STUDY OF ELECTRICAL DISCHARGE EFFECT ON THE NATURAL ESTER INSULATING OILS PROPERTIES

MOHD AIZZAT BIN AZMI

A report submitted in fulfilment of the requirements for the degree of Bachelor of Electrical Engineering With honours

Faculty of Electrical Engineering
UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2018

“I hereby declare that I have read through this report and in my opinion this project is sufficient in terms of scope and quality for the award of the degree of Bachelor of Electrical Engineering with Honours”

Signature :

Supervisor’s Name : Nur Hakimah Binti Abd Aziz

Date :

"I declare that this report entitled " *Study of Electrical Discharges Effects on The Natural Ester Insulating Oils Properties* " is the result of my own research expect as cited in the references. The report has not been accepted for any degree and is not currently submitted in candidature of any other degree".

Signature : ....

Name : Mohd Aizzat Bin Azmi

Date : 6th June 2018

DEDICATION

To my beloved mother, father and family

For their boundless love, prayer and support

ACKNOWLEDGEMENT

Firstly, I would like to express my gratitude to my Final Year Project supervisor Madam Nur Hakimah Binti Abd Aziz for her invaluable guidance, suggestions, encouragement and continued support during the course of this research work. Special thanks also to Mr. Sharin Bin Ab. Ghani, Mr. Imran Bin Sutan Chairul and Dr Hidayat bin Zainuddin, for their sincere guidance especially about this research works scope and background. Next, a very great appreciation to Faculty of Electrical Engineering, Universiti Teknikal Malaysia Melaka for the given opportunity to implement my knowledge during 4 years of my study in this research work.

Besides that, I would like to extend my gratitude to Mr Wahyudi, technician in high voltage lab. His assistance during the experiments is greatly appreciated. Extremely grateful to classmates for their helps in completing this study. Without their encouragement and assistance, I would never accomplish this research work. Special thanks to all staff, and lecturers for their helps and motivation during my undergraduate life.

Last but not least, I would also like to express my profound gratitude to my family for their moral support and encouragement during my study in UTeM.

ABSTRACT

Natural Ester Insulating oil NEI is an environmental friendly source due to its high rate biodegradability which is very environmental friendly. Although, the cooling properties of the natural ester oil are poorer than mineral oil it is still widely used because of its flash point and fire point and lower fire risk. The natural ester insulating (NEI) oil PFAE and Midel eN had been chosen to investigate the effect of electrical discharge. In this research work, the oil sample need to pass the initial condition by following ASTM D974, D6871, D1816 and D1533 standards in terms of acidity, moisture and breakdown (BDV) before proceed to the next stage. Hence, the treatment process will be performed if the moisture content of the oil sample does not achieve the standard. Next, partial discharges inception voltage (PDIV) will be tested on the test cell to determine the PDIV value of oil sample. All the equipment and setup for PDIV is according to IEC 61294 standard. Next, the electrical discharges will apply from 200 until 1000 discharges with a constant voltage based on the PDIV value. Electrical discharge is the emission and transmission of electricity in an electric field applied to the medium such as liquid. Hence, this study will determine the effect of the electrical discharge to the physicochemical properties in terms of moisture content, acidity level and breakdown voltage of the Midel eN and PFAE. The properties of PFAE and Midel eN after electrical discharge test also will be analyzed to compare with initial properties of natural ester insulating (NEI) oil. According to the experiment conducted, the PFAE oil change is not significant in terms moisture content, acidity and breakdown voltage (BDV) compared to Midel eN oil. This result is due to its saturated fatty chain acid with good oxidation stability. The BDV of PFAE oil also not much affected by the high electrical discharges. Hence, it shows that PFAE has better performance in terms of good oxidation stability and also the BDV value that can withstands.

ABSTRAK

Natural Ester Insulating oil NEI adalah minyak daripada sumber mesra alam kerana kadar biodegradasi yang tinggi yang sangat mesra alam. Walaupun, sifat penyejuk minyak ester semulajadi lebih rendah daripada minyak mineral yang masih digunakan secara meluas kerana titik kilat dan titik api dan risiko kebakaran yang lebih rendah. Penebat ester semula jadi (NEI) minyak PFAE dan Midel eN telah dipilih untuk menyiasat kesan pelepasan elektrik. Dalam kajian ini, sampel minyak perlu melepasi keadaan awal dengan mengikuti piawaian ASTM D974, D6871, D1816 dan D1533 dari segi nilai asid, kelembapan dan kerosakan voltan (BDV) sebelum meneruskan ke peringkat seterusnya. Oleh itu, proses rawatan akan dilakukan jika kandungan lembapan sampel minyak tidak mencapai standard. Seterusnya, voltan penunaikan pelepasan separa (PDIV) akan diuji pada sel ujian untuk menentukan nilai PDIV sampel minyak. Semua peralatan dan persediaan untuk PDIV adalah mengikut piawaian IEC 61294. Seterusnya, pelepasan elektrik akan dikenakan dari 200 hingga 1000 pelepasan dengan nilai voltan yang tetap berdasarkan nilai PDIV. Pelepasan elektrik adalah pelepasan dan penghantaran elektrik di medan elektrik yang digunakan untuk medium seperti cecair. Oleh itu, kajian ini akan menentukan kesan pelepasan elektrik kepada sifat-sifat fizikokimia dari segi kandungan lembapan, tahap keasidan dan kerosakan voltan Midel eN dan PFAE. Sifat-sifat PFAE dan Midel eN selepas ujian pelepasan elektrik juga akan dianalisis untuk membandingkan dengan sifat awal penebat ester semula jadi (NEI). Menurut eksperimen yang dijalankan, perubahan minyak PFAE tidak signifikan dalam kandungan kelembapan, keasidan dan voltan kerosakan (BDV) berbanding minyak Midel eN. Keputusan ini disebabkan oleh asid rantai lemak tepu dengan kestabilan pengoksidaan yang baik. BDV minyak PFAE juga tidak banyak dipengaruhi oleh pelepasan elektrik yang tinggi. Oleh itu, ia menunjukkan bahawa PFAE mempunyai prestasi yang lebih baik dari segi kestabilan pengoksidaan yang baik dan juga nilai BDV yang boleh bertahan

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

FIGURE	TITLE	PAGE

FIGURE	TITLE	PAGE

LIST OF ABREVIATION

HV	-	High Voltage
AC	-	Alternating Current
MI	-	Mineral Insulation
NEI	-	Natural Ester Insulation
ASTM	-	American Society for Testing and Materials
IEC	-	International Electrotechnical Commission
BDV	-	Breakdown Voltage
FPO	-	Fresh Palm Oil
VDE	-	Verband Deutsher Elektrotechniker
PDIV		Partial Discharge Inception Voltage

CHAPTER 1

INTRODUCTION

1.0 Introduction

This chapter highlights the importance of the study through the description on the background of the research and the related issues that motivate this research work. The objectives of this study are explained which limited to the scopes stated here. Lastly, the structure of the thesis is given, showing the connection between the chapters.

1.1 Research Background

A transformer is one of the vital asset in power network system which that transfer electric energy from one alternating current circuit to one or more other circuits, either increasing or reducing the voltage [1]. For more than hundred years, liquid-immersed transformers have been mainly filled with mineral oil due to its wide availability, good properties, and low cost. However, there have also been major environment concerns over the toxic effects of uncontained mineral oil spills. Thus, it is crucial to use insulating oil with a high biodegradability and more environmental friendly. The recent availability of natural ester fluids based on vegetable oils has provided a new insulating liquid for use with transformers [2].

The reliability of a power transformer is largely determined by its insulation condition [1]. The insulation fluids of the transformer able to lengthen the transformer life span by protecting solid insulation and serve as a coolant by absorbing the temperature heat occurring produced in transformer's winding and core [2,3,4]. Most of the distribution transformers in Malaysia are oil filled that has capability to dissipate heat more efficiently compared to gas and solid materials [5]. Mineral oil has been widely used in Malaysia for a long time due to its reasonable price. Moreover, mineral oil has high dielectric strength, medium viscosity, less susceptible to oxidation, able to running well in low temperature and good heat transfer capacity [6].

However, mineral oil has some disadvantages such as low fire point, consist of toxic substance and poorly biodegradable [7]. The usage of mineral oil also not environmental friendly because it is a toxic waste thus affect the fertile of soil. The environment will get influenced if mineral oil spill occur because 20% of mineral oil can only biodegrade within 28days. Then, it could contaminate the soil and ground water [8]. Furthermore, the mineral oil sources do not last long because it's from fossil like petroleum and gas.

More research work has been conducted to find other available sources that better than mineral oil. Natural ester is a better alternative and has been use since early 1990's [6]. In 2000's, a number of companies in US, UK and Japan such as LION, MIDDLE and FR3 have started to commercialised the vegetable oil. Ester oil is increasing due to its properties that are better than mineral oil i.e non-toxic, biodegradable, lower fire risk, high flash point and fire point [7].

1.2 Motivation

Natural ester insulation (NEI) oil are the derivatives from the naturally available plants and seeds of the plants for example sunflower, palm oil and rapeseed. The natural ester characteristics depend on the fatty acids compositions.

Recently, there is researcher study about the partial discharges characteristics on Palm Fatty Acid Ester (PFAE) oil as High Voltage Insulating Material and as biodegradable oil for power transformer oil application. Besides that, the characteristics of PFAE also compare to other insulation oil which is mineral oil, to show the PDIV value between two oil samples. It also shows the result revealed that the partial discharges number of PFAE is lower than mineral oil. On top that, it makes as a catalyst for this research work by comparing the properties of Midel eN and PFAE. This give an idea to investigate the electrical discharges effect to natural ester insulating (NEI) oil by using palm oil and rapeseed based. Hence, this research work also to study on it physicochemical properties as the insulator for transformer. Therefore, this research work aims to investigate the capability of Midel eN and PFAE oil as the transformer insulation oil after the electrical discharge.

This research work studies the effect of electrical discharge on the natural ester insulating oil in terms of the acidity, water content and BDV in the transformer. Hence, to strengthen use the Midel eN and PFAE insulation oil transformer and to reduce pollution to the environment. Finally, to compare the PDIV, acidity, moisture and BDV between Midel eN and PFAE before and after electrical discharges.

1.3 Problem Statement

Power transformer is one of the expensive and important equipment in power generation and transmission system that no exceptions to faced serious failure for example insulation breakdown. One of the causes of insulation failure is electrical discharge. Electrical discharge is the release and transmission of electricity in an electric field applied to a medium such liquid in transformer. The electrical discharges usually happen many times in transformer oil and it will cause the breakdown. The electrical stress in unavoidable in power equipment and the capability of oil to resist decomposition under electrical stress is one of the important things for the safety of the transformer. However, there is still lack of data and needs to study for further analysis in terms of the effect to the physicochemical properties after electrical discharges to the natural ester insulating (NEI) oil. On top of that, by investigating the effect of electrical discharges on MIDEL eN and PFAE, it can show the best result to choose the best performance of the NEI. It also a lesser extent, to support a green campaign by using rapeseed and palm oil. Does the Midel eN can perform better than PFAE oil ?

Therefore, this research work will focus on the PDIV, breakdown voltage and the effect of electrical discharge on natural ester insulating (NEI) oil which is Midel eN and PFAE in terms of acidity, moisture content and BDV. It is widely known that ester oil has better quality as transformer oil compared to mineral oil. The characteristic of PFAE also are similar with commercially mineral oil. Even though, vegetable oil is more expensive but it can be an alternative to reduce the environmental pollution resulting from the use of transformer and also reducing the dependence on mineral oil use. Moreover, Malaysia is one of the main producers of palm oil in the world.

1.4 Objectives

- To investigate the effect of electrical discharge on the natural ester-based insulating oil.
- To compare the properties of natural ester insulating oil after electrical discharges.

1.5 Scope

The scopes of this work are given as follow:

- The effect to the natural ester-based insulating oil in terms of acidity, moisture and BDV
- Types of natural ester insulation oil (NEI) used are Midel eN 1204 (rapeseed based) and PFAE.
- The ASTM D974 & D1533 Standard with compliance of D6871 standard is followed for acidity and moisture experiment.
- The ASTM D1816 Standard is followed for Breakdown Voltage BDV experiment.
- Partial Discharges Inception Voltage procedure for insulating liquid is followed the IEC 61294 standard.
- Type of electrode used in the experiment to analyse the electrical discharge stability of the Midel eN and PFAE is point to plane with 10mm gap.
- Type of needle used in the experiment to analyse the electrical discharge stability of the Midel eN and PFAE is needle of 50mm length and 1mm diameter from Ogura Jewel Industry Co Ltd.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter gathers all the related information to this research work from the previous researchers such as type of transformer oil, properties of insulating oil, and electrical discharge. The published results on the properties of natural ester oil (NEI) also are attached for comparison. On top of that, there are also the detail of method and type of insulation oil that been used by other researcher in this chapter. The most appropriate method is selected for this study.

2.2 Transformer

Transformers are one the precious equipment in a substation and power plant. There are a few types of transformer which is power transformer and distribution transformer. Transformer also known as important component of high voltage equipment for power generation plants, transmission systems and big industrial plant. In 1884, the early transformer produce is a dry type transformer. In 1891, the petroleum oil was used on an experiment basis in the insulation of three phase transformer. Due to the higher demand of electric energy and the growing power transmission network, air- insulated transformer become large. To reduce the volume, petroleum oil application in power transformer was generalized from 1905 but mineral oil did not provide the necessary fire protection [9]. Almost 60% of the total cost power station covered by power transformer. There is a few type of distribution transformer, for example hermetically sealed and free breathing with rating 11/0.433kV, 22/0.433Kv and 33/0.433kV [10].

2.3 Insulation System of the Transformer

In general, one of the factors electrical faults occurring in power transformers increase due to its weak insulating performance. Hence, the life span of transformer also depends on its insulating condition. There are three different states of insulation system in transformer; gas, solid and liquid (transformer oil) [11].

2.3.1 Gas Insulation

Air is one of the examples of gas insulation that commonly used as the insulating medium in the most of the electrical apparatus. Other than that, nitrogen (N^2), carbon dioxide (CO^2), freon ($CC1_2F_2$) and sulphur hexafluoride (SF_6) are also used as the insulating medium. Therefore, there are several preferred properties of a gaseous insulation for practical use in high voltage applications. The properties includes high dielectric strength, thermal and chemical stability inactivity towards materials of construction, non-flammability, low temperature of condensation, and good heat transfer. Regarding to the previous research, it state that SF_6 seem to possess most of these requirements [12].

2.3.2 Solid Insulation

Solid insulation is one of the type insulation that needs to consider in the system of transformer. However, there are few factors that affected the degradation solid insulation (cellulose based products) in transformer which is temperature, moisture content, oxygen and acid exists in the insulation system. The degradation of solid insulation produces chemical products such as furanic derivatives, carbon monoxide (CO) and carbon dioxide (CO_2) which dissolved in the transformer oil. These chemical products are produced due to the chains of cellulose molecular are getting shorter after the degradation process occurs. Degree of polymerization (DP) is an average measurement of the number of glucose units per molecule chain. Besides, the location of cellulosic materials that need to be replaced can be identified with the help of DP analysis. Moreover, cellulose materials are the weakest link in the insulation system. Hence, the life span of the transformer will end when the paper reaches DP of 200 or less where the paper becomes very brittle [13].

2.3.3 Liquid Insulation

Insulation oil is one of the important parts in transformer that need to consider during do the maintenance of transformer. The right way to ensure the insulating oil able to operate well is choosing the oil that fulfils the standard requirement. Liquid insulation also has been used to operate the transformer rating more than 10-25kV and also been used in power distribution equipment as the dielectric liquids. Liquid insulation also widely used because of its capable of long service life with low cost, less maintenance issue, good dielectric and compability with other materials. There are some characteristics that need to consider in selecting liquid dielectric system which are dielectric strength, dielectric constant value, liquid viscosity, thermal characteristics, flammability and physical environmental conditions [14]. However, there are several factors that affect the quality of insulating oil such as oxidation, contamination, and excessively high temperature. Based on these factors, oxidation is the most factors that give influence to cause of oil deterioration. Hence, many transformer manufacturers overcome this problem by sealing the transformer from the atmosphere. Besides, moisture is the main contaminants in the oil where its presence can reduce the dielectric properties of the insulating oil. Apart from that, an excessively high temperature in transformer will cause decomposition of the oil and will increase the rate of oxidation. The best way to overcome this situation is by preventing the transformer from overloading [15].

2.4 Types of Insulating Oil in Transformer

There are two type of insulating oil in transformer which is mineral and natural ester oil.

2.4.1 Mineral insulating Oil

The mineral insulating oil produced from crude oil has been widely used in power transformer, since the development of the power sector was newly used. Besides that, mineral oil compatible and reliable used for over a century. Mineral oil also has been used all over the world due to its ageing behaviour, good dielectric strength, low viscosity, less susceptible to oxidation and able to operate in low temperature. However, mineral oil has low biodegradability [16]. Mineral oil has some characteristics that unfavourable properties which are low fire point, toxicity and poor biodegradable[7]. Hydrocarbon in molecules mainly found in mineral oils. Hydrocarbon composed only of a carbon backbone more or less saturated with hydrogen[16]. There are three different types in crude oils which are paraffinic, naphthenic and mix crudes [5]. However, in varying amounts, molecules sizes and structures, the composition affects the physical properties such as viscosity, viscosity index, density, cloud point, pour point and solving power [16].

2.4.2 Natural Esters Insulating (NEI) Oil

Recently the use of ester oil are increasing in order to support the green technology. Natural ester liquids which known as vegetables oil is seen as an alternative to mineral oil because it is non-toxic and good biodegradable. Natural ester also has lower fire risk, higher flash point and fire point compared with mineral oil [6]. Besides that, natural ester rarely used in industrial applications because of the lack of oxidative stability. The viscosity of mineral oil also is three to four times less than natural ester insulating (NEI) oil [17].

Natural ester oil consists of triglycerids which are naturally synthesized by esterification of the tri-alcohol with three fatty acids that from the plant [18,19].

Triglycerides esters of fatty acids presents both fats and oils where fats has relatively high percentage of saturated triglycerides and can be freeze to solid at below room temperature. Usually, the oily part remains as liquid when temperature is above 0°C.. Besides that, oil with high unsaturation remains as liquid at temperature 15°C and 30°C. The molecules of triglycerides ester may be presented as CH2-OOCR1, CH-OOCR2 and CH-OOCR3 where R1, R1 and R3 are same or different types of fatty acid and chains [20]. Basically, the fatty acids are composed through the linear hydrocarbon chains and it is ended by a carboxylic function [18]. The molecular structure of Triglyceride and Mono ester has shown in figure 2.1 and the fatty acid composition of some vegetable oils can refer to table 2.1.

The natural ester insulating (NEI) oil also subjected to thermal stresses and different chemical reaction take place when transformer is operate for example oxidation and hydrolysis. According to this reaction, it will lead to formation of different product such as gasses, acids and water [21]. Hence, the higher the unsaturation level of the vegetable oil, the more easily to oxidation because the oxygen molecule disrupts the fatty acid at the carbon double bond [22]. The hydrolysis reaction will occur between triglycerides and water and. Next, it will forms glycerol and free fatty acids [23]. Based on the hydrolysis reaction which form acids, the vegetables have higher acidity than mineral oil and also different chemical structure. The vegetables oil also contain high molecular acid such as oleic and steraic [24].

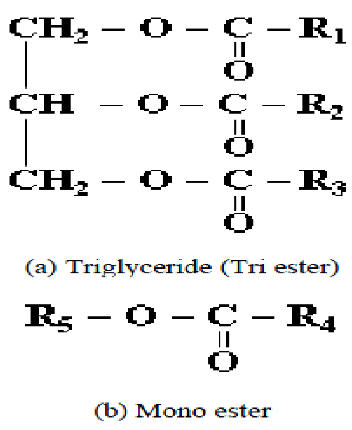


Figure 2.1: Molecular structure of Triglyceride and Mono ester [19]

Table 2.1: Typical fatty acid composition of some vegetable oils [20]

Vegetables oil	Saturated fatty acids (%)	Unsaturated fatty acids (%)		
		Mono	Di	Tri-
Canola Oil	7.9	55.9	22.1	11.1
Corn Oil	12.7	24.2	58	0.7
Cottonseed Oil	25.8	17.8	51.8	0.2
Peanut Oil	13.6	17.8	51.8	0.2
Olive Oil	13.2	73.3	7.9	0.6
Safflower Oil	8.5	12.1	74.1	0.4
Safflower oil, high Oleic Content	6.1	75.3	14.2	-
Soybean Oil	14.2	22.5	51	6.8
Sunflower Oil	10.5	19.6	65.7	-
Sunflower Oil, High oleic content	9.2	80.8	8.4	0.2
*Low erucic acid variety of rapeseed oil. Recently canola oil with more than 75% monounsaturated content has been delayed				

2.4.3 Palm Fatty Acid Ester (PFAE) oil.

Palm Fatty Acid Ester known as PFAE is palm-oil based transformer oil that manufactured by Lion Corporation and Japan Ae Power System Corporation. PFAE has close dielectric properties to mineral oil regarding the modification on it and also has advantageously derived from natural ester. PFAE also has a few characteristics that gives an advantage as insulation oil which is high flash point, high cooling ability, good oxidation stability, providing a better match of oil-paper insulation system due to its high relative permittivity and enhances the insulation system due to its high water solubility. However, the usage of PFAE as transformer oil is only limited in Japan and not globally commercialize . Besides that, PFAE also is a fatty acid alkyl ester (R_1COOR_2) derived from palm oil. PFAE also has great potential for use as an insulating medium for transformer and environmentally friendly [25].

