UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF PALM-BASED NEOPENTYL GLYCOL DIESTER FOR TRANSFORMER OIL APPLICATION

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DEVELOPMENT OF PALM-BASED NEOPENTYL GLYCOL DIESTER FOR TRANSFORMER OIL APPLICATION

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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DEVELOPMENT OF PALM-BASED NEOPENTYL GLYCOL DIESTER FOR TRANSFORMER OIL APPLICATION

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Faculty : Engineering

Transformer oil plays an important role as electrical insulator of transformers. Mineral-based transformer oil has relatively high toxicity, low biodegradability and low fire point. The use of vegetable oils has constraints because the presence of beta-hydrogen in its structure renders it susceptibility to oxidation. The polyol esters have a unique feature that can overcome the oxidation problem faced by the vegetable oils. Hence the focus of this project is to study the potential of neopentyl glycol (NPG) diesters to be used as transformer oil. The synthesis of NPG diester was successfully optimized via transesterification of palm oil methyl ester (PME) with neopentyl glycol. The present study investigated the application of low-pressure technology as a new synthesis method which is able to shorten the reaction time. The optimum reaction conditions obtained by manual and response surface methodology (RSM) optimization were molar ratio of 2:1.3, reaction time of 1 hour, temperature at 182°C, pressure at 0.6 mbar and catalyst concentration of 1.2 wt%. The ester exhibited better properties than the commercial transformer oil especially with regards to the breakdown voltage, flash point and moisture content. The synthesized NPG diester was then formulated with anti-oxidant and pour point depressant to enhance its oxidative stability and low temperature properties. While 2,6-di-tert-butyl-p-cresol (DBPC) has proven to be useful and effective anti-oxidant for mineral oil, present studies indicate that it is not suitable to be used as additive in NPG diester, or in polyol ester as general. The pour point depressant on the other hand, has successfully increased the pour point of NPG diester from -14°C to -48°C. The laboratory thermal aging studies have also been developed to study the effect of temperature and aging time on selected properties and were compared with commercial mineral and refined, bleached and deodorized palm oil (RBDPO) at 90°C, 110°C and 130°C. It was found that aging has profound effect on the moisture content and acidity of the oil due to degradation of both oil and insulating paper. The

result indicated that throughout the aging period, NPG diester exhibits low acid value and no significant changes to viscosity and breakdown voltage. The study on tensile properties of insulating paper aged in NPG diester at 130°C shows higher tensile strength than paper aged in mineral oil and RBDPO. The aging rate calculated based on tensile strength indicated that at high temperature, insulating paper degraded faster in mineral oil and RBDPO than in NPG diester. The synthesized NPG diester has high potential to be used as transformer oil.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PEMBANGUNAN NEOPENTIL GLIKOL DIESTER DARIPADA MINYAK KELAPA SAWIT UNTUK KEGUNAAN MINYAK TRANSFORMER

