" I hereby declare that I have read through this report entitle *"Study Of Adsorbent Effectiveness On Reclaimed Transformer Oil"* and found that it has comply the partial fulfillment for awarding the degree of Bachelor of Electrical Engineering (Industrial Power)

Signature :……

Supervisor's Name : MR. IMRAN BIN SUTAN CHAIRUL

Date :…….

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First and foremost, all praise to Allah for giving us health, patience and strength in completing this project until the end. Without health permission given by Allah and, most likely I cannot complete this project with successfully.

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STUDY OF ADSORBENT EFFECTIVENESS ON RECLAIMED TRANSFORMER OIL

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A thesis submitted in fulfillment of the requirements for the degree of Bachelor of Electrical Engineering (Industrial Power)

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2016

I declare that this report entitle *"Study Of Adsorbent Effectiveness On Reclaimed Transformer Oil"* is the result of my own research except as cited in the references. The report has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :……..

Name : AMINUR HAZIEQ BIN ZULKEFLI

Date :……

Dedication
To my beloved mother, father and whole family

ABSTRACT

Mineral oil is being widely used in oil-immersed power transformer for generation, transmission and distribution in electrical system network. It plays important role in a transformer as liquid insulation and coolant. To maintain a normal operation of a transformer, it is essential for insulating oil to has a good dielectric properties, excellent heat transfer and chemically stable under various range of application. Degradation of transformer oil due to ageing process can decrease the efficiency of transformer's operation. High heat, oxidation and presence of moisture are the catalyst of the ageing process. Presently, aged transformer oil will be drained out from the long period service transformer. Then the transformer will be immersed with new mineral oil. These disposing and changing process of aged mineral oil are highly cost. In addition, the aged mineral oils can bring harmful to living life and ecosystem if disposed freely. Thus, this project's aim is to study the effectiveness of adsorbent on aged transformer oil using fuller's earth by reclamation technique. The method used for this reclamation process is by contact process. Then the mixed compound is filter using Whatman filter paper No.42 with the aid of vacuum pump. Results of Dissolved Decay Product (DDP), AC breakdown voltage and total acid number test and will be analyzed. These results will reveal the significant of applying adsorbent on aged transformer oil. From the results obtained, it shows that the relative amount of dissolved decay product has been removed by 25% after reclaimed. As the by-product being decreased, the breakdown voltage has achieved in increment by 50%. In the meantime, the effectiveness of fuller's earth as adsorbent for reclamation process has enhanced of total acid number value by 83.7%. The achievement of these results has proved the effectiveness of fuller's earth as adsorbent for reclamation process

ABSTRAK

Minyak mineral digunakan secara meluas dalam pengubah kuasa untuk penjanaan, penghantaran dan pengagihan. Minyak ini memainkan peranan penting dalam pengubah kuasa iaitu sebagai penebat cecair dan penyejuk. Untuk mengekalkan operasi dalam keadaan normal bagi sesebuah pengubah kuasa, adalah penting bagi minyak penebat mempunyai sifat dielektrik yang tinggi, pemindahan haba yang sangat baik dan stabil di bawah pelbagai keadaan. Degradasi minyak pengubah yang disebabkan oleh proses penuaan boleh mengurangkan kecekapan operasi pengubah. Pemangkin kepada proses penuaan minyak pengubah kuasa ialah haba yang tinggi, pengoksidaan dan kehadiran kelembapan. Pada kebiasaanya, minyak pengubah kuasa yang telah degradasi akan ditukar kepada minyak yang baru. Proses penukaran dan pelupusan minyak ini akan melibatkan kos yang tinggi. Di samping itu, minyak mineral boleh membawa bahaya kepada kehidupan dan ekosistem jika dilupuskan secara bebas. Oleh itu matlamat ini projek itu adalah untuk mengkaji keberkesanan adsorben pada minyak pengubah yang telah degradasi menggunakan *fuller's earth* melaui proses reklamasi. Jenis kaedah yang digunakan untuk proses reklamasi ini adalah jenis kacauan antara minyak dengan adsorben dan kemudian sebatian campuran ditapis menggunakan kertas turas Whatman No.42 dengan bantuan pam vakum. Keputusan ujian mendapan larutan (DDP), kekuatan dielektrik dan jumlah asid dalam minyak akan dianalisis. Keputusan ini akan menunjukkan keberkesanan adsorben pada minyak pengubah yang telah degradasi. Dari keputusan yang diperolehi , ia menunjukkan bahawa jumlah relatif produk pereputan yang dibubarkan telah dikeluarkan sebanyak 25 % selepas proses reklamasi. Apabila jumlah produk asing menurun dalam minyak, kekuatan dielektrik telah mencapai kenaikan sebanyak 50%. Dalam pada itu , keberkesanan 'fuller's earth sebagai penjerap untuk proses reklamasi, telah menurunkan jumlah asid dalam minyak sebanyak 83.7 %. Pencapaian keputusan ini telah membuktikan keberkesanan 'fuller's earth sebagai penjerap bagi proses reklamasi

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LIST OF ABBREVIATIONS

AC	-	Alternating Current
UV	-	Ultraviolet
DDP	-	Dissolved Decay Product
PSAC	-	Palm Shell Activated Carbon
IFT	-	Interfacial Tension
kV	-	kilovolt
ASTM	-	American Society for Testing and Materials
TAN	-	Total Acid Number
IEE	-	Institute of Electrical and Electronics Engineers
CFR	-	Code of Federal Regulation
IEC	-	International Electrotechnical Comission
µm	-	micrometer
mm	-	milimeter
l	-	litre
g	-	gram
℃	-	degree celcius
ml	-	mililitre
nm	-	nanometer
a.u	-	absorbance unit

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CHAPTER 1

INTRODUCTION

1.1 Overview

The column of this chapter will explain about the research background, problem statement, project objectives, scopes and contribution of this project. The description and overview of this project will be explained in project background. Problem statement is highlighting issues that need to be solve by propose several ways and solution. Besides, the objectives and scope will explain about the purpose and area covered for this project. Lastly contribution of project is benefit that can be achieved at the end of this project.

1.2 Research Background

The lifespan of transformer functionality are depending on its insulation. One of the major and important part in power transformer is insulating oil or known as transformer oil. This oil contributes as cooling system and act as liquid insulation in power transformer [1]. The normal operation of electric transformer depends on the high dielectric strength and insulating properties. The various electrical and thermal stress can cause the transformer oil ageing but the major factors that speed up the ageing of the insulation transformer oil are humidity, high temperature, oxidation and the acidity of its oil [2]. Since the transformer oil come from mineral oil which extract from fractional distillation and the treatment of crude petroleum, it is highly cost and additional it's not sustainable product [1]. Moreover the ecosystem and the living life will be destruct and threatened when age mineral oil are disposed [3]. Recently there has method to overcome

this problem, which is by oil reclamation. It has been proven that this method is helps to improve the quality of mineral oil by removing and eliminating the contaminants as well as sludge in the age mineral oil. Several parameters can be observed and measured to differentiate between aged mineral oil before reclaim and after reclaimed. The parameters are the breakdown voltage, viscosity, flashpoint, firepoint as well as their colour. Thus this project are proposed and aim to carry out the reclamation of aged transformer oil in laboratory condition by using adsorbent which is fuller earth. Then UV-visible spectrophotometer is used to evaluate the performance of transformer oil in terms of dissolved decay product (DDP) [4]. In addition high voltage apparatus for breakdown voltage test are used to determine the breakdown voltage. Meanwhile the titrator for the total acid number test is used to determine the total acid number that presence in the oil samples

1.3 Problem Statement

Transformers are the most important and valuable electrical equipment. The failure of this component will lead to disruption in electric system network and then will cause the loss either in finance and energy. Generally, under the normal operation, the insulation of transformer can be degraded by a several factors such high temperature, moisture, oxidation and acidity of its oil [2,3]. As water is electrical conductor, it may increase the risk of electrical breakdown if it appears as free water in highly stressed regions [5]. Moreover the ecosystem and the living life will be destructs and threatened when age mineral oil are disposed freely Since mineral oil commonly used for transformer oil, which is highly cost in its production, thus this project will point on how to reused the aged transformer oil by using adsorbent fuller earth in reclamation process..