Table 2.2: Composition of fatty acid in oils [26]

Fatty Acid	Soy-bean oil	Rapeseed oil	Sunflower oil	Palm		Coconut oil	Beef fat
				Palm oil	Palm kernel oil		
C10 Capric acid	-	-	-	-	3.3	7.8	-
C8 Caprylic acid	-	-	-	-	3.1	7.6	-
C12 Lauric acid	-	-	-	-	45.7	44.8	-
C14 Myristic acid	-	-	0.2	1.1	16.4	18.1	2.0
C16 Palmitic acid	6.7	5.4	7.1	44.4	8.9	9.5	32.5
C18 Stearic acid	3.3	2.0	2.8	4.3	2.3	2.4	14.5
C18 Oleic acid	41.7	68.0	30.0	39.9	17.1	8.2	48.3
C18 Linoleic acid	41.3	21.0	59.5	9.4	2.7	1.5	2.7
C18 Liolenic acid	5.9	-	-	-	-	-	-
Others	1.1	3.6	0.4	0.9	0.5	0.1	-

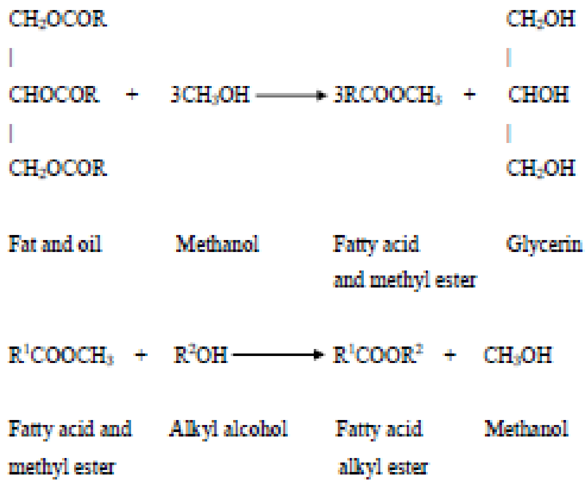


Figure 2.2 : Chemical reaction of PFAE [26]

By referring composition fatty acids as shown in Table 2.2, the palm derived oil has carbon 8 to 18. Triglycerides of C16 and C18 acid are presents for palm oil. Hence, figure 2.2 shows the chemical reaction of PFAE. Besides that, 99% purity of fatty acid methyl ester is using palm derived oil as a raw material [26]. In order to improve the liquidity, Fatty acid methyl ester has been through trans-esterification with other alkyl alcohol at low temperature. Based on the equation in figure 2.3, it also shows PFAE that representing or declare as fatty acid alkyl ester R_1COOR_2. Moreover, it improves liquidity at low temperature when trans esterification from various fatty acid methyl ester with fatty acid moieties of 8 to 18 carbon atoms. PFAE also has good biodegradability and environmental friendly in case if spilled or polluted to soil and water [26].

2.4.4 Midel eN

Midel eN is a natural ester oil, it particularly good for those seeking an insulating oil that environmentally friendly in term of dielectric fluid requirement. It also made from renewable material which rapeseed based and in the case of a leak or spillage its biodegradability greatly reduces the potential damage caused to the surrounding area. It give advantages for environmental protection by it properties that non-toxic and no corrosive sulphur. Midel eN has been prove that non-corrosive by independent laboratories and also contains lower carbon footprints. Moreover, it has very high fire point more than 300°C and is fully RoHS compliant.

Besides that, Midel eN also offers superior moisture tolerance and has the potential to increase the lifetime of cellulose based solid insulation which in turn can extend the transformer life. It able to absorbs large amount of moisture with no reduction of breakdown voltage (up to 300ppm) and also has high saturation limit making precipitation of free water virtually impossible. In the other hand, Midel eN also has slower rate of cellulose ageing than mineral oil and able to longer the cellulose lifetime at standard temperature.

This insulating oil is suitable for sealed transformer and good operating in temperate climate, and making it a particular good choice for distribution transformer in built-up areas and indoors [27]. Hence, all the characteristic of Midel eN are based on the Midel eN fluid properties in table 2.3 and the detail also shown in appendix B.

Table 2.3: Midel eN fluid properties

Property	Standard Test Methods		MIDEL eN 1215	MIDEL eN 1204	MIDEL 7131	Mineral Oil
	ASTM	ISO/IEC				
Physical						
Colour	D1500		0.5	0.5		0.5
Flash Point PMCC (°C)	D93	ISO 2719	> 260	> 260	260	150
Flash Point COC (°C)	D92	ISO 2592	> 315	> 315	275	160
Fire Point (°C)	D92	ISO 2592	> 350	> 350	316	170
Net Calorific Value (MJ/kg)	D240-2		37.2	37.5	30.8	46.0
IEC Classification		IEC 61039	K2	K2	K3	O1
Pour Point (°C)	D97	ISO 3016	-18	-31	-56	-50
Density at 20°C (g/cm³)		ISO 3675	0.92	0.92	0.97	0.88
Viscosity (mm²/sec)	D445	ISO 3104				
@100°C			7.6	8.3	5.3	2.6
@40°C			32	37	29	8.7
@0°C			206	232	233	70
@-20 (°C)			Solid	1485	1440	400
Biodegradation - OECD 301			Readily Biodegradable	Readily Biodegradable	Readily Biodegradable	Non Biodegradable
Electrical						
Dielectric Breakdown (kV)	D877		≥ 30	≥ 30	47	43
Dielectric Breakdown (kV)						
1 mm gap	D1816		30	45	46	47
2 mm gap	D1816		51	57	71	65
2.5 mm gap		IEC 60156	> 75	> 75	> 75	>70
Gassing Tendency (µl/min)	D2300		-31.9	-46.1	+26.0	<+30.0
Dissipation Factor at 90°C		IEC 60247	< 0.03	< 0.03	< 0.008	<0.001

2.5 Importance of Transformer Insulating Oil Testing

2.5.1 Water Content

Water content or moisture in the power transformer insulating oil is one of the factor that need to be considered accordance to the standard ASTM D6871 where the permissible water content in NEI oil is $\leq$ 200ppm. Water content is usually measured in parts per million (ppm) or mg/l [15]. The available standards for water content test are ASTM D1533 [18] and IEC 60814 [28]. Presence of moisture in transformer can affect its dielectric properties. With a decrease in oil temperature, any dissolved water will become free water. Besides that, the ability of water molecule to polarize is responsible for its attraction to each other polar molecules for example hydrocarbons in fatty acids, breaking the weak hydrogen bond of the water molecules. Hence, the BDV of transformer insulation oil also will affect and lower as the moisture content increase. Higher moisture also gives an influence to bubbling effect in transformer. On top of that, one of the routine maintenance procedures is to monitor the moisture content. These standards provide the standard of procedures (SOP) to measure the water content in the oil using Coulometric Karl Fischer Titration. The increasing of moisture in insulation oil can affect the leading causes of electrical breakdown because it increases the ionic conductivity of the oil and speeds up the insulation to breakdown [29].

2.5.2 Acidity

The natural ester insulating liquid contains higher neutralization number (acid value). Acid number usually was used to assess the oxidation and hydrolysis performance. Oxidation and hydrolysis reaction, generate acid in natural ester insulating oil [30] . In order to investigate the performance of insulation oil between Midel eN and PFAE, the acidity test shall be done by referred to ASTM D974 standard with total acid number must below 0.06 mg KOH/g. The acidity content must be measured using 848 Titrino Plus before the insulating liquid do electrical discharge test as shown in figure 2.3. Hence, to reduce electrical conduction and metal corrosion and to increase the life of the insulation system, the natural ester insulating oil need to make sure it content low total acid number.

Figure 2.3: 848 Titrino Plus

2.5.2 Breakdown Voltage

Breakdown voltage test on the insulating oil is also known as dielectric strength test. Breakdown voltage also determines the ability of the insulating oil to withstand electric stress without failure. Hence, breakdown voltage test is very important process before do electrical discharge to ensure the applied voltage not the exceed the breakdown value. The breakdown voltage also assists to indicate the presence of contaminating agents such as water, dirt, cellulosic fibers, or conducting particles in the liquid [15]. This test is measured by applying a voltage through the oil sample between two electrodes under certain prescribed conditions in the oil. The standards that are frequently used for breakdown voltage test are ASTM D1816, ASTM D877 and IEC 60156.

The devices that can be used to measure the breakdown voltage of the transformer oil are Megger OTS60PB, Megger OTS60AF/2, Megger OTS80AF/2 and Megger OTS100AF/2 [31]. The Megger OTS60PB that available in high voltage lab in UTeM shows in figure 2.8. The features between these devices are shown in Table 2.4

Figure 2.4 : Megger OTS60PB

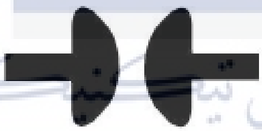
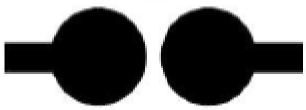
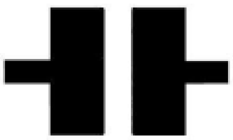
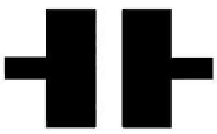
Table 2.4 shows the specifications and features of Megger OTS60PB

Table 2.4: Feature of Megger OTS60PB[31]

Specifications	Megger OTS0PB
Test Voltage	-30 to +30 Vrms / 60 kV (Max)
Operating Temperature	0℃ to 40℃
Operating Humidity	80% Relative Humidity at 40℃
Voltage Resolution	1 kV, ±1%, ±2 digits

Table 2.5 shows three different electrode shape; mushroom-type (shape A), spherical-type (shape B), and cylindrical-type (shape C).

Table 2.5: Type of shape of oil testing electrode [31]

Types of Electrode	Figures of Electrode Shape	Description
Electrode Shape A		• Mushroom / semispherical • 36 mm diameter
Electrode Shape B		• Spherical • 12.7 mm diameter
Electrode Shape C		• Cylindrical • 25.4 mm diameter
Electrode Shape D		• Cylindrical • 25.44 mm diameter with 0.5 mm edge radius

2.6 Electrical discharges

Electrical discharge is the release and transmission of electricity in an electric field applied to medium such as gas and liquid. Electrical discharges also recognise as electrical stress that one of the ageing factor. Insulating oil is reveal to electrical stress and may be experience electrical discharges under certain circumstances. Hence, the electrical stress in unavoidable in power equipment and the capability of oil to resist decomposition under electrical stress is one of the importance things for the safety of the transformer. Electrical stress consist of heat and moisture in the presence of oxygen, oxidise the oil producing free radicals, acid and sludge that are deleterious to the transformer[32].

The electrical discharge initiation in dielectric liquid has been research for many years. Most of the previous studies had been focused on mineral and there is still lack of research regarding electrical discharge to natural ester insulating oil.. Based on the previous research, it state that electrical discharge initiation and propagation are very complicated process involving diverse physical phenomena. Hence, according the physical properties and chemical composition of the liquids, pressure and temperature, type of testing voltage, the process determining discharge initiation may be bubble formation by joule heating. Besides that, the discharge initiation in oil also influence by the volume of oil. Electrical discharges in oil also may be started in oil being under electrical field stress which exceeds 90% of maximum value for electrode preparation. The higher the value impurities or gas bubbles in oil volume, the higher the chance of discharges initiation[33].

2.6.1 Partial Inception Voltage (PDIV)

Partial Discharge is localised gaseous breakdown which can occur within any plant system provided the electrical stress appropriately. Partial discharge activity is both symptoms of degradation in the insulating system of power plant, irrespective of the causative stress and stress mechanism in itself. Based on the partial discharge mechanism, the electron ions atoms, radicals and excited molecular species produced because of the influence of the thermal excitation, the electric field, electrostatic forces, and the electric wind, generated by the collision of the ionic species, moving under influence of the electric field with the molecules of the surroundings gas.

Hence, partial discharge (PD happen when electrical discharge or spark that bridges a small portion of the insulation between two conducting electrodes. Partial discharge activity also can occur at any point in the insulation system especially when the electric field strength exceeds the breakdown strength of that portion of the insulating material. Generally, PD occurs at the defect sites such as delamination, cavities, joins or void in insulation system of high voltage component. The defects can influence the performance of the insulation system in service because the frequently PDs causes the degradation of insulation system, which may lead to breakdown of insulation system. The voltage during ionization and the starting partial discharge is called Partial Discharge Inception voltage (PDIV). There are two type of energy emit by partial discharge which is electromagnetic emissions and acoustic emissions. Electromagnetic emissions is the emit energy in the form of radio waves, light and heat while acoustic emissions emit energy in the audible and ultrasonic ranges. Ozone and oxide of nitrogen gases also one of energy emit by partial discharge [34]. Figure 2.5 shows partial discharge inception voltage (PDIV) circuit set-up.

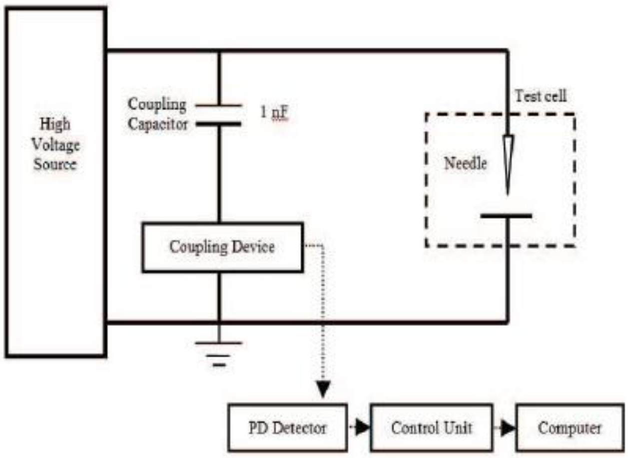


Figure 2.5 : partial discharge inception voltage (PDIV) circuit set-up.[35]

The long term persistent occurrence of partial discharges (PD) may influence the long term stability of insulating liquid. There are different test methods and electrode configuration that can be tested for PDIV. The difference of partial discharge inception voltage (PDIV) and partial discharge extinction voltage (PDEV) are PDIV is the lower voltage at which discharge recur while PDEV is the lower voltage at which discharge appear when applied AC voltage is applied [12].

Partial Discharge also one of the factors that affects the thermal ageing and degradation of the insulating oil. The ageing process is attributed to the decomposition of the hydrocarbon molecules of oil, so the detection of partial discharge (PD) will influence the lifetime of power transformer oil. The charge injection or the occurrence of the bubble effect also makes the partial discharge take place at field intensification. Partial discharge also occurs due to chemical and physical degradation in transformer oil. PD initiation process is also influenced by the space charge at the electrode vicinity, thus PDIV is sensitive to its definition used. [7]. Based on figure 2.6, it shows the partial discharges in the simulation. The only PD occur in quadrant 1 and 3 will be accepted.

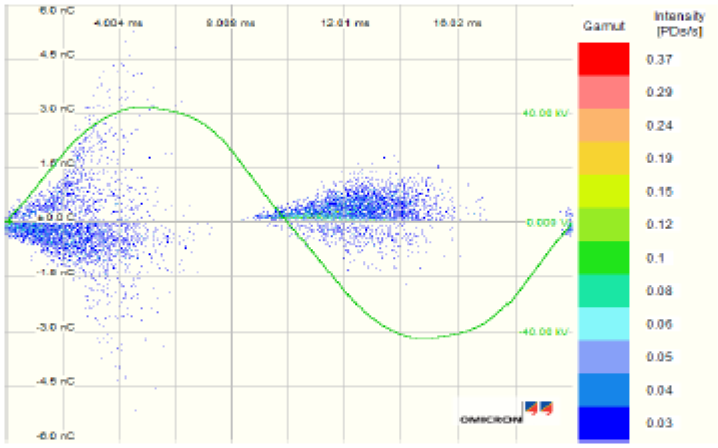


Figure 2.6: Partial discharges pattern [36].

2.7 Analysis of Oil Properties

Transformer insulation oil need to have chemical and physical properties as follows with the main function of transformer. There are 3 main characteristics for transformer insulation oil must be handle which are as insulation barrier, heat transfer medium and to preserve the transformer over time[16]. All these characteristics are under standard specification IEE 60296 and ASTM 3487 which require minimum and maximum limits. Besides that, the properties of dielectric insulation also needed for this application other than the physical and chemical properties which depending on the base and its degree of refining because insulating oil give around 80% dielectric strength to transformer. In order to get potential long in service, the understanding of properties of transformer is also one of the important criteria during selecting the best insulation oil for the transformer. Besides that, the proper maintenance of the insulation system also one of the factors that can longer the life span of transformer and power transformer can function more than 50 years. The insulation oil also act as a coolant and able to allows the transformer to operate in high temperature for a long time because of the oxidation stability of the insulating oil [13]. Furthermore, during electrical energy step up or step down in transformer it will produces heat and the temperature must not higher than the limit to avoid negative effect to the transformer.

To make the accurate choice of the transformer insulation oil, choose the good electrical insulation properties on insulation oil to design a compact transformer construction. Next, choose the insulation oil that excellent as heat transfer medium to ensure the transformer temperature in minimum condition, minimize cellulose and oil degradation. Besides that, the oil that has good oxidation stability for application and loading pattern is the right choice to preserve the fluids properties over a long period [16]. Hence, to use natural ester insulating oil as the transformer insulation liquid, the researcher need to follow the D6871 standard in table 2.6.

Table 2.6: ASTM Standard for natural(vegetables) ester fluids.

D6871 – 03 (2008)

TABLE 1 As-Received New Fluid Property Requirements

Property	Limit	ASTM Test Method
Physical:		
Color, max	1.0	D1500
Fire point, min, °C	300	D92
Flash point, min, °C	275	D92
Pour point, max, °C	−10	D97
Relative density (specific gravity) 15°C/15°C, max	0.96	D1298
Viscosity, max, cSt at:		D445 or D88
100°C (212°F)	15	
40°C (104°F)	50	
0°C (32°F)	500	
Visual Examination	Bright and Clear	D1524
Electrical:		
Dielectric breakdown voltage at 60 Hz		
Disk electrodes, min, kV	30	D877
VDE electrodes, min, kV at:		D1816
1 mm (0.04 in.) gap	20	
2 mm (0.08 in.) gap	35	
Dielectric breakdown voltage, impulse conditions 25°C, min, kV, needle negative to sphere grounded, 1 in. (25.4 mm) gap	130	D3300
Dissipation factor (or power factor) at 60 Hz, max, %		D924
25°C	0.20	
100°C	4.0	
Gassing tendency, max, μl/min	0	D2300
Chemical:		
Corrosive sulfur	not corrosive	D1275
Neutralization number, total acid number, max, mg KOH/g	0.06	D974
PCB content, ppm	not detectable	D4059
Water, max, mg/kg	200	D1533[A]

[A] As stated in Test Methods D1533 Annex A1 "Alternative Solvent Systems," alternate reagents may be needed for certain natural ester formulations. Consult the manufacturer for recommendations. Reagents for aldehydes and ketones (such as Coulomat AK and CG-K) should be used if the additives are unknown. When alternate reagents are needed, using the Test Methods D1533 reagents may yield elevated and erratic water content results.

2.8 Weibull

Weibull probability plot is to measure the best value of its data. It has been used widely in high voltage experiment and measured breakdown voltage. On top of that, the probability value of the data falls at 63.2% where it is the point corresponds to the time of failure of the oil under test. The Weibull distribution also provides the best fit for life data and simple graphical solution. The preference of using Weibull because it can provide a reasonably accurate failure forecast and failure analysis with extremely small samples in which the small samples are allowed cost effective component testing.

