Journal Homepage: <http://ijmr.net>*. in, 6(05).
- Jariyanurat, K., Potivejkul, S., Pattanadech, N., & Chotigo, S. (2017). Dielectric properties of natural ester based nanofluid. In *2017 IEEE 19th International Conference on Dielectric Liquids (ICDL)*, (pp. 1–4)
- Jeong, J. Il, An, J. S., & Huh, C. S. (2012). Accelerated aging effects of mineral and vegetable transformer oils on medium voltage power transformers. *IEEE Transactions*

- on *Dielectrics and Electrical Insulation*, 19(1), 156–161.
- Judendorfer, T., Karius, V., Adamietz, G., Beck, W., Rapp, K. & Badent, R. (2014). Development and Testing of HV Instrument Transformers using Natural Ester Fluid.
- Kadim, E. J., Noorden, Z. A., Adzis, Z., & Azis, N. (2021). Nanoparticles application in high voltage insulation systems. *IEEE Transactions on Dielectrics and Electrical Insulation*, 28(4), 1380–1399.
- Kanoh, T., Iwabuchi, H., Hoshida, Y., Yamada, J., Hikosaka, T., Yamazaki, A., Hatta, Y., & Koide, H. (2008). Analyses of electro-chemical characteristics of palm fatty acid esters as insulating oil. In *2008 IEEE International Conference on Dielectric Liquids* (pp. 28–31).
- Karambar, S., & Tenbohlen, S. (2021). Compatibility study of silicone rubber and mineral oil. *Energies*, 14(18).
- Katim, N. I. A., Ishak, M. T., Amin, N. A. M., Hamid, M. H. A., Ahmad, K. A., & Azis, N. (2018). Lightning breakdown voltage evaluation of palm oil and coconut oil as transformer oil under quasi-uniform field conditions. *Energies*, 11(10).
- Kaur, K., Bhalla, D., & Singh, J. (2022). Hybridized and complementary comparative analysis of dissolved gases in a power transformer. *IOP Conference Series: Materials Science and Engineering*, 1228(1), 012003.
- Khaled, U., & Beroual, A. (2019). Statistical investigation of ac dielectric strength of natural ester oil-based Fe₃O₄, Al₂O₃, and SiO₂ nano-fluids. *IEEE Access*, 7, 60594–60601.
- Khaled, U., & Beroual, A. (2020). DC breakdown voltage of natural ester oil-based Fe₃O₄, Al₂O₃, and SiO₂ nanofluids. *Alexandria Engineering Journal*, 59(6), 4611–4620.
- Khan, Q., Hussain, R., & Khan, A. A. (2018). Dielectric characterisation of Fe₃P nanoparticles based ester oil. *IET Nanodielectrics*, 1(4), 132–136.

- Khan, S. A., Khan, A. A., & Tariq, M. (2021). Measurement of tan-delta and dc resistivity of synthetic ester based oil filled with Fe₂O₃, TiO₂ and Al₂O₃ nanoparticles. *Smart Science*, 9(3), 216–225.
- Kittikhuntharadol, Y., Pattanadech, N., Maneerot, S., Jongvilaikasem, K., Jariyanurat, K., Jeenmuang, S., Udomluksananon, P., Chumpiboon, K., & Bunlaksananusorn, C. (2023). Physical and chemical properties' comparison of natural ester and palm oil used in a distribution transformer. *Energy Reports*, 9, 549–556.
- Koutras, Konstantinos N, Tegopoulos, S. N., Charalampakos, V. P., Kyritsis, A., Gonos, I. F., & Pyrgioti, E. C. (2022). Breakdown performance and partial discharge development in transformer oil-based metal carbide nanofluids. *Nanomaterials*, 12(2), 269.
- Koutras, K. N., Naxakis, I. A., Antonelou, A. E., Charalampakos, V. P., Pyrgioti, E. C., & Yannopoulos, S. N. (2020a). Dielectric strength and stability of natural ester oil based TiO₂ nanofluids. *Journal of Molecular Liquids*, 316.
- Koutras, K.N., Naxakis, I. A., Antonelou, A. E., Charalampakos, V. P., Pyrgioti, E. C., & Yannopoulos, S. N. (2020b). Dielectric strength and stability of natural ester oil based TiO₂ nanofluids. *J. Mol. Liq.*, 316, 113901.
- Lewand, L. R. (2004). Laboratory testing of natural ester dielectric liquids. *Neta World*, 1–4.
- Li, Y., Zhou, J., Tung, S., Schneider, E., & Xi, S. (2009). A review on development of nanofluid preparation and characterization. *Powder Technology*, 196(2), 89–101.
- Lv, Y., Rafiq, M., Li, C., & Shan, B. (2017). Study of dielectric breakdown performance of transformer oil based magnetic nanofluids. *Energies*, 10(7).
- Madavan, R., Kumar, S. S., & Willjuice Iruthayarajan, M. (2018). A comparative

- investigation on effects of nanoparticles on characteristics of natural esters-based nanofluids. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 556, 30–36.
- Mahanta, D. K., & Laskar, S. (2017). Electrical insulating liquid: A review. *Journal of Advanced Dielectrics*, 7(4), 1–9.
- Maharana, M. (2019). Development of alternative dielectric fluid for power and distribution transformer.
- Maharana, M., Baruah, N., Nayak, S. K. S. K., Meher, N., & Iyer, P. K. P. K. (2019). Condition assessment of aged ester-based nanofluid through physicochemical and spectroscopic measurement. *IEEE Transaction on Instrumentation and Measurement*, 68(12), 4853–4863.
- Maharana, M., Nayak, S. K., & Sahoo, N. (2018). Karanji oil as a potential dielectrics liquid for transformer. *IEEE Transactions on Dielectrics and Electrical Insulation*, 25(5), 1871–1879.
- Makmud, M. Z. H., Illias, H. A., Chee, C. Y., & Sarjadi, M. S. (2018). Influence of conductive and semi-conductive nanoparticles on the dielectric response of natural ester-based nanofluid insulation. *Energies*, 11(2), 333.
- Maneerot, S., & Pattanadech, N. (2020). The comparative study of physical and chemical properties of palm oil and mineral oil used in a distribution transformer. In *2020 8th International Conference on Condition Monitoring and Diagnosis (CMD)*, (pp. 385–388).
- Maneerot, S., Vittayakorn, W., Pattanadech, N., Soubol, N., & Yodrayub, N. (2020). Effect of ZnO and BaTiO₃ nanoparticle on partial discharge characteristics of palm oil based nanofluids. In *2020 8th International Conference on Condition Monitoring and*

Diagnosis (CMD), (pp. 389–392).

