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CHARACTERISTICS OF POLYOL ESTER AS TRANSFORMER INSULATING OIL

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By

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CHARACTERISTICS OF POLYOL ESTER AS TRANSFORMER INSULATING OIL

By

NURLIYANA BINTI ABDUL RAOF

January 2021

Chairman : Prof Robiah binti Yunus, PhD
Faculty : Engineering

Mineral-based transformer oil has long served the purposes of insulating and cooling in an electrical power transformer. However, its low flash and fire point constitute a high risk for fire and explosion and not suitable to be used in densely-populated and environmentally sensitive areas. The overall objective of this study is to develop an environmentally friendly polyol ester that is practical and suitable for the transformer application. Specifically, the effect of polyol esters with different chemical structures on the electrical properties and oxidative degradation were investigated. Different chemical structures that can be produced from the transesterification of various methyl esters (C8/C10, C12 and C18) with neopentylglycol (NPG)/ trimethylolpropane (TMP) alcohols were synthesized and compared with those of commercial synthetic ester PFAE and mineral oil. Based on the evaluation of physicochemical and electrical properties, TMPE C18 exhibited the most optimum insulation properties with excellent flash point and moisture content, excellent breakdown voltage, relative permittivity, dissipation factor and resistivity. Experimental investigations on the oxidative characteristics and catalytic effects of copper/iron on different esters were also conducted by using the turbine oil oxidation test (TOST). The unadditived TMPE C18 ester showed a comparable oxidative performance to the commercial ester PFAE and the most viable option for ester-based transformer oil. TMPE C18 exhibited high stability against oxidation by having almost similar oxidation lifetime as PFAE, maintains low acidity even after oxidation duration and does not corrode copper or iron. The effects of different ester and mineral oil blending composition were further investigated to improve the current properties of base oil. C18 TMPE sample was blended and homogenized with 20 to 50% of mineral oil. It was found that the 20MO (80/20 of C18 TMPE/mineral oil) blending sample has the properties near to the standard values for transformer liquid insulation. The effects of different antioxidants were studied too. Blends containing Irganox L57 antioxidant showed a remarkable oxidation lifetime compared with other antioxidants. However, the use of Irganox L57 contributed to increased concentration of copper and iron after oxidation in oil thus gives comparable performance to untreated 20MO blend. The thermal aging behavior of insulating paper aged in unadditived 20MO

blend at 110, 130 and 150°C for 14, 28, 42, 56 and 84 days were evaluated and compared with that of mineral oil. The overall results showed that paper aged in mineral oil degraded significantly faster than in the 20MO blend. The kinetics study on the Kraft paper degradation predicted the loss of tensile strength and degree of polymerization aged in blend and mineral oil and the proposed kinetics model fitted well with the experimental data. The blend showed good thermal ageing characteristics compared to the conventional mineral oil.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

CIRI-CIRI ESTER POLIOL SEBAGAI MINYAK PENEBAT PENGUBAH

Oleh

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Minyak pengubah berasaskan galian telah lama berfungsi sebagai penebat dan penyejuk dalam pengubah kuasa elektrik. Walau bagaimanapun, titik kilat dan api yang rendah memberi risiko tinggi untuk kebakaran dan letupan dan tidak sesuai digunakan di kawasan yang berpenduduk padat dan sensitif terhadap persekitaran. Objektif keseluruhan kajian ini adalah untuk membangunkan ester poliol yang mesra alam yang praktikal dan sesuai untuk aplikasi pengubah. Secara khususnya, kesan struktur kimia ester poliol yang berbeza terhadap sifat-sifat elektrik dan penurunan beroksida telah disiasat. Perbezaan struktur kimia yang boleh dihasilkan daripada transesterifikasi pelbagai ester metil (C8/C10, C12 dan C18) dengan alkohol neopentil glikol (NPG)/ trimetilolpropana (TMP) telah disintesis dan dibandingkan dengan ester sintetik dagangan PFAE dan minyak galian. Berdasarkan penilaian sifat-sifat kimiafizik dan elektrik, TMPE C18 telah mempamerkan sifat-sifat penebat yang optimum dengan titik kilat dan kandungan lembapan yang sangat baik, voltan keruntuhan, kebertelusan nisbi, faktor lesapan dan keberintangan yang sangat baik. Siasatan ujikaji mengenai ciri-ciri oksidatif dan kesan mangkin kuprum/besi pada ester poliol yang berbeza juga telah dilakukan dengan menggunakan ujian pengoksidaan minyak turbin (TOST). Ester TMPE C18 tanpa bahan tambah telah menunjukkan prestasi pengoksidaan yang setanding dengan ester sintetik dagangan PFAE dan menjadi pilihan yang paling sesuai untuk minyak mengubah berasaskan ester. TMPE C18 telah menunjukkan kestabilan tinggi terhadap pengoksidaan dengan mempunyai masa hayat pengoksidaan yang hampir sama dengan PFAE, mengekalkan keasidan yang rendah walaupun selepas tempoh pengoksidaan dan tidak menghakis kuprum atau besi. Kesan kerencaman pengadunan ester dan minyak galian yang berbeza telah disiasat lebih lanjut untuk memperbaikkan sifat-sifat minyak asas sedia ada. Sampel C18 TMPE telah diadun dan dihomogenkan dengan 20 hingga 50% minyak galian. Telah didapati bahawa sampel adunan 20MO (80/20 daripada C18 TMPE/minyak galian) mempunyai sifat-sifat yang paling hampir dengan nilai piawai untuk cecair penebat pengubah. Kesan antibahan pengoksidaan yang berbeza telah dikaji juga. Adunan yang mengandungi antibahan pengoksidaan Irganox L57 menunjukkan masa hayat pengoksidaan yang luar biasa berbanding dengan antibahan pengoksidaan lain. Walau bagaimanapun, penggunaan

Irganox L57 telah menyumbang kepada peningkatan kepekatan kuprum dan besi didalam minyak selepas pengoksidaan dan ini memberikan prestasi yang setanding dengan adunan 20MO tanpa antibahan pengoksidaan. Tingkah laku penuaan haba kertas penebat di dalam minyak adunan 20MO yang tidak ditambah antibahan pengoksidaan telah dinilai dan dibandingkan dengan minyak galian pada suhu 110, 130 dan 150°C selama 14, 28, 42, 56 dan 84 hari. Hasil keseluruhan menunjukkan bahawa kadar penurunan kertas penebat jauh lebih cepat di dalam minyak galian berbanding dengan di dalam adunan 20MO. Kajian kinetik pada penurunan kertas Kraft meramalkan kehilangan tegasan tegangan dan darjah pempolimeran di dalam minyak adunan dan minyak galian dan model kinetik yang dicadangkan sesuai dengan data ujikaji. Adunan 20MO juga telah menunjukkan sifat-sifat penuaan haba yang baik berbanding dengan minyak galian lazim.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

NPG	Neopentylglycol
TMP	Trimethylolpropane
2EHE	2-Ethyl Hexyl Ester
NPGD	Neopentylglycol Diester
TMPE	Trimethylolpropane Triester
PFAE	Palm Fatty Acid Ester
MO	Mineral Oil
RBDPO	Refined, Bleached and Deodorized Palm Oil
TOST	Turbine Oil Oxidation Stability Test
TGA	Thermogravimetric Analysis
OIT	Oxidation Induction Time
BHA	Butylated Hydroxyanisole
BHT	Butylated Hydroxytoluene
NAPA	*N*-Phenyl-1-Naphthylamine
DGA	Dissolved Gas Analysis
FTIR	Fourier-Transform Infrared Spectroscopy
TAN	Total Acid Number
BDV	Breakdown Voltage
DDF	Dielectric Dissipation Factor
OL	Oxidation lifetime
DBPC	2,6-Ditertiary-Butyl Paracresol
GC	Gas Chromatography
HPLC	High-Performance Liquid Chromatography

ED-XRF	Energy Dispersive X-ray Fluorescence
CED	Copper (II) Ethylenediamine
KOH	Potassium Hydroxide
HMWA	High Molecular Weight Acid
LMWA	Low Molecular Weight Acid
DP	Degree of Polymerization
TS	Tensile Strength

LIST OF NOTATIONS

$\tan \delta$	Dielectric dissipation factor
k	Rate constant
k_{TS}	Tensile strength degradation rate constant
k_{DP}	Degree of polymerization degradation rate constant
A	Pre-exponential factor
E_A	Activation energy
t	Ageing time
TS_0	Initial tensile strength
DP_0	Initial degree of polymerization
ω^*_{TS}	Capacity of tensile strength degradation reservoir
ω^*_{DP}	Capacity of degree of polymerization degradation reservoir

CHAPTER 1

INTRODUCTION

1.1 Research background

Transformer is basically one of the most critical, expensive and strategic assets in electrical transmission and distribution. The performance of transformer depends heavily on the performance of its insulation system which consists of insulating oil and insulating paper. Mineral oil has been used for cooling and insulation purposes in power transformers for over ten decades (Evangelista Jr. et al., 2017; Gnanasekaran and Chavidi, 2017). The modern insulating liquid development is driven by a number of considerations, such as increased fire protection, material sustainability, environmental friendliness and prolonged service life of the transformer (Fofana, 2013; Rafiq et al., 2016).

In recent years, substantial efforts have been explored to develop high performance eco-friendly insulating liquids to replace conventional petroleum-based mineral oil. Polychlorinated biphenyl (PCB)-based insulating liquids were once marketed for high fire safety requirements, but were outlawed in the 1970s due to the health and environmental risks (Fofana, 2013). The phasing out of a PCB led to development of other halogenated insulating fluids, such as benzyltoluene, perchorethylene, trichlorobenzene and dichlorotoluene, which are considered non-flammable. 'Less flammable' insulating liquids have been established for transformers at major fire risk locations and are primarily high molecular weight hydrocarbons, silicone oil, synthetic esters and natural (vegetable) esters (McShane et al., 1999).

Natural esters originate from plants and seeds. Natural ester has been one of the most interesting research topics in recent decades thanks to its 'green' credential (Rycroft, 2014). However, despite their many benefits relative to mineral oils and synthetic esters, natural esters have two main drawbacks; inferior oxidative stability and high pour point (Salimon et al., 2014). The chemical modification by elimination of beta-hydrogen in the natural ester molecule is an option to improve their thermal characteristics and this modification is called esterification/transesterification (Liu et al., 2014). The transesterification reaction involves the replacement of the glyceride moiety in the natural ester molecule with a polyhydric alcohol (such as neopentylglycol (NPG) and trimethylolpropane (TMP)) to produce esters called polyol esters, widely known as synthetic esters (Liu et al., 2014). Polyol esters which were invented in the beginning of 1960s are now applied for various applications such as refrigeration compressors, aviation, greases, air compressors, metalworking, fire resistant and biodegradable hydraulic liquids and chain oils (Lucazeau, 2016). Polyol ester exhibits exceptional stability particularly as it does not have beta-hydrogens and is thus beta-hindered (Rudnick, 2013; Totten et al., 2003; Yunus et al., 2005).

The ester-based insulating liquids synthesized from vegetable oil can mitigate the disadvantages of mineral transformer oil, such as its adverse environmental effects and high flammability. Esters are more environmentally friendly and have lower risk of fire and explosion. However, owing to the high polarity of ester, the dielectric properties of the ester liquids are not as excellent as the mineral oil. Mixing esters with mineral oil is mostly performed to acquire the most of the properties of ester and mineral oil. The use of mineral oil/ester blend as transformer dielectric liquid would benefit the utilities in terms of performance and life cycle cost over traditional mineral oil-based fluids.

Like all other organic materials in archives and libraries, old books and documents, insulation papers in oil-filled transformers are subject to several fundamental degradation processes. The degradation of paper reduces its mechanical strength, making it susceptible to damage and failure. Other changes include the change in chemical structure and the formation of degradation by-products (Ding, 2009). There are several published studies that demonstrated the aging of insulation paper in transformer oil (Abdelmalik et al., 2013; Feng et al., 2019; Hao et al., 2018; Liao et al., 2019, 2011a). Most of the studies revealed slower ageing rate of paper in vegetable-based esters than conventional mineral oil. The present study explores the influence of temperature and time as primary aging factors with the presence of copper in the prolonged thermal aging experiments.

1.2 Problem Statement

Mineral oils have been used in liquid-filled transformers for over hundred years. These oils, which are a mixture of different types of hydrocarbon compounds, may pose a significant ecological danger in the event of spillage or leakage (Rozga, 2013). The mass of oil in a standard 110kV power transformer is about 7-8 thousand kilograms while in the 400kV transformer such mass is nearly 80 thousand kilograms. There is no doubt that the effects of oil spillage are harmful especially if the oil spills into soil, water and drain. Adequate oil containment systems or installation of oil panes at the base of transformer could avoid or reduce the impact, but the anticipated cost of installing them is worth considering (Rozga, 2013). Although the occurrence and severity of oil spills may be considered minimal, the danger persists, and the problem of controlling the spill can be very challenging and costly (McDonald, 2016). Apart from leakage issues, great emphasis has been put on disposal of waste or mineral transformer oil due to its non-biodegradability (Mehta et al., 2015). The combustibility of mineral oil was another major problem, because of its comparatively low flash and fire point (Rozga, 2013).

There are several studies in the literature reporting the application of polyol ester as the insulating fluid (Hof et al., 2008; Kanoh et al., 2012, 2008; Qiu and Brown, 2013; Raof et al., 2016). However, for transformer applications, most research centred on esters' physical and chemical characteristics such as viscosity, flash point, pour point, moisture level, and acidity. Due to the constant exposure of these natural esters to air and moisture, issues of oxidative and hydrolytic stabilities of these esters are of great concern. In addition, most of these esters are made up of long carbon chain molecules ranging from C30 to C56, hence the chemical structure may affect the chemical, mechanical and electrical properties of the intended applications. No reports have been found so far on the effect of chemical structure on the electrical properties of transformer oil. The earlier report on the conductivities and breakdown of aromatic compounds with different number of benzene rings have underlined the importance of studying the effect of chemical structures on electrical properties (Forster et al., 1991). Thus, as observed from prior studies, the chemical structure plays a significant role in electrical properties than was originally postulated.

Oxidation is the main degradation reaction that accelerates the aging of transformer oil (Filho et al., 2019; Rouabeh et al., 2019). The oxidation of transformer oil often produces insoluble sludge that will result in reduced heat transfer capabilities, overheating and reduced service life. It has been established that the relative stability towards oxidation in decreasing order is as follows: silicone oil>synthetic ester>mineral oil>natural ester (CIGRE, 2010; Fernández et al., 2013). Whilst mineral oil is relatively a stable substance, studies have found that mineral oil will start to degrade if it is heated above 175°C (Santos et al., 2006). In the presence of oxygen and metal, the oxidation could occur at a much lower temperature (Filho et al., 2019; Randles, 2013; Ushakova et al., 2017). However, due to the difference in the chemical structures, the oxidation process and oxidation products of synthetic polyol esters are slightly different and more complicated compared to the mineral oil and natural esters (Wu et al., 2013a).

There are limited literatures on the effects of blending ester and mineral oil in transformer oil particularly on the electrical properties and oxidative stability of the blends. As the demand for cleaner fuel grows, the insulating liquid (transformer oil) manufacturer will be forced to look for environmentally friendlier liquid and blending with esters is a commercially viable option. However, there are many esters in the market with different chemical structures and performance characteristics. The oxidation behavior of these liquids is directly linked to its compositions and structures. However, it should be noted that the oxidation behavior of for mineral oil-ester blends could be different from that of their original compounds. While it is possible to estimate certain properties that tend to blend linearly, there is no general rule of additivity which applies to oxidation of blends (Stepina and Vesely, 1992; Totten et al., 2003). So far, the research on ester-based transformer oils and its blend with mineral oil only focused on the physical, chemical and electrical stability of these individual oils only. However, far too little attention has been given to the detailed study on the effects of chemical structure of those blends. Futhermore, the effects of different antioxidants on the oxidative degradation of the mineral oil-ester blends have yet been assessed.

As transformer oil ages, the insulation paper is also subjected to irreversible ageing phenomena. The combined effects of heat, oxygen and moisture which are all present in an operating transformer oil will accelerate the degradation/ageing process (Munajad et al., 2017). Over time, the paper tends to degrade and loses its mechanical strength as well as its ability to insulate the windings (Lelekakis et al., 2014b). Due to the hygroscopicity nature of polyol ester, it can easily absorb the moisture from the insulation paper, hence the hydrolytic degradation of paper can be minimized. In addition, polyol ester can convert cellulose in the paper into blocking groups that further inhibit the paper degradation process (Rapp et al., 2012). However, very few studies have been conducted to elucidate the thermal aging mechanism of polyol ester to convince the utilities to use ester-based insulating oil as an alternative to mineral oil. Moreover, to estimate the degradation rate of new insulating fluid and insulation paper, the kinetic models need to be established, taking into account the properties of the new materials. Particularly, very few reports detailing the effect of copper on the oil-paper insulation system.

1.3 Objectives

The main objective of this study is to assess the potential of vegetable oil-based polyol esters as a transformer oil. The specific objectives are;

1. To assess effect of different chemical structure of polyol esters on physicochemical properties, electrical insulation properties and oxidative stability
2. To investigate effects of blending ratio of selected polyol ester with mineral oil and additives on physicochemical properties, electrical insulation properties and oxidative stability of selected blends
3. To elucidate mechanism of ageing behavior of ester and mineral oil blends and to identify suitable kinetic models to represent the degradation of Kraft insulating paper

1.4 Scopes of study

The scope and limitations of this research work are as follows:

1. This research only considers 6 types of polyol esters namely neopentyl glycol dicaprylate/dicaprate (NPGD C8/C10), neopentyl glycol dilaurate (NPGD C12), neopentyl glycol dioleate (NPGD C18), trimethylolpropane tricaprylate/ tricaprate (TMPE C8/C10), trimethylolpropane trilaurate (TMPE C12), and trimethylolpropane trioleate (TMPE C18) in assessing the effect of chemical structure on physicochemical properties, electrical insulation properties and oxidative stability of various polyol esters.
2. The blending ratio of ester/mineral oil used in this study are as follows only: 100% ester, 100% mineral oil, 20% mineral oil/80% ester, 30% mineral oil/70% ester, 40% mineral oil/60% ester, and 50% mineral oil/50% ester
3. In the formulation study, this research only considered 5 types of antioxidants with 1 wt% concentration of each. The antioxidants used are Irganox L115, Irganox L57, T531, NAPA and BHT.
4. In the ageing study only the effects of temperature, ageing time and copper were covered.
5. This study only focused on the physical, chemical and electrical properties such as viscosity, acidity, moisture content, breakdown voltage, dissipation factor, resistivity, permittivity, etc.

1.5 Contribution of the research

It is important to study the structure-properties relationship and the oxidative degradation of ester to properly design a new ester-based insulating oil. The knowledge of how different chemical structures of polyol esters affect the insulation properties are useful for utilities and other related research. This study provides a comprehensive understanding on the effect of chemical structure to the physicochemical and electrical properties of various polyol esters for ester-based insulating oils.

To better understand the nature behind the interaction between ester and mineral oil blending and how they differ from pure mineral oil, the fundamental aspects of physicochemical and electrical properties were explored. This information can benefits key utilities, end users and transformer manufacturers on the retro-filling of existing mineral oil transformers with esters (CIGRE, 2010; Perrier et al., 2006; Zimmerman and Bass, 2014). This study will provide a little insight on the amount of residual mineral oil that may be accepted until the properties of the new ester are compromised, during retro-filling. It is worth mentioning that the use of palm-based ester and mineral oil blends for the potential replacement of conventional mineral oil in the transformer is novel and has not been reported elsewhere.

In the thermal ageing study, a unique approach has been used to enable the investigation of the effect of copper on the degradation of ester/ mineral oil blending and blend-paper insulation system. As far as it is concerned, only three authors had studied the copper catalytic effect on the thermal degradation of mineral oil-paper insulation system. The existing kinetic models, a new degradation rate for papers aged in ester/mineral oil blending was obtained from the measurement of tensile strength and degree of polymerization. The knowledge obtained from the ageing study can be used to understand the ageing behaviors of ester and mineral oil blending especially in the presence of copper and Kraft paper.

1.6 Thesis layout

Chapter 2 provides the literature review on the chemistry of esters, the mechanism of oxidative degradation and the applications of additives in developing ester-based dielectric fluid. This chapter also briefly reviews the physical, chemical and electrical properties which have been established to evaluate the properties of the dielectric fluid. Moreover, experimental studies conducted for understanding the ageing behavior of oil-paper insulation system and kinetics of ageing themselves are reviewed.

Chapter 3 clearly discusses all the materials and methods used in the study. The transesterification methods to produce the esters, the turbine oil oxidation stability test (TOST) and the blending procedures are deeply discussed in this chapter. The pretreatment procedures of the oil/paper/copper systems, the thermal ageing experiments and the analyses conducted after the ageing are elucidated here.

Chapter 4 presents the results following each of the objective in the study. Chapter 4 is divided into five main sub chapters. The results on the effect of different chemical structures to the physical, chemical and electrical properties of dielectric fluid are explained in Section 4.1. In section 4.2, the effect of different chemical structures on the oxidative degradation of polyol esters are presented. The chapter continues with the discussion on the effects of different blending ratio of ester/mineral oil to the insulation properties and their respective oxidative degradation study in section 4.3. The oxidative degradation studies are further continued with the effects of different types of antioxidants based on TOST experiment. The results and discussion are presented in section 4.4. Lastly, section 4.5 presents the results of the thermal ageing experiment. In this section the ageing behavior of Kraft insulation paper in mineral and ester insulating oils is compared. The effects of aging time and temperature on the moisture content, acidity, breakdown voltage, tensile strength and degree of polymerization are studied. The ageing rate and the kinetics of ageing are calculated.

Chapter 5 is the conclusion chapter. The summary of the main points and the concluding remarks are included here. This chapter also briefly provides the recommendations for the future works and the study contribution to the body of knowledge.

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