1.4 Objectives

The objectives of this project are:

1. To prepare the reclamation oil by using contact process method.
2. To compare the oil before and after reclamation by using UV-visible test, AC breakdown voltage test and total acid number test.
3. To analyze the UV-visible, AC breakdown voltage and total acid number result.

1.5 Scope of Research

The scopes of research are:

1. Prepare the reclamation oil from aged transformer oil by using adsorbent, which is fuller's earth.
2. Total Acid Number test by using 848 Titrino Plus test set made by Metrohm.
3. AC Breakdown Voltage Test according to ASTM D1816 by using Megger Oil Test Set OTS60PB.
4. Mineral oil used is aged mineral oil from service transformer 11kV/433V made by ACEC transformer on 1986.
5. UV-Visible Spectrometer made by Shimadzu UV mini 1240 for the UV-vis spectro test.

1.6 Contribution of Research

Reclamation of aged transformer oil has give advantage in improving the efficiency and dielectric strength. This research also can reduce the cost in replacing the aged transformer oil to new insulation mineral oil. Significantly the reclamation process can eliminate the contaminants in transformer oil without change the naturality of the oil.

1.7 Report Outlines

This thesis is covered in five chapters. Chapter 1 is explains about the research background, problem statement, project objectives and scopes of project. While Chapter 2 will describe the theory or general concept that related to the project and review the previous research works. This review will more focus on the performances of the liquid insulation oil on reclamation process by using adsorbent. Then, Chapter 3 will illustrate methodology applied in order to get the required output. The flow of project will be explained by a flow chart. Chapter 4 are explained the results by present table and graph. Analysis about the result will be discussed. Lastly, Chapter 5 is about the conclusions for the project.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter will discuss and review about several of previous studies regarding to this project. The subject that related to this project are more on the theory, basic principal electrical properties of transformer oil and application of reclamation to the ageing oil by using the adsorbent. The basic theory of the method used on the previous research also been highlighted in this chapter.

2.2 Mineral Oil

Mineral oil has been used for insulating oil for transformer for a long time either in generation, transmission or distribution. This oil has several main abilities, which are provide dielectric strength, provide heat transfer (cooling system), protect the paper insulation and lastly act as a diagnostic tool for the condition of the equipment. It significantly obtains from extraction and fractional distillation and filtration treatment of petroleum [1]. This oil has consists chemical compound which is the hydrocarbon compounds such as paraffin, naphthene and aromatic series, but also consists a minor scale amounts of sulfur, nitrogen, and oxygen compounds. As this mineral oil has this compound bring the important characteristics of insulating oil such as the immune to corrosive sulfur and oxidation stability [5].

2.2.1 Chemical structure of mineral oil

As explanation above state that this oil is mainly consists of hydrogen and carbon molecule with different structure. About three different structures contain in this molecule, which are paraffinic, naphthenic and aromatic structure. Paraffinic also can be known as normal-alkane or the other words waxes, which is it-saturated element and has only single bonds. This structure has give advantages to the mineral oil as the content of normal alkane have low solubility for water and oxidation products but has low thermal stability compare to the aromatic and naphthenic molecules. Naphthenic are came from group cycloalkane and it has the best low-temperature properties [6]. Lastly aromatic structure has present in all naturally oil. It has give good electrical properties and gas absorption of the oil as well as it has stable in oxidation. Figure 2.1 show the molecular structure of paraffinic, naphthenic and aromatic.

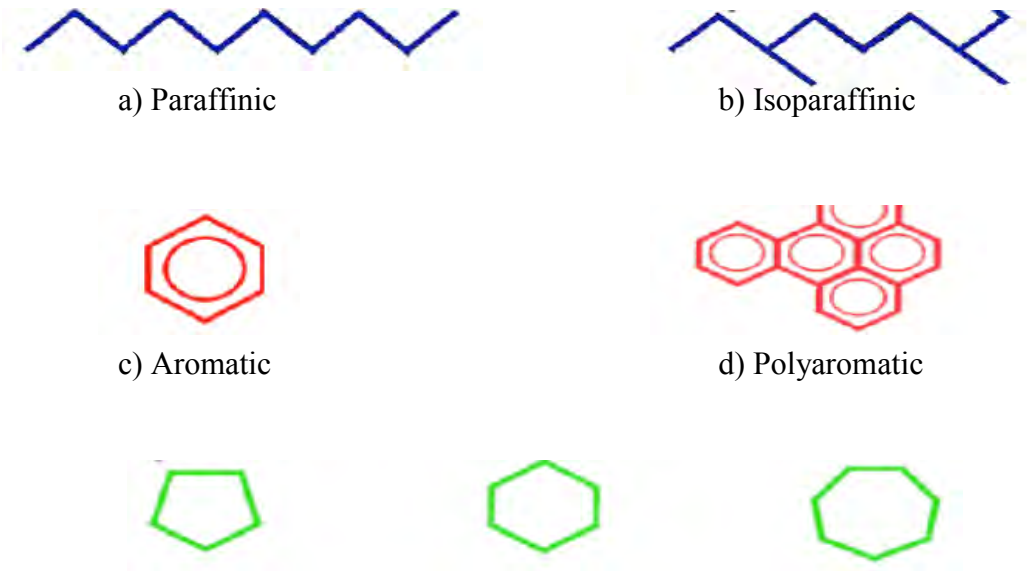


Figure 2.1: Basic hydrocarbon structure in mineral oil molecules (a),(b),(c),(d),(e) [6]

2.2.2 Ageing Transformer Oil

Transformer oil can be degrade or decrease in efficiency because of ageing process. The high temperature and chemical reaction such as oxidation presence of moisture are the element that catalyst of the ageing process [7]. Besides, the high

temperatures of the transformer are due to overloading. Aged transformer oil can give high percentage of transformer failure. Through roughly, the ageing transformer oil can be recognized by see the changing from its colour. Normally the colours of aged transformer oil are turn to yellow as in Figure2.2 but this are not appropriated method to determined ageing oil.

Figure 2.2: The colour of aged transformer oil [3]

There are several parameter that can be considered for determine the aged of the transformer oil such as the decreases in dielectric strength and interfacial tension (IFT) also increases in the total acid number (TAN) [8]. The cause for all this problem are due to contaminants in the oil such as water content and foreign particle like sludge from insulation paper. By referring to ASTM standard there has limit value for classified the service-aged oil with different voltage class. Table 2.1 shows the suggested limit breakdown voltage by group.

Table 2.1: Suggested Limits for In-Service Oils Group I by Voltage Class [9]

Property	Limit			ASTM Test Method
Voltage Class	69 kV and below	Above 69V through 288kV	345 kV and above	
Dielectric breakdown voltage,60 Hz, 0.100 gap 1 min, kV, min	26	26	26	D877 [6]
Dielectric breakdown voltage, 0.040 gap, kV, min	23	26	26	D1816 [16]
Dielectric breakdown voltage, 0.080 gap, kV, min	34	45	45	D1816 [16]
Neutralization number max, mg KOH/g	0.2	0.2	0.1	D974 [9]
Interfacial tension, min, mN/m	24	26	30	D971 [8]
Water max, ppm	35	25	20	D1533 [14]
Gas content when specified, max %	-	-	-	D831 [5] D1847 [26] or D2945 [22]

2.3 Reclamation

Refer to IEEE standard reclaiming or regeneration can be defined as the restoration to usefulness by the removal of contaminants and products of degradation such as polar, acidic, or colloidal materials from used electrical insulating liquids by chemical or adsorbent [10]. The terms of reclaim are differ from the term reconditioning because reconditioning just used mechanical process without engaged with chemical reaction. Since the limitation of transformer oil are not sustainable substances and harm to environment when it is dispose directly, the reclamation or regeneration process are the best method to overcome this problem. The reclaiming process can help to remove the contaminants that exist in aged oil transformer such as water and sludge without changing the naturality of the oil itself [3]. Subsequently it can improve the dielectric strength,

interfacial tension (IFT) and total acid number (TAN). Instead of doing reclamation on transformer oil, there are some selection of substances in the oil that must be considered for doing reclamation, which are askarel, substances that must be dispose and handling according to Federal Regulation 40 CFR, Part 761 or with local regulation law [10]. Then oil containing silicone fluid, which can cause foam excessively, and suspended carbon that must through filtration process first, mean it must process separately. However there has several type for reclamation process such as vacuum dehydrator, mechanical filter (blotter or filter press), coalescing filter, precipitation settling, contact process percolation by gravity, percolation by pressure, thermo-siphon bypass. The table 2.2 below shows the different type of contaminant that can be removed by using different type of process. The common type of process that industrial sector practice for reclaiming transformer oil is percolation by pressure and the vacuum dehydrator.