Oleh

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Minyak transformer memainkan peranan yang penting sebagai penebat elektrik didalam transformer. Minyak transformer mineral mempunyai ketoksikan yang agak tinggi, tahap biodegredasi dan titik api yang rendah. Penggunaan minyak sayuran mempunyai kekangan kerana kehadiran beta-hidrogen dalam struktur yang menjadikannya terdedah kepada pengoksidaan. Ester poliol mempunyai ciri-ciri unik yang boleh mengatasi masalah pengoksidaan yang dihadapi oleh minyak sayuran. Oleh sebab itu fokus projek ini adalah untuk mengkaji potensi neopentil glikol (NPG) diester untuk digunakan sebagai minyak penebat. Sintesis NPG diester telah berjaya dioptimumkan melalui transesterifikasi metil ester minyak sawit (PME) dengan alkohol neopentil glikol. Penemuan ini mendedahkan keadaan tindak balas optimum yang diperolehi dengan optimasi secara manual dan permukaan respons (RSM) adalah nisbah molar 2: 1.3, masa tindak balas 1 jam, suhu pada 182 °C, tekanan pada 0.6 mbar dan kepekatan pemangkin sebanyak 1.2%. Ester yang disintesis mempamerkan ciri-ciri yang lebih baik daripada minyak penebat komersial terutamanya berkaitan dengan voltan jatuhan, takat kilat dan kandungan kelembapan. NPG diester kemudian dirumuskan dengan anti-oksida dan penurun titik tuang untuk meningkatkan kestabilan oksidatif dan sifat-sifat suhu rendahnya. Walaupun 2,6-di-tert-butil-p-cresol (DBPC) telah terbukti sebagai anti-oksida yang berguna dan berkesan untuk minyak mineral, kajian ini menunjukkan bahawa ia tidak sesuai untuk digunakan sebagai bahan tambahan dalam NPG diester, atau dalam poliol ester sebagai umumnya. Penurun titik tuang walaubagaimanapun telah berjaya meningkatkan titik tuang NPG diester daripada -14°C kepada -48°C. Kajian makmal termal penuaan juga telah dibangunkan untuk mengkaji kesan suhu dan masa penuaan ke atas ciri-ciri yang terpilih dan telah dibandingkan dengan minyak mineral dan minyak sawit halus, luntur dan dinyahbau, (RBDPO) pada suhu 90°C, 110°C dan 130°C. Kajian telah mendapati bahawa penuaan mempunyai kesan

mendalam kepada kandungan kelembapan dan keasidan minyak akibat degradasi kedua-dua minyak dan kertas penebat. Hasil menunjukkan bahawa sepanjang tempoh penuaan, NPG diester mempamerkan nilai asid yang rendah dan tidak ada perubahan ketara kepada kelikatan dan voltan jatuhan. Kajian ke atas ketegangan kertas penebat yang direndam dalam NPG diester menunjukkan kekuatan tegangan yang lebih tinggi daripada kertas penebat yang direndam dalam minyak mineral dan RBDPO. Kadar penuaan yang dikira berdasarkan kekuatan tegangan menunjukkan bahawa pada suhu yang tinggi, penebat kertas mempunyai tahap degradasi yang lebih cepat dalam minyak mineral dan RBDPO berbanding dengan NPG diester. NPG diester yang telah disintesis mempunyai potensi yang tinggi untuk digunakan sebagai minyak transformer.

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I certify that a Thesis Examination Committee has met on 1 December 2015 to conduct the final examination of Nurliyana binti Abdul Raof on her thesis entitled "Development of Palm-Based Neopentyl Glycol Diester for Transformer Oil Application" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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This thesis was submitted to the senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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This is to confirm that:

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LIST OF ABBREVIATIONS

α-HE	α-acyloxy-α-hydroperoxyalkanes
ANOVA	Analysis of Variance
ASTM	American Standard Testing Method
BDV	Breakdown Voltage
BHA	Butylated Hydroxyanisole
BHT	Butylated Hydroxytoluene
BS	British Standard
BSTFA	N,O-Bistrifluoroacetamide
BTA	1H-Benzotriazole
CCD	Central Composite Design
DF	Degree of Freedom
DBPC	2,6-di-tert-butyl-p-cresol
DGA	Dissolved Gas Analysis
DP	Degree of Polymerization
FAME	Fatty Acid Methyl Ester
FDS	Frequency Dielectric Spectroscopy
FFA	Free Fatty Acid
G3	Grade 3 Paper
GC	Gas Chromatography
HMWA	High Molecular Weight Acids
IEC	International Electrotechnical Commission
IEEE	Institute of Electrical and Electronics Engineers
IFT	Interfacial Tension
KOH	Potassium Hydroxide
KP	Kraft Paper
LMWA	Low Molecular Weight Acids
MO	Mineral Oil
MPOB	Malaysian Palm Oil Board
NPG	Neopentyl Glycol
PFAE	Palm Fatty Acid Ester
PKOME	Palm Kernel Oil Methyl Ester
PME	Palm Oil Methyl Ester
PCB	Polychlorinated Biphenyls
PPD	Pour Point Depressant
RBