# EFFECT OF NEUTRALIZATION ON THE PROPERTIES OF METHYL ESTER DIELECTRIC LIQUID

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## DECLARATION

I declare that this thesis entitled "EFFECT OF NEUTRALIZATION ON THE PROPERTIES OF METHYL ESTER DIELECTRIC LIQUID is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Date : 5/7/2021

**APPROVAL**

I hereby declare that I have checked this report entitled “Effect of neutralization on the properties of methyl ester dielectric liquid” and in my opinion, this thesis it complies the partial fulfillment for awarding the award of the degree of Bachelor of Electrical Engineering with Honours

Signature : .....

Supervisor Name : Imran bin Sutan Chairul

Date : 05-07-2021

## DEDICATIONS

This report is dedicated to my parents, Latif Bin Omar and Noorazlina Binti Abdullah for always support of finishing this project report for the final year. In addition, they also encouraged me all the way and taught me to believe in Allah in whatever condition we had, especially during the completion of this report. Furthermore, to my family, who always support me with unconditional love, which motivates me to set a higher goal to complete this final year project 1. Next, I devote this study to my supervisor, Ir. Imran Bin Sutan Chairul, who have given opportunity and guided me to finish my final year project 1.

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## ABSTRACT

Previously, waste cooking oil methyl ester have been produced via transesterification process [I. S. Chairul, N. Abu Bakar, M. N. Othman, S. A. Ghani, and M. N. Deraman, "Development Of waste cooking oil methyl ester as potential electrical insulating fluid for power transformer," *ARPN J. Eng. Appl. Sci.*, vol. 13, no. 20, 2018]. However, its acidity is more than the standard requirement of 0.06 mgKOH/g. Thus, this study aim to firstly lower the oil acidity so that it can achieve the standard. Therefore, this research proposes to neutralize waste cooking oil with caustic soda (normality: 3.0 N) for develop methyl ester using sodium hydroxide (NaOH) with percentage of NaOH are 0.2%, 0.4%, 0.6%, 0.8%, 1.0% and 1.2%. It will reduce usage of mineral transformer oil as insulants for industrials, power transmission and distribution transformers insulation system. The waste cooking oil was neutralized using caustic soda with normality 3.0 N at a temperature of 70° and stirring speed at 400 rpm for 5 minutes before do the transesterification process. The suitable weight for NaOH is 0.6% or 3 g in the 500 ml of neutralized WCO and 125 ml of methanol because lowest acidity was achieved for neutralized WCOME. The acidity of neutralized WCOME is 0.2320 mgKOH/g and WCOME from the previous research is 0.25652 mgKOH/g. The WCOME with neutralization and without neutralization effect in acidity measure. The results show that neutralization process give effect in the acidity of the oil. But as for the requirement new natural ester fluid, the acidity of neutralized WCOME not achieve the standard which is 0.06 mgKOH/g. Furthermore, the breakdown voltage of oil was achieve the standard requirement which is more than 20 kV. The neutralization process also change the colour of the oil from dark brown to clear yellowish. As for the transesterification process, the weight of NaOH give influence the yield of oil.