Table 2.2 - Type of Oil Purification Process [9]

Type of contamination remove							
Oil-Purification Practice	Solid	Free water	Soluble water	Air and Gas	Volatile	other	Page no
Vacuum dehydrator	No	Yes	Yes	Yes	Most	No	12
Mechanical filter (blotter or filter press)	Yes	Partial	Partial	No	No	No	12
Centrifuge	Yes	Yes	No	No	No	No	14
Coalescing filter	Yes	Yes	No	No	No	No	14
Precipitation settling	Yes	Yes	No	No	No	No	14
Contact process	Yes	Yes	Yes	No	No	Yes	18
Percolation by gravity	Yes	Yes	Partial	No	No	Yes	19
Percolation by pressure	Yes	Yes	Partial	No	No	Yes	20
Thermo-siphon bypass	No	No	Partial	No	Partial	Partial	23
Activated carbon sodium silicate process	Yes	No	No	No	Yes	Yes	23
Trisodium phosphate process	Yes	No	No	No	Yes	Yes	25

2.4 Adsorbent

Adsorbent are the common phrases that used in the reclamation of transformer oil. It has bought a meaning a material or substance attracts and holds the other substance tenaciously to its surface area [10]. Differ from absorbent phrases are not used because adsorbent can adsorb only 50% of other material or substance while absorbent can absorb 100% of substances. From this properties reclamation process can maintain the naturally of the oil itself. The ability of adsorbent used for reclamation is it can remove the contaminant that presence in the oil without changing the naturallity structure of the oil. Moreover there has many material of adsorbent that used in reclamation of transformer oil. The common material used in industry of reclamation process is a fuller earth. Other materials are bentonite, attapulgite, activated carbon, and molecular sieves.

2.4.1 Fuller's Earth

Fuller's earth as in Figure 2.3 is class of naturally occurring adsorbent clays, rather than to specific mineralogical specie as regarding to IEEE standard 637 [3]. The word fuller earth has established after the practice of healing the grease and stains from wool and cloth [10]. Fuller earth is a silicate anions (Si_2O_5)n and containing internal and external polar active sites. When fuller earth mixes with oil, it allowed the non-polar substances in the oil to flow but block or adsorb the polar contaminants that dissolved in the oil [5]. It also contains the layer of calcium or magnesium as exchangeable ion, which give high performances in decolourized colour of the aged transformer oil [11]. Other than that, it give advantage which can neutralizes carboxylic acids and adsorb polar [10]. As fuller earth is naturally from earth, it not gives a harmful to the environment as it can dispose in landfill safely.

Figure 2.3 : Fuller Earth [12]

2.4.2 Bentonite

Bentonite ($Al_2O_3 4SiO_2 H_2O$) or hydrated aluminum silicate as in Figure 2.4 is a clay form and it has a little similarity properties with fuller earth, which can absorb contaminant in the oil. Furthermore the presence of calcium or magnesium ions is good in decolorized color. Bentonite can be found naturally with combination of volcanic ash minerals called montmorillonite and ocean water [10]. Bentonite has been used widely for water treatment which removing toxic metal ions [12] and involve on process of drilling soil and muds. The adsorption of bentonite can be improved by activation treatment with acid. The ion such as Ca2+, Na2+ is replaced by H+ when acid (sulphuric acid or hydrochloric acid) was added to bentonite.

Figure 2.4 : Bentonite Powder [10]

2.4.3 Activated Carbon

Activated carbon are widely used in various industries which include, filtration of water, gas purification, air purification, spill cleanup and also in regenerating of lubricating oil [13]. Moreover it also has ability to remove the organic and inorganic pollutants. Physical structure of activated carbon is it has high surface area, porous structure and surface adsorption capacity which means the structure can be modified by enhancing with chemical and physical treatment [13]. Figure 2.5 shows the pore structure of activated carbon that used to adsorb organic molecules.

Previous research was used palm shell activated carbon (PSAC) as adsorbent for reclamation of transformer oil process [14]. This adsorbent gives result in improvement of

aged mineral oil since the adsorbent remove higher relative content of dissolve decay product (DDP) during the first reclamation. In addition this adsorbent has give some advantages such are economical, easily to get since it manufactured locally in Malaysia and it can be reactivated after use [14].

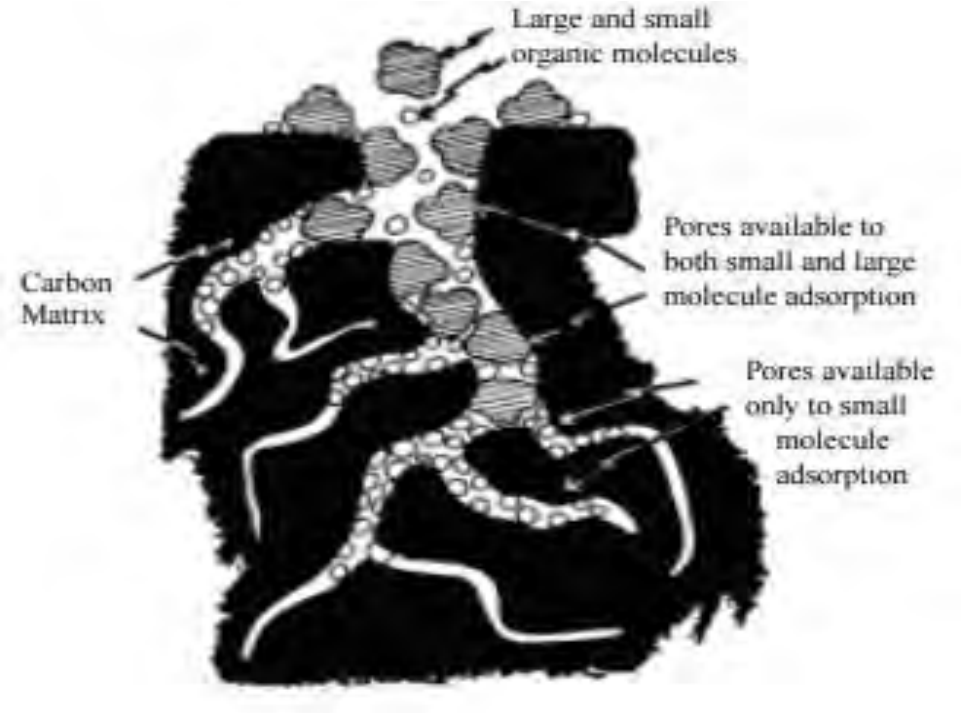


Figure 2.5 : Activated carbon particles pore and structures [14]

2.5 Related Previous Work

2.5.1 Principles Method Used

From the previous, there are some methods about the reclamation of aged mineral oil by using adsorbent. Different researchers have carried out different method for reclamation process and measure different parameter value for test such as breakdown voltage, total acid number (TAN), interfacial tension (IFT), and the dissolved decay product. Based on the previous research, there are several adsorbent used for reclamation such as fuller's earth. Fuller's earth is used since when it mixes with oil, it allowed the non-polar substances in the oil to flow and block or adsorb the polar contaminants that dissolved in the oil. It also contains the layer of calcium or magnesium as exchangeable ion, which give high performances in decolourized colour of the aged transformer oil

[11]. Previous research made by J.S.N'Cho and has conducted the experiment of reclamation-aged oil by using adsorbent fuller earth. The reclamation has made by percolation by gravity with 15 cycles. The oil has treated with temperature at 60 ℃ before pass through to the fuller's earth cartridge. Then, the improvement of the aged oil has been tested using UV-Vis spectro set which conclude the result that the dissolved decay product removal are gradually decreases after each passes [6]. While refers on research made by H.M Wilhem [15], the method used is contact process and filter by vacuum. About 1 litre of aged mineral oil with 100g of adsorbent was used in contact process to agitate the adsorbent with the oil. The contact process take 4 hour with maintain temperature at 25℃

However, according to A.Raymon and R.Karthik's research, bentonite has been introduced. The bentonites are improved by acid activation with added concentrated sulphuric acid (H_2SO_4). The activation takes place is to enhance the bleaching efficiency of the adsorbent when used in the oil [3]. The contact process are used for reclamation process and conducted by using 5g of the activated bentonite and 500 ml for aged oil. The agitation of the mixture has used magnetic stirrer as in Figure 2.6 under 750 rpm speed and heated up to temperature 80℃. After that the adsorbent are filter using Whatman filter paper no.42.