- Mariappan, Y., & Rengaraj, M. (2021). A comprehensive review on nanotechnology for enhancement in performance of transformer liquid insulation. *Materials Today: Proceedings*, 47(1), 229–234.
- Matharage, B. S. H. M. S. Y., Fernando, M. A. R. M., Bandara, M. A. A. P., Jayantha, G. A., & Kalpage, C. S. (2013). Performance of coconut oil as an alternative transformer liquid insulation. *IEEE Transactions on Dielectrics and Electrical Insulation*, 20(3), 887–898.
- McShane, C. P., Corkran, J. L., Harthun, R. A., Gauger, G. A., Rapp, K. J., & Howells, E. (2000). Vegetable oil based dielectric coolant. *Cooper Industries Inc. Houston, Tex.*. United States. Pat. 6,037,537.
- McShane, C. P. (2000). New safety dielectric coolants for distribution and power transformers. *IEEE Ind. Appl. Mag.*, 24–32.
- McShane, C. P. (2002). Vegetable-oil-based dielectric coolants. *IEEE Industry Applications Magazine*, 8(3), 34–41.
- Méndez, C., Fernández, I., Ortiz, A., Ortiz, F., Olmo, C., Santisteban, A., Delgado, F., & Renedo, C. J. (2020). Effect of magnetic and non-magnetic nanoparticles on insulation and cooling behaviour of a natural ester for power transformers. In *2020 International Symposium on Electrical Insulating Materials (ISEIM)*, (pp. 111–114).
- Menkiti, M. C., Agu, C. M., Ejikeme, P. M., & Onyelucheya, O. E. (2017). Chemically improved *Terminalia catappa* L. oil: A possible renewable substitute for conventional mineral transformer oil. *Journal of Environmental Chemical Engineering*, 5(1), 1107–1118.
- Mentlik, V., Trnka, P., Hornak, J., & Totzauer, P. (2018). Development of a biodegradable

- electro-insulating liquid and its subsequent modification by nanoparticles. *Energies*, 11(3), 508.
- Mohamad, M. S., Zainuddin, H., Ghani, S. A., & Chairul, I. S. (2017). AC breakdown voltage and viscosity of palm fatty acid ester (PFAE) oil-based nanofluids. *Journal of Electrical Engineering and Technology*, 12(6), 2333–2341.
- Mohamad, N., Azis, N., Jasni, J., Kadir, M. Z. A. A., Yunus, R., & Yaakub, Z. (2021). Experimental study on the partial discharge characteristics of palm oil and coconut oil based Al₂O₃ nanofluids in the presence of sodium dodecyl sulfate. *Nanomaterials*, 11(3), 786.
- Mohamad, N. A., Azis, N., & Jasni, J. (2019). Impact of Fe₃O₄, CuO and Al₂O₃ on the ac breakdown voltage of palm oil and coconut oil in the presence of CTAB. *Energies*, 12(9), 1605.
- Mohamad, N. A., Azis, N., Jasni, J., Kadir, M. Z. A. A., Yunus, R., Ishak, M. T., & Yaakub, Z. (2016). Investigation on the dielectric, physical and chemical properties of palm oil and coconut oil under open thermal ageing condition. *Journal of Electrical Engineering and Technology*, 11(3), 690–698.
- Mohamad, N. A., Azis, N., Jasni, J., Kadir, M. Z. A. A., Yunus, R., & Yaakub, Z. (2018). Ageing study of palm oil and coconut oil in the presence of insulation paper for transformers application. *Materials*, 11(4).
- Mohamad, N. A., Azis, N., Jasni, J., Mohd Zainal, M. Z. A., Yunus, R., & Yaakub, Z. (2020). Effect of surfactants on the lightning breakdown voltage of palm oil and coconut oil based Al₂O₃ nanofluids. *Nanotechnology*, 31(42).
- Mohd, N. K., Wen-Huei, L., Abu Hassan, N. A. S., & Shoot-Kian, Y. (2021). Potential application of palm oil products as electrical insulating medium in oil-immersed

- transformers. *Environmental Progress and Sustainable Energy*, 40(6).
- Montero, A., Garcia, B., Burgos, J. C., & Gonzalez-Garcia, C. (2022). Dielectric design of ester-filled power transformers: ac stress analysis. *IEEE Transactions on Power Delivery*, 37(3), 2403–2412.
- Münster, T., Werle, P., Hämel, K., & Preusel, J. (2021). Thermally accelerated aging of insulation paper for transformers with different insulating liquids. *Energies*, 14(11).
- Murad, N. S., Muhamad, N. A., Suleiman, A. A., & Jamail, N. A. M. (2013). A study on palm oil-based oil moisture absorption level and voltage breakdown. In *2013 Annual Report Conference on Electrical Insulation and Dielectric Phenomena*, (pp. 925–928).
- Murugesan, S., Raj, R. A., Sarathi, R., & Amizhtan, S. K. (2023). Experimental investigation of electrical and rheological properties of modified punga oil. *IEEE Transactions on Dielectrics and Electrical Insulation*, 30(4), 1422–1431.
- Musa, I. A. (2016). The effects of alcohol to oil molar ratios and the type of alcohol on biodiesel production using transesterification process. *Egyptian Journal of Petroleum*, 25(1), 21–31.
- Nkouetcha, E. T., Mengounou, G. M., & Imano, A. M. (2019). Elaboration and performance analysis of a bio-based insulating liquid from castor oil for power transformers. *Open Access Library Journal*, 06(05), 1–16.
- Nor, S. F. M., Azis, N., Jasni, J., Ab Kadir, M. Z. A., Yunus, R., & Yaakub, Z. (2017). Investigation on the electrical properties of palm oil and coconut oil based TiO₂ nanofluids. *IEEE Transactions on Dielectrics and Electrical Insulation*, 24(6), 3432–3442.
- Okikiola, S., Andrew, O., Adekunle, A., Ene, V., Ismaila, A., Abdelghaffar, G., & Abdelmalik, A. (2022). An experimental investigation on composite methyl ester as a

- solution to environmental threat caused by mineral oil in transformer insulation. *Biomass Conversion and Biorefinery*, 1–11.
- Oommen, T. V. (2002). Vegetable oils for liquid-filled transformers. *IEEE Electrical Insulation Magazine*, 18(1), 6–11.
- Oommen, T. V., Claiborne, C. C., & Mullen, J. T. (1997). Biodegradable electrical insulation fluids. In *Proceedings of the Electrical/Electronics Insulation Conference*, 465–468.
- Oparanti, S. O., Khaleed, A. A., Abdelmalik, A. A., & Chalashkanov, N. M. (2020). Dielectric characterization of palm kernel oil ester-based insulating nanofluid. In *Proceedings of IEEE Conference on Electrical Insulation and Dielectric Phenomena (CEIDP)*, USA, 211–214.
- Oparanti, S. O., Rao, U. M., & Fofana, I. (2022). Natural esters for green transformers : Challenges and keys for improved serviceability. *Energies*, 16(61).
- Oparanti, S. O., Abdelmalik, A. A., Khaleed, A. A., Abifarin, J. K., Suleiman, M. U., & Oteikwu, V. E. (2022). Synthesis and characterization of cooling biodegradable nanofluids from non-edible oil for high voltage application. *Materials Chemistry and Physics*, 277(125485).
- Peppas, G. D., Charalampakos, V. P., Pyrgioti, E. C., Danikas, M. G., Bakandritsos, A., & Gonos, I. F. (2016). Statistical investigation of ac breakdown voltage of nanofluids compared with mineral and natural ester oil. *IET Science, Measurement and Technology*, 10(6), 644–652.
- Peppas, G. D., Charalampakos, V. P., Pyrgioti, E. C., Bakandritsos, A., Polykrati, A. D., & Gonos, I. F. (2018). A study on the breakdown characteristics of natural ester based nanofluids with magnetic iron oxide and SiO₂ nanoparticles. In *2018 IEEE International Conference on High Voltage Engineering and Application (ICHVE)*, (pp.

1–4).

- Pérez-Rosa, D., García, B., & Burgos, J. C. (2022). Influence of nanoparticles on the degradation processes of ester-based transformer insulation systems. *Energies*, 15(4), 1520.
- Perrier, C., & Beroual, A. (2009). Experimental investigations on insulating liquids for power transformers: mineral, ester, and silicone oils. *IEEE Electrical Insulation Magazine*, 25(6), 6–13.
- Perrier, C., Marugan, M., & Beroual, A. (2012). DGA comparison between ester and mineral oils. *IEEE Transactions on Dielectrics and Electrical Insulation*, 19(5), 1609–1614.
- Piotrowski, T., Rozga, P., & Kozak, R. (2019). Comparative analysis of the results of diagnostic measurements with an internal inspection of oil-filled power transformers. *Energies*, 12(11).
- Pyrgioti, E. C., Koutras, K. N., Charalampakos, V. P., Peppas, G. D., Bakandritsos, A. P., & Naxakis, I. A. (2020). An experimental study on the influence of surface modification of TiO₂ nanoparticles on breakdown voltage of natural ester nanofluid. In *The International Symposium on High Voltage Engineering*, (pp. 279–288). Cham: Springer International Publishing.
- Rabelo, S. N., Ferraz, V. P., Oliveira, L. S., & Franca, A. S. (2015). FTIR analysis for quantification of fatty acid methyl esters in biodiesel produced by microwave-assisted transesterification. *International Journal of Environmental Science and Development*, 6(12), 964–969.
- Rafiq, M., Shafique, M., Anam, A., & Ateeq, M. (2021). Transformer oil-based nanofluid: The application of nanomaterials on thermal, electrical and physicochemical properties of liquid insulation-A review. *Ain Shams Engineering Journal*, 12(1), 555–576.

- Rafiq, Muhammad, Lv, Y., Li, C., & Sun, Q. (2020). Effect of Al₂O₃ nanorods on the performance of oil-impregnated pressboard insulation. *Electrical Engineering*, 102(2), 715–724.
- Rafiq, M., Lv, Y. Z., Zhou, Y., Ma, K. B., Wang, W., Li, C. R., & Wang, Q. (2015). Use of vegetable oils as transformer oils-a review. *Renewable and Sustainable Energy Reviews*, 52, 308–324.
- Raj, R. A., Samikannu, R., Yahya, A., & Mosalaosi, M. (2021). Investigation of survival/hazard rate of natural ester treated with al₂o₃ nanoparticle for power transformer liquid dielectric. *Energies*, 14(5).
- Raof, N. A. (2021). Characteristic of polyol ester as transformer insulating oil. In *PhD's Thesis, Universiti Putra Malaysia*.
- Ravulapalli, S., Ravindhranath, K., & Ramamoorthy, M. (2019). Preparation, characterization and feasibility analysis of methyl ester of Sesbania seeds oil (MESSO) as alternate liquid dielectrics in distribution transformers. *RSC Advances*, 9(6), 3311–3319.
- Reffas, A., Idir, O., Ziani, A., Moulai, H., Nacer, A., Khelfane, I., Ouagueni, M., & Beroual, A. (2016). Influence of thermal ageing and electrical discharges on uninhibited olive oil properties. *IET Science, Measurement and Technology*, 10(7), 711–718.
- Rooney, D., & Weatherley, L. R. (2001). The effect of reaction conditions upon lipase catalysed hydrolysis of high oleate sunflower oil in a stirred liquid-liquid reactor. *Process Biochemistry*, 36(10), 947–953.
- Rozga, P. (2013). Properties of new environmentally friendly biodegradable insulating fluids for power transformers. *European Scientific Journal*, 9(21).
- Saenkhumwong, W., & Suksri, A. (2017). The improved dielectric properties of natural ester oil by using ZnO and TiO₂ nanoparticles. *Engineering and Applied Science Research*,

44(3), 148–153.

- Shen, Z., Wang, F., Wang, Z., & Li, J. (2021). A critical review of plant-based insulating fluids for transformer: 30-year development. *Renewable and Sustainable Energy Reviews*, 141, 110783.
- Sheppard, H. R. (1986). Century of Progress in Electrical Insulation 1886-1986. *IEEE Electrical Insulation Magazine*, 2(5), 20–30.
- Shukri, M. S., Azis, N., Jasni, J., Yunus, R., & Yaakub, Z. (2021). Statistical analysis of AC dielectric strength for palm oil under the influence of moisture. *Pertanika Journal of Science and Technology*, 29(1), 77–94.
- Sinan, S. S., Shawaludin, S. N., Jasni, J., Azis, N., Ab Kadir, M. Z. A., & Mohtar, M. N. (2014). Investigation on the ac breakdown voltage of rbdpo olein. In *2014 IEEE Innovative Smart Grid Technologies - Asia (ISGT ASIA)*, (pp.760–763).
- Singha, S., Jr, R., Frimpong, G., Claiborne, C., & Cherry, D. (2014). Comparative aging characteristics between a high oleic natural ester dielectric liquid and mineral oil. *IEEE Transactions on Dielectrics and Electrical Insulation*, 21(1), 149–158.
- Sitorus, H. B. H., Setiabudy, R., Bismo, S., & Beroual, A. (2016). Jatropha curcas methyl ester oil obtaining as vegetable insulating oil. *IEEE Transactions on Dielectrics and Electrical Insulation*, 23(4), 2021–2028.
- Srivastava, M., Goyal, S. K., & Saraswat, A. (2021). Ester oil as an alternative to mineral transformer insulating liquid. *Materials Today: Proceedings*, 43, 2850–2854.
- Stockton, D. P., Bland, J. R., McClanahan, T., Wilson, J., Harris, D. L., & McShane, P. (2007). Natural ester transformer fluids: safety, reliability & environmental performance. In *2007 IEEE Petroleum and Chemical Industry Technical Conference*, (pp. 1–7).

- Suhaimi, Nur S., Ishak, M. T., Rahman, A. R. A., Muhammad, M. F., Abidin, M. Z. Z., & Khairi, A. K. (2022a). A review on palm oil-based nanofluids as a future resource for green transformer insulation system. *IEEE Access*, 10, 103563–103586.
- Suhaimi, N. S., Din, M. F. M., Ishak, M. T., Rahman, A. R. A., Wang, J., & Hassan, M. Z. (2022b). Performance and limitation of mineral oil-based carbon nanotubes nanofluid in transformer application. *Alexandria Engineering Journal*, 61(12), 9623–9635.
- Suhaimi, N. S., Din, M. F. M., M. T. Ishak, A., & Rahman, A. R. A. (2018). Statistical analysis on ac breakdown voltage of CNT nanofluid with mineral oil and palm oil. In *Proceedings of IEEE 7th International Conference Power Energy*, (pp. 111–116).
- Suhaimi, S. N., Rahman, A. R. A., Din, M. F. M., Hassan, M. Z., Ishak, M. T., & Jusoh, M. T. (2020). A review on oil-based nanofluid as next-generation insulation for transformer application. *Journal of Nanomaterials*. 2020, 1–17.
- Suhaimi, S. N. (2021). Properties improvement of disposed transformer oil by dispersing multi-walled carbon nanotube for transformer application. In *PhD's Thesis, Universiti Pertahanan Nasional Malaysia*.
- Suleiman, A. A., & Muhamad, N. A. B. (2014). Feasibility study on the use of vegetable oil (natural ester) in Malaysia power system transformers. *Transactions on Electrical and Electronic Materials*, 15(3), 113–116.
- Suleiman, A. A., Muhamad, N. A., Bashir, N., Murad, N. S., Arief, Y. Z., & Phung, B. T. (2014). Effect of moisture on breakdown voltage and structure of palm based insulation oils. *IEEE Transactions on Dielectrics and Electrical Insulation*, 21(5), 2119–2126.
- Suwarno, Sitinjak, F., Suhariadi, I., Imsak, L., Sitinjak, S. F., Suhariadi, I., & Imsak, L. (2003). Study on the characteristics of palm oil and it's derivatives as liquid insulating materials. In *Proceedings of IEEE International Conference on Properties and*

Applications of Dielectric Materials, 2, (pp. 495–498).

- Tenbohlen, S., & Koch, M. (2010). Aging performance and moisture solubility of vegetable oils for power transformers. *IEEE Transactions on Power Delivery*, 25(2), 825–830.
- Thakur, S., Sarathi, R., & Danikas, M. G. (2019). Investigation on thermal ageing impact on dielectric properties of natural ester oil. *Electrical Engineering*, 101(3), 1007–1018.
- Thakur, S., & Sarathi, R. (2018). Understanding incipient discharge characteristics in nano ester oil under AC/DC voltages adopting UHF technique. In *2018 Condition Monitoring and Diagnosis*, (pp. 1–5).
- Tokunaga, J., Koide, H., Mogami, K., & Hikosaka, T. (2016). Comparative studies on the aging of thermally upgraded paper insulation in palm fatty acid ester, mineral oil, and natural ester. *IEEE Transactions on Dielectrics and Electrical Insulation*, 23(1), 258–265.
- Tokunaga, J., Nikaido, M., Koide, H., & Hikosaka, T. (2019). Palm fatty acid ester as biodegradable dielectric fluid in transformers: a review. *IEEE Electrical Insulation Magazine*, 35(2), 34–46.
- Torres, A., Fuentes, B., Rodríguez, K. E., Brito, A., & Díaz, L. (2020). Analysis of the content of fatty acid methyl esters in biodiesel by Fourier-transform infrared spectroscopy: Method and comparison with gas chromatography. *Journal of the American Oil Chemists' Society*, 97(6), 651–661.
- Totzauer, P., & Trnka, P. (2019). Different ways to improve natural ester oils. *Transportation Research Procedia*, 40, 102–106.
- Von Thien, Y., Azis, N., Jasni, J., Ab Kadir, M. Z. A., Yunus, R., Ishak, M. T., & Yaakub, Z. (2016). Evaluation on the lightning breakdown voltages of palm oil and coconut oil under non-uniform field at small gap distances. *Journal of Electrical Engineering*

Technology, 11(1), 184–191.

- Wajanasoonthon, K., & Suksri, A. (2022). Long term thermal performance of palm oil and nano graphene filler in nanofluids application on transformer insulating oil and electrical breakdown voltage. *Key Engineering Materials*, 931, 9–15.
- Wajanasoonthorn, K., & Suksri, A. (2021). Electrical breakdown voltage of palm oil and nano graphene filler in nanofluids application on transformer insulating oil. *Key Engineering Materials*, 902, 59–63.
- Wang, X., Tang, C., Huang, B., Hao, J., & Chen, G. (2018). Review of research progress on the electrical properties and modification of mineral insulating oils used in power transformers. *Energies*, 11(3), 487.
- Xing, Y., Hu, Y., Ma, Y., Liu, Y., Dong, M., & Xu, Y. (2023). Difference analysis of dissolved gas in natural ester insulating fluids under typical electrical and thermal faults: an experimental study. *High Voltage*, 1–9.
- Yaacob, M., & Alsaedi, M. (2015). Use palm oil as alternative with insulation oil in high voltage equipment. *Physical Science International Journal*, 5(3), 172–178.
- Yahya, M., & Amirrazli, F. A. (2018). Study of breakdown voltage of vegetables oil with SiO₂ nanoparticle additive. *Indones. J. Electr. Eng. Comput. Sci.*, 12(1), 296–302.
- Yang, L., & Hu, Y. (2017). Toward TiO₂ Nanofluids—Part 1: Preparation and properties. In *Nanoscale Research Letters*, 12(147).
- Yu, W., & Xie, H. (2012). A review on nanofluids: preparation, stability mechanisms, and applications. *Journal of Nanomaterials*, 2012, 1–17.
- Yue, Y., Yang, D., & Han, D. (2022). Application of Duval pentagon in state diagnosis of on load tap changer. *Journal of Physics: Conference Series* (Vol. 2247, No. 1, p. 012021). IOP Publishing

- Zafeiris, K., Bertrand, Y., Hipszki, G., Kuffel, M., Rehorek, C., & Seidel, S. (2011). Investigation on natural ester fluids concerning gassing and compatibility with transformer materials. In *International Symposium on High Voltage Engineering*.
- Zhou, Y., Sui, S. Y., Li, J., Wang, Z. Y., Cui, W., Lv, Y. Z., & Li, C. R. (2018). Statistical analysis of moisture's effect on ac breakdown strength of TiO₂ nanofluids. *Journal of Molecular Liquids*, 249, 420–428.

APPENDICES

Appendix A: Publications

(i) ISI Indexed Journal

1. **Masra, S. M. W.**, Arief, Y. Z., Sahari, S. K., Muhammad, M. S., Rigit, A. R. H., & Rahman, M. R. (2022). A systematic review on promising development of palm oil and its nanofluid as a biodegradable oil insulation alternative. *IEEE Transactions on Dielectrics and Electrical Insulation*, 29(1), 302-318.
2. **Masra, S. M. W.**, Arief, Y. Z., Rigit, A. R. H., Sahari, S. K., Rahman, M. R., Muhammad, M. S., & Eteruddin, H. (2023). Assessing electrical and physicochemical performance of chemically modified palm oil as an alternative transformer liquid. *Journal of Oil Palm Research*, 35(1), 33-44.
3. **Masra, S. M. W.**, Arief, Y. Z., Sahari, S. K., Rigit, A. R. H., Rahman, M. R., & Suhaimi, N. S. (2023). Thermal aging effects on the electrical breakdown voltage of TiO₂ and MWCNT nanofluids based on POME. *IEEE Transactions on Dielectrics and Electrical Insulation*, 30(6), 2522-2531.

(ii) Conference Proceeding/Newsletter

1. **Masra, S. M. W.**, Arief, Y. Z., and Berinau, G. B., “Performance analysis of palm oil methyl ester as an alternative dielectric liquid for oil-immersed transformers," MyHVnet Newsletter, Issue 7, ISSN 2462-1994, January 2022. (Extended Abstract)
2. **Sharifah Masniah Wan Masra**, Yanuar Zulardiansyah Arief, Siti Kudnie Sahari, Ernieza Musa, Andrew Ragai Henry Rigit, and Md. Rezaur Rahman, “Exploring the impact of accelerated thermal aging on POME-based MWCNT nanofluid,” Proceedings of the 12th International Conference on Robotics, Vision, Signal Processing and Power Applications, Lecture Notes in Electrical Engineering 1123, https://doi.org/1007/978-981-99-9005-4_13, pp. 101-107.

Appendix B: Data Collected on AC BDV of POME-based TiO₂

No. of measurement	Pure POME	0-h				50-h				250-h		
		0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L
1	23.15	42.3	27.7	18.3	21.9	20.4	22.5	33.7	21.3	48	40	77
2	22.3	31.6	24.1	22.4	24.8	24.3	25.9	41.5	23.1	47.6	32.7	73.4
3	21.4	57.4	26.3	19.1	21.3	15.5	25.5	40	24.8	46.5	39.8	73.7
4	23.85	58.3	27.1	19.8	28.6	15.8	24.3	42.8	25.5	48.7	31.2	68.4
5	22.15	31.9	26.3	23.6	29.1	21.6	24.8	34.1	24.8	42.9	34.1	76
6	22.5	24.7	26.3	28.1	29.5	16.2	22.7	31.2	24.9	42.5	29.7	77.7
7	23.55	28	27.2	22.2	19.4	19.1	20.7	20.2	26.3	43.1	27.8	80.2
8	22.1	31.1	19.8	30	27.8	18.3	21.9	30.9	25.4	45.8	28.2	77.3
9	22.9	28.8	28.4	25.3	22	19.4	19.3	33.5	24.1	39.9	31.9	78.7
10	23.35	25.5	28	22.9	32	22	19.4	31.5	23.3	44.6	29.3	65.5
11	22.9	23.6	19.4	31.2	32.6	27.7	20.7	29.1	18.5	50	50.5	63.9
12	22.85	32.7	27.5	31	33	19.8	19	36.8	23.9	45.9	50.6	78.9
13	23.4	28.8	29.1	32.6	32.7	27	23.1	29.4	22.3	48.7	57	79.8
14	24.05	27.4	28.6	35.9	34.9	28.3	19.7	35	22.5	44.7	57.4	78.1
15	22.55	28.7	20.4	36.3	32.5	24	18.4	35.8	21.4	38.9	56.7	55.9
16	21.7	28.1	28.2	40	28	30	27	28.1	21.7	43.3	65.8	62
17	22.25	26.6	26.3	38.4	32	27.8	22.4	30.4	23.8	43.8	65.2	53.3
18	21.6	25.1	25.5	31.2	31.8	35.9	24.9	35.7	21.7	42.4	52.8	49.4
19	21.65	25.4	31.2	36.7	31.8	18.3	21.5	26.5	24	40.2	47.7	47.7
20	21.35	24.3	29.1	34.5	33.5	15.7	25.4	31.9	24	46	50.9	47.6
21	23.65	26.2	25.2	32.9	31.9	15.7	22.6	35.2	25.2	47.8	49	48.5
22	22.55	21.6	28	39.1	35.3	14.9	19.2	27.7	22.5	45.3	47.8	44.4
23	20.95	28.7	25.5	35.5	31.4	15.7	26.2	33.5	24.3	47	43.2	49.7
24	21.25	25.1	27.1	34.4	32.3	19.8	26.2	30.2	24.8	44.5	39	57.4
25	22.7	22.7	29.6	35	35.2	16.6	19.9	33.5	24.5	40.2	43.6	45.3
26	22.25	24.7	26.7	35.2	30.7	22.4	20.9	35.1	22.2	44.7	39.6	43.4
27	21.4	27.7	31.5	35.4	34.9	16.6	18.2	38.7	18.2	41.5	35.9	36.9
28	22.7	29.1	27.8	30.4	34.2	24.3	23	35.8	23.3	38.9	36.4	43.3
29	22.4	24.3	27.3	37.6	31.5	20.6	18.9	28.7	18.4	39.8	40	42.9
30	22.95	28.4	25.2	31.9	32.7	24.2	19.7	35.4	23	42.7	29.9	35.9
31	22.1	25.4	29.1	37.7	31.3	23.9	19.9	34.4	23.2	40.2	43.4	48.3
32	21.55	33.4	27.2	31.2	31.1	30.2	20.8	23.8	23	40.4	44.7	45.2
33	21.4	24.7	30.4	33.8	33.7	21.7	20.8	34.8	22.5	37.7	47.8	58.1
34	19.75	31.6	29.1	30.4	33.5	29.7	21.6	32.7	21.6	35.4	46	45.4
35	19.15	31.9	30.4	34.8	34.2	20.9	25.5	34.4	21.4	34	46.1	43.9
36	19.15	35	28.7	34.8	34.4	22.3	22.5	30.3	22.5	36.1	43.9	40.2

No. of measurement	500-h				1000-h			
	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L
1	77.3	80.2	54.5	79.6	48.5	63.55	66.35	59.75
2	75.1	74.4	74.6	79.8	42.5	64.35	68.7	57.35
3	74	79.7	65.5	80	30.9	57.75	66.15	62.95
4	75.7	80.2	74.4	80.2	33.2	62.1	70	71.55
5	72.1	80	48.1	79.6	36.4	65.6	70.4	62.15
6	71.5	78.5	74.4	80	36.95	65.55	74.05	61.4
7	72.9	79.9	73.8	79.8	38.45	65.5	67.8	70.45
8	77	74.3	74.3	78.7	37.65	61.2	72.2	65
9	63	79.8	76.2	74.4	35.45	57.9	64.2	56.2
10	54	67.7	74.4	63.9	35.8	60.3	64.1	49.4
11	40.6	80	62.7	66.4	37.9	63.9	69.5	53.5
12	39.1	64.7	70.8	62	37.85	60.5	71.55	52
13	45	80.2	64.7	52.9	35.55	60.8	66.7	47
14	43.8	64.5	63	51.7	35.1	58	65	45.65
15	60.6	64.7	61.6	50.5	34.35	54.85	67.1	52.1
16	37.6	58.4	59.7	47.8	34.6	55.5	61.35	49.7
17	33.2	60.4	59.3	45.4	38.7	49.75	54.75	43.1
18	33.9	47.8	51.7	37.1	32.5	50.65	64.45	51.55
19	24.8	52.9	48.1	35.9	36.4	48.55	64.4	43.5
20	26.3	48.5	48.1	24.3	33.2	48.8	58.9	37.95
21	23.7	47.8	45.3	24	35.1	41.65	52.65	39
22	20.7	56.7	51.9	23.1	35.65	48.25	55	37.05
23	26.3	51	55.4	21.6	34.75	48.95	51.65	33.9
24	31.2	53.9	59.1	22	33	36.15	56.75	31.5
25	16.4	51.7	56.8	25.2	35.2	33.25	45.45	31.85
26	28	49.2	58.7	22.4	33.6	35.4	43.2	31
27	28.7	44.9	56.6	21.6	30.95	35.7	46.3	31.4
28	37.6	36.8	52.8	18.3	31.85	36.1	43	31.35
29	40	36	54.5	20.7	32.15	31	41.25	32.6
30	34.2	40.3	43.2	19.1	30.3	33.15	44.65	32.85
31	37.1	39	50.8	17.9	34.05	38.35	41.75	41.45
32	33.8	39.1	40.4	17.9	29.1	47	40.1	41.2
33	37.6	35.8	50.7	24	30.2	38.15	36.85	40.75
34	38.4	35.9	42.7	22	29.4	34.05	38.5	39.1
35	39	34.4	50.4	21.1	31.65	43.65	40.6	44.3
36	33.2	35	41.6	21.1	25.45	36.05	49.4	43

Appendix C: Data Collected on AC BDV of POME-based MWCNT

No. of measurement	Pure POME	0-h				50-h				250-h			
		0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L
1	23.15	23.5	17.5	17.4	15.6	52.4	44.6	63.3	39.2	66.8	61.4	63.2	56.7
2	22.3	22.7	18.3	17.5	17.4	48.5	62.3	64.7	42.3	74.4	51	61.5	46.6
3	21.4	23.6	18.7	17.5	16.6	46.2	51.3	43.2	43.7	65.5	63.9	59	38.6
4	23.85	23.1	19.4	18.3	16.6	44.2	60.6	63.9	40.8	65.3	59.4	48.5	45.3
5	22.15	24.5	18.8	16.8	16.2	45.4	54.9	60.8	47.9	56	53.7	47.3	42.4
6	22.5	24	18.1	19.6	17.1	45.4	60.4	63.2	43.9	61.4	50.6	46.6	52.5
7	23.55	24.2	18.3	16.2	16.5	39.1	40	61.8	43.2	60.4	47.4	46	44.6
8	22.1	20.9	18.3	18.9	17.6	54.1	47	61.7	39.6	61.9	47	47	50.1
9	22.9	23.8	20.6	18.3	18.1	48.9	54.3	57	45.9	61	48.1	43.4	50.2
10	23.35	24.8	20.7	18.3	18.4	46.5	58.9	54.5	50.2	56.7	42.3	41.4	47.7
11	22.9	24.1	21.1	17.8	18.3	54.4	54	52.4	47.8	54.8	36.4	43.8	40.2
12	22.85	21.6	19.4	16.6	17.4	51.3	50.2	65.5	33.7	51.2	45	41.2	46.7
13	23.4	23.8	19.8	15.8	17.1	48.3	47.8	63.2	43.7	53.4	38	28.7	51.5
14	24.05	19.3	21.4	15.5	18.7	52.4	47	56.5	42.9	46.8	36.3	38.4	41.4
15	22.55	18	20.2	16.4	18	55.9	52.4	61.4	50.4	49.7	28.9	29.9	43.3
16	21.7	23.6	21.4	18.1	19.8	52.9	48.7	62	45.6	46.1	32.7	31.6	38.1
17	22.25	23.1	19.4	18.1	17.8	55.9	49.7	60	48.3	45	31.2	30.5	44.7
18	21.6	23.6	21.4	17.8	18.7	58.9	55.5	58.9	50.1	40.1	30.2	39.6	35.9
19	21.65	23.1	19.1	17.7	16.6	50.2	52.5	60.4	51.1	38.7	34.4	34.3	38
20	21.35	23.6	19.4	17	19.4	54.5	54.5	67.8	54.9	57.8	29.5	29.1	41.2
21	23.65	22.7	18.7	17.4	17.8	23.8	54.5	64.7	54.8	38	31	41.4	51.7
22	22.55	22.1	22.4	18.7	19.1	20.6	49.4	60.8	50.6	67	32.7	38	49.7
23	20.95	23.1	22.5	15.8	18.6	19.6	54.1	64.7	52.5	46.8	34.8	43.2	34.4
24	21.25	22.7	20.4	18.3	18.2	46.4	58.9	66.1	52.9	53.4	29.1	41.1	43.3
25	22.7	22.4	19.1	15.8	18.4	21.6	58.2	67.1	52.5	49.2	29.1	40	39.6
26	22.25	23.7	18.3	15.8	18.9	46.8	59	63.9	52.2	49.5	32.5	32	40.3
27	21.4	23.1	20.4	18	18.2	45	51.7	67.1	50.8	49.8	31.9	35.2	51
28	22.7	22.9	20.7	17.7	20.7	51.7	60	57.3	52.5	51.3	39.1	39	49.2
29	22.4	23.2	19.1	18.1	19.4	37.6	61.5	56.7	52.9	39.6	38.4	43.1	40.6
30	22.95	23.6	17.3	16.2	19.8	49.1	55.1	66.8	51.7	48.5	37.6	37.9	39.6
31	22.1	22.4	19.6	17.4	17.6	54.1	60.4	69.5	52.2	47.1	37.6	39.8	44.3
32	21.55	24	20.4	16.6	18.3	51.9	58.9	66.1	54	58.8	39.8	39	49.5
33	21.4	23.6	19.2	15.8	18	53.7	57.8	63.2	51.8	59.6	41.7	34.2	48.4
34	19.75	22.4	19.2	16.2	21.4	54.5	60	66.7	50.5	56.7	33.9	41.5	40.8
35	19.15	22.2	21.3	17	18	53.4	62	66.8	52.5	38.4	43.2	44.7	44.2
36	19.15	23.6	17.3	17.1	19.4	52.9	60.6	58.2	51.7	39.1	40.4	33.6	47

No. of measurement	500-h				1000-h			
	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L	0.01-g/L	0.02-g/L	0.05-g/L	0.10-g/L
1	43.4	40.9	53.2	56.7	42.6	40.65	40.6	46.9
2	53.3	47	46.3	59	46.2	47.7	43.65	37.9
3	50.8	58.4	53.4	44.7	42.3	47.95	42.35	51.1
4	53.2	51.2	47	55.9	45.85	44.1	45.65	49.45
5	54.1	51.6	46.9	60.8	50.15	43.25	35.55	41.35
6	54.9	45.4	52.7	39.1	46.05	39.9	45.2	48
7	50.1	47.1	52.9	57.1	46.3	43.3	45.65	48.95
8	45	44.7	47	47.1	48.75	43.15	41.4	45.1
9	32.6	45.2	47.3	43.2	50.45	44.35	46.6	45.75
10	35.9	41.5	47	30.7	47.35	40.55	48.4	54.2
11	32.6	37.3	47.3	41.2	52.1	41.5	48.3	47.3
12	38.7	45.9	47	38.5	53.9	39.95	47.9	53.15
13	28.5	41.2	35.2	28	55.6	34.3	45.7	47.05
14	24.9	42.4	40.5	26.9	44.35	37.4	49.1	42.7
15	32	42.2	40.3	26.3	42.2	33.1	40.85	45.35
16	30.6	40.7	36.8	27.5	54.1	36.95	35.9	45.4
17	25.5	42.7	32.8	22.1	45.05	35.15	37.15	47.15
18	28.7	40.7	37.1	28.6	44.8	34.95	40.3	46.15
19	32.3	29.5	31.7	21.7	40.05	32.65	32.8	38.1
20	30.1	31.6	28.7	21.7	38.05	28.2	30.4	38.5
21	29.4	39	34.3	28.6	37.9	30.35	30.05	37.05
22	32.2	46.2	39.5	26.2	39.9	27.1	29.25	35.35
23	24.8	27.2	31.1	37.6	37.4	32.1	29.2	33.8
24	32.2	37.6	31.1	32.7	38.75	31.5	28.45	32.15
25	28.6	38.3	30	34.8	38.7	28.4	26.95	34.65
26	26.3	39.1	31.7	33.6	41.85	32.75	26.3	32.5
27	31.2	45	31	32.5	41.7	27.5	25.8	32.3
28	26.3	40	29.5	38.3	35.35	24.25	23.75	29.5
29	35.9	39.8	25.3	34.8	39.9	34.35	24.15	26.6
30	32.7	40.8	33.4	36.6	32	31.9	24.95	27.1
31	39.1	40.8	26.1	37.6	34.4	32.7	23.2	27.75
32	43.2	38	25.3	35.7	43.2	32.55	22.75	26.6
33	37.6	40.8	27.5	35.9	34.55	40.75	28.7	26.35
34	37.3	46.6	28	37.6	38.05	31.15	31.05	28.7
35	36.8	35.5	28	35.5	44.7	32.2	22.25	26.95
36	42.3	34.4	28.4	44.3	46.7	28.35	28.5	28.9

Appendix D: Data Collected on DGA, Acidity, Moisture, Viscosity, and Resistivity from TNBR

DSM Report

Client : UNIVERSITI MALAYSIA SARAWAK

Date Received : 09/01/2023

Job No. : TNBR-AC-48873-23-0075

Job Status : Complete

No.	Sample No.	Certificate No.	Sampling Date	Mnemonic	Site	TX No.	DGA (ppm)								NV	Moisture	Viscosity	Resistivity	
							H ₂	O ₂	N ₂	CH ₄	CO	CO ₂	C ₂ H ₄	C ₂ H ₆					C ₂ H ₂
1	AC-TXOIL-23-00205	TNBR-AC-ECP001-23-00583	09/01/2023	POME J	UNIMAS	POME 0.02TiO2/50HR	6	17783	41376	1	17	1199	NIL	4	NIL	0.7338	233	4.69	0.36
2	AC-TXOIL-23-00206	TNBR-AC-ECP001-23-00584	09/01/2023	POME K	UNIMAS	POME 0.05TiO2/50HR	6	19099	42756	2	23	1649	NIL	15	NIL	0.501	251	4.56	0.52
3	AC-TXOIL-23-00207	TNBR-AC-ECP001-23-00585	09/01/2023	POME L	UNIMAS	POME 0.10TiO2/50HR	6	15788	37025	2	18	1198	NIL	7	NIL	0.3819	220	4.58	0.41
4	AC-TXOIL-23-00208	TNBR-AC-ECP001-23-00586	09/01/2023	POME M	UNIMAS	UNAGED PURE POME	3	6572	27194	NIL	6	437	NIL	1	NIL	0.0985	657	4.45	0.52
5	AC-TXOIL-23-00209	TNBR-AC-ECP001-23-00589	09/01/2023	POME N	UNIMAS	POME 0.01TiO2/0HR	NIL	5163	13002	1	6	416	NIL	7	NIL	0.1314	284	4.46	0.1
6	AC-TXOIL-23-00210	TNBR-AC-ECP001-23-00587	09/01/2023	POME O	UNIMAS	POME 0.02TiO2/0HR	10	18827	46084	3	20	1247	NIL	4	NIL	0.1966	271	4.59	0.08
7	AC-TXOIL-23-00211	TNBR-AC-ECP001-23-00588	09/01/2023	POME P	UNIMAS	POME 0.05TiO2/0HR	6	14234	42539	2	23	1194	NIL	12	NIL	0.1454	253	4.57	0.06
8	AC-TXOIL-23-00212	TNBR-AC-ECP001-23-00590	09/01/2023	POME Q	UNIMAS	POME 0.10TiO2/0HR	7	17484	47935	2	20	1384	NIL	5	NIL	0.1454	268	4.59	0.05

DSM Report

Client : UNIVERSITI MALAYSIA SARAWAK

Date Received : 09/01/2023

Job No. : TNBR-AC-48873-23-0074

Job Status : Waiting For Approval

No.	Sample No.	Certificate No.	Sampling Date	Mnemonic	Site	TX No.	DGA (ppm)									NV	Moisture	Viscosity	Resistivity
							H ₂	O ₂	N ₂	CH ₄	CO	CO ₂	C ₂ H ₄	C ₂ H ₆	C ₂ H ₂				
1	AC-TXOIL-23-00196		09/01/2023	POME F	UNIMAS	POME 0.02CNT/1000HR	39	17454	55511	2	63	2914	2	27	1	1.6738	322	4.89	0.04
2	AC-TXOIL-23-00197		09/01/2023	POME H	UNIMAS	POME 0.10CNT/1000HR	41	18966	63328	2	60	2696	1	24	NIL	1.6048	273	4.88	0.08
3	AC-TXOIL-23-00198		09/01/2023	POME I	UNIMAS	PURE POME/50HR	10	20313	59420	2	22	1207	NIL	24	NIL	0.3728	280	4.58	0.16
4	AC-TXOIL-23-00199		09/01/2023	POME A	UNIMAS	POME+0.01TiO2/1000HR	33	18589	61629	2	52	2562	1	21	NIL	1.8587	258	4.87	0.11
5	AC-TXOIL-23-00200		09/01/2023	POME B	UNIMAS	POME+0.02TiO2/1000HR	34	23353	67430	2	67	3331	1	19	NIL	2.1485	318	5.01	0.04
6	AC-TXOIL-23-00201		09/01/2023	POME C	UNIMAS	POME+0.05TiO2/1000HR	27	21047	61938	1	45	2337	1	15	NIL	1.8125	264	4.9	0.05
7	AC-TXOIL-23-00202		09/01/2023	POME D	UNIMAS	POME+0.10TiO2/1000HR	18	23266	65488	1	36	2189	1	19	NIL	1.7831	287	4.92	0.04
8	AC-TXOIL-23-00203		09/01/2023	POME E	UNIMAS	POME+0.01CNT/1000HR	31	23601	69138	1	67	3342	1	19	NIL	2.3889	384	5.04	1570
9	AC-TXOIL-23-00204		09/01/2023	POME G	UNIMAS	POME 0.05CNT/1000HR	34	21091	66945	1	59	2611	1	23	NIL	1.4265	313	4.86	0.04

4

Client : UNIVERSITI MALAYSIA SARAWAK

5

Date Received : 24/08/2023

6

Job No. : TNBR-AC-48873-23-3118

7

Job Status : Waiting For Approval

8

9

10

No.	Sample No.	Certificate No.	Sampling Date	Mnemonic	Site	TX No.	Sampling Point	NV	Moisture	Power Factor	Flash Point	Pour Point	Viscosity	Resistivity	
								mg KOH/g	ppm		°C	°C	mm ² /s		
14	1	AC-TXOIL-23-09782	TNBR-AC-ECP001-23-12506	21/08/2023	POME A	UNIMAS	NON-AGED POME	Others	0.1772	477	0.09135	175	13	4.54	1.26
15	2	AC-TXOIL-23-09783	TNBR-AC-ECP001-23-12505	21/08/2023	POME B	UNIMAS	0.01TiO2/50HR	Others	0.2508	278	0.1318	180	13	4.68	0.95
16	3	AC-TXOIL-23-09784	TNBR-AC-ECP001-23-12511	21/08/2023	POME C	UNIMAS	0.02TiO2/50HR	Others	0.2568	290	0.4095	174	13	4.61	0.49
17	4	AC-TXOIL-23-09785	TNBR-AC-ECP001-23-12510	21/08/2023	POME D	UNIMAS	0.05TiO2/50HR	Others	0.2889	258	0.412	180	13	4.61	0.32
18	5	AC-TXOIL-23-09786	TNBR-AC-ECP001-23-12512	21/08/2023	POME E	UNIMAS	0.10TiO2/50HR	Others	0.3195	262	0.471	172	13	4.6	0.25
19	6	AC-TXOIL-23-09787	TNBR-AC-ECP001-23-12513	21/08/2023	POME F	UNIMAS	0.01TiO2/250HR	Others	0.5401	337	0.2663	174	13	4.81	0.41
20	7	AC-TXOIL-23-09788	TNBR-AC-ECP001-23-12509	21/08/2023	POME G	UNIMAS	0.02TiO2/250HR	Others	0.4811	295	0.28195	175	13	4.71	0.41
21	8	AC-TXOIL-23-09789	TNBR-AC-ECP001-23-12508	21/08/2023	POME H	UNIMAS	0.05TiO2/250HR	Others	0.4258	272	0.3533	182	13	4.69	0.35
22	9	AC-TXOIL-23-09790	TNBR-AC-ECP001-23-12504	21/08/2023	POME I	UNIMAS	0.10TiO2/250HR	Others	0.6564	300	0.2059	167	13	4.77	0.52
23	10	AC-TXOIL-23-09791	TNBR-AC-ECP001-23-12507	21/08/2023	POME J	UNIMAS	0.01TiO2/1000HR	Others	2.561	387	0.452	168	13	5.46	0.29
24	11	AC-TXOIL-23-09792	TNBR-AC-ECP001-23-12503	21/08/2023	POME K	UNIMAS	0.02TiO2/1000HR	Others	0.711	277	1.1305	172	13	4.8	0.13
25	12	AC-TXOIL-23-09793	TNBR-AC-ECP001-23-12502	21/08/2023	POME L	UNIMAS	0.05TiO2/1000HR	Others	0.9837	296	0.35105	174	13	4.89	0.31

26

4

5

6

7

8

9

Client : UNIVERSITI MALAYSIA SARAWAK

Date Received : 24/08/2023

Job No. : TNBR-AC-48873-23-3119

Job Status : Waiting For Approval

No.	Sample No.	Certificate No.	Sampling Date	Mnemonic	Site	TX No.	Sampling Point	NV	Moisture	Power Factor	Flash Point	Pour Point	Viscosity	Resistivity
								mg KOH/g	ppm		°C	°C	mm²/s	
1	AC-TXOIL-23-09794	TNBR-AC-ECP001-23-12541	21/08/2023	POME M	UNIMAS	0.10TiO2/1000HR	Others	1.7356	407	0.9745	170	13	5.03	0.26
2	AC-TXOIL-23-09795	TNBR-AC-ECP001-23-12542	21/08/2023	POME N	UNIMAS	0.01CNT/0HR	Others	0.1836	357	0.7035	174	13	4.54	
3	AC-TXOIL-23-09796	TNBR-AC-ECP001-23-12543	21/08/2023	POME O	UNIMAS	0.02CNT/0HR	Others	0.162	290	0.8685	173	13	4.53	
4	AC-TXOIL-23-09797	TNBR-AC-ECP001-23-12544	21/08/2023	POME P	UNIMAS	0.05CNT/0HR	Others	0.192	289	1.4565	171	13	4.57	
5	AC-TXOIL-23-09798	TNBR-AC-ECP001-23-12545	21/08/2023	POME Q	UNIMAS	0.10CNT/0HR	Others	0.1742	283	2.3695	178	13	4.51	
6	AC-TXOIL-23-09799	TNBR-AC-ECP001-23-12554	21/08/2023	POME R	UNIMAS	0.01CNT/50HR	Others	0.2528	260	0.727	176	13	4.59	
7	AC-TXOIL-23-09800	TNBR-AC-ECP001-23-12546	21/08/2023	POME S	UNIMAS	0.02CNT/50HR	Others	0.2568	294	0.3111	178	13	4.62	
8	AC-TXOIL-23-09801	TNBR-AC-ECP001-23-12547	21/08/2023	POME T	UNIMAS	0.05CNT/50HR	Others	0.2675	284	0.28835	180	13	4.6	
9	AC-TXOIL-23-09802	TNBR-AC-ECP001-23-12548	21/08/2023	POME U	UNIMAS	0.10CNT/50HR	Others	0.2189	294	0.35435	176	13	4.66	
10	AC-TXOIL-23-09803	TNBR-AC-ECP001-23-12549	21/08/2023	POME V	UNIMAS	0.01CNT/250HR	Others	0.592	329	1.093	178	13	4.74	
11	AC-TXOIL-23-09804	TNBR-AC-ECP001-23-12556	21/08/2023	POME W	UNIMAS	0.02CNT/250HR	Others	0.372	221	1.2535	170	13	4.63	
12	AC-TXOIL-23-09805	TNBR-AC-ECP001-23-12550	21/08/2023	POME X	UNIMAS	0.05CNT/250HR	Others	0.3849	225	0.9945	182	13	4.67	

DSM (2)

DSM Report

Client : UNIVERSITI MALAYSIA SARAWAK

Date Received : 24/08/2023

Job No. : TNBR-AC-48873-23-3120

Job Status : Waiting For Approval

No.	Sample No.	Certificate No.	Sampling Date	Mnemonic	Site	TX No.	Sampling Point	NV	Moisture	Power Factor	Flash Point	Pour Point	Viscosity	Resistivity
								mg KOH/g	ppm		°C	°C	mm ² /s	
1	AC-TXOIL-23-09806	TNBR-AC-ECP001-23-12524	21/08/2023	POME Y	UNIMAS	0.10CNT/250HR	Others	0.5095	352	0.977	178	13	4.71	
2	AC-TXOIL-23-09807	TNBR-AC-ECP001-23-12525	21/08/2023	POME Z	UNIMAS	0.01CNT/500HR	Others	0.5542	240	1.711	179	13	4.69	
3	AC-TXOIL-23-09808	TNBR-AC-ECP001-23-12526	21/08/2023	POME a	UNIMAS	0.02CNT/500HR	Others	0.5277	219	2.245	176	13	4.68	
4	AC-TXOIL-23-09809	TNBR-AC-ECP001-23-12533	21/08/2023	POME b	UNIMAS	0.05CNT/500HR	Others	0.511	257	3.147	176	13	4.7	
5	AC-TXOIL-23-09810	TNBR-AC-ECP001-23-12527	21/08/2023	POME c	UNIMAS	0.10CNT/500HR	Others	0.5515	241	1.9355	177	13	4.69	
6	AC-TXOIL-23-09811	TNBR-AC-ECP001-23-12539	21/08/2023	POME d	UNIMAS	0.01CNT/1000HR	Others	2.3164	513	1.181	162	13	5.3	
7	AC-TXOIL-23-09812	TNBR-AC-ECP001-23-12528	21/08/2023	POME e	UNIMAS	0.02CNT/1000HR	Others	2.2449	557	1.157	178	13	5.27	
8	AC-TXOIL-23-09813	TNBR-AC-ECP001-23-12535	21/08/2023	POME f	UNIMAS	0.05CNT/1000HR	Others	1.398	480	1.1055	175	13	4.99	
9	AC-TXOIL-23-09814	TNBR-AC-ECP001-23-12530	21/08/2023	POME g	UNIMAS	0.10CNT/1000HR	Others	2.0027	478	2.803	172	13	5.19	

Appendix E: Duval Triangle and Pentagon Coordinates Calculation

Duval Triangle Method

Nanofluid samples	%CH ₄	%C ₂ H ₄	%C ₂ H ₂	Fault type
0.01-g/L TiO ₂	66.7	33.3	0	T1
0.02-g/L TiO ₂	66.7	33.3	0	T1
0.05-g/L TiO ₂	50	50	0	T1
0.10-g/L TiO ₂	50	50	0	T1
0.01-g/L MWCNT	50	50	0	T1
0.02-g/L MWCNT	50	50	0	T1
0.05-g/L MWCNT	50	50	0	T1
0.10-g/L MWCNT	66.7	33.3	0	T1

Duval Pentagon Method

Nanofluid samples	(x_{H_2}, y_{H_2})	$(x_{C_2H_6}, y_{C_2H_6})$	(x_{CH_4}, y_{CH_4})	$(x_{C_2H_4}, y_{C_2H_4})$	$(x_{C_2H_2}, y_{C_2H_2})$	(C_x, C_y)
0.01-g/L TiO ₂	(0, 57.9)	(−35.0, 11.4)	(−2.1, −2.8)	(1.1, −1.5)	(0, 0)	(−7.2, 13.0)
0.02-g/L TiO ₂	(0, 60.7)	(−32.3, 10.5)	(−2.1, −2.9)	(1.0, −1.4)	(0, 0)	(−6.7, 13.4)
0.05-g/L TiO ₂	(0, 61.4)	(−32.3, 10.5)	(−1.4, −1.9)	(1.4, −1.9)	(0, 0)	(−6.46, 13.62)
0.10-g/L TiO ₂	(0, 46.2)	(−46.2, 15.0)	(−2.1, −2.9)	(−1.5, −2.1)	(0, 0)	(−9.24, 11.4)
0.01-g/L MWCNT	(0, 59.6)	(−34.8, 11.3)	(−1.1, −1.5)	(1.1, −1.5)	(0, 0)	(−6.96, 13.58)
0.02-g/L MWCNT	(0, 55.6)	(−36.7, 11.9)	(−1.7, −2.3)	(1.7, −2.3)	(0, 0)	(−7.34, 12.58)
0.05-g/L MWCNT	(0, 61.8)	(−32.9, 10.7)	(−1.1, −1.5)	(1.1, −1.5)	(0, 0)	(−6.58, 13.9)
0.10-g/L MWCNT	(0, 60.3)	(−33.6, 10.9)	(−1.7, −2.3)	(0.9, −1.2)	(0, 0)	(−6.88, 13.54)