## *ABSTRAK*

Sebelum ini, sisa minyak masak metil ester telah dihasilkan melalui proses transesterifikasi [I. S. Chairul, N. Abu Bakar, M. N. Othman, S. A. Ghani, and M. N. Deraman, "Development Of waste cooking oil methyl ester as potential electrical insulating fluid for power transformer," *ARPN J. Eng. Appl. Sci.*, vol. 13, no. 20, 2018]. Walau bagaimanapun, keasidannya lebih daripada 0.06 mgKOH/g mengikut piawaian. Oleh itu, kajian ini bertujuan untuk menurunkan keasidan minyak terlebih dahulu sehingga dapat mencapai piawaian yang telah ditetapkan. Oleh itu, penyelidikan ini mencadangkan untuk mengaslikan sisa minyak masak dengan soda kaustik (normaliti: 3.0 N) untuk menghasilkan metil ester menggunakan natrium hidroksida (NaOH) dengan peratusan NaOH adalah 0,2%, 0,4%, 0,6%, 0,8%, 1,0% dan 1,2 %. Ia akan mengurangkan penggunaan minyak mineral sebagai penebat untuk industri, sistem penebat pengubah dan transmisi kuasa. Sisa minyak masak diaslikan menggunakan soda kaustik dengan normaliti 3.0 N pada suhu 70° dan kelajuan pengadukan pada 400 rpm selama 5 minit sebelum melakukan proses transesterifikasi. Berat yang sesuai untuk NaOH ialah 0.6% atau 3 g dalam 500 ml WCO yang diaslikan dan 125 ml methanol kerana keasidan terendah dicapai untuk WCOME yang diaslikan. Keasidan WCOME yang diaslikan adalah 0.2320 mgKOH/g dan WCOME dari penyelidikan sebelumnya ialah 0.25652 mgKOH/g. WCOME dengan peneutralan dan tanpa kesan peneutralan dalam ukuran keasidan. Hasil kajian menunjukkan bahawa proses peneutralan memberi kesan keasidan minyak. Tetapi untuk keperluan cecair ester semula jadi baru, keasidan WCOME yang dinetralkan tidak mencapai standard iaitu 0.06 mgKOH/g. Seterusnya, voltan minyak telah mencapai keperluan standard yang melebihi 20 kV. Proses peneutralan juga mengubah warna minyak dari coklat gelap menjadi kekuningan. Bagi proses transesterifikasi, berat NaOH memberi pengaruh kepada penghasilan dalam minyak.

## TABLE OF CONTENTS

## LIST OF TABLES

## LIST OF FIGURES

## LIST OF SYMBOLS AND ABBREVIATIONS

| | | |
|---|---|---|
| WCO | - | Waste Cooking Oil |
| WCOME | - | Waste Cooking Oil Methyl Ester |
| NaOH | - | Sodium hydroxide |
| PCBs | - | Polychlorinated Biphenyls |
| BdV | - | Breakdown Voltage |
| DDF | - | Dielectric Dissipation Factor |
| FAME | - | Fatty Acid Methyl Ester |
| DDP | - | Dissolved Decay Product |

## LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

## 1.1 Overview

Transformers in electric power distribution and transmission systems are expected to perform reliably and efficiently in order to meet human needs. The features of transformer oil have been studied and reported on for decades, and the quality of transformer oil plays an important role in completing this function [1]. Transformer oil is use for insulator as well as coolant medium of transformers. One of the best types of fluid is polychlorinated biphenyls (PCBs) as it is not flammable but PCBs compounds do not breakdown when released into the environment. The PCBs can build up in plant and animal tissues that can cause cancer, serious deformities and also can release other highly toxic compounds when burned. As a result, PCBs were banned for used as insulator in electrical component. Other than PCBs, petroleum based mineral oil is also being used as insulating oil for transformer. Mineral transformer oil is cheap and reliable insulants for industrial, power transmission and distribution transformers insulation system. Due to its good dielectric properties, mineral oil has been used as heat transfer medium and insulating liquid in the power transformers for more than 100 years. However, mineral oil still had a negative effect on the environment as dissolve PCBs so it is often became contaminated. Mineral oil also has low fire point can easily cause explosion. There are two types of transformer oil that are commonly used which are paraffin oil and naphtha oil. Mineral oil is now slowly being replace with ecofriendly insulating liquids, derived from vegetable oil. Vegetable oil have two types which are triglyceride and low viscosity natural ester. There a lot of benefit if vegetable oil can replace mineral oil as insulator on transformer. The advantages vegetable oil as insulator can definite environmental, safety gain and high fire point compare to mineral oil.

## 1.2 Motivation

Due to regular enhance the use and demand of cooking oil day by day that want to fulfill human needed, it was produced many waste cooking oil (WCO). In 2019, approximately 574.48 thousand metric tons of WCO was produced in Malaysia [3]. A lot of people not know that WCO can be sell because not many campaign about WCO were held. The average price for WCO is RM1.00 – RM1.50 perkg. So, WCO not only can be recycle but also can get money from selling it and can prevent from clogged sink. Instead of throw away or just keep it, the WCO can be reused as transformer insulating oil with several process.

## 1.3 Problem Statement

Apart from that, WCO can be renewable to use as transformer oil with process neutralization and transesterification. After several process WCO will become waste cooking oil methyl ester (WCOME). Previously, waste cooking oil methyl ester have been produced via transesterification process by I. S. Chairul, N. Abu Bakar, M. N. Othman, S. A. Ghani, and M. N. Deraman from paper "Development Of waste cooking oil methyl ester as potential electrical insulating fluid for power transformer" [4]. However, its acidity is more than the standard requirement of 0.06 mgKOH/g. Thus, this study aim to firstly lower the oil acidity so that it can achieve the standard. By means of that, first is neutralization waste cooking oil (WCO) and second is variation weight of sodium hydroxide (NaOH). Other than definite environmental, WCOME also have high flash point and high fire point rather than use mineral oil. It will reduce usage of mineral transformer oil as insulants for industrials, power transmission and distribution transformers insulation system. The study of WCOME can improve the properties, especially to get the acidity below than 0.06 mgKOH/g to achive the specification and can get high quality of WCOME.



## 1.4 Objective

The objective that must be accomplish in this project are:

1) To neutralize waste cooking oil using caustic soda with normality 3.0 N.

2) To develop methyl ester using sodium hydroxide (percentage of used NaOH: 0.2%, 0.4%, 0.6%, 0.8%. 1.0%, 1.2%).
3) To measure the acidity of new develop waste cooking oil methyl ester.

## 1.5 Scope of Study

To achieve an aim of the research, the specification based on standard BS EN 62770 has been used. This study should focusing on properties transformer oil of waste cooking oil methyl ester which are acidity. The scope of this properties are acidity complying ASTM D664, breakdown voltage complying D1816 and colour complying ASTM D1500.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Overview

This chapter will discuss the effect of neutralization on the properties of methyl ester dielectric liquid to gain the knowledge for this study. This chapter will compare the method to produce WCOME that involve in order to get the best outcome. This study is start by studying transformer oil and the method to produce WCOME before know the specification of transformer oil.

## 2.2 Transformer Oil

Transformer oil is the oil that can withstand high temperatures. It also has the best insulating properties, the best degeneration stability, a low number of vitality, which is a substance's propensity to evaporate at normal temperatures, and the least amount of grease formation. Transformer oil, also known as coolant, is used in oil-filled electrical power transformers as an insulant, to prevent arcing and corona discharge, and to dissipate the heat generated by the transformer [5]. Cade [6] briefly states that the transformer oil mainly serves two purposes: first, it acts as liquid insulation in electrical power transformers, and second, it eliminates heat produced by the transformer and serves as a coolant. Furthermore, if an oil transformer is a good insulator, the performance of the transformer improves, lowering maintenance costs while increasing travel distance. Transformer oil is also used to keep the transformer's core and windings in good working condition because they are completely absorbed by oil. Another significant advantage of insulating oil is its ability to prevent cellulose oxidation. Figure 2.1 shows an insulating oil that acts as a barrier between atmospheric oxygen and the cellulose, preventing direct contact and thus reducing oxidation at the cellulose's wall.

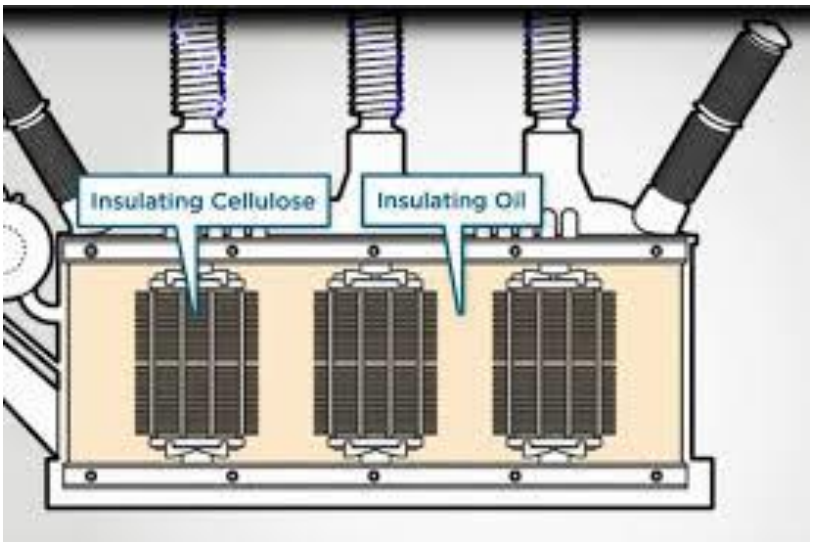


**Figure 2.1:** Transformer

### 2.2.1 Mineral Oil

All country used mineral oil for variation purpose along with innovation from time to time. Mineral oil widely used such as in biomedicine, mechanical, electrical, and food preparation. Mineral oil, as compared to typically edible vegetable oil, has various colourless, odourless, light mixtures of higher alkanes from a mineral source and petroleum. Veterinarian use mineral oil as mild laxative for pets and livestock. As an adjuvant, a livestock vaccine is used to stimulate a cell-mediated immune response to the vaccinating agent. In other field just like cosmetic used mineral oil to make baby lotion, cool creams and etc.



Mineral oil cannot conduct electricity and has poor conductor heat because of that mineral oil used as transformer oil in electrical component. In addition, mineral oil acts as a preservative for lithium and other alkali metals by preventing moisture absorption from the air [7]. Two types mineral oil commonly used as transformer oil which are paraffin oil and naphtha oil. In comparison to naphtha oil, paraffin-based oil is still widely used in many countries due to its wide availability. The main reason why mineral oil widely used in many countries because low cost. Mineral oil also has high voltage insulation and can solve the problem of transformer heat dissipation due to its large capacity [8]. The disadvantages of mineral oil is low biodegradability potential because oil will leakage at serous in operation place and may affect the safe of equipment. Mineral oil also is flammable cause burn and explode when it comes to

high temperature. The disadvantages of mineral oil have made it necessary to look for other insulating liquids such as vegetable oil.

### 2.2.2 Natural Ester

Vegetable oils were produced natural ester which are manufactured from plant crops. Vegetable oil-based transformer fluids are gradually replacing mineral oil-based products on the market [9]. Vegetable oils, particularly marine lubricants, can be utilized as lubricants in natural forms for industrial and mechanical lubrication. Using vegetable oils can be advantageous because it is provide better lubrication when compared to mineral oil. Vegetable oil can perform better than mineral oil products because it can provide definite environmental, safety gains, high flash point and less flammable compare to mineral oil. Vegetable oils are being studied by many researchers and industries as an alternative to insulating oils in transformers. Soybean, corn, rapeseed, and sunflower oil are the most popular sources of natural esters for electrical applications. Due to considerations like as availability, pricing, and performance qualities, this is the situation [10]. However, crude vegetable oil have disadvantages properties such as high pour point. High pour point of transformer oil mainly depends upon wax in the oil that associated with high paraffin content. Paraffin oil has a higher value of pour point compared to Naphtha oil. However, due to India's warm environment, it has no effect on the use of paraffin oil [5]. So, the using of oil with high pour point in electrical device is based on temperature condition.



## 2.3 Processing Waste Cooking Oil for Insulating Purpose

As discuss in other section, WCO should be done several process before can measure the properties of transformer oil. WCO is obtained after frying using eatable vegetable oils multiple times. WCO was chosen for this investigation because it is three times cheaper than crude vegetable oil, therefore lowering the cost. To achieve best quality of WCOME, the process must be done with carefully and cautiously. If the process not done with care and caution, the oil can failure to flow at the pour point. This could be due to viscosity or the specimen's previous heat history. As a result, the pour point may deceive consumers about the oil's handling capabilities. The base oil is either hydrogenated or has a greater oleic acid content to provide optimal oil

stability. To eliminate crystallized lipids and enhance the pour point of the base oil, the oil can be winterized [11]. For better pour point, dielectric stability, and cooling qualities, the vegetable oil can be blended with an additive package combining ingredients and several processes.

### 2.3.1 Triglycerides - Purification

A triglyceride is an ester made up of three fatty acids and glycerol. Figure 2.2 shows that chemical structure of a triglyceride has chain of glycerol, ester bond and three fatty acid. Triglyceride is the main constituents in vegetable fat, body fat in human and etc. Triglyceride has to do purification to remove free fatty acid. Purification have three process which are degumming, neutralization and bleaching.

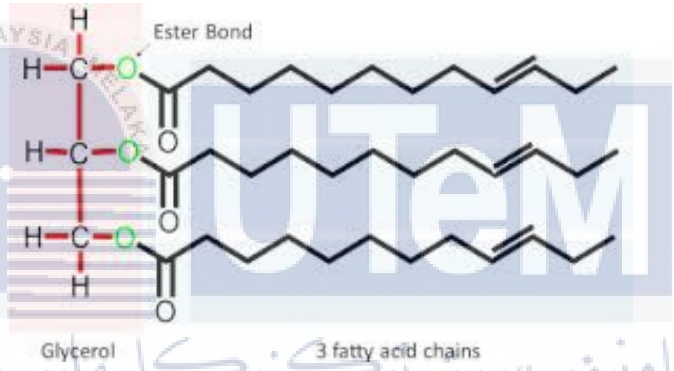


**Figure 2.2:** Chemical structure of a triglyceride



The removal of phosphatides, gum, or lecithin from crude soybean and other vegetable oils is known as degumming. Phosphatides make up the majority of the gums, although gums also contain entrained oil and meal particles. Water absorbs into the oil, causing some phosphatides to become hydrated and hence oil insoluble. As a result, before storing the oil, drying the gums and removing the hydrated gums from the oil can help prevent the formation of a gum deposit. Water degumming is the term for this procedure. It is never used on fruit oils such as olive and palm oil since these oil have already come into contact with water during manufacture [12]. Since the water degumming process involves more water the gums resulting from the water degumming process can remove hydrophilic substances such as sugar from the oil. Quality issues can occur if gums are not removed before the oil is exposed to the high temperatures required in deodorization or deacidification. This is because the gum can

make troublesome emulsions, resulting in a considerable waste of oil that could otherwise be used to generate money.

After degumming completion, second process to do purification is neutralization. Neutralization is the action of making something chemically become neutral. A chemical reaction in which an acid and a base react quantitatively with each other is known as neutralization. Neutralization is carried out to reduce the amount of free fatty acids in the oil because the high content of free fatty acids will accelerate the hydrolysis process cause an aging mechanism of vegetable oil [13]. The base is used to neutralize free fatty acids and eliminate acidity from the oil. The resultant soaps will be dilute in a water phase as a result of this method. To safonify the free fatty acid, caustic soda is commonly employed as the base in the oil business. In addition, some processors use potassium hydroxide as a base. The free fatty acid is stearic acid, which separates from a triglyceride molecule in the oil to form a diglyceride. The feedstock is introduced into the neutralization process, where the heating temperature is kept between 55°C and 70°C depending on the quality of the crude vegetable oil. To maintain consistency, the oil must be constantly agitated [14]. The H+ of the carboxyl group gives the free fatty acid (RCOOH) its acidity. This H+ of the stearic acid functional group reacts with the OH- group of the caustic soda (NaOH) to produce soap (RCOONa) and water. Figure 2.3 shows that reaction of neutralization.




**Figure 2.3:** Neutralization reaction of free fatty acids

Neutralization is an important step in determining the WCO's quality. If the neutralization isn't done correctly, the rest of the processes will be incorrect. The oil will be affected by acidity removal, colour removal, moisture removal, and gum removal. After that, the neutral oils are bleached and deodorized. Chemical refining process steps can be adjusted according to crude oil quality to produce a satisfactory end oil quality with acceptable oil losses.

Bleaching process is one of the important processes after complete effective neutralization in WCO refining. In the palm oil refining business, an adsorbent known as bleaching clay is widely used to absorb undesirable colour pigments inside vegetable oil [15]. The neutralised WCO is heated to a higher temperature using

thermic boilers, bringing the oil temperature up to 120°C to 130°C. The oil is next bleached with clays that absorb the colour pigments and lower the chlorophyll concentration. The amount of bleaching clay added varies depending on the oil and the colour level of the crude oil. The bleaching process is considerably important because can achieving high quality refined oil products and the economic viability of the oil purification process. Efficient bleaching can remove traces of phosphatides, remove certain pigment, partially remove oxidation product and remove other pollutants [16]. The bleaching have several type such as dry bleaching and wet bleaching. Dry bleaching is done in the presence of sparking steam and under vacuum to prevent oxidation. The bleaching reactor operates at a vacuum of around 70 torr, which considerably lowers the humidity in the oil, making this a dry bleaching process. Water is introduced to the oil while it is in contact with the bleaching agent in the bleaching reactor for wet bleaching. The water used in wet bleaching is introduced in the form of a citric acid solution or by transferring wet oil from the separation line. Adjustment is carried out by careful control of the operating vacuum in the bleaching reactor to determine how much of the water subsequently evaporates. As a result, before filtration, the oil must be dried. After the bleaching process is completed, the crude vegetable oil is filtered. The goal of filtration is to remove used chemicals from the oil. Another example is bleaching, which consists of two steps which are the contact process and the filtration process. A certain amount of adsorbent is mixed with the used transformer oil during the contact process [17]. A digital hot plate magnetic stirrer was used to mix the used transformer oil in this bleaching. Table 1 shows how the weight of the Fuller's Earth adsorbent, stirring speed, and oil temperature for the contact process were determined using the L4 orthogonal array. Following that, the oil mixture was separated using filter paper in the filtration process to remove sludge such as adsorbents and dissolved decay products found in used transformer oil. The filtration process employs Whatman No. 42 filter paper with a pore size of 2.5 µm.

| Experimental run | Reclamation process parameters | | |
|---|---|---|---|
| | Weight of Fuller's Earth adsorbent (g) | Stirring speed (rpm) | Oil temperature (°C) |
| 1 | 50 | 500 | 60 |
| 2 | 50 | 1000 | 80 |
| 3 | 100 | 500 | 80 |
| 4 | 100 | 1000 | 60 |

**Table 1:** L4 ($\mathbf{2^3}$) orthogonal array for the reclamation process [17]

### 2.3.2 Low viscosity natural ester - Transesterification

Transesterification is the reaction of a fat or oil with an alcohol to produce esters and glycerol. A catalyst is used to increase the rate and yield of the reaction. The addition of an acid or alkali catalyst frequently catalyzes these reactions. It can be also be done with the help of enzymes such as lipases. The transesterification process will convert the free fatty acids of waste cooking oil in the present of methanol to methyl ester and modifies the properties of waste cooking oil to become suitable oil for transformers [18]. Enzyme transesterification is an attractive method since it's can be performed under low temperature, do not form soaps, reduced environmental impact and energy requirement in chemical industrial processes. However, enzyme transesterification has several disadvantages such as high-cost production, low reaction rate, longer reaction time and higher catalyst cost. The acid catalyst offers a low-cost catalysts, no soap formations and higher yield. This provides advantages compared to enzyme catalyst in terms of yield and catalyst cost. Though, the main drawback of acid catalyzed are very slow reaction rate, higher cost of equipment, environmental effect and higher temperature required [19]. However, alkali catalysts are potentially to overcome the limitation of the acid catalyst. Alkaline catalyst includes sodium hydroxide (NaOH), sodium methoxide (NaOCH3), potassium hydroxide (KOH) and potassium methoxide (KOCH3). NaOH and KOH catalysts are the most common catalysts used in the transesterification reaction process because of the cheaper making process, economical catalyst cost in the market. Alkaline catalyst also able to react at low temperature and atmospheric pressure, reacts faster and can achieve a high conversion yield in a short time [20]. Alkaline catalyst is considered as