The mathematical of Weibull distribution can be defined as follows:

$$F(t) = 1 - e^{(t/n)ß}$$

Where;

F(t)= Fraction falling
t= Failure time
n=Characteristic life or scale parameter
ß= Sloper or shape parameter

By referring the equation above, it can be state that F(t) is always 63.2% when t is equal to n and it does not matter what the real value of β. It also show the feature that occur at the 63.2% regardless of the weibull distribution. Thus, it can be proved with the Weibull equation shown below [37].

$$F(t) = 1 - e^{(t/n)ß}$$

$$= 1\text{-}0.368$$
$$= 0.632 \text{ regardless the value of ß}$$

2.9 Review of related research work

Table 2.7 show the review of related research work that been used as the literature review for this research work.

Table 2.7 : Review of related research work

NO	Title/Author	Statement
1	S. bin A.Ghani, " Enchancing the Dielectric strength and Oxidation Of Natural Ester Insulation Oil By OptimizationOf Antioxidants Mixtures"	• According to the type of transformer used in Malaysia power system, oil-immersed transformer is oil-filled tank complete with laminated iron core and paper wrapped copper winding are one of. • mineral oils have high dielectric strength, medium viscosity, less susceptible to oxidation, able to running well in low temperature, good heat transfer capacity, cost benefits
2	M. Jaroszewski and K. Rakowiecki, "Partial discharge inception voltage in transformer natural ester liquid — Effect of the measurement method in the presence of moisture," IEEE Trans. Dielectr. Electr. Insul., vol. 24, no. 4, pp. 2477–2482, 2017.	• Mineral oil have some characteristics that unfavourable properties which are low fire point, toxicity and poor biodegradable. • Mineral oil also have been used all over the world due to cast benefit effectively, its adequate ageing behaviour, good dielectric strength, low viscosity, less susceptible to oxidation and able to operate in low temperature. • Ester oil for distribution and traction are increasing to support the green technology which is on its good biodegradable. • Natural ester liquids are seen as an alternative to mineral oil because it non-toxic, biodegradable, lower fire risk, higher flash point and fire point compared with mineral oil.

4	I.Fofana, N'cho, A.Beroual. ' Effect of Exposure to Electrical Discharge on Transformer Oil Properties" G. Jishu, H. V. Engineering, and J. Sylvestre	• Electrical discharge is the release and transmission of electricity in an electric field applied to medium such as gas and liquid • Electrical stress consist of heat and moisture in the presence of oxygen, oxidise the oil producing free radicals, acid and sludge that are deleterious to the transformer

2.10 Summary

This chapter have been discussed about type of insulation in transformer, types and properties of insulation oil, and electrical discharges of insulation liquids. This research work focusing on electrical discharges on PFAE and Midel eN. Natural Ester Insulating(NEI oil for distribution and traction are increasing to support the green technology which is on its good biodegradable. Natural ester liquids also are seen as an alternative to mineral oil because it non-toxic, biodegradable, lower fire risk, higher flash point and fire point compared with mineral oil. After through the electrical discharges test, the properties of insulation oil in terms of moisture, acidity and BDV will be investigate. Next, chapter 3 will show the methodology used in this research work.

CHAPTER 3

METHODOLOGY

3.0 Introduction

This chapter discussed the flow of this project to show the overall methodology this research work and give the detail of the process of this project which is illustrated in Figure 3.0. In this chapter also provide experiment that will be used and descriptive techniques used for the project implementation.

3.1 Flowchart of Project Implementation

The flow of project implementation shows in Figure 3.1. There are several processes will be discussed in details which is starting from a selection of natural ester oil, treatment process, PDIV test and finally with electrical discharge test. The procedures used for moisture test, acidity test, PDIV and Electrical discharge study for this research work are referred to related previous researcher and follow ASTM D974, D6871, D1816 and D1533 standards.

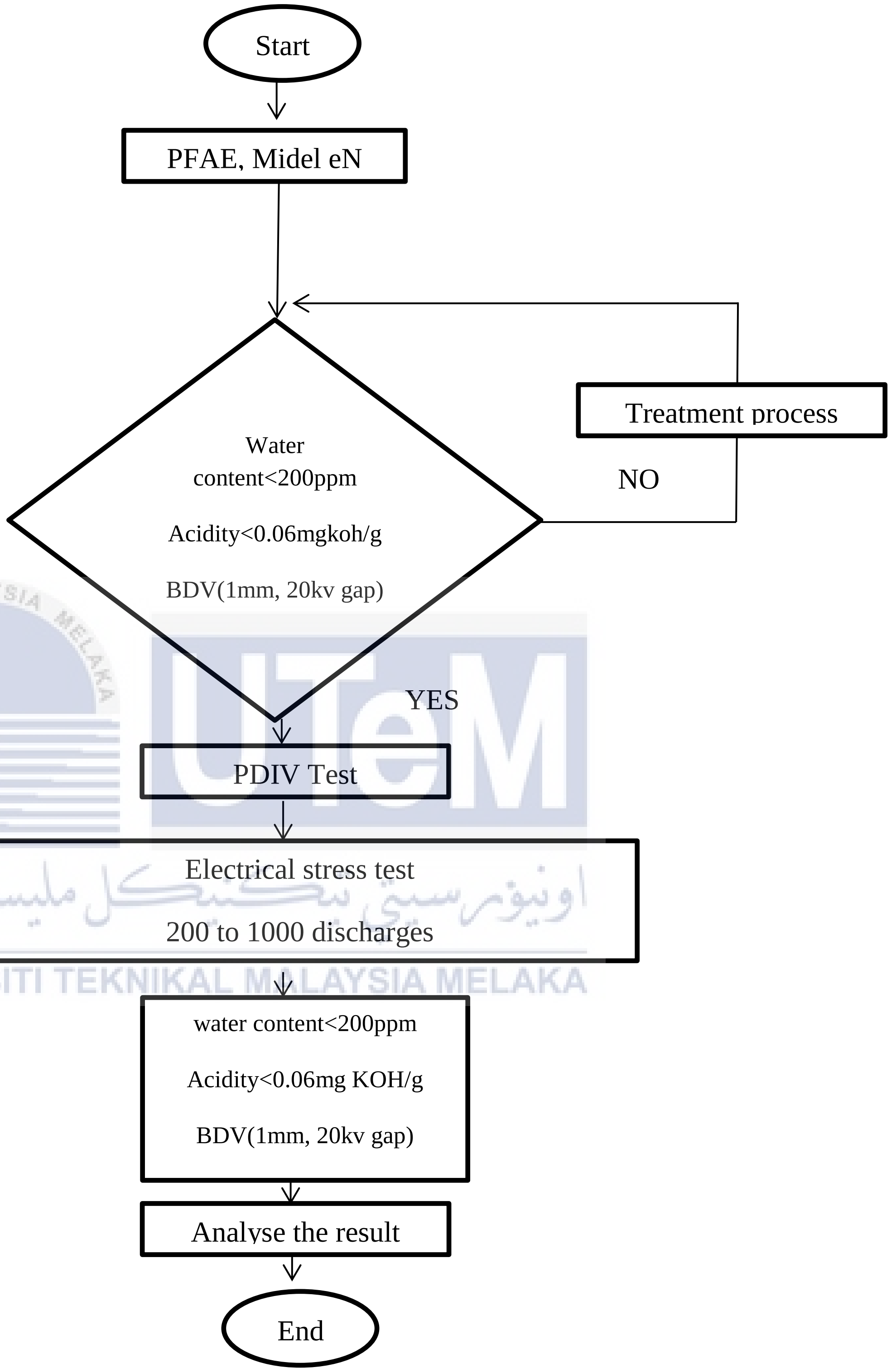


Figure 3.1: Flowchart of project implementation for PFAE, and Midel eN oil.

3.2 Selection of Natural Ester Oil

Palm Fatty Acid (PFAE) and Midel eN ester are the natural ester that selected for this research work. Natural ester oil using vegetable oil which is readily biodegradable that give less impact on environment in case of a spillage and leakage.. The natural ester widely used in transformer according to the characteristic above even though it has poor cooling properties than mineral oil. Besides that, the palm oil has stabilized chemical combinations. PFAE also has a few characteristic that give an advantage as insulation oil which is high flash point, high cooling ability, good oxidation stability, providing a better match of oil-paper insulation system due to its high relative permittivity and enhances the insulation system due to its high water solubility.

Although, Midel eN also offers superior moisture tolerance and has the potential to increase the lifetime of cellulose based solid insulation which in turn can extend the transformer life. It able to absorbs large amount of moisture with no reduction of breakdown voltage (up to 300ppm) and also has high saturation limit making precipitation of free water virtually impossible. In the other hand, Midel eN also has slower rate of cellulose ageing than mineral oil and able to longer the cellulose lifetime at standard temperature [31]. Hence, this two type of oil which is from palm oil bases and rapeseed based will be investigate in this research work. Based on figure 3.2, blue barrel is contain Midel eN while green barrel is containing PFAE oil.

Figure 3.2 : Midel eN and PFAE oil

There are 5 sample of each oil need to be prepared for this study. 400ml each of sample palm fatty acid ester (PFAE) and Midel eN. Each oil sample will be test for the moisture content, acidity and breakdown voltage before and after electrical discharges applied. Next, all of the test sample will be test for electrical discharge with different number of discharges. Hence, the acidity level, moisture content and breakdown voltage will be measured again to show the effect of the electrical discharge the oil sample.

Figure 3.3 : PFAE oil sample

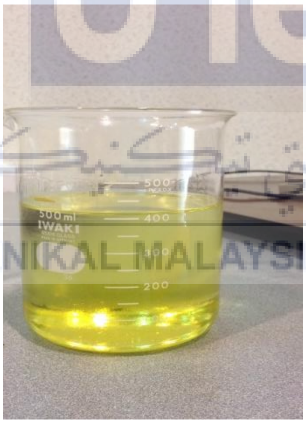


Figure 3.4: Midel eN oil sample

3.3 Pre-processing of the PFAE and Midel eN oil

There are a few tests that PFAE and Midel eN need to consider to make sure it follow with requirement of D6871 and IEC 61294 standard . The test includes water content test, acidity test and breakdown voltage test before all the samples go to next step which is electrical discharge test.

3.3.1 Water Content Test

All of sample oil will be tested by using Coulometric Karl Fisher Titration as shown in figure 3.5 to measure the moisture contain in the insulating oil. This test must refer and follow the ASTM D1533 standards which method for water in natural ester insulating liquids. This test method is based on the reduction of iodine containing reagent. An automatic titration with commercially available reagents are needed in the Coulometric Karl Fischer test method. The apparatus, reagents, preparation of apparatus and procedure for this test are all according to ASTM D1533 standards. According to ASTM D6871 standards, the water content for natural ester fluid must < 200ppm. A low water content of natural ester insulating liquid is compulsory to attain adequate electrical strength and low dielectric loss characteristic, to increase the insulation system life and to reduce metal corrosion.

Figure 3.5: Coulometric Karl Fischer Titrator

The procedures for water content are follows:

Step 1 Firstly, filled 6ml of the PFAE oil volume into syringe tube.

Figure 3.6: Syringe tube fill with natural ester oil.

Step 2 Then, removed the air bubble inside the syringe tube.

Step 3 Next, by using digital analytical balance, record the mass of 6ml NEI.

Figure 3.7: Digital analytical balance to measure weight the oil.

Step 4 Filled 1 ml volume each of the NE1 oils into Coulometric Karl Fisher Titration for one test. To start the titration, press the [START] button on screen of Coulometric Karl Fisher Titration.

Step 5 Repeat this process for next sample oils.

According to ASTM D6871 standards, the water content for natural ester fluid must < 200ppm. Otherwise, the sample need to through treatment process to reduce the water content value.

Figure 3.8: Filled NE1 sample into Coulometric Karl Fisher Titration tube.

Figure 3.9: Results displayed the moisture content of the oil sample.

3.3.2 Treatment Process

The treatment process is to lower the level of moisture content in oil. In this process, nitrogen gas is selected for treatment process. 500ml of Midel eN oil will put in a conical flask. Next, the nitrogen will on channel in the oil sample for 30 minutes. A pump will be used to pass out the oxygen through the flask tube in order to remove the moisture content. Finally, after finish the treatment process, the sample need to through water content test to achieve the standard. Finally, put the oil sample in the amber glass bottle with label and sealed until it used. The glass bottle needs to fills with nitrogen gas and seal tightly to prevent any oxygen trap inside the bottle. This treatment process has shown in figure 3.8 and table 3.1 shows material for Treatment process.

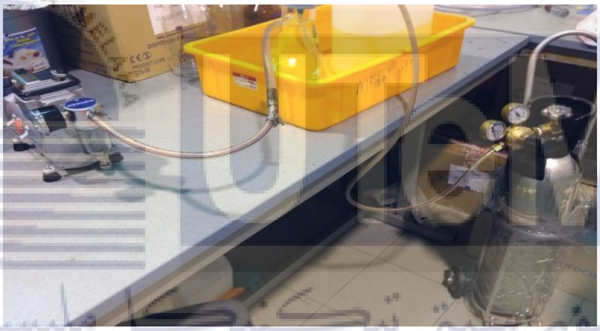


Figure 3.10: Treatment process

Table 3.1: Material for Treatment process

Sample Oil	500 ml
Gas	Nitrogen gas
Equipment	Vacuum pump
Time	30 Minutes

3.3.2 Acidity Test

The acidity content must be measured using 848 Titrino Plus before the insulating liquid do electrical discharge test. There are several procedure and calibrate that must be consider during use the 848 Titrino Plus which are procedure of conducting TAN test, Loading a method, carrying out a determination, preparing burette unit (PREP) and calibration of electrode. Hence, to reduce electrical conduction and metal corrosion and to increase the life of the insulation system, the natural ester insulating oil need to make sure it content low total acid number. Usually, the natural ester insulating liquid contains higher neutralization number than the mineral insulating oil. The natural ester insulating oil also incline to form long-chain fatty acid while the mineral insulating oil incline to form shorter chain organic acid. The acidity test was referred to ASTM D974 standard with total acid number must below 0.06 mg KOH/g.

The procedures are follows:

Figure 3.11 848 Titrino Plus

Procedure for conduct Tan Test:

Step 1 Firstly, set up the apparatus (electrode, tubing, stirrer)

Step 2 Next, rinse and fill the burette with KOH IN IPA (0.1 mol/L).

Step 3 Standardize the KOH in IPA 0.1 mol/L.

Figure 3.12: Calibration process using buffer solution

Step 4 After that, calibrate the electrode with buffer solution and slope must around 97%-103%.

Step 5 Next, for standardization put the Potassium Hydrogen Phthalate (KHP) with weight around of 0.1g into beaker, add approximately 80mL of DI water, titrate with KOH in IPA 0.1 mol/L.

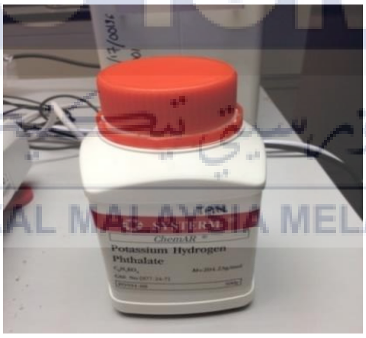


Figure 3.13: Potassium Hydrogen Phthalate (KHP)

Figure 3.14: The result of standardise in KOH

Step 6 Duplicate the blank titration and the bank value will be auto save at common variable.

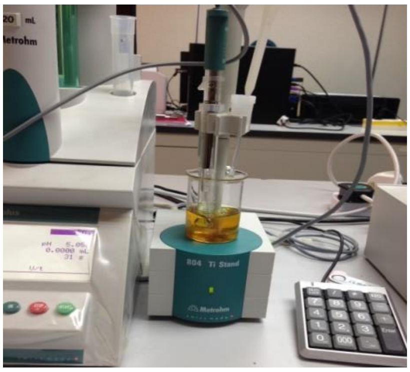


Figure 3.15: Oil sample Titration

Step 7 For sample titration, put the 5g of sample into the titration vessel and add 20ml IPA solvent. Next, titrate the sample with KOH in IPA until pH11.5 and TAN result will show at screen.

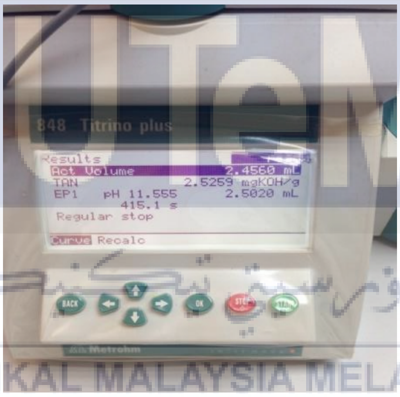


Figure 3.16: The acidity result of oil sample

Step 8 Lastly, rinse the electrode with titration solvent IPA then followed by the DI water. Keep the electrode moist with the electrolyte.

3.3.3 Breakdown voltage test

The breakdown voltage for this research work complies with ASTM D1816 standards by using method for determination of dielectric breakdown voltage of insulating liquids by verband duetscher elektrotechniker (VDE) electrodes using megger OTS60PB. Besides that, the water content of the insulating liquids also need less than 200ppm by follow D1533 standards. According to the ASTM D6871, the breakdown voltage of the natural ester oil for 1mm gap must be at least 20kV. The oil sample of PFAE and Midel eN oil will be test for breakdown voltage to analyse the effect before and after electrical discharge applied.

The procedures are follows:

Step 1	Firstly, prepared the 500ml NEI into oil test vessel and ensure there is no formation of bubble in the oil, sample, check the temperature and the room humidity.

Step 2	Next, set the electrode shape mushroom/semisphere (36mm diameter) with gap 1mm according to ASTM D6871.

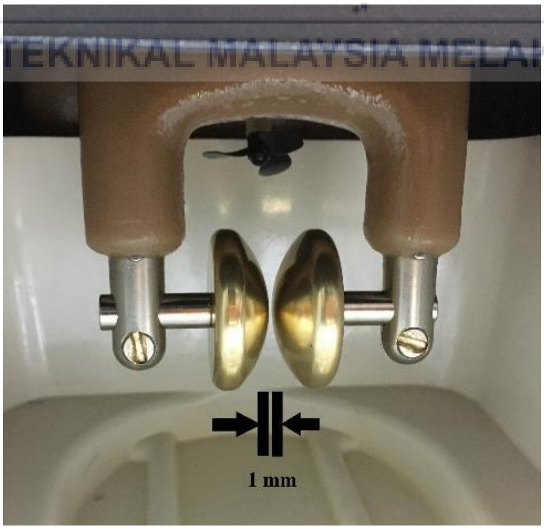


Figure 3.17 : Electrode shape

Figure 3.18: Measure the initial oil temperature.

Step 3 After that, measure the oil temperature, room temperature and humidity before put the oil sample into Megger OTS60PB. Make sure the sample is at room temperature which is 20°C to 30°C as in Figure 3.18

Step 4 Next, turn on the Megger OS60PB, and select the standard ASTM D1816.

Figure 3.19: Oil sample inside the test chamber.

Figure 3.20: Selected type of standard of test.

Step 5 Finally, the breakdown voltage test will be run between 15-17 minutes for 5 test and show the result. The process in step 3 and 4 is repeated to get 50 data of BDV test for each sample for the purpose of large sample of date that give highest accuracy.

Figure 3.21: The monitor to shows 5 test result.

Table 3.2 : Parameters Breakdown voltage test using 0TS60PB

PARAMETER	SPECIFICATIONS
STANDARD	ASTM D1816
Type of electrode shape	A
Initial stand	3 min.
Rate of voltage rise	0.5kV/s
Intermediate Stir Time	Continous
Intermediate Stand Time	1 min
Maximum Duration test	17 min.
Number of test	5

3.4 PDIV Test

Partial Discharge Inception Voltage need tests to both of the oil samples to determine the initial value of the discharges occur in the insulating oil. PDIV test is one of the methods that recently develop to describe the behaviour of insulating liquids when subjected to high electrical stress. PDIV test also is applicable to all types of insulating liquids used in medium and high voltage equipment. Nevertheless, the equipment described in this procedure may not be adapted to liquids with values of PDIV more than 70kV.

3.4.1 Apparatus for PDIV test

There are list of apparatus that need to follow according to 61294 standard:

Step 1 Firstly, the test cell shall consist of a vessel containing a vertical gap and the volume of liquid in this cell shall be of the order of 300ml.

Step 2 Secondly, the insulating materials used in the cell shall be high dielectric strength, compatible with the insulating liquids to be tested and resistant to solvents and cleaning agents that usually used for these liquid.

Step 3 Third, metal parts shall be restricted to the electrode and their supports. The test cell also shall be discharge free at 70kV.