Figure 2.6 : Magnetic Stirrer [14]

2.5.2 Breakdown Voltage Test

Insulating oil has been used in transformer because of it has good electrical parameter such as high flash point and high dielectric strength. Breakdown voltage test is

the way in determining the dielectric strength. Breakdown voltage test can be divided into three type of test, which are AC breakdown voltage (figure 2.7), DC breakdown voltage AC Impulse breakdown voltage. Factor such as moisture, bubble, sludge and acidity can affect the breakdown voltage result [16]. Breakdown voltage test are measure is by immersed the two electrode with specific gap in the oil and record the voltage level, when sparking between two electrodes happen. Figure 2.7 show the type of breakdown voltage test set that used to test insulating oil. Several standard are used by worldwide which from ASTM International (USA) and IEC (Europe). There are two standard codes for ASTM in breakdown voltage test which ASTM D877 and ASTM D1816 while for code for IEC is IEC 60156. Appendix A shows the differences between ASTM and IEC standard with respect to the limit value for test.

Figure 2.7: Breakdown Voltage Test set (Megger OTS60PB/80PB) [17]

2.5.3 UV-Visible Spectrocospy

UV-visible spectro is the technique that measures the absorption radiation UV as function of frequency or wavelength due to the interaction with a sample, which means it, can determine the amount of substance or dissolved decay product (DDP) that presence in sample. The conduction of UV-Vis spectro test with respect to the transformer oil is based on ASTM D6802 (Test Method for Determination of the Relative Content Of Dissolved Decay Products in Mineral Insulating Oils by Spectrophotometry) [18]. Based on previous study UV-Vis spectro can be implemented to determine the efficiency and estimate the interfacial tension (IFT) of transformer oil. This research can help to make alternative

method for the ring method (ASTM D971 Interfacial Tension of Oil against Water by the Ring Method) that used to measure the interfacial tension of oil [19]. The working principal for UV-Vis sepectro test is when the light radiation is emitted through the oil sample containing various contaminations, the light are going to decrease and is detected as a function of wavelength. The amount of light absorbed by a solution can be calculated by using Beer-Lambert Law:

$$A_\lambda = - log_{10}(\frac{S_\lambda - D_\lambda}{R_\lambda - D_\lambda}) \epsilon_\lambda . c.l \quad [2.1]$$

Where :

A_λ : The light absorbance

S_λ : The sample intensity

R_λ : The reference intensity

D_λ : The dark intensity

ε : The absorbance coefficient of the absorbing species at wavelength λ

c : The concentration of the absorbing species (gram/liter)

l : The path length traversed by the light.

2.5.4 Total Acid Number Test

Generally the formations of acid in transformer oil are due to reaction between the oxygen that dissolved in the oil and with molecule of the oil itself. The molecules mainly consist of hydrogen and carbon. These molecules interact with oxygen to form carboxylic acids [20]. As mentioned by Bronsted-Lowry acid and Lewis base theory, they stated that carboxylic acids can react with the hydroxyl group (-OH) and water with donating proton (H+) [21]. The hydroxyl group can be found commonly in insulating paper, which is made from cellulose. The reactions will produce soluble oxidation by-products such as low molecular weight acid like acetic and levulinic acids [22]. Over long term of this decay product reaction, it can form the insoluble compounds or the other words, sludge. Then the sludge will accumulate and attach on the winding of transformer. This thing will lead to reduction of heat dissipation in transformer causes the winding become hotter. Higher temperature can cause additional oil decomposition [23]. The continuous of this phenomenon will cause the insulation of oil to degrade under normal operations. To

overcome of this problem some test can be made to monitor the content of acid in transformer oil. It can be measured by chemical titration by using the potassium hydroxide (KOH). The mass of potassium hydroxide is required to neutralize the acids in the oil sample (in mg of KOH/g) [20]. By referring to ASTM D974 the suggested limit of the amount neutralization number for the transformer oil is between 0.1 to 0.2 (mgKOH/g) [24] as shown in table 2.1 and the limit for level of running transformer oil acid number is about 0.25 mg KOH/g [25]. Figure 2.8 shows the titrator for volumetric titration, which used to analyze the total acid number (TAN) in the transformer oil.

Figure 2.8 Titrator (Metrohm 848 Titrino Plus) [26]

2.6 Summary and Discussion Review

This chapter has review some important information about the effect of adsorbent to the aged transformer oil by reclamation process. Thus the performances of aged mineral oil after reclamation by using adsorbent will be discussed by taking into account all the importance factors that will be affect the results.

For this experiment, the materials of adsorbent are clearly plays an importance action in improving the aged transformer oil. Fuller's earth is been chosen as adsorbent for reclamation of aged transformer oil process. Based on the previous research, mostly of the experiment choose the fuller earth since it mixes with oil, it allowed the non-polar substances in the oil to flow and block or adsorb the polar contaminants that dissolved in the oil. It also has potential to decolurized colour of aged oil since it contains the layer of

calcium or magnesium as exchangeable ion. Previous research made by J.S.N'Cho and the team, the colour are closely same as new insulating mineral oil and based on the result of UV-Vis spectro test, it gives higher percentage to remove the dissolve decay product (DDP). Furthermore, fuller earth is readily available compare to the other adsorbent. It also can safely disposed on landfill after being used.

The different of type reclamation process also can affect the efficiency of reclaim oil. Referring to the IEE guide for reclamation of insulating oils, mostly the type of reclamation cannot fully remove the soluble water and solid except on by using contact process. So this project will used the contact process to agitate the oil with the adsorbents. Then the adsorbents are filter by vacuum with Whatman filter paper no 42.

There are several test that are introduce from previous research to determine the improvement oil by compare before and after reclaim such as AC breakdown test, Total Acid Number (TAN) test, UV-Vis spectro test, moisture test by Karl-fischer method and colour test by using chromometer. AC breakdown, Total Acid Number and UV-Vis spectro are choosing for the test because this equipment is readily available in the laboratory.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter contains the principles of methods or procedures that used in this research. The procedure such as preparation of oil sample, type of reclamation method used, UV-visible test, breakdown voltage and total acid number will be discussed briefly.

3.2 Flow Chart of Methodology

This project's objective is to study the adsorbent effectiveness on reclaimed transformer oil. Fuller's earth has been choosing as adsorbents for the reclamation process. This fuller's earth will be mixing with aging mineral oil under contact process method and filtering by filter paper. This method is chosen based on the availability that present in laboratory. After that, several test will be done in order to study the effectiveness of adsorbents on reclaimed transformer oil. The test is Ac Breakdown Voltage test, Total Acid Number test and UV-Visible test.

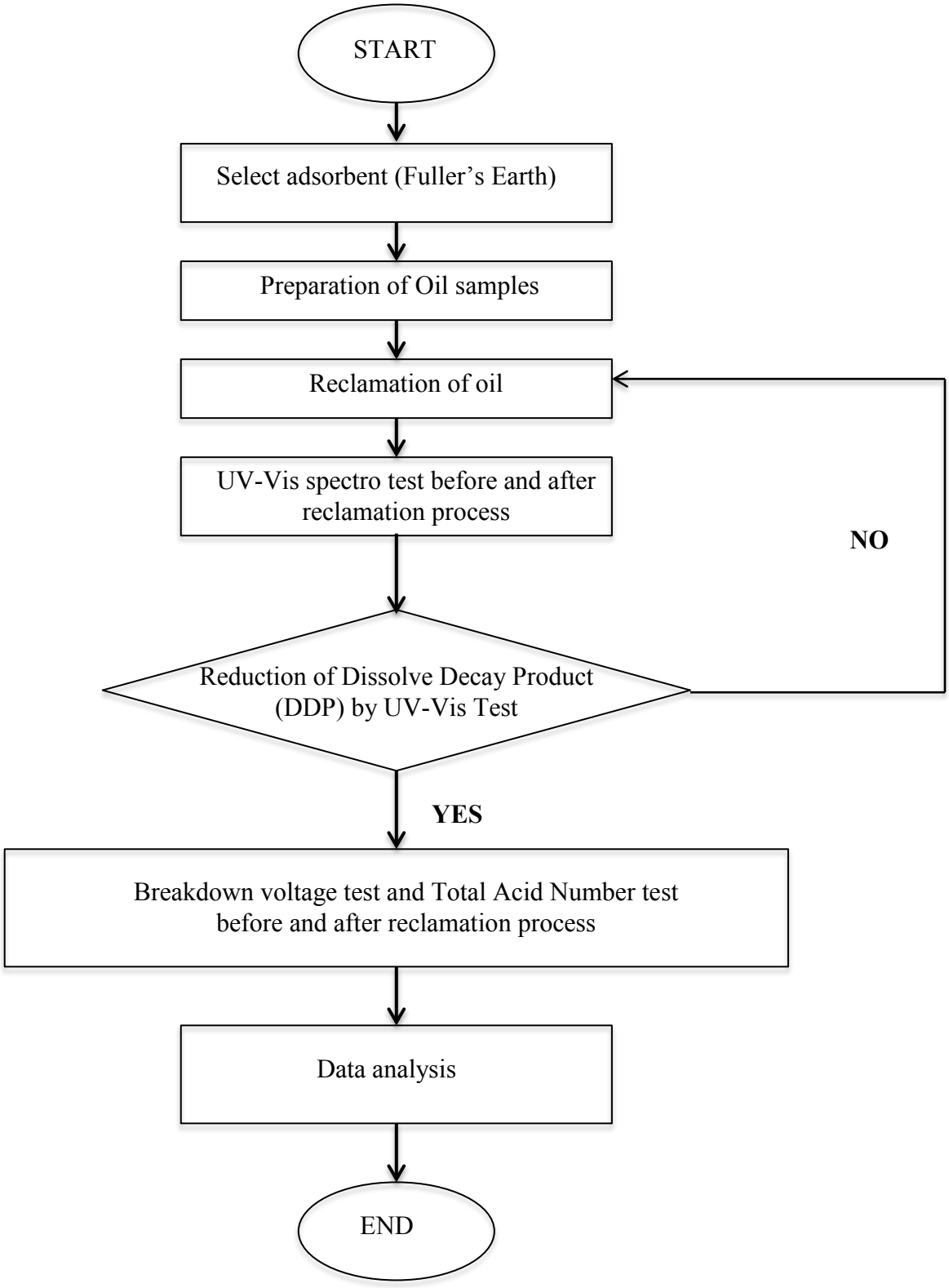


Figure 3.1 : Flowchart of Methodology

3.2.1 Select Adsorbent

Fuller's Earth has been chosen for this reclamation project. About 100g of fuller's earth powder will be dispersing into 1 litre aging mineral oil.

3.2.2 Preparation of oil samples

1 litre of aging mineral oils is taken from the services transformer. It is taken from a distribution transformer 500 kVA, 11kV/433V made by ACEC transformer, which made on 1986.

3.2.3 Reclamation Process

Reclamation process is done in the laboratory condition. The adsorbents are mixed into the aged oil by contact process methods. It needs to be stirrer for few hours. Then the mixture of solution are filter though filter paper with the aid of vacuum pump.

3.2.4 UV-Vis Spectro Test

UV-Vis test is made on the Shimadzu UV mini 1240. This spectro test is conducted accordance with the ASTM D6802 standard in order to determine the relative content of DDP for each mineral insulating oil [18].

3.2.5 AC Breakdown Voltage and Total Acid Number Test

Megger Oil Tester OTS60PB was used for AC Breakdown Voltage test. The standard used for this test is referring to ASTM D1816 [27]. Then for Total Acid Number (TAN) test, the apparatus used is from Titrator (Metrohm 848 Titrino Plus) [26]. This test is conducted based on ASTMD974 [24].

3.2.6 Result

After complete the test, the result of the AC breakdown voltage, Total Acid number in the oil and the dissolved decay product by UV-Vis test is collected in order to

monitor the performance of aging mineral oil before and after reclaiming. If there is no improvement on the result after reclamation, the process of reclamation is going to repeat again.

3.2.7 Data Analysis

The data is analyzed to determine the effectiveness of adsorbents on reclaimed transformer oil. The analyses of this experiment are based on the objective of this research. Then the conclusion can be made.

3.3 Reclamation Process

The oil samples were taken from the services transformer 500 kVA, 11kV/433V made by ACEC. These oils are undergoes aging for 30 years in the transformer and it doesn't have been reclaimed since it reveal on 1986. By referring to the reclamation standard by IEEE [9], the methods that been used for this reclamation process project is by contact process and filter the oil by vacuum with Whatman filter paper No 42. This project has used two different quantity of adsorbent and oil sample, which the first sample is about 1 litre of oil samples were mixes and stirs with 100g of adsorbent and the other one was used about 500 ml of aged mineral oil and mixes with 5g of fuller's earth with repeated for five cycle of reclamation process. To agitate or stir the mixture, magnetic stirrer is used with maintain speed at 750 rpm. The contact process take 4 hour with temperature remain at 60℃. Then the mixture is filter through 2.5 μm Whatman No. 42 filter paper to remove the adsorbent with the aid of vacuum. Figure 3.2 shown the flowchart for reclamation process.

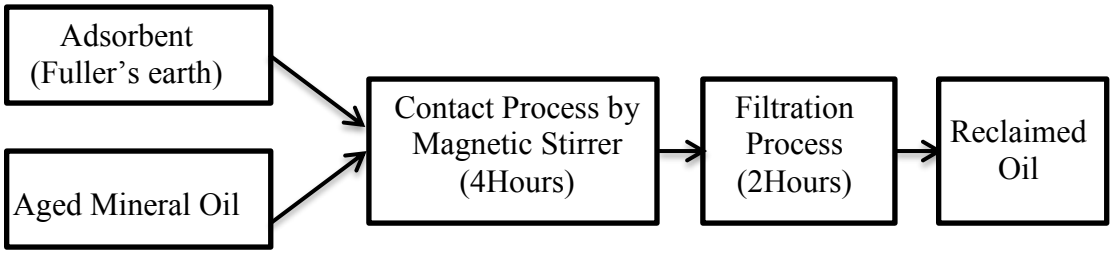


Figure 3.2 : Flowchart of Reclamation process

3.3.1 Weighing and Mixing Process

Fuller's earth used for this project is from Sigma-Aldrich [28]. Some precaution must take during weighing and mixing process because fuller's earth powders are dangerous when inhale it and same as mineral oil, which is it can bring harmful when the skin is exposed to it. The safety items that must be used are masks, rubber gloves and safety glasses. Digital analytical balance was used in order for weighing the fuller's earth. Figure 3.3 shows the weighing of fuller's earth by using digital analytical balance.

Figure 3.3 : Weighing of Fuller's earth

For the mixing process, the digital hotplate magnetic stirrer by Fisher Scientific was used. The speed of stirrer is set on 750 rpm constantly. To homogenize the concentrations of the mixture, the temperature is maintained at 60℃ for a period of 4 hours. Figure 3.4 shows the mixing and stirrer process of mineral oil and fuller's earth by using digital hotplate magnetic stirrer.

Figure 3.4 : Stirrer and mixing process

3.3.2 Filtration Process

After the completion of mixing process, the mixture is undergoing the filtration process by through to the filter paper with the aid of vacuum pump as to remove the sludge of the adsorbents that form in the mineral oil during mixing process. Filter paper that has been used is from Whatman filter paper No 42 that has 2.5 μm in pore sizes. Since the pore sizes of filter paper are to small, the time taken for filtration process took a several day for complete filter. It also can cause the mineral oil exposed to moisture. As refer to the IEEE guide for reclamation mineral oil [9], the aid of vacuum pump can be used to speed up the process as well to remove the moisture in the flask.

Figure 3.5 shows the filtration process, whereby the mixtures from the mixing process are poured directly to the fleaker. The oil will go through to the filter paper that has been put between the fleaker and the flask. The vacuum pump is connected to the flask by rubber hose in order to speed up the flow rate of mineral oil as well to remove the air and moisture in the flask. With the aid vacuum pump, this process took about 2 hours for complete filtration.

Figure 3.5 : The filtration process by filter paper with the aid of vacuum pump

3.4 UV-Visible Spectro Test

Ultraviolet-visible (UV-Vis) spectro test is used to determine the relative content of dissolved decay product. Figure 3.10 shows UV-Vis test set made by Shimadzu mini

1240. The optical absorption of the samples was measured over a wavelength range of 360–600 nm in accordance with the ASTM D6802 [18]

Figure 3.6 : UV-Vis Spectro by (Shimadzu mini 1240)

3.4.1 Preparation of Oil Sample in Cuvette

Before test, the cuvette is cleaned until there is no dust or other substances. Then, 5 ml of oil sample is placed into cuvette for UV-Vis test. Figure 3.11 shows 5 ml of oil sample is placed into cuvette by using syringe.

Figure 3.7 : The placement of oil sample into cuvette using syringe

3.4.2 Preparing the Oil Test Set

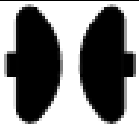
After the cuvette is placed into the test vessel, the ranges of wavelength UV light are choose from the menu at the test set. This test is used range of wavelength between 360–600 nm in accordance with the ASTM D6802 [18]. When the light radiation interact with oil sample, the passing light is carried by output fiber to the spectrometer, which is connected with a screen of test set to display and analyze the spectral response of the oil sample. The curve from graph absorbance against wavelength that shows on the screen will be recorded. Figure 3.12 shows the example of absorption spectrum recorded using the Shimadzu UV Mini 1240 instrument with the range of wavelength between 360 nm-600 nm.

Figure 3.8 : Example of absorption spectrum curve by UV-Vis test

3.5 AC Breakdown Voltage Test

The next step is to conduct the AC breakdown voltage test for aged oil before and after reclamation process. It is a necessary to know the dielectric strength by through breakdown voltage test. The breakdown voltage test kit with measuring capacity 60 kV is used for breakdown voltage test. According to ASTM D1816 the electrodes is spherical in shape and the gap set up between two electrodes is 1mm. Table 3.1 shows a standard with their shape and the gap size.

Table 3.1 : A standard with their shape and the gap size [27]

Standard	ASTM D1816
Electrode Shape	
Gap Size	1mm

3.5.1 Preparation of Electrode and Oil Test Set

First step before run the test, the electrodes are ensured to be free from the contaminant or other substances. The electrodes are cleaned by using acetone, and then wipes using the dry cloth. Then the electrode are set up with the gap between electrodes is 1mm according to ASTM D1816. About 500 ml is taken from the oil samples to place into AC breakdown voltage test set. There is need to make sure that the electrode must fully immersed into the oil and there is no air bubbles present in the oil sample for the test. Before operate the test, the humidity and temperature of the room and oil is recorded. Then a standard ASTM D1816 are chosen from the display at the test set. After selecting, the AC breakdown voltage test will be started. During the test, voltage will raised up automatically for 2kV until the breakdown. Average values are taking from 25 tests made. The oil samples were given a delay time of 2 minutes in between tests to enable them to cool to the desired temperature of 30°C [20]. Figure 3.6 show the gap setting between the electrodes, while Figure 3.7 shows the placed of the sample in test set where the electrodes are fully immersed into the oil.

Figure 3.9 : The gap setting between both electrodes

Figure 3.10 : The placed of the sample in test set

3.6 Total Acid Number Test

Total acid number was tested by using titrator (Metrohm 848 Titrino Plus). This test is required several chemical substances in its process to determine the number of acid in the mineral oil. The chemical substances that involve in this test are Potassium Hydroxide (KOH), Isopropyl Alcohol (IPA), buffer solution and Potassium Hydrogen Phthalate (KHP). Figure 3.8 shows the titrator (Metrohm 848 Titrino Plus) that used to determine the total acid number. The procedure for this test is referred to guide for preparation of total acid number test by Metrohm. Appendix B shows the details about the preparation

Figure 3.11 : Titrator (Metrohm 848 Titrino Plus)

3.6.1 Apparatus Set Up and Calibration

The apparatus such electrode, tubing and stirrer are connected to the test set. Then the burette is rinsed and filled with the KOH in IPA (0.1 mol/L) as titrant.

3.6.2 Calibration and Standardization

The electrode must be calibrated with buffer solution as buffer solution can maintain the pH at constant value. The electrode can be used as the slope with range 97%-103%. This slope value will be displayed on the screen of titrator. When the slope is out of range value, the electrode must be calibrated again with the buffer solution.

3.6.3 Blank Titration

About 20 ml of solvent IPA is measured into titration vessel. Then the solution is titrating with KOH in IPA (0.1 mol/L). The blank titration is performed in order to get the value of titration without any oil sample. This value will be automatically saved into the system as it is used for calculation of Total Acid Number.

3.6.4 Sample Titration

Firstly, 5g of mineral oil are weighed into titration vessel and 20 mL of IPA solvent is added into it. Secondly, the solution is titrating with KOH in IPA (0.1 mol/L). Lastly the result total acid number (TAN) of oil sample will be showed on the screen and been recorded. Figure 3.9 shows the example results of total acid number by using titrator Metrohm (848 Titrino Plus).

Figure 3.12 : Example of Total Acid Number(TAN) results

3.7 Summary

This chapter is discussed about the procedure and methodology of entire experiment test. The experiment that has been done is following the flowchart in order to achieve the objective of the project. At the same time, all the experiment is done in laboratory condition with referring to the national standard test, which is ASTM D6802 for UV-Vis test, ASTM D1816 for AC Breakdown test and ASTM D974 for Total Acid number test. While the method used for reclamation process is based on IEEE Guide for Reclamation of Insulating Oil.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter shows the results recorded during the experiment. To accomplish the objective of this project, the aged mineral oil samples before and after reclamation are prepared for the UV-Vis test, AC breakdown voltage test and Total Acid Number (TAN) test. This chapter also shows the physical change of mineral oil before and after reclamation.

4.2 Comparison of UV-Vis test results between aged mineral oil before and after reclamation process.

The wavelength range between 360 nm until 600 nm was chosen by referring to ASTM D6802. The absorbance unit is a value that indicates the amount of UV-light absorb by absorbing compound, which is the dissolved decay product. As the value of absorbance increase it will indicated the high of dissolved decay product.

At first, there is three preparation of oil sample. First the aged oil before reclamation while secondly the aged oil that undergoes reclamation process for five cycle with used about 5g of fuller's earth and mix with 500ml aged oil. Lastly, the oil sample that undergoes reclamation process for just one cycle but with different quantity of adsorbent and oil which is used about 100g of fuller's earth are mix with 1000ml of aged oil. The different quantity that been chosen is by referring to previous research that made by H.M.Wilhelm et.al and A.Raymond et.al [3,15]. The relative content of dissolved decay product of the oil sample is determined by integrating the area under the curve. Figure 4.2 shows the curve that obtained from the result of

absorbance unit with respect to the wavelength. The wavelength at 420 nm is chosen as references in order to analyse and make comparison between oil sample before and after reclamation process. From the curve obtained that shows in Figure 4.2, the absorbance unit of oil before reclaimed at wavelength 420nm is 2.9929 a.u, while the oil after reclaimed by five cycle with 5g of fuller's earth is 2.771a.u and for the oil that reclaimed for one cycle with using 100g fuller's earth is give value at 2.6919 a.u. From this value, it can be summarised that the amount of absorbing compound that absorb UV light is decreases. This decreases in value of absorbance unit prove and support the statement that the by-products or the dissolved decay product will decrease after reclamation process. The dissolved decay products or by products is formed under oxidation process. The hydorxyl group can be found commonly in insulating paper, which is made from cellullose. The reactions will produce soluble oxidation by-products such as low molecular weight acid. As further reaction, the decay product will form the insoluble compound.

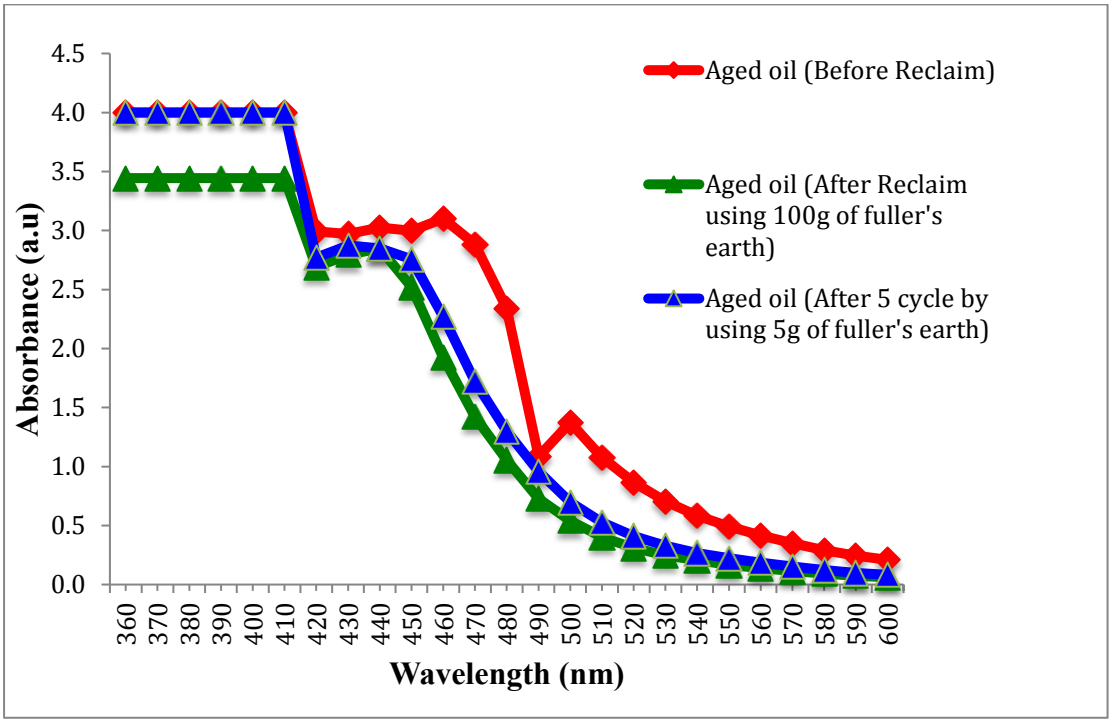


Figure 4.1: Absorbance curves against wavelength (nm)

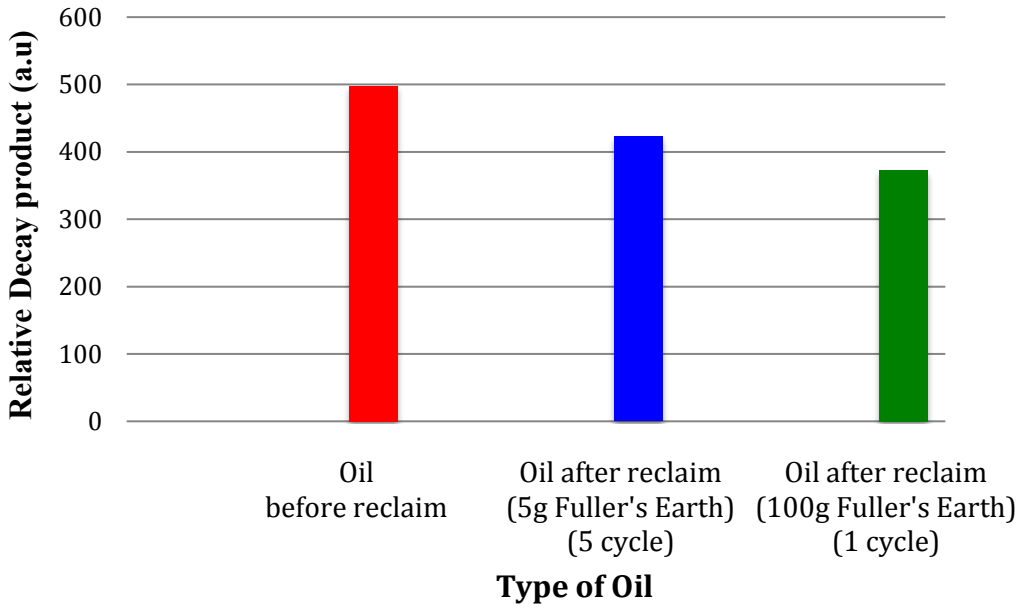


Figure 4.2 : The relative content of dissolved decay product (DDP)

The relative dissolved decay products are represented the areas under the absorbance curve with range at 0 - 4.5 for the absorbance and 360 - 600 nm for the wavelength. Generally, the relative content of dissolved decay product (DDP) is high for the used transformer oil before reclamation, with give value at 498.34 a.u. This has indicated the oil has undergone oxidation during service. Besides after the aged oil is being reclaimed, the results show improvement by decrease gradually in value but the result shown by method that used 100g of fuller's earth for one cycle is more effectiveness than the method that used 5g of fuller's earth with 5 cycle reclaimed, which give value at 372.57 a.u. rather than 422.8625 a.u. This is due to different amount of adsorbent used. For method that used 100g fuller's earth there is much amount of fullers earth that can absorb polar contaminants compare to method that used 5g but repeated with five cycles. So the method that used 100g of fulle's earth with one cycle is chosen to proceed for the AC breakdown voltage test and total acid number test. With referring on result for method used 100g fuller's earth it can prove that reclamation process has reduced 25% of by-product that contain in the oil samples. Already known that the ability of fuller's earth can absorb and block the polar contaminants or polar molecule that dissolved in the oil. For this project, reclamation process

4.3 Comparison of AC Breakdown Voltage Test result between aged mineral oil before reclamation and after reclamation

Before the test, the value of humidity and temperature of room and oil sample is recorded on Table 4.1. The breakdown voltage test result for the base aged mineral oil before undergoes reclamation process and after reclamation processes are provided in in appendices 3. There were 25 test has been made and the Megger oil tester OTS60PB will delivered out average value of breakdown voltage for every 5 test made.

Table 4.1: Humidity and Temperature of room and oil

	Room	Oil
Humidity	70.5%	70.9%
Temperature	30.1℃	30.1℃

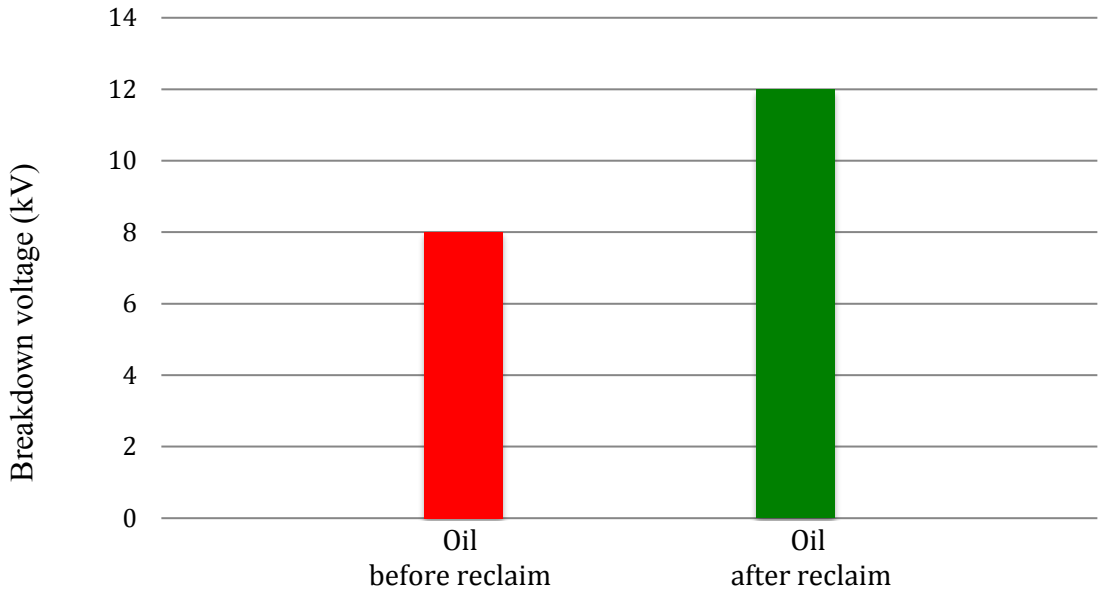


Figure 4.3: Average Breakdown Voltage between aged mineral oil before reclaim and after reclaimed

Based on the Figure 4.1, the graph clearly show that the breakdown voltage value for aged mineral oil are very low compare to the standard limit suggested by IEEE which for the type of transformer that run below 69kV, the limit should 23 kV and above [9], but for the mineral oil after reclaim has shown the increment in breakdown voltage value. The increases of breakdown voltage are approximately 4 kV. It is showed that the breakdown voltage has achieved in increment by 50 %.

From the result shows in Table 4.2 there has 25 tests has been made in order to obtain average value. For every test the result will shown the different value in breakdown voltage. This is because the oil is affected by the humidity, as the humidity of room is not constant for every single time. It is must realize that the breakdown voltage of transformer oil is primarily depending on four factors, which are moisture content, the void present or bubbles, sludge or suspended particles and the acidity of the oil [14]. This increment has proven that the effectiveness of fuller's earth as adsorbent in reclamation process. The percentage of increment in breakdown voltage, depending on the removal of the relative content of dissolved decay product, moisture and contaminants during the reclamation process. It is believed that if the reclamation process is done repeatedly for several cycles, the breakdown voltage will increase steadily. This means that more relative content of dissolved decay product, moisture and contaminants can be remove from aging mineral oil.

4.4 Comparison of Total Acid Number Test result between aged mineral oil before and after reclamation process.

The titrator from Metrohm 848 Titrino Plus was used for determined the total acid number (TAN) in the mineral oil. This test was repeated for three times as to get the average value. Figure 4.4 shows the result of total acid number for aged mineral oil before and after reclamation process.

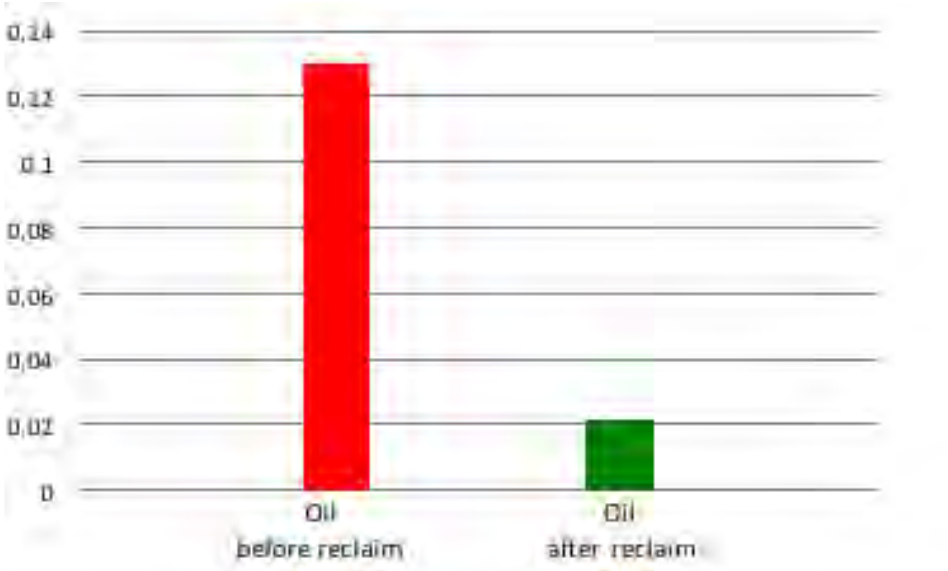


Figure 4.4 : Total Acid Number for oil before reclaimed and after reclaimed

From the results showed that, the average value from three test of total acid number is 0.1302 mgKOH/g, whereas this value is higher than the suggested limit which is 0.1 mgKOH/g for transformer [9]. The higher value of total acid number has proved that there is formation of acid in this aged mineral oil. The formation of acid is due to oxidation process that usually occurred between the dissolved oxygen presence in the oil with the mineral oil molecule itself or with the cellulose that came from the Kraft paper in the transformer. The molecules of the mineral oil mainly consist of hydrogen and carbon and interact with oxygen to form carboxylic acids [20], while insulating paper commonly made from cellulose that contains the hydroxyl group (-OH). According to Bronsted-Lowry acid and Lewis, the hydroxyl group can react with carboxylic acid and water to produce donating proton (H+). This donating proton (H+) can react with other molecule to form acid such as acetic and levulinic acids. Subsequently this decay product reaction will produce sludge, which can cause the dielectric strength become decreases as the breakdown voltage will occurred at the sludge.

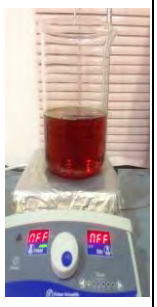
Eventually, after this aged mineral oil undergoes the reclamation process the result of total acid number has been decreased to 0.0212 mgKOH/g of the average value. The effectiveness of fuller's earth as adsorbent for reclamation process has enhanced of total acid number value by 83.7%. This enhancement is helped by the chemical structure of fuller's earth that containing internal and external polar active

sites. It allowed the non-polar substances in the oil to flow but block or adsorb the polar contaminants that dissolved in the oil. All type of acid is a polar molecule as they have opposing charge on their molecule [29]. The decrement of the total acid number that contain in the aged mineral oil after undergoes reclamation process has indicated the effectiveness of fuller's earth as adsorbent in reclamation process.

4.5 Physical Changes of Oil Samples

Commonly the colour of the transformer oil is used as indicator presence of oxidation and the degree of deterioration of the service-aged transformer oil [30]. Usually, service transformer oil is highly exposed to the oxidation due to presence of dissolved oxygen in the oil. Table 4.2 shows the colour of the aged oil for different method and quantity of reclamation process.

Table 4.2 : The colour of oil samples

Reclaim by using 100g of fuller's earth and mixed with 1litre aged mineral oil					
	Before Reclamation			After Reclamation	
Reclaim by using 5g of fuller's earth and mixed with 500 ml aged mineral oil (5 cycle)					
	1st cycle	2nd cycle	3rd cycle	4th cycle	5th cycle

For the reclamation method that used 100g of fuller's earth, the result shows that the aged mineral oil before reclaim has show improvement in colour, which turn from dark brown to light brown while for the method that used 5g of fuller's earth, the colour does not shows further changes compare from the aged mineral oil before reclamation. This is due to lack in amount of fuller's earth as fuller's earth contains magnesium and calcium ion, which is gives high performance in decolourized colour of the aged transformer oil

4.6 Summary

This chapter is discussed about the present result obtain from entire experiment. All the discussions are done in order to achieve the objective of this project. This topic has discussed the effectiveness of the fuller's earth as adsorbents in reclamation process by analyzed the AC breakdown voltage, total acid number and UV-Vis result of the aged mineral oil.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Based on this experiment, investigation on the breakdown voltage, total acid number and the dissolved decay products using UV-Vis spectrometer result for the based aged transformer oil can be concluded that the low performances of the aged oil can cause the failure of transformer. To improve and extend lifecycle of the aged oil, reclamation process is the best way since the mineral oil is a non-renewable resources and highly cost to produced from refining of crude petroleum oil. The effectiveness of the adsorbent has helped in reclamation process to improve the aged transformer oil. From the analysis that has been made, fuller earth has been proved to remove the acid number as well as the dissolved decay product by showing decreasing in total acid number and relative content of dissolved decay product. It also give an improvement of the breakdown voltage. Since fuller's earth has active polar site in its structure molecule, it can attract or adsorb the polar molecule or contaminant that present in the oil. Furthermore the containing of calcium or magnesium as exchangeable ion, which give high performances in decolourized colour of the aged oil. Other than that this adsorbent cannot harmful to environment, so it can be disposed safely on landfill.

5.2 Recommendations

Nevertheless, this project is expected that the improvement of the aged transformer oil can be enhanced and improve by repeated of the reclamation process by using the other adsorbent such as bentonite and activated carbon as to ensure that

the acid, dissolved decay product and insoluble compound is optimally removed. Other than that, this project is proposed to do the reclamation process on the ester oil instead of on mineral oil only. Besides, in order to speed up the time for filtration process, it is suggested to use the fleaker that has bigger in diameter size so that it can be filled with more mineral oil.

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APPENDICES

APPENDIX A- DIFFERENCES BETWEEN IEC AND ASTM STANDARDS [17]

APPENDICES B :GUIDE FOR PREPARATION TOTAL ACID NUMBER TEST BY METROHM

APPENDICES C: RESULTS OF AC BREAKDOWN VOLTAGE TEST