Step 4 Lastly, use suitable needle of 50 mm length and 1 mm diameter from Ogura Jewell Industry Co. Ltd. Figure 3.20 and 3.321 show the needle and test cell that been used in this research work.

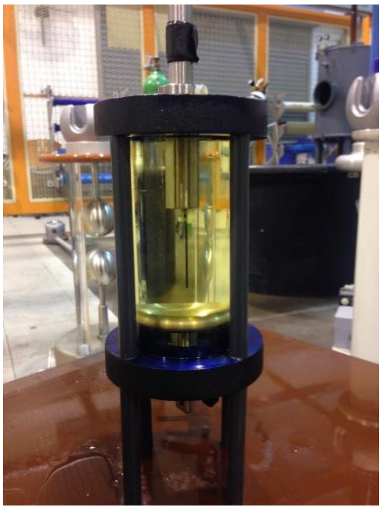


Figure 3.22: test cell for electrical discharges test

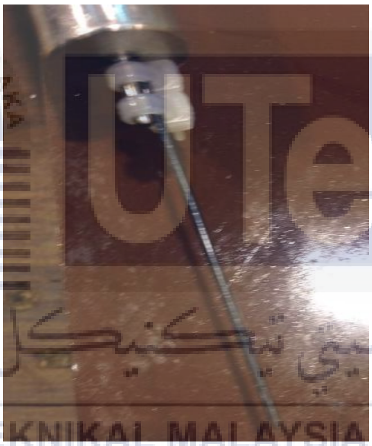


Figure 3.23: Needle from Ogura Jewell Industry Co. Ltd.

3.4.2 Experiment procedure for PDIV test.

There are list of procedure that need to follow according to 61294 standard before PDIV test:

Step 1 Firstly, the PD test will be test by applied voltage without test cell to ensure there is no corona discharges or partial discharge occur from AC supply to rod before connect to test cell.

Step 2 Secondly, the PD test will be run by applied maximum required voltage for 5 minutes but without needle and insulating oil to ensure there is no corona discharges occur around the test cell. During this period, there is no discharges exceeding 50pC are to be registered. It is permissible to use electrical and acoustic technique to discriminate between discharges in the sample and background interference.

Step 3 Before start the PD test, the connection and the measurement device need to calibrate in terms of apparent charge magnitude with an appropriate calibration device as shown in figure 3.24.

Figure 3.24: Calibration of measurement device

Step 4 After, the PD test in step 1 and 2 had been done and the equipment had been prove that free from partial discharges the PDIV test for Midel eN and PFAE oil can be proceed.

Next, the PDIV test for the selection insulating oil which is Midel eN and PFAE :

Step 1 Firstly, fill the clean test cell by gently pouring the insulating oil into the test cell and allow the liquid to stand for 15 min before the first voltage application.

Step 2 Next, increase the voltage from zero at a rate of 1 kV/s until a partial discharge inception voltage (PDIV) occurs of apparent charge equal to or greater than 100 pC. Record the voltage and rapidly decrease the voltage down to zero. The PDIV display can refer in figure 3.25.

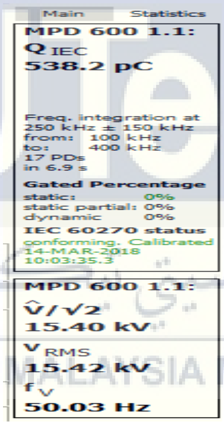


Figure 3.25 : PDIV value of Midel eN

Step 3 After that, make repeated measurements on the same filling of the cell to record at least 10 values get the accurate value of PDIV of Midel eN and PFAE. Allow an interval of 1 min between each voltage application. Only the voltage at which the apparent charge of the discharges is equal to or larger than 100 pC is recorded. At least 10 PDIV measurements are made on each of two different fillings of the cell. All the test are using MPD 600 in figure 3.26 to detect the partial discharges and refer to figure 3.27block diagram for PD measurement system.

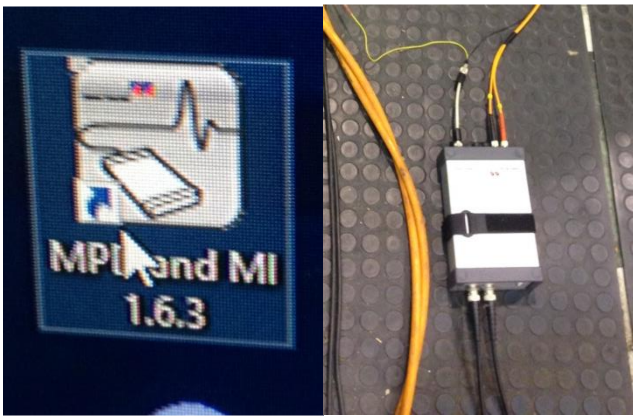


Figure 3.26 : MPD 600 software and detector

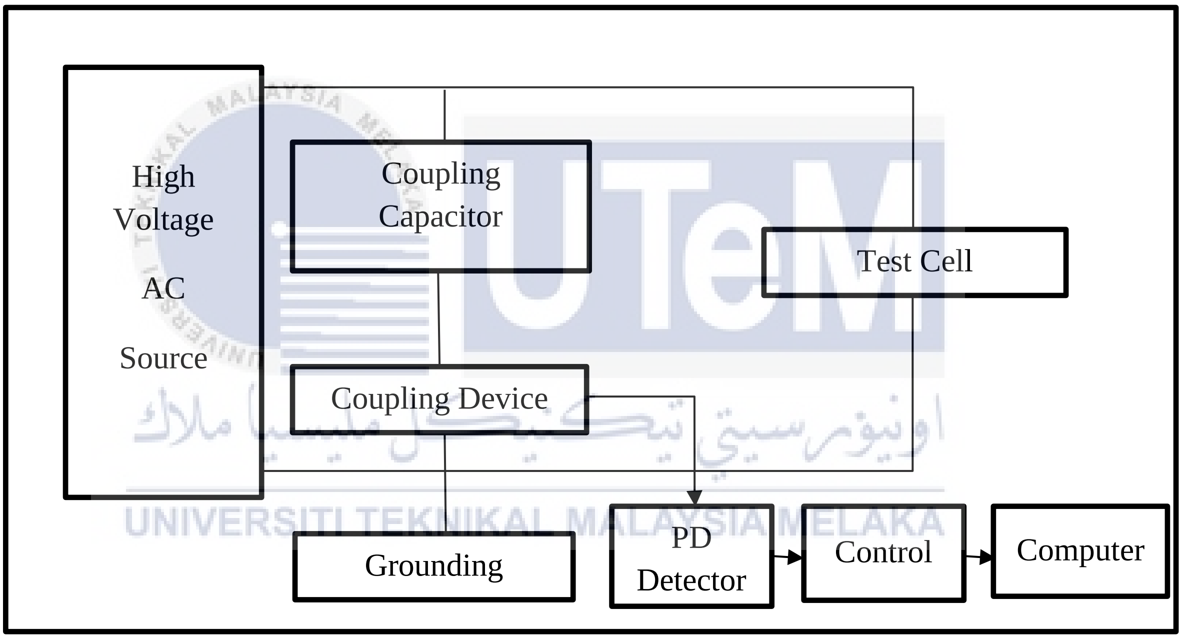


Figure 3.27: Block diagram for PD measurement system

3.5 Electrical Discharge Test

Electrical discharge stability test are to determine the effect that occurs when electrical discharge have applied to the oil sample Midel eN and PFAE. Electrical discharge is the emission and transmission of electricity in an electric field applied to the medium such liquid . This study will determine the effect of the electrical discharge to the moisture content, acidity level and breakdown voltage.

There are list of procedure that need to follow according to 61294 standard for electrical discharges:

Step 1 The oil samples will be placed in 400ml test cell with containing a point to plane electrodes arrangement made of steel. Hence, the 10mm electrode gap will be set with 10µm radius of the point electrodes.

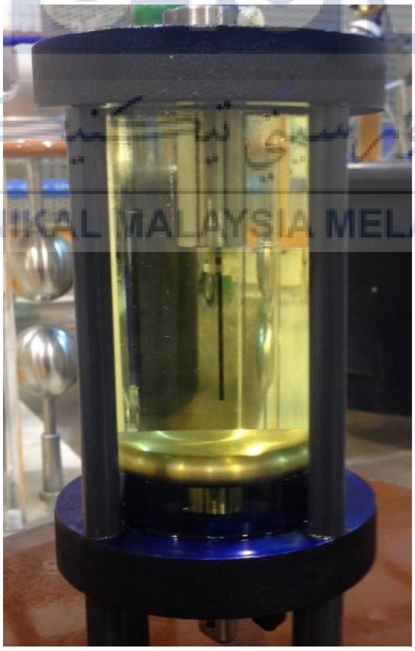


Figure 3.28: 400ml test cell with containing a point to plane electrodes with 10mm electrode gap

Step2 Next, 200 up to 1000 discharges at 50hz sine –wave with constant voltage based on the PDIV value of Midel En and PFAE are applied to the same amount of oil sample using Operating Terminal OT276 as shown in figure 3.29. All the electrical discharges equipment setup can refer to figure 3.31 and its same with partial discharges equipment setup.

Figure 3.29: Operating Terminal OT276

Step 3 The electrical and physicochemical properties will be measured after series discharges (around discharges measured using monitor display). The electrical discharges test are used the same equipment and setting of the PDIV test. Figure 3.32 also show the oil sample were place in the amber glass before the electrical and physicochemical properties will be measured,

Table 3.3: Table for parameters of test specification

PARAMETER	**SPECIFICATIONS**
Electrode Shape	Point to plane electrodes
Rate of Test Voltage of Midel eN(kV)	15.42
Rate of Test Voltage of PFAE(kV)	17.87
Number of Tests	200-1000 discharges

Figure 3.30: Digital Measurement DM 1551

Figure 3.31 : Electrical discharges setup

Figure 3.32 : Test sample place in amber glass

3.6 Analysis of Results

Based on the result of the experiment, the value of data collection of electrical and physiochemical will be analysed and compared with the initial results to show the effect of electrical discharges to the oil samples. On top of that, there will be analysis and discussion according to the PDIV, acidity, moisture content and BDV result of the oil sample. The properties of Midel eN and PFAE after electrical discharges process also will be showed to prove whether there is any improvement or decrement of electrical discharge performance of Midel eN and PFAE oil.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview

This chapter shows the result and analysis of research work that have been done in Chapter 3 in order to fulfill the objectives of this study. The experiment that successfully been done are Breakdown voltage (BDV), Partial Discharges Inception Voltage (PDIV), electrical discharges test, water content, acidity and BDV.

4.2 Results of the Fresh Midel eN and PFAE

Water content and BDV test on fresh transformer oil (Midel eN) and PFAE have been carried out to determine its properties. The permissible water content for natural ester insulation oils is ≤ 200ppm as according to ASTM D6871 using Coulometric Karl Fisher Titration complies with ASTM D1533 standard while BDV test was conducted using Megger OTS60PB with 1 mm gap following the ASTM D1816 standard. Figure 4.1 shows the test results on fresh Midel eN oil. By referring table 4.2 and 4.3 it’s shows that the water content of the fresh Midel eN and PFAE oil fulfils the requirements sets by the ASTM D6871 where the permissible water content in NEI oil is ≤ 200ppm.

4.2.1 Moisture content Fresh Midel eN and PFAE

Firstly, the experiment start by using Coulometric Karl Fisher Titration to achieve water content standard. According to ASTM D1533 standard, the result should <200 ppm and the insulating oil need to through treatment process if not fulfill the standard . The result moisture content for fresh Midel eN and PFAE before and after treatment process with hydrogen are recorded in table 4.1, 4.2 and table 4.3.

Table 4.1 : Moisture of fresh Midel eN before treatment process with hydrogen

Type Of Samples	No. of test sample	Sample Size (g)	Moisture content (ppm)	Average (ppm)
Fresh Midel En	Test 1	0.9337	222.0	218.2
	Test 2	0.9254	217.9	
	Test 3	0.9111	217.6	
	Test 4	0.9866	220.8	
	Test 5	0.9525	212.7	

Table 4.2 : Moisture of Fresh Midel eN after treatment process with hydrogen

Type Of Samples	No. of test sample	Sample Size (g)	Moisture content (ppm)	Average (ppm)
Fresh Midel En	Test 1	0.9987	186.8	184.56
	Test 2	0.9960	168.0	
	Test 3	0.9971	180.8	
	Test 4	0.8975	189.6	
	Test 5	1.0820	197.6	

Table 4.3 : Moisture of Fresh PFAE

Type Of Samples	No. of test sample	Sample Size (g)	Moisture content (ppm)	Average (ppm)
PFAE	Test 1	0.9273	142.5	147.9
	Test 2	0.9268	151.7	
	Test 3	0.9466	151.2	
	Test 4	0.9098	143.0	
	Test 5	0.9233	151.1	

From table 4.1, the initial moisture content of Midel En is 218.2ppm which is more than 200 ppm and not follow the standard ASTM D1533. Thus, the moisture content needs to be reduces via treatment process with hydrogen. The Fresh PFAE oil on the other hand, no need to through the treatment process since the moisture content is below 200ppm. PFAE need to be sealed properly and placed it dark because it has capability to absorb much more moisture content.

4.2.2 Breakdown voltage (BDV) Fresh Midel eN and PFAE

In this experiment, the breakdown voltage test (BDV) is conducted by using Megger OTS60PB with follow the ASTM D1618 standard. The spherical electrodes with the diameter of 36mm and the electrode gap spacing 1mm were used in this experiment. Hence, the applied voltage was increased continuously at the rate of 0.5kv/s until breakdown occurs. The result of BDV test is plotting using Weibull probability plot as shown in figure 4.4 and 4.5 below. A total of 30 breakdown voltages were recorded in BDV template in Appendix A for each sample and the data were analyzed based on Weibull probability plot.

.

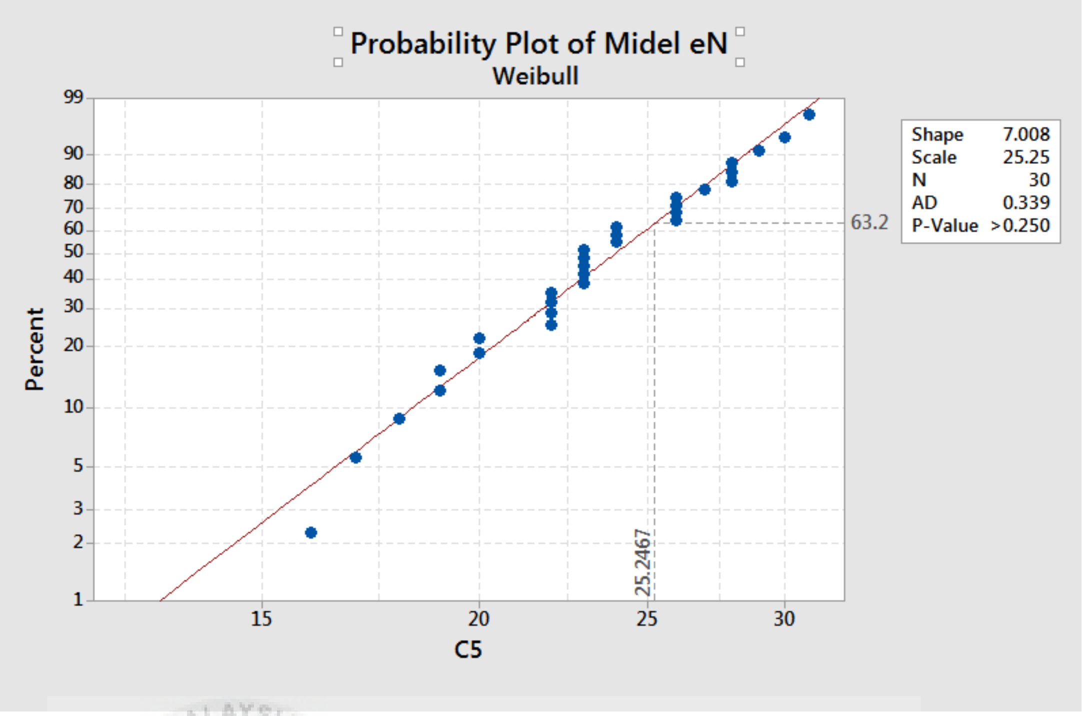


Figure 4.0: Result of BDV using weibul probability for Fresh Midel eN oil

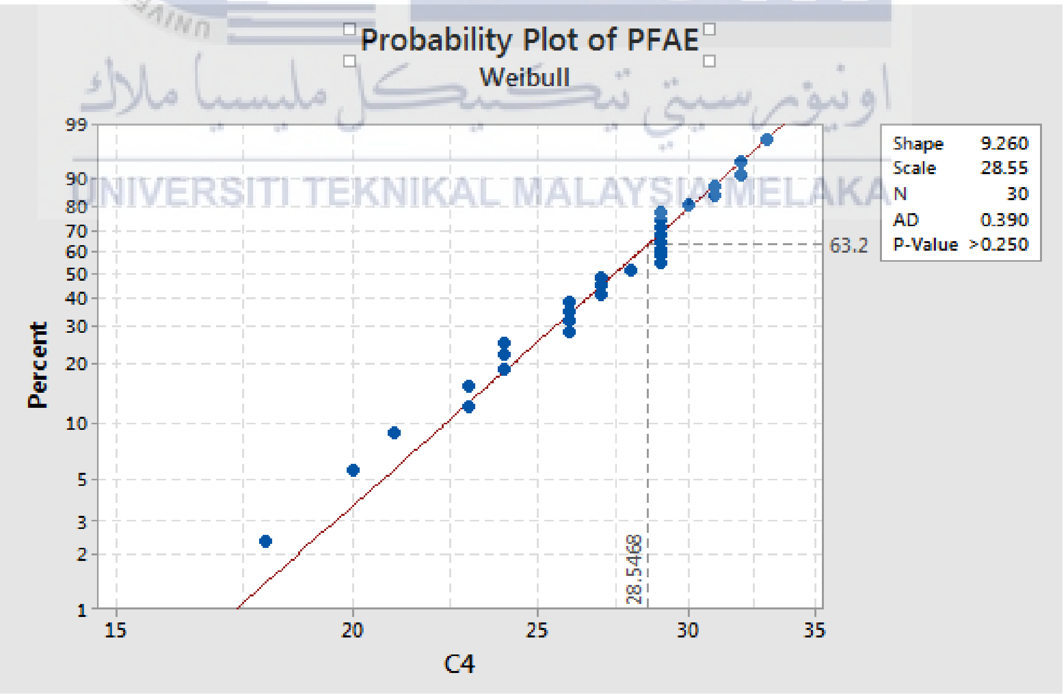


Figure 4.1 : Result of BDV using weibul probability for Fresh PFAE oil

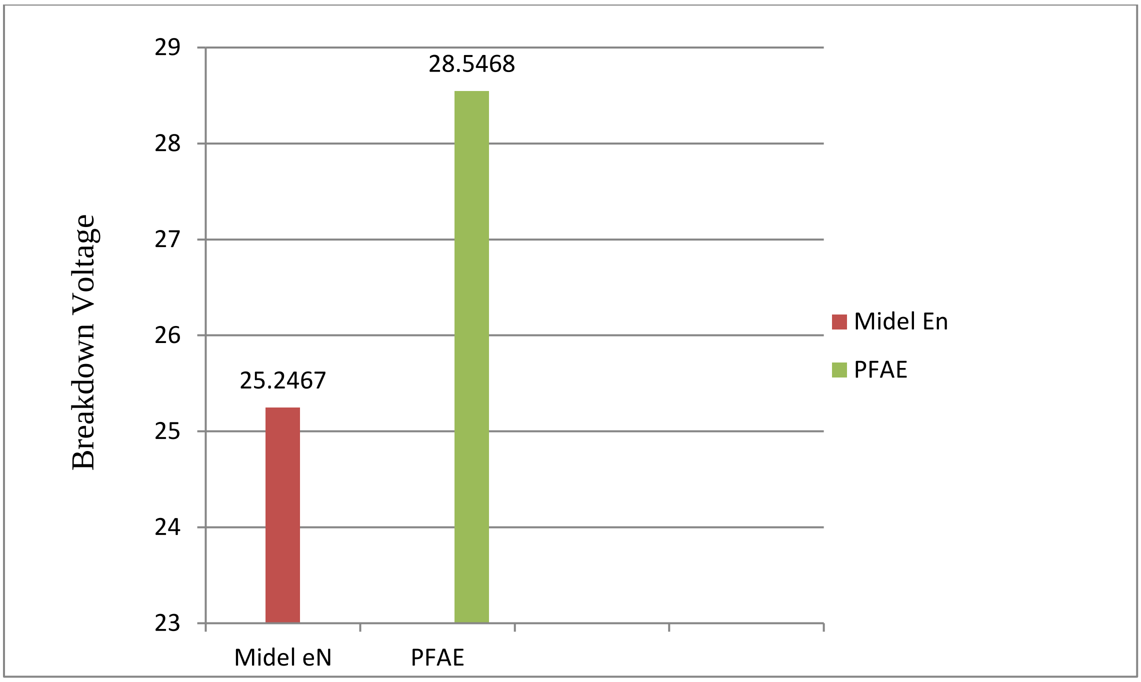


Figure 4.2: Result of BDV Fresh Midel En and PFAE oil

The purpose of using Weibull probability plot is to measure the best value of its data. It has been used widely in high voltage experiment and measured breakdown voltage. On of that, the probability value of the data falls at 63.2% where it is the point corresponds to the time of failure of the oil under test.

Based on the result show above, the BDV value for PFAE oil is higher than Midel En. The average BDV value by using Weibull probability plot for PFAE is 28.5468kv while Midel eN 25.2467kv after intersect the 63.2% unreliability line. This result is due to Midel eN that has highest of moisture compared to PFAE oil. Both Midel eN and PFAE test by using Megger OTS69PB with follow the requirement and their standard for BDV which are ≥ 20kV with using 1mm of gap spacing of electrode.

4.3 PDIV Fresh Midel eN and PFAE

PDIV is a test to recognise the voltage that initial electrical discharges occur. PDIV also an alternatively important indicator that can be used to represent the integrity of the insulation. The PDIV test procedure is followed the IEC 61294 standard where pd occur only recorded at threshold 100pc and above. For PDIV test, type of electrode used in this experiment is point to plane with 10mm gap. The PDIV result for Mide eN and PFAE oil are shown in figure 4.3 and 4.4 below.

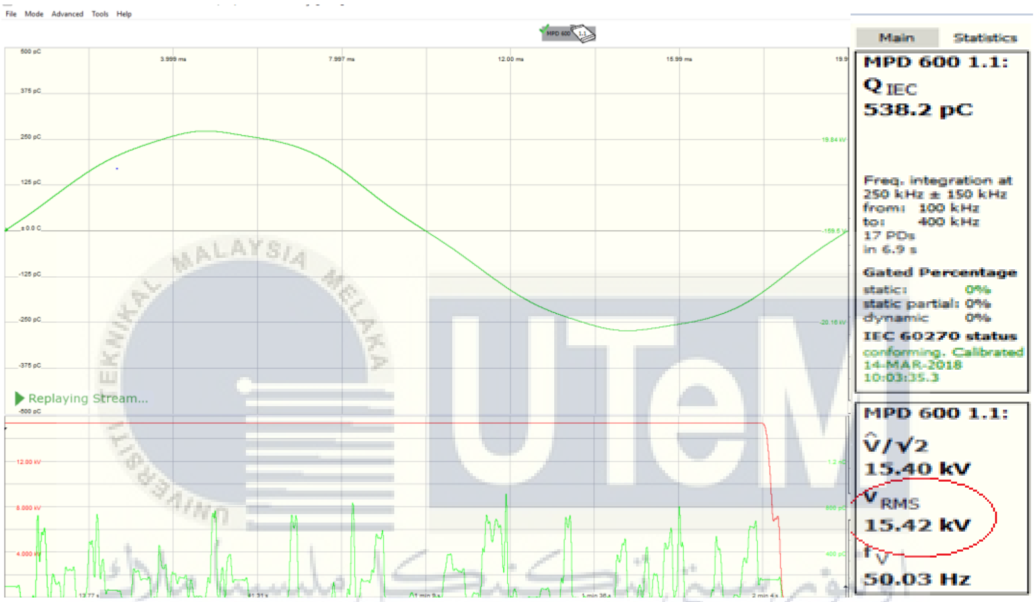


Figure 4.3 : PDIV of Fresh Midel eN oil

Figure 4.4 : PDIV of PFAE oil

Based on the result PDIV of Midel En and PFAE show that the PDIV of PFAE is higher than Midel En. It also expected that BDV of PFAE is higher and it one of the reason why its PDIV occur at higher value than Midel En. PFAE has low vicosity compare to Midel En with viscosity of PFAE is 8cST while Midel eN is 35cST. PFAE also has through many esterification processes that reacts with alcohol in order to make it high performance.

4.4 Result of electrical discharge test for insulating oil Midel En.

Figure 4.5: 200 electrical discharges of Fresh Midel eN oil

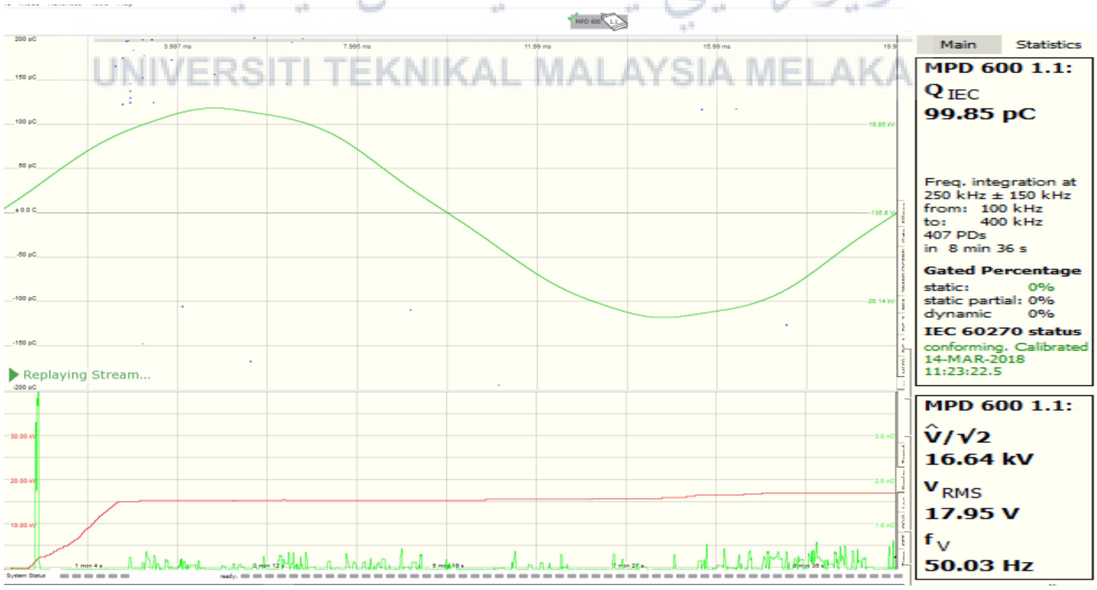


Figure 4.6 : 400 electrical discharges of Fresh Midel eN oil

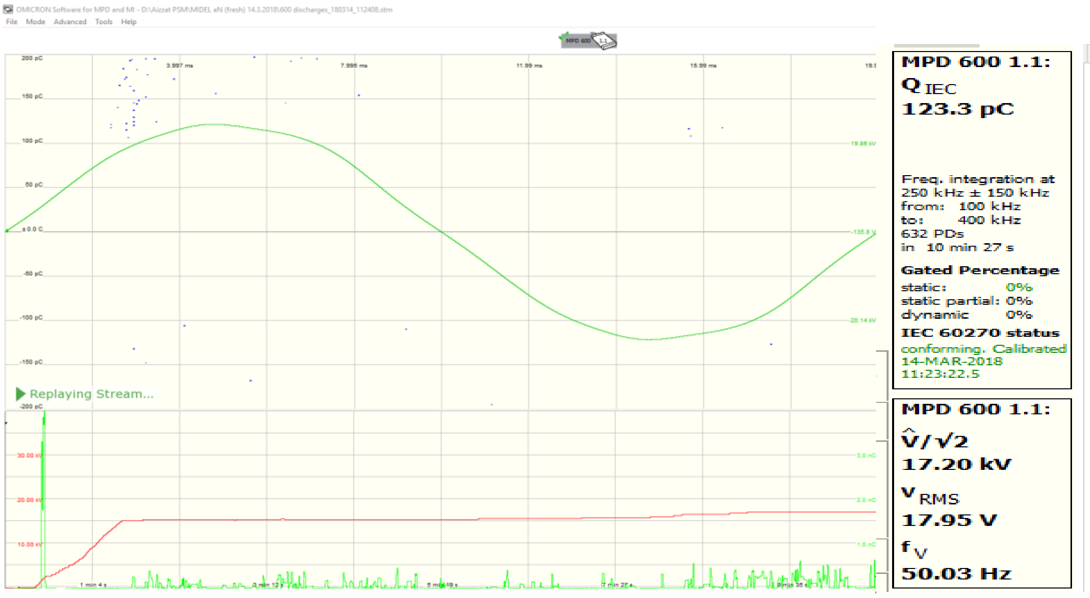


Figure 4.7 : 600 electrical discharges of Fresh Midel eN oil

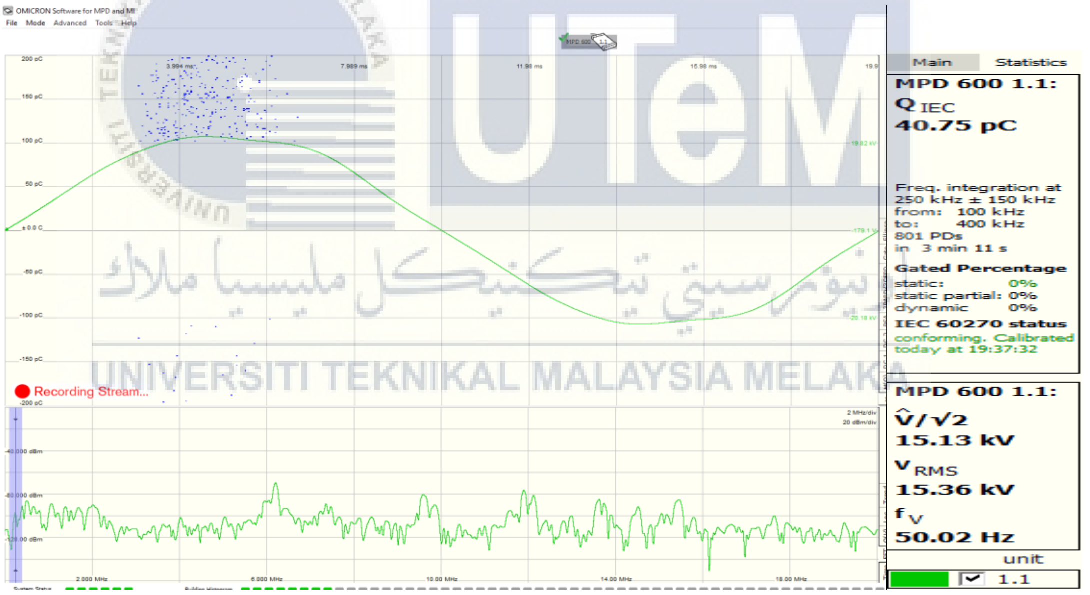


Figure 4.8: 800 electrical discharges of Fresh Midel eN oil

Figure 4.9: 1000 electrical discharges of Fresh Midel eN oil

4.5 Result of electrical discharge test for insulating oil PFAE.

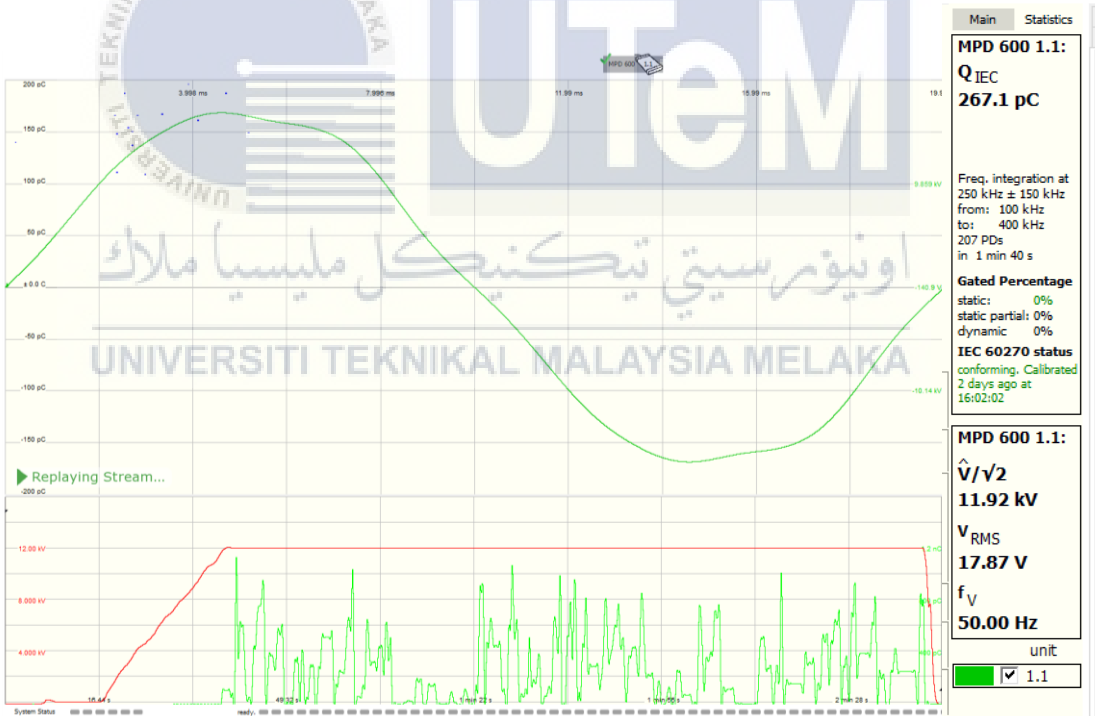


Figure 4.10: 200 electrical discharges of Fresh PFAE oil

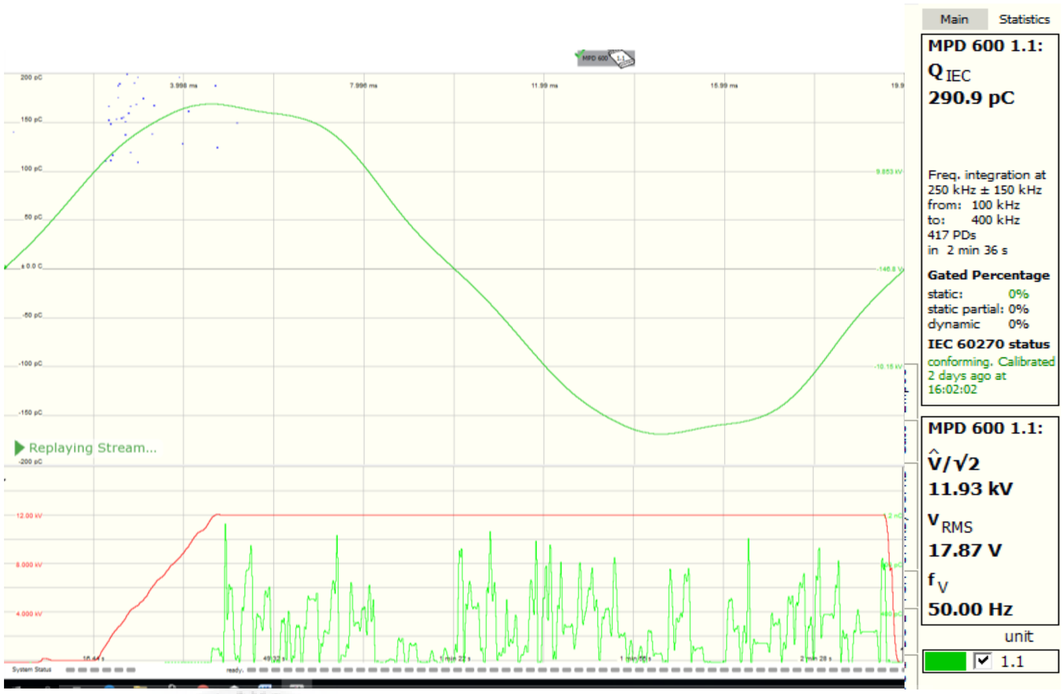


Figure 4.11: 400 electrical discharges of Fresh PFAE oil

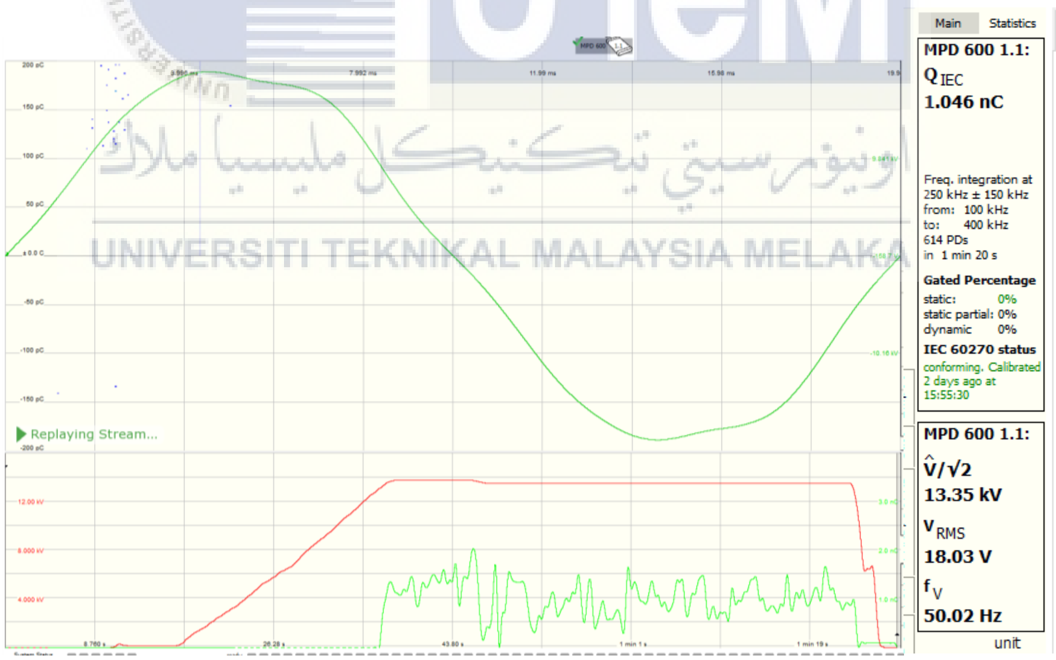


Figure 4.12: 600 electrical discharges of Fresh PFAE oil

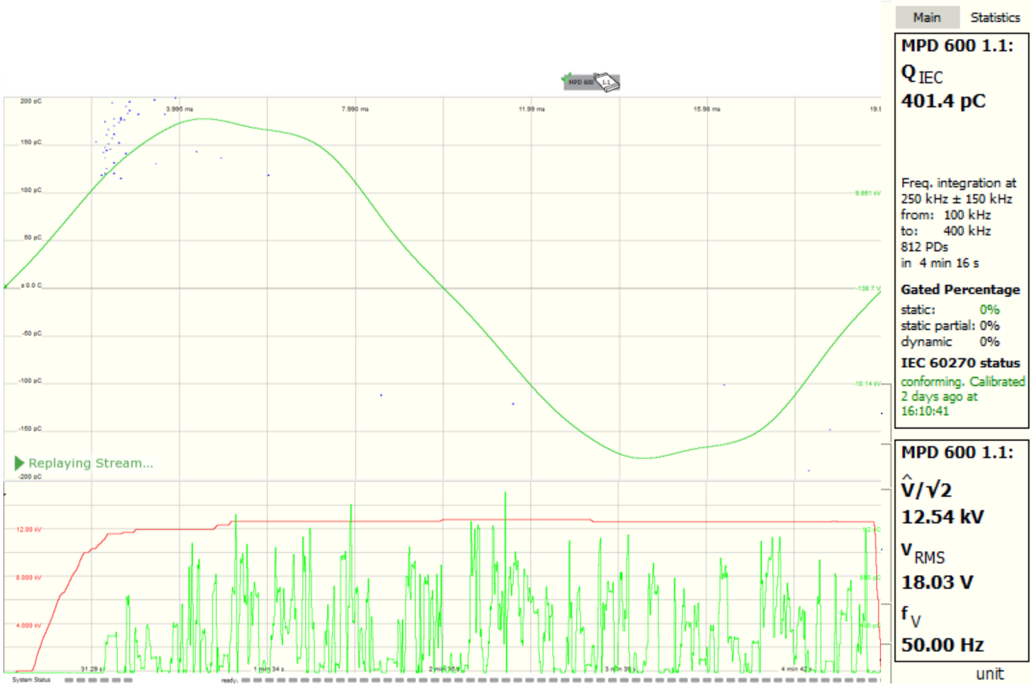


Figure 4.13: 800 electrical discharges of Fresh PFAE oil

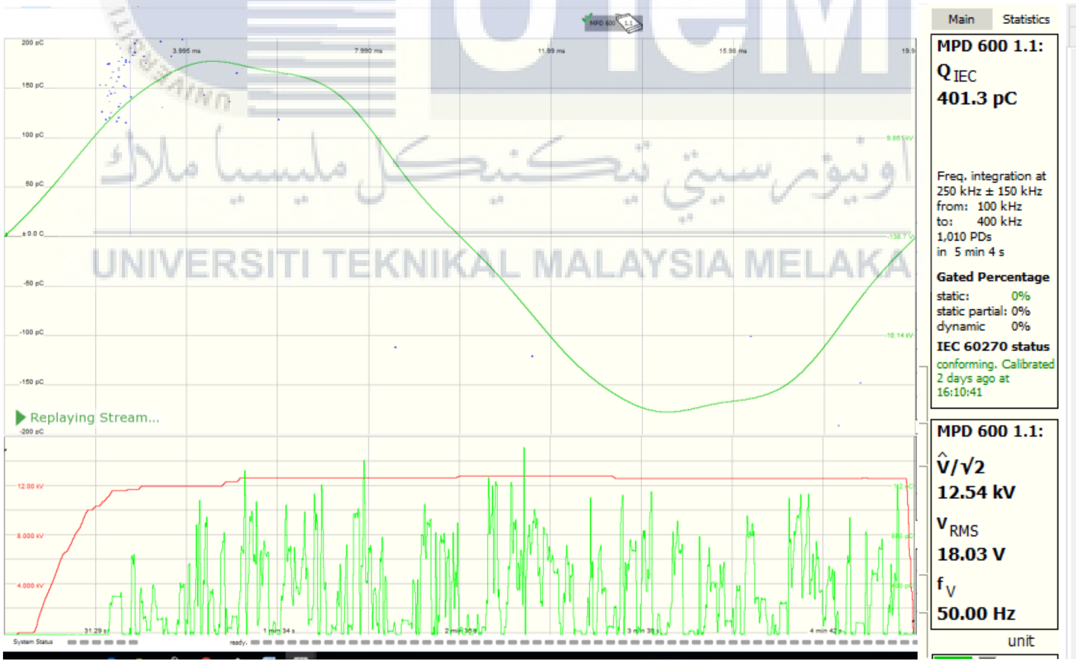


Figure 4.14: 1000 electrical discharges of Fresh PFAE oil

4.6 Result of moisture of Midel En and PFAE oil after electrical discharge.

Figure 4.15: Moisture of fresh Midel eN and PFAE oil

Based on figure 4.15, it shows that the result of moisture content of all samples after through electrical discharges test from 200 until 1000 discharges. The graph result of water content present a fluctuated variation and the most significant changes of moisture content is Midel eN where the moisture before electrical discharge is 150.2ppm but it continues decrease until 73.74ppm after finished the all the electrical discharges test .There is a clear tendency with the application of electrical discharges. Besides, the reduction in water content also with the application of discharges is mainly due to evaporation of water traces that are present in the zone of discharges occurrence. Moreover, Midel eN contain unsaturated fatty acid which is very easy influence to the dielectric and physicochemical properties. The molecules in unsaturated esters is not fully saturated with hydrogen atoms. The structures also are relatively weak and in the presence of water and heat, it show that the Midel eN has poor hydrolytic stability. On top of that, when there is increase of electrical discharges applied to the oil sample, it will allow more free radical to cut the molecules in the oil. Hence, this process known as autocatalytic reaction. Midel eN also has superior moisture tolerance that can absorbs large amounts of moisture with no reduction of breakdown up to 300ppm. Besides that, high saturation limit (1100ppm@20 ˚C) making precipitation of free water virtually impossible.

However, the moisture content of PFAE not affected by the electrical discharges and it shows that the value of moisture content is between 141.56 until 147.54ppm. By referring the result of moisture content of PFAE there are small increasing value and it maybe occur with increasing time of during electrical discharges test from 200 to 1000 discharges. PFAE contain saturated fatty acid hydrocarbon chain that contain a lot of hydrogen with the oxidation stability moisture and its not easy to give a chance hydrocel group to absorb water. The chemical modification conducted on PFAE has resulted to the close dielectric properties of PFAE to mineral oil with some advantageous derived from natural esters. The fatty acid alkyl esters which known as PFAE that produced by LION Corporation of Japan also had been prove exhibit dielectric breakdown voltage of about 80kV/2.5mm.

PFAE oil also have low viscosity compared to Midel eN and it has through a few esterification process that mixed with alcohol so that it become very good properties of insulation oil in terms of high flash point, high cooling ability, good oxidation stability, providing a better match of oil paper insulation system due to its high relative permittivity and enhanced the insulation life due to its high water solubility.

4.7 Result of Total acid Number of Midel En and PFAE oil after electrical discharges.

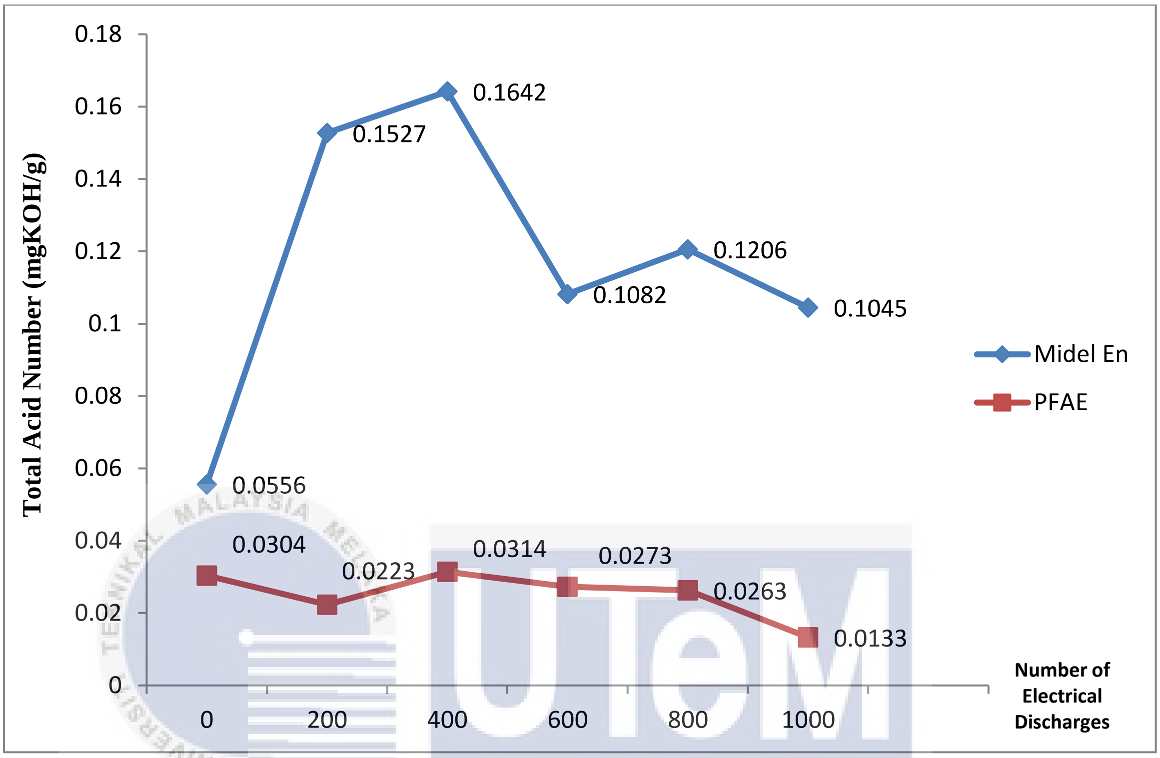


Figure 4.16: Total acid number of fresh Midel eN and PFAE oil

By referring figure 4.16, it shows that the total acid number of Midel eN decreases with the number of occurred discharges. However, there was a little change of total acid number of PFAE compared to Midel eN oil due to its saturated fatty chain acid with good oxidation stability. It shows that PFAE not much effected by ageing factor. Electrical discharges also an electrical stress that belong to ageing factor. Hence, it can be contributed to the molecules structures changes in the oil. When the breakdown occurs, it will modify locally the oil. However, in chemical point of view, long- chain fatty acids produced by natural ester fluids are less reactive and the degradation of by products of natural ester are mostly are high molecular weight acid. These acid can be non-aggressive for cellulosic material and even beneficial.

Based the result, it also can relate with the properties of this oil sample. Midel eN is a unsaturaed esters that has unfully saturated molecules with hydrogen atoms. Hence, it also relatively weak and expected can be poor hydrolytic stability in the presence of water and heat. The acidity value of Midel eN after discharges also exceed the standard requirement which need below 0.06. Meanwhile, the PFAE is a saturated ester and the hydrolytic stability is improved. Hence, the presence of water in the oil does not effect the acidity content.

4.8 Result of Breakdown Voltage of Midel eN and PFAE oil after electrical discharges.

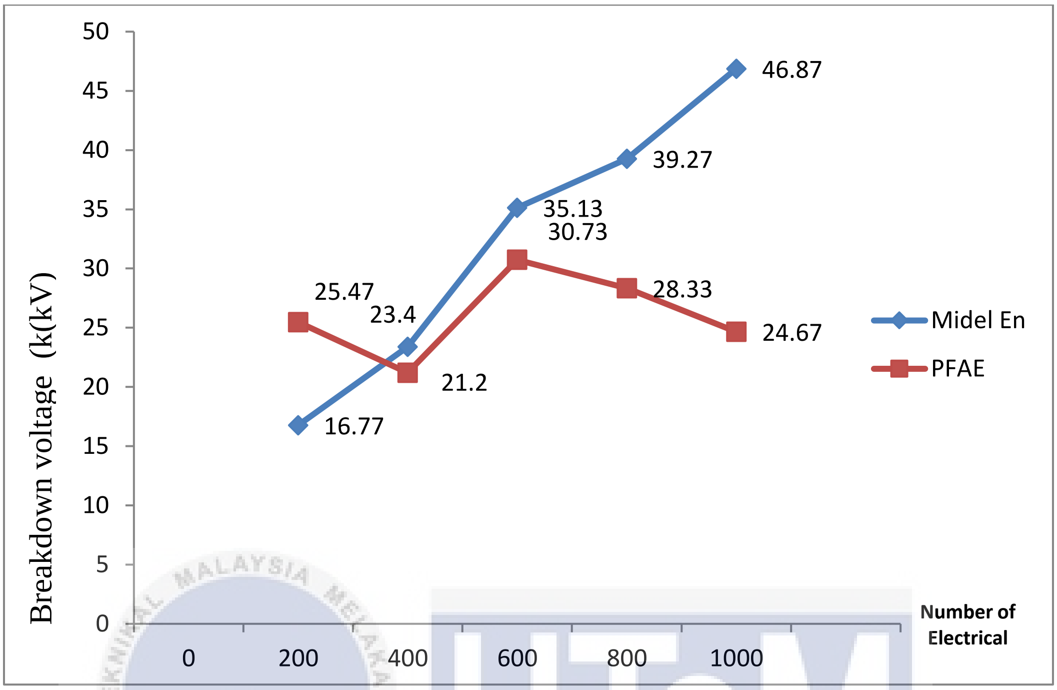


Figure 4.17: Breakdown Voltage of Midel eN and PFAE oil for 5 sample after electrical discharges.

The breakdown voltage (BDV) plays an important role in determining the voltage value that can withstand of the insulation oil with different electrical discharges that applied. Based on the figure 4.17, it shows that the BDV of Midel eN oil is increasing as the moisture content is reduced after electrical discharges were applied from 200 until 1000 discharges.

Regarding figure 4.17, it shows that the BDV value is of PFAE oil decrease from 25.47 to 21.2kV. However, its start to increase to 30.73kV at 600 discharges and it reduce again until 24.67kV. Hence, it can be concluded that the presence of moisture in PFAE is one of the leading causes of electrical breakdown because it increases the ionic conductivity of the oil hence dropping the breakdown voltage. On top of that, this result also due to electrical discharges that changes the molecules structures and increase the number of an electron. Hence, it also reduces the BDV level of PFAE after through electrical discharges. Although, based the result shown in the figure PFAE can be proved that PFAE has a good oxidation stability oil and not much affected by the electrical discharges because of its saturated fatty chain and not easy to form free radical from molecules structure.

4.9 Summary

Table 4.4: Summary of the result Midel eN and PFAE

Oil Sample	Moisture (ppm)	Acidity (mgKOH/g)	PDIV(kV)	BDV (kV)
Fresh Midel eN	150.2	0.0556	15.42	24.92
Midel En (200 Discharges)	145.4	0.1527	-	16.77
Midel En (1000 Discharges)	73.34	0.1045	-	46.87
Fresh PFAE		0.0304	17.87	26.66
PFAE (200 Discharges)	140.76	0.0223	-	25.47
PFAE (1000 Discharges)	147.54	0.0133	-	24.67

Regarding on the result of this chapter, there is two type of natural ester insulating oil has been in this experiment which is PFAE and Midel eN. The physicochemical properties of the oil are dependent on the stability of the fatty acid chains. Hence, moisture absorbed by the oil will either break the fatty acid chains, break C=C unsaturated double bonds, or ionize some of the acid and alcohol molecules in the chain. When the carbonyl groups in this oil react with water molecules, it will enhance the formation of hydroxyl radicals (hydrolysis reaction). Besides that, the position of all the radical molecules in the Carboxylic acid and alcohol in the waveband in the spectrum or their intensities/relative intensities can be used to explain the structural arrangement contributing to improve the conductivity of the oil and thus lower the PDIV and BDV value. Then, based on the result it shows that the properties of PFAE oil in terms of moisture, acidity and BDV before and after electrical discharges are not significantly change compared to the Midel En.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.0 Introduction

This chapter will provide the conclusion of this project which discussed about the summary of result of the experiment.

5.1 Gantt Chart

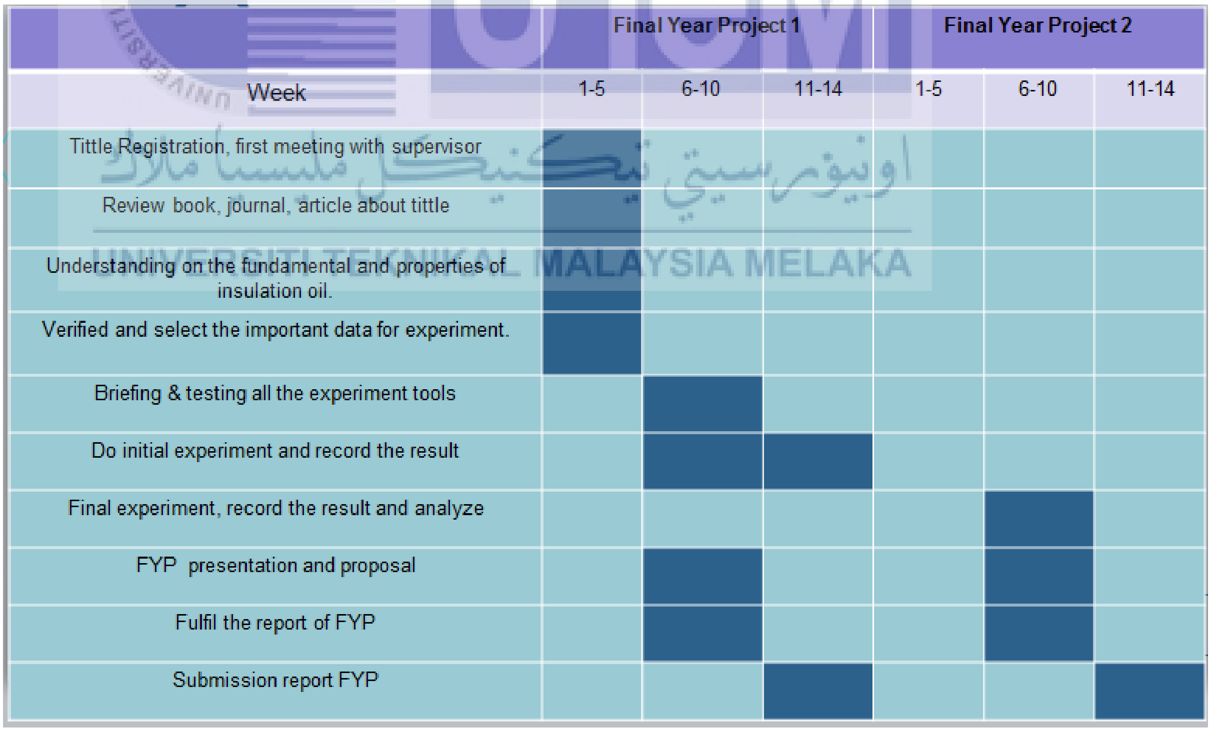


Figure 5.1 : Gantt Chart of the research work

5.2 Conclusion

In this research work, the natural ester insulating oil which is PFAE and Midel eN had been chosen to investigate the effect of electrical discharge on the natural ester-based insulating oil. In this study, the initial state of the oil sample is followed ASTM D974, D6871, D1816 and D1533 standards in terms of acidity, moisture and breakdown (BDV) before proceed to the next stage. PDIV test will be started first before the electrical discharges. All the equipment and setup for electrical discharges is according to IEC 61294 standard. By applied more than 200 discharges test and following the IEC 61294 standard, the properties of PFAE and Midel eN had been showed in this research work. Hence to fulfill the objectives of this research work, all the sample has been analyzed after electrical discharge test by measure it physicochemical properties to compare with initial properties of natural ester insulating (NEI) oil. After experiments have been done, the result shows that the intensity of the chemical bonds in the molecular structure of the oil is weakened by moisture it absorbs. Besides that, the absorbed moisture causes instability in their physical stability. The total acid number of Midel eN decreases with the number of occurred discharges. Hence, it can be contributed to the molecules structures changes in the oil. When the breakdown occurs, it will modify locally the oil. However, there was only a little change of the total acid number of PFAE compared to Midel eN oil due to its saturated fatty chain acid with good oxidation stability. The BDV of PFAE oil also not much affected with the high electrical discharges. Hence, it can be conclude that PFAE has better performance in terms of good oxidation stability and also the BDV value that can withstands.

5.3 Recommendation

Based on this study, it is evidently proves that the PFAE oil has a good performance in terms of moisture, acidity, PDIV and BDV after through electrical discharges. Hence, there are some recommendations for future studies to be considered as the improvement of this study:

1. In this study, the only oil used is PFAE and Midel eN to enhance the physicochemical properties and BDV level of the NEI after electrical discharges test. Therefore, other type of oil can be used such as mineral oil might be used to compare their significant difference in the physicochemical properties and BDV enhancement results.

2. Other chemical properties such as the effect of moisture and acidity increase on other physical properties of the oils like viscosity, flash point, and resistivity should be studied to gain more comprehensive understandings of the oil behaviour as electrical discharge applied.
3. FTIR analysis test should be perform in order determine the characteristic and mechanism of the NEI oils before and after the electrical discharge applied.

REFERENCES

[1] B. Vishal, M. Saurabh, T. Vikas and K. Prashant, “Transformer’s History and its Insulating Oil”, 5th National Conf., INDIACom, Computing for Nation Development, 2011

[2] K. Celal et al., “Breakdown Voltage Analysis of Insulating Oils Under Different Conditions”, IEEE Conference on Electric Power and Energy Conversion Systems (EPECS), pp. 1-4, 2013.

[3] C. Perrier and A. Beroual, “Experimental investigation on insulating liquids for power transformers: mineral, ester and silicone oil”, IEEE Electr. Insul. Mag., Vol. 25, No. 6, pp. 6-13, 2009.

[4] I. Fofana, “of Insulating Liquids,” vol. 29, no. 5, 2013.

[5] I. Fofana, H. Borsi, and E. Gockenbach, “Transformer Fluids Under Thermal Ageing,” *Behaviour*, pp. 360–363.

[6] S. bin A. Ghani, “Enhancing the Dielectric Strength and Oxidation Stability of Natural Ester Insulation Oil by Optimization of Antioxidants Mixtures.”

[7] M. Jaroszewski and K. Rakowiecki, “Partial discharge inception voltage in transformer natural ester liquid — Effect of the measurement method in the presence of moisture,” *IEEE Trans. Dielectr. Electr. Insul.*, vol. 24, no. 4, pp. 2477–2482, 2017.

[8] O. Idir *et al.*, “Influence of thermal ageing and electrical discharges on uninhibited olive oil properties,” *IET Sci. Meas. Technol.*, vol. 10, no. 7, pp. 711–718, 2016.

[9] I. Fofana and J. Sabau, "Application of Petroleum-based oil in PowerTransformer," 23 pages, In *Natural Gas Research Progress*, Editors: Nathan David and Theo Michel, © 2008 Nova Science Publishers, Inc., ISBN: 978-1-60456-700-7.].

[10] “Distribution-Transformer, MTM.pdf.” .

[11] A. Raymon and R. Karthik, “Reclaiming Aged Transformer Oil with Activated Bentonite and Enhancing Reclaimed and Fresh Transformer Oils with Antioxidants”, IEEE Trans. Dielectr. Electr. Insul., pp. 548-555, Vol. 22, No. 1, February 2015.

[12] M.S Naidu and V. Kamaraju, High Voltage Engineering, 4th Edition, McGraw-Hill Publishing Company Limited, 2009.

[13] Rashmi Sanghi, “Chemistry Behind the Life of a Transformer:, General Article, June 2003.

[14] H. Voltage, S. Transformer, and G. E. Company, “Insulation systems for liquid filled transformers,” pp. 13–15.

[15] Allen Filters, Inc., Transformer Oil, Part 1: Maintenance of Mineral Transformer Oil, 2009.

[16] J. Weesmaa, P. Wiklund, B. Pahlavanpour, L. Bergeld, and J. Nunes, “Transformer oil specifications and important properties for optimal in-service life,” *ICHVE 2014 - 2014 Int. Conf. High Volt. Eng. Appl.*, 2014.

[17] A. Raymon, P. S. Pakianathan, M. P. E. Rajamani, and R. Karthik, “Enhancing the Critical Characteristics of Natural Esters with Antioxidants for Power Transformer Applications,” vol. 20, no. 3, pp. 899–912, 2013.

[18] Y. Bertrand and L. Hoang, “Vegetable Oils as Substitute for Mineral Insulating Oils in Medium-Voltage Equipments,” France, 2004.

[19] M. Rafiq, Y. Z. Lv, Y. Zhou, K. B. Ma, W. Wang, C. R. Li, and Q. Wang, “Use of Vegetable Oils as Transformer Oils-A review,” *Renew. Sustain. Energy Rev.*, vol. 52, no. October 2016, pp. 308–324, 2015.

[20] U. U. Abdullahi, S. M. Bashi, R. Yunus, Mohibullah, and H. A. Nurdin, “The Potentials of Palm Oil as a Dielectric Fluid,” in *National Power and Energy Conference, PECon 2004 - Proceedings*, 2004, pp. 224–228.

[21] T.V. Omen, “Vegetables Oils for Liquid- Filled Transformers”, IEEE Electrical Insulation Magazine, Vol 18, No.1, 2002, pp 6-11.

[22] R. Liao. J. Hao, G Chen, Z. Ma and L. Yang.” A comparative Study of physicochemical, Dielectric and Thermal Properties of Pressboard Insulation Impregnante with Natural Ester and Mineral Oil” IEEE Transaction on Dielectrics and Electrical Insulation, Vol. 18, No 5, 2011, pp. 1626-1637

[23] S. Tembohlen and M. Koch , " Aging Performance and Moisture Solubility of Vegetables Oils for Power Transformer” IEEE Transaction on Power Delivery, Vol. 25, No 2, 2010, pp. 825-830.

[24] N. Azis and Z.D. Wang, “Acid Generation Study of Natural Ester” XVII International Symposium on High Voltage Engineering. Hannover, Germany, August 22-26, 2011

[25] A. Rajab, M. Tsuchie, M. Kozako, M. Hikita, and T. Suzuki, “PD properties and gases generated by palm fatty acids esters (PFAE) oil,” *Proc. 2016 IEEE Int. Conf. Dielectr. ICD 2016*, vol. 2, pp. 816–819, 2016.

[26] T. Kanoh, H. Iwabuchi, Y. Hoshida, J. Yamada, T. Hikosaka, A. Yamazaki, Y. Hatta, and H. Koide, “Analyses of Electro-Chemical Characteristics of Palm Fatty Acid Esters as Insulating Oil,” in *2008 IEEE International Conference on Dielectric Liquids, ICDL 2008*, 2008, no. 1, pp. 2–5.

[27] www.midel.com/productsmidel/midel-en/moisture-tolerant

[28] IEC 60814 Second edition, "Insulating liquids - Oil-impregnated paper and pressboard - Determination of water by automatic coulometric Karl Fischer titration", 1997.

[29] A. A. Suleiman1,2, N. A. Muhamad1, N. Bashir1, N. S. Murad1 and Y. Z. Arief1"Effect of Moisture on Breakdown Voltage and Structure of Palm Based Insulation "Oils IEEE Transactions on Dielectrics and Electrical Insulation Vol. 21, No. 5; October 2014

[30] Coulibaly, M.L., Perrier, C., Marugan, M., et al.: 'Aging behavior of cellulosic materials in presence of mineral oil and ester liquids under various conditions', IEEE Trans. Dielectr. Electr. Insul., 2013, 20, (6), pp. 1971–1976

[31] Megger OTS60PB, 60AF/2, 80AF/2, 100AF/2, VCM100, Automatic Oil Test Sets.

[32] I.Fofana, N'cho, A.Beroual. ' Effect of Exposure to Electrical Discharge on Transformer Oil Properties" G. Jishu, H. V. Engineering, and J. Sylvestre G. Jishu, H. V. Engineering, and J. Sylvestre, no. April 2014, 2011.

[33] P. Rozga and D. Hantsz, "Influence of volume effect on electrical discharge initiation in mineral oil in the setup of insulated electrodes," *Electr. Eng.*, vol. 99, no. 1, pp. 179–186, 2017

[34] A. Cavallini and C. G. Azcarraga, "Comparison of partial discharge inception voltages for ester and mineral oils under divergent fields," *Annu. Rep. - Conf. Electr. Insul. Dielectr. Phenomena, CEIDP*, pp. 1254–1257, 2013.

[35] M.S. Mohamad*, H. Zainuddin, S. A. Ghani and I.S. Chairul " Breakdown and Partial Discharge Performance of Palm Fatty Acid Ester (PFAE) Oil - based Fe3O4 Nanofluids" 978-1-5090-2547-3/16/$31.00 ©2016 IEEE

[36] Nur Amirah Othman et al. / Jurnal Teknologi (Sciences & Engineering) 78: 10–3 (2016) 31–36 "Investigation on the degradation behavior of creepage discharge on pressboard immersed in palm fatty acid ester (PFAE) oil"

[37] W. P. Beach, *Weibull Analysis Handbook.*

APPENDICES

APPENDIX A: BREAKDOWN VOLTAGE RESULT

Date: 14.3.2018 Time: 9.00AM

Type of oil : Midel eN

	Room	**Oil**
Humidity	**43.2**	**-**
Temperature	**26.5**	**26.8**

Test Run	**BDV**	**Average**	**Test Run**	**BDV**	**Average**
1	**22**		16	**22**	
2	**23**	**20kv**	17	**30**	**25kV**
3	**16**		18	**26**	
4	**20**		19	**26**	
5	**19**		20	**23**	
6	**28**		21	**20**	
7	**26**	**25kV**	22	**22**	**24kV**
8	**22**		23	**28**	
9	**29**		24	**23**	
10	**19**		25	**26**	
11	**28**		26	**8**	
12	**17**	**25kV**	27	**24**	**23kV**
13	**31**		28	**23**	
14	**24**		29	**27**	
15	**23**		30	**24**	
Average:	23.3kV				

Date: 14.3.2018 **Time:** 2.00PM

Type of oil :_PFAE

	Room	**Oil**
Humidity	**44.4**	**-**
Temperature	**25.2**	**26.4**

Test Run	**BDV**	**Average**	**Test Run**	**BDV**	**Average**
1	**23**		16	**28**	
2	**29**	**26.8kV**	17	**29**	**26.8kV**
3	**24**		18	**24**	
4	**29**		19	**32**	
5	**29**		20	**21**	
6	**31**		21	**27**	
7	**31**	**31.4kV**	22	**23**	**25.8kV**
8	**33**		23	**26**	
9	**32**		24	**26**	
10	**30**		25	**27**	
11	**29**		26	**20**	
12	**18**	**25.6kV**	27	**27**	**24.6kV**
13	**29**		28	**25**	
14	**26**		29	**24**	
15	**26**		30	**27**	
Average :	26.83kV				

Appendix B: Midel eN Natural Ester Dielectric Insulating Fluid Overview

MIDEL® MIDEL® eN

Natural Ester Dielectric Insulating Fluid Overview

September 2014 Page 2 of 2

Table 1 - Characterisation of Natural Ester Dielectric Fluid

	Unit	Test Method	Requirement as per IEEE C57.147	MIDEL eN Typical values
Physical Properties				
Appearance	-	IEC 61099 7.1.2 / ASTM D 1524	-	clear, free from suspended matter and sediment
Density at 20°C	kg/dm^3	ISO 3675 / ASTM D 1298	max. 0.96	0.92
Kinematic Viscosity at 0°C 40°C 100°C	mm^2/s (cSt)	ISO 3104 / ASTM D 445	max. 500 max. 50 max. 15	232 37 9.3
Flash Point	°C	ISO 2592 / ASTM D 92	min. 275	327
Fire Point	°C	ISO 2592 / ASTM D 92	min. 300	360
Pour Point	°C	ISO 3016 / ASTM D 97	max. -10	-31
Colour	ASTM unit	ASTM D 1500	≤ 1.0	1.0
Chemical Properties				
Water Content	mg/kg	IEC 60814 / ASTM D 1533	max. 200	50
Neutralisation Value	mg KOH/g	IEC 62021 / ASTM D 974	max. 0.06	<0.05
Net Calorific Value	MJ/kg	ASTM D 240-02	-	37.5
Corrosive Sulphur	-	IEC 62535 / ASTM D 1275B	-	Non corrosive
Dielectric Properties				
Breakdown Voltage at 1 mm 2 mm 2.5 mm Breakdown Voltage, Impulse at 25.4 mm	kV	ASTM D 1816 ASTM D 1816 IEC 60156 ASTM D 3300 / IEC 60897	min. 20 min. 35 min. 130	45 57 >75 134
Dielectric Dissipation Factor Tan δ at 90°C and 50 Hz 25 °C 100 °C	-	IEC 60247 ASTM D 924 ASTM D 924	 max. 0.2% max 4.0%	<0.005 0.023% 0.767%

Data quoted above are typical values, may be altered without notice and do not constitute a specification

www.midel.com

Appendix C: ASTM D6871

Designation: D6871 – 03 (Reapproved 2008)

Standard Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus[1]

This standard is issued under the fixed designation D6871; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers a high fire point natural vegetable oil ester insulating fluid for use as a dielectric and cooling medium in new and existing power and distribution electrical apparatus such as transformers and attendant equipment.

1.2 Natural vegetable oil ester insulating fluid differs from conventional mineral oil and other high fire point (or "less-flammable") fluids in that it is an agricultural product derived from vegetable oils rather than refined from petroleum base stocks or synthesized from organic precursors.

1.3 This specification is intended to define a natural vegetable oil ester electrical insulating fluid that is compatible with typical materials of construction of existing apparatus and will satisfactorily maintain its functional characteristic in this application. The material described in this specification may not be miscible with some synthetic electrical insulating liquids. The user should contact the manufacturer of the natural ester insulating fluid for guidance in this respect.

1.4 This specification applies only to new insulating fluid as received prior to any processing. The user should contact the manufacturer of the equipment or fluid if questions of recommended characteristics or maintenance procedures arise.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D88 Test Method for Saybolt Viscosity

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

D97 Test Method for Pour Point of Petroleum Products

D117 Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D877 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes

D923 Practices for Sampling Electrical Insulating Liquids

D924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids

D974 Test Method for Acid and Base Number by Color-Indicator Titration

D1275 Test Method for Corrosive Sulfur in Electrical Insulating Oils

D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D1524 Test Method for Visual Examination of Used Electrical Insulating Oils of Petroleum Origin in the Field

D1533 Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration

D1816 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using VDE Electrodes

D1903 Practice for Determining the Coefficient of Thermal Expansion of Electrical Insulating Liquids of Petroleum Origin, and Askarels

D2300 Test Method for Gassing of Electrical Insulating Liquids Under Electrical Stress and Ionization (Modified Pirelli Method)

D2717 Test Method for Thermal Conductivity of Liquids

D2766 Test Method for Specific Heat of Liquids and Solids

D2864 Terminology Relating to Electrical Insulating Liquids and Gases

D3300 Test Method for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Under Impulse Conditions

[1] This specification is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.02 on Gases and Non-Mineral Oil Liquids.

Current edition approved Oct. 1, 2008. Published December 2008. Originally approved in 2003. Last previous edition approved in 2003 as D6871-03. DOI: 10.1520/D6871-03R08.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 As-Received New Fluid Property Requirements

Property	Limit	ASTM Test Method
Physical:		
Color, max	1.0	D1500
Fire point, min, °C	300	D92
Flash point, min, °C	275	D92
Pour point, max, °C	−10	D97
Relative density (specific gravity) 15°C/15°C, max	0.96	D1298
Viscosity, max, cSt at:		D445 or D88
100°C (212°F)	15	
40°C (104°F)	50	
0°C (32°F)	500	
Visual Examination	Bright and Clear	D1524
Electrical:		
Dielectric breakdown voltage at 60 Hz		
Disk electrodes, min, kV	30	D877
VDE electrodes, min, kV at:		D1816
1 mm (0.04 in.) gap	20	
2 mm (0.08 in.) gap	35	
Dielectric breakdown voltage, impulse conditions 25°C, min, kV, needle negative to sphere grounded, 1 in. (25.4 mm) gap	130	D3300
Dissipation factor (or power factor) at 60 Hz, max, %		D924
25°C	0.20	
100°C	4.0	
Gassing tendency, max, µl/min	0	D2300
Chemical:		
Corrosive sulfur	not corrosive	D1275
Neutralization number, total acid number, max, mg KOH/g	0.06	D974
PCB content, ppm	not detectable	D4059
Water, max, mg/kg	200	D1533[A]

[A] As stated in Test Methods D1533 Annex A1 "Alternative Solvent Systems," alternate reagents may be needed for certain natural ester formulations. Consult the manufacturer for recommendations. Reagents for aldehydes and ketones (such as Coulomat AK and CG-K) should be used if the additives are unknown. When alternate reagents are needed, using the Test Methods D1533 reagents may yield elevated and erratic water content results.

D4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography

2.2 *National Fire Protection Association Standard:*

National Electrical Code Article 450-23[3]

3. Terminology

3.1 *Definitions*—Definitions of terms related to this specification are given in Terminology D2864. Vegetable oil natural ester: vegetable oil containing ester linkages, typically triglycerides. Most often obtained from seed crops (a "natural" source of esters, as opposed to synthesized esters).

4. Sampling and Testing

4.1 Take all fluid samples in accordance with Test Methods D923.

4.2 Perform each test in accordance with the ASTM test method specified in Table 1.

5. Property Requirements

5.1 Natural ester insulating fluid, as received, shall conform to the requirements of Table 1. The significance of these properties is covered in Guide D117 and Appendixes X2.1-X2.3.

6. Keywords

6.1 electrical insulating fluid; fire point; flammability; insulating fluid; natural ester

[3] *National Electrical Code*, NFPA 70, National Fire Protection Association Inc.

Appendix D: ASTM D1816

Designation: D1816 – 12

Standard Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using VDE Electrodes[1]

This standard is issued under the fixed designation D1816; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers the determination of the dielectric breakdown voltage of insulating liquids (oils of petroleum origin, silicone fluids, high fire-point mineral electrical insulating oils, synthetic ester fluids and natural ester fluids). This test method is applicable to insulating liquids commonly used in cables, transformers, oil circuit breakers, and similar apparatus as an insulating and cooling medium. Refer to Terminology D2864 for definitions used in this test method.

1.2 This test method is sensitive to the deleterious effects of moisture in solution especially when cellulosic fibers are present in the liquid. It has been found to be especially useful in diagnostic and laboratory investigations of the dielectric breakdown strength of insulating liquid in insulating systems.[2]

1.3 This test method is used to judge if the VDE electrode breakdown voltage requirements are met for insulating liquids. This test method should be used as recommended by professional organization standards such as IEEE C57.106.

1.4 This test method may be used to obtain the dielectric breakdown of silicone fluid as specified in Test Method D2225 and Specification D4652, provided that the discharge energy into the sample is less than 20 mJ (milli joule) per breakdown for five consecutive breakdowns.

1.5 Both the metric and the alternative inch-pound units are acceptable.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)

D923 Practices for Sampling Electrical Insulating Liquids

D2225 Test Methods for Silicone Fluids Used for Electrical Insulation

D2864 Terminology Relating to Electrical Insulating Liquids and Gases

D3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus

D4652 Specification for Silicone Fluid Used for Electrical Insulation

D6871 Specification for Natural (Vegetable Oil) Ester Fluids Used in Electrical Apparatus

2.2 *IEEE Standard:*

Standard 4 IEEE Standard Techniques for High Voltage Testing[4]

C57.106 Guide for Acceptance and Maintenance of Insulating Oil in Equipment[4]

3. Significance and Use

3.1 The dielectric breakdown voltage of an insulating liquid is of importance as a measure of the liquid's ability to withstand electric stress without failure. The dielectric breakdown voltage serves to indicate the presence of contaminating agents such as water, dirt, cellulosic fibers, or conducting particles in the liquid, one or more of which may be present in significant concentrations when low breakdown voltages are obtained. However, a high dielectric breakdown voltage does not necessarily indicate the absence of all contaminants; it may merely indicate that the concentrations of contaminants that are present in the liquid between the electrodes are not large enough to deleteriously affect the average breakdown voltage of the liquid when tested by this test method (see Appendix X1.)

---

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.05 on Electrical Test.

Current edition approved June 15, 2012. Published July 2012. Originally approved in 1960 as D1816 – 60 T. Last previous edition approved in 2004 as D1816 – 04. DOI: 10.1520/D1816-12.

[2] Supporting data is available from ASTM Headquarters. Request RR:D27-1006.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

[4] Available from the Institute of Electrical and Electronic Engineers, Inc., PO Box 1331, Piscataway, NJ 08855.

3.2 This test method is used in laboratory or field tests. For field breakdown results to be comparable to laboratory results, all criteria including room temperature (20 to 30°C) must be met.

4. Electrical Apparatus

4.1 In addition to this section, use IEEE Standard 4 to determine other requirements necesary for conducting test measurements, and maintaining error limits using alternating voltages. Procedures to ensure accuracy should follow the requirements of IEEE Standard 4. Calibration(s) shall be traceable to national standards and calibration should be verified annually or more often to ensure accuracy requirements. IEEE Standard 4 is required during the manufacturing of the test apparatus and utilized during calibration of the equipment.

4.1.1 *Test Voltage*—The test voltage shall be an alternating voltage having a frequency in the range from 45 to 65 Hz, normally referred to as power-frequency voltage. The voltage wave shape should approximate a sinusoid with both half cycles closely alike, and it should have a ratio of peak-to-rms values equal to the square root of 2 within ±5 %.

4.1.2 *Generation of the Test Voltage*— The test voltage is generally supplied by a transformer or resonant circuit. The voltage in the test circuit should be stable enough to be unaffected by varying current flowing in the capacitive and resistive paths of the test circuit. Non-disruptive discharges in the test circuit should not reduce the test voltage to such an extent, and for such a time, that the disruptive discharge (breakdown) voltage of the test specimen is significantly affected. In the case of a transformer, the short-circuit current delivered by the transformer should be sufficient to maintain the test voltage within 3 % during transient current pulses or discharges, and a short circuit current of 0.1 A may suffice.

4.1.3 *Disruptive Voltage Measurement*— Design the measurement circuit so the voltage recorded at the breakdown is the maximum voltage across the test specimen immediately prior to the disruptive breakdown, with an error no greater than 3 %.

4.2 *Circuit-Interrupting Equipment*— Design the circuit used to interrupt the disruptive discharge through the specimen to operate when the voltage across the specimen has collapsed to less than 100 V. It is recommended that the circuit design limit the disruptive current duration and magnitude to low values that will minimize damage to the electrodes and limit formation of non-soluble materials resulting from the breakdown, but consistent with the requirements of 4.1.2, but in no case should the short-circuit current exceed 1 mA/kV of applied voltage.

4.3 *Voltage Control Equipment*—Use a rate of voltage rise of 0.5 kV/s. The tolerance of the rate of rise should be 5 % for any new equipment manufactured after the year 2000. Automatic equipment should be used to control the voltage rate of rise because of the difficulty of maintaining a uniform voltage rise manually. The equipment should produce a straight-line voltage-time curve over the operating range of the equipment. Calibrate and label automatic controls in terms of rate-of-rise.

4.4 *Measuring Systems*—The voltage shall be measured by a method that fufills the requirements of IEEE Standard No. 4, giving rms values.

4.5 Connect the electrode such that the voltage measured from each electrode with respect to ground during the test is equal within 5 %.

4.6 *Accuracy*—The combined accuracy of the voltmeter and voltage divider circuit shall be such that measurement error does not exceed 3 % at the rate-of-voltage rise specified in 4.3. For equipment manufactured prior to 1995 the maximum allowable error is 5 %.

5. Electrodes

5.1 The electrodes shall be polished brass spherically-capped electrodes of the VDE (Verband Deutscher Elektrotechniker, Specification 0370) type having the dimensions shown in Fig. 1 ±1 %, mounted with axes horizontal and coincident within ±1 mm.

6. Test Cell

6.1 The test cell shall be designed to permit easy removal of the electrodes for cleaning and polishing, verification that the shape is within the specified tolerance, and to permit easy adjustment of the gap spacing. The vector sum of the resistive and capacitive current of the cup, when filled with oil meeting the requirements of Specification D3487, shall be less than 200 µA at 20 kV, at power frequency. Mount the electrodes rigidly from opposite sides with the spacing axially centered within ±1 mm. Clearance from the electrodes to all sides, bottom, cover or baffle, and any part of the stirring device is at least 12.7 mm (½ in.). Provide the test cell with a motor-driven two-bladed impeller and drive shaft, constructed of a material having high dielectric strength. The two-bladed impeller is 35 mm (1⅜ in.) ±5 % between the blade extremities, having a

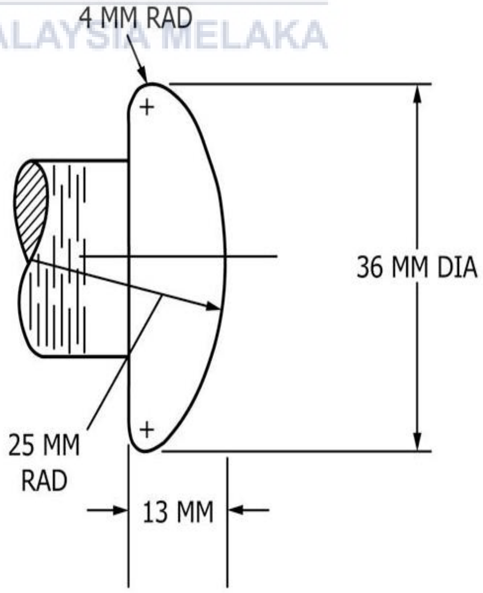


FIG. 1 VDE Electrode

pitch of 40 mm (1.57 in.) ±5 % (blade angle of twenty degrees (20°) ± 5 %), operating at a speed between 200 and 300 rpm. The impeller, located below the lower edge of the electrodes, rotates in such a direction that the resulting liquid flow is directed downward against the bottom of the test cell. Construct the test cell of a material of high dielectric strength, that is not soluble in or attacked by any of the cleaning or test liquids used, and is nonabsorbent to moisture and the cleaning and test liquids. So that the breakdown may be observed, transparent materials are desirable, but not essential. In order to preclude stirring air with the sample, provide the cell with a cover or baffle that will effectively prevent air from contacting the circulating liquid.

7. Adjustment and Care of Electrodes and Test Cell

7.1 *Electrode Spacing*—With the electrodes held firmly in place, check the electrodes with a standard round gage for 2 ± 0.03-mm (0.079-in.) spacing. If a dielectric breakdown does not occur during any of the consecutive breakdown tests using the 2 mm spacing or the sample is not adequate for the 2 mm spacing test cell a 1 ± 0.03-mm (0.039-in.) spacing should be used to determine the breakdown voltage and the spacing reported. Flat "go" and "no-go" gages may be substituted having thicknesses of the specified value ±0.03 mm for electrode spacing of 1 or 2 mm. If it is necessary to readjust the electrodes, set the electrodes firmly in place and check the spacing. For referee tests or tests that will be used for close comparisons, the laboratories shall agree in advance on the spacing for the tests and ensure that all other requirements of this test method are met. The spacing agreed upon shall be measured with the gage that corresponds exactly to the selected spacing within tolerance stated above for the gage.

7.2 *Cleaning*—Wipe the electrodes and cell clean with dry, lint-free tissue paper, or a clean dry chamois. It is important to avoid touching the electrodes or the cleaned gage with the fingers or with portions of the tissue paper or chamois that have been in contact with the hands. After adjustment of the spacing, rinse the cell with a dry hydrocarbon solvent, such as kerosine or solvents of Specification D235. Do not use a low boiling point solvent, as its rapid evaporation may cool the cell, causing moisture condensation. If this occurs, before using, warm the cell to evaporate the moisture. Avoid touching the electrodes or the inside of the cell after cleaning. After thorough cleaning, flush the cell with new insulating liquid of the type to be tested that is filtered through a 5-micron filter or smaller and containing less than 25 ppm moisture. Conduct a voltage breakdown test on a specimen of this insulating liquid in the manner specified in this test method. If the breakdown voltage is in the expected range for this conditioned insulating liquid, the cell is considered properly prepared for testing other samples. A lower than anticipated value is considered as evidence of cell contamination; then repeat the cleaning and the breakdown test with clean dry insulating liquid.

7.3 *Daily Use*—At the beginning of each day's testing, the electrodes shall be examined for pitting and carbon accumulation, and the spacing checked. If the test of any sample is below the breakdown value being used by the operator as a minimum satisfactory value, drain the cell and flush the cell with new insulating liquid of the type to be tested that is filtered through a 5-micron filter and containing less than 25 ppm moisture before testing the next specimen. When not in use, keep the cell filled with oil that meets the requirements of Specification D3487 of the type normally tested. Alternatively, the cell may be stored empty in a dust-free cabinet. At the beginning of each days testing, clean according to 7.2.

7.4 *Polishing of Electrodes*—When electrodes show slight etching, scratching, pitting, or carbon accumulation, they should be removed from the test cup and polished by buffing with jeweler's rouge using a soft cloth or soft buffing wheel. The residue from the buffing should be removed by repeated wiping with lint-free tissue paper saturated with a suitable solvent, followed by solvent rinsing or ultrasonic cleaning. After careful inspection, any electrodes from which pitting cannot be removed by light buffing should be discarded, as more refinishing would destroy the electrode contour and dimensions shown in Fig. 1. Reinstall the electrodes in the test cup and adjust spacing and clean in accordance with 7.1 and 7.2.

8. Sampling

8.1 Obtain a sample of the insulating liquid to be tested using appropriate ASTM sampling apparatus. Insulating liquid sampling procedures are detailed in Practice D923. Particular reference should be made to the general precaution statement of this test method. The sample shall be taken in a dry, clean, non-permeable bottle. Tightly seal and shield from light until ready to be tested. Plastic bottles are permeable and moisture content of the sample may change resulting in a measurable difference when compared to samples collected in non-permeable containers. Prior to starting the test, the sample shall be inspected for the presence of moisture, sludge, metallic particles, or other foreign matter. If the sample shows evidence of free water, the dielectric breakdown test should be waived, and the sample reported as unsatisfactory.

9. Test Procedure

9.1 Allow the sample and the test cup to equilibrate to ambient temperature. Laboratory and referee tests shall be conducted at room temperature (20 to 30°C).

9.2 To ensure a homogenous sample, gently invert and swirl the sample container several times. Rapid agitation is undesirable, since an excessive amount of air may be introduced into the liquid. Within 1 min after agitation, use a small portion of the sample to rinse the test cell. Drain the rinse. Within 30 s of the rinse, fill the cell slowly with the remaining portion of the sample. The cell is full when closing the cover or baffle allows no air to be in contact with the insulating liquid.

9.3 Wait at least 3 min but no more than 5 min between filling the cup and application of voltage for the first breakdown. For high fire-point electrical insulating oils, natural esters and synthetic esters, the hold time before the initial application of voltage shall be at least 30 min. Apply the voltage increasing from zero at the rate of 0.5 kV/s ± 5 % until breakdown occurs as indicated by operation of the circuit-interrupting equipment; record the highest rms voltage value that occurred immediately prior to each breakdown. Occasional momentary discharges may occur which do not result in operation of the interrupting equipment; these shall be disregarded until the voltage across the specimen collapses to less than 100 V. Conduct four additional breakdowns waiting at least 60 s but no more than 90 s before applications of voltage for successive breakdowns. (During the intervals before voltage application, between breakdowns, and at the time voltage is being applied, the propeller shall be circulating the insulating liquid.)

9.4 Calculate the mean of the five breakdowns as follows:

$$\bar{X} = \frac{1}{5}\sum_{i=1}^{5} X_i$$

where:

$\bar{X}$ = mean of the five individual values,
X_i = ith breakdown voltage.

9.5 Using the breakdown voltage values determined in 9.3, determine that the range of the five (5) breakdown voltages does not exceed the values indicated as follows:

$$\text{Range} = X_{Highest} - X_{Lowest}$$

where:

$X_{Highest}$ = the highest breakdown voltage of the five readings, and
X_{Lowest} = the lowest breakdown of the five readings.

1 mm gap setting	Range must be less than 120 % of the mean of the five (5) breakdown voltages
2 mm gap setting	Range must be less than 92 % of the mean of the five (5) breakdown voltages

If this range is exceeded and there is sufficient volume of test specimen, repeat the analysis on a new cup filling. If an insufficient volume of test specimen is available, the test result can be reported with an additional comment that the range of the breakdowns exceeds the allowable range.

9.6 When it is desired merely to determine if the dielectric strength is above or below a specified level, five breakdowns are required, provided the five values are all above or all below this level. Otherwise, follow the procedure described in 9.3 – 9.5

10. Report

10.1 Include in the report the following:

10.1.1 The test method used,

10.1.2 The volts (rms value) at each breakdown, and the mean of all breakdowns reported to two significant digits,

10.1.3 The approximate temperature of the insulating liquid at the time of the test, and

10.1.4 The electrode spacing.

11. Precision and Bias[5]

11.1 *Single-Operator Precision*—The single operator percent coefficient of variance of a single test result comprised of 5 breakdowns has been found for the 1 mm gap to be 14 % and for the 2 mm gap 11 %. Therefore, results of two properly conducted tests by the same operator on the same sample should not differ by more than 40 % and 30 % respectively. The maximum allowable range for the series of 5 breakdowns should not exceed 120 % for the 1 mm gap and 92 % for the 2 mm gap.

11.2 *Multilaboratory Precision*—The multilaboratory percent coefficient of variance has been found for the 1 mm gap to be 17 % and for the 2 mm gap 14 %. Therefore, two separate test results conducted by separate laboratories on the same sample of insulating liquid should not differ by more than 47 % of the mean of the two tests if the 1 mm gap was used or differs by more than 40 % of the mean of the two tests if the 2 mm gap was used. The precision statement for the 1 mm gap is temporary, because sufficient laboratories were not available and testing is planned to replace the precision statement before 2009.

11.3 *Bias*—No statement can be made about the bias of this test method because a standard reference material is not available.

12. Keywords

12.1 breakdown voltage; dielectric strength; insulating liquids; insulating oils; test cell; VDE electrodes

[5] Supporting data is available from ASTM International Headquarters. Request RR:D27-1014.

Appendix E: IEC 61294 PDIV Test Procedure

INSULATING LIQUIDS - DETERMINATION OF THE PARTIAL DISCHARGE INCEPTION VOLTAGE (PDIV) - TEST PROCEDURE

1 Scope

This technical report describes a test procedure to measure the partial discharge inception voltage (PDIV). It is one of a group of methods which have been recently developed to describe the behaviour of insulating liquids when subjected to high electrical stress.
The method is applicable to all types of insulating liquids used in medium and high voltage equipment. Nevertheless the equipment described in this report may not be adapted to liquids with values of PDIV greater than 70 kV (see annex A).
Complementary information may be obtained by the gassing properties (IEC 628) and the lightning impulse breakdown voltage (IEC 897) in addition to the widely used measurement of electric strength (IEC 156).

2 Reference documents

IEC 156 : 1963, Method for the determination of the electric strength of insulating oils

IEC 270 : 1981, Partial discharge measurements

IEC 897 : 1987, Methods for the determination of the lightning impulse breakdown voltage of insulating liquids

IEC 1072 : 1991, Methods of test for evaluating the resistance of insulating materials against the initiation of electrical trees

ISO 5725 : 1986, Precision of the test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests

3 Definition

According to this technical report, the partial discharge inception voltage (PDIV) of an insulating liquid is the lowest voltage at which a partial discharge occurs of an apparent charge equal to or exceeding 100 pC when the sample is tested under the specified conditions.

NOTE - All details related in partial discharge measurement and definitions are to be found in IEC Publication 270.

4 General notes on the partial discharge inception voltage

Experience of tests with needle electrodes has shown that partial discharges occur in the test liquid at a threshold voltage known as the PDIV, thereafter the number of partial discharges is found to increase logarithmically with applied voltage. The test described in this report determines the PDIV of a liquid. Further classification of liquids in terms of the increase in discharge number with voltage may be performed with the same test arrangement but the procedure is not covered in this document.

At the present state of knowledge, PDIV in liquids is considered to be mostly related to their chemistry, and relatively little affected by the conditioning. The opposite is usually found with breakdown voltage measurements in a more uniform field at power frequency (IEC 156).

5 Outline of the method

After treatment under the specified conditions the insulating liquid is poured into the test cell described in this report. A power frequency a.c. voltage is increased at a constant rate and the appearance of discharges is monitored by an appropriate partial discharges (PD) measuring device. The voltage at which the apparent charge of the discharges is equal to or larger than 100 pC is recorded. At least 10 PDIV measurements are made on each of two different fillings of the cell. The average of the mean values of the two series is taken as the result.

6 Apparatus

6.1 Test Cell

An example of the test cell illustrated in figure 1 is the same as the one described in IEC 897 with a different needle electrode and electrode spacing.

6.1.1 The test cell shall consist of a vessel containing a vertical gap. The volume of liquid in this cell shall be of the order of 300 ml. Insulating materials used in the cell shall be high dielectric strength, compatible with the insulating liquids to be tested, and resistant to solvents and cleaning agents commonly used for these liquids. Metal parts shall be restricted to the electrode and their supports. The test cell shall be discharge free at 70 kV using the method described in subclause 7.2.2.

6.1.2 The cell shall contain two electrodes forming an adjustable point to sphere configuration. The spherical electrode shall be a 12,5 mm to 13 mm diameter steel ball bearing. The point electrode shall be a needle of 3 µm radius at the tip as described in the IEC 1072.

NOTE - Suitable needle of 50 mm length and 1 mm diameter may be obtained from Ogura Jewell Industry Co Ltd., 7-12 Omori-kita 5 - Chome-Ota-Ku, Tokyo, 143 Japan.

The distance from needle to sphere is set at 50 ± 1 mm. The extension of the needle from the holder is set at 25 ± 1 mm. These dimensions are shown in figure 1.

NOTE - Care must be taken not to damage the tip of the needle when measuring the gap. A small error in the gap length does not affect the PDIV but a small change in the diameter of the needle may significantly affect the result. The test needle shall not be brought into contact with the sphere electrode.

7 Preparation of the test sample and equipment

7.1 Test Sample

The liquid is to be treated by passing through a Rachig or similar column and appropriate filter under vacuum.

Typical conditions are as follows :

Filter :	5 µm sintered glass
Temperature :	approximately 60 °C
Absolute pressure :	100 Pa (or less)

At the end of the treatment the liquid is to be cooled down to room temperature under vacuum. It may be allowed to stay in the treatment vessel for a few hours if needed, still under vacuum. Bottles (brown glass), of 2 litres each, will be completely filled from the treatment vessel and then sealed.

7.2 Equipment

7.2.1 Calibrate the PD measurement device in terms of apparent charge magnitude with an appropriate calibration device (see IEC 270, clause 5).

7.2.2 Check that the equipment is free from partial discharges as follows : fill the cell with an insulating liquid. Remove the test needle and apply the maximum required test voltage for 5 min. During this period no discharges exceeding 50 pC are to be registered. It is permissible to use electrical, acoustic and optical techniques to discriminate between discharges in the sample and background interference.

8 Test procedure

1) Fill the clean test cell by gently pouring the liquid into the cell.

2) Allow the liquid to stand for 15 min before the first voltage application.

3) Connect the cell to the ac (power frequency) transformer. Increase the voltage from zero at a rate of 1 kV/s until a partial discharge occurs of apparent charge equal to or greater than 100 pC. Record the voltage *V*1. Rapidly decrease the voltage down to zero.

4) Make repeated measurements on the same filling of the cell to record at least 10 values (*V*1 to *V*10). Allow an interval of 1 min between each voltage application. Calculate the mean value and the standard deviation of the individual measurements.

5) Empty the cell.

6) Insert a new needle.

7) Refill the test cell with a new portion of the test sample and repeat steps 2 to 4.

8) If the difference between the two mean values obtained exceeds the repeatability, *r* (see clause 10), then one further test shall be made. Without regard to the result of the third test the measurements are finished.

9 Report

Report the average of the two mean values which meet the repeatability requirement as the PDIV value of the test sample. Otherwise report the average of the mean values for the three series of measurements and the spread.

The report should also include the following :

- the sample identification and preparation;
- a description of the cell if different from the one described in this report;
- a description of the type of PD measuring device;
- the number of test series;
- the mean value of the PDIV (kV) and the mean standard deviation for each serie;
- any departure from the procedure described in this report.

APPENDIX A

TEST PROCEDURE FOR THE DETERMINATION OF THE PARTIAL DISCHARGE INCEPTION VOLTAGE OF INSULATING LIQUIDS

Insulating liquids in service in electrical apparatus may be required to withstand very high stress to avoid the inception of partial discharges at points where the field is divergent and therefore enhanced in magnitude. This characteristic of liquids to prevent or suppress partial discharge is important in many applications and wide variation is found between the performances of liquids of different compositions. In this field of application for liquids and liquid additives a standardised method of assessing the partial discharge inception characteristics is required for the purpose of quality assurance, specification, product development and condition monitoring. For many users a PDIV measurement is more relevant to the application of the liquid than the currently available tests for say electric strength or lightning impulse breakdown voltage. In one respect the PDIV test is easier to perform than breakdown voltage tests. This is because the test is virtually non destructive, hence a large number of individual measurements can be made on the single sample of liquid giving a precise result without the need for frequent cleaning of the test cell and refurbishing electrodes.

The discharge detection equipment needed to carry out the test is widely available in electrical test laboratories and considerable latitude is allowed in the choice of apparatus. Likewise, the test cell is based on an existing type.

The required measurement sensitivity of 100 pC is not difficult to achieve and is shown to be effective in tests on a wide range of insulating liquids. No advantage is apparent in tests made at higher sensitivity than 100 pC.

PDIV measurements are influenced by the field configuration and the test requires the use of a needle electrode of specified radius of curvature at the tip. Microscopic techniques are available to measure the radius of curvature but require very high grade equipment and in practice it may be preferable to rely on the precision with which the needles are manufactured. An examination, using a scanning electron microscope, of needles sampled from batches supplied by Ogura Jewel Industry Co. Ltd.; Tokyo, suggests that they are manufactured to a close tolerance. Needles from this source were used in the Round Robin and other tests without any preselection.

Unlike breakdown voltage test at power frequency (IEC 156), the PDIV measured using point-sphere electrodes is relatively independant of such contaminants as moisture and particles. This is because the critical high field conditions are present within only a very small volume of the sample under test. In the test procedure a basic treatment of the liquid is described to ensure that any contamination is held within the normal limits of serviceability of insulating liquids.

The PDIV test procedure has been successfully applied to a wide range of liquids including mineral oils, esters, silicone oils, and halogenated liquids which give partial discharge inception voltages in the range 20 kV to 70 kV. The test has not proved satisfactory for measurements on some highly aromatic hydrocarbon impregnants used in capacitors which have PDIV above the specified limit of 70 kV.

The precision of the test method has been investigated for mineral insulating oils only. In a Round Robin Test two samples of oil, one naphthenic and other paraffinic were tested in five laboratories. For each oil five groups of twenty determinations were used, each group corresponding to a separate filling of the cell. The two oils give very similar results nevertheless they are ranked in the same order in all laboratories. From the results summarised in table 1, the repeatability r and the reproducibility R (see ISO Standard 5725) are calculated as r = 5 kV and R = 7 kV which, set against a mean value of approximately 30 kV, provides sufficient precision for a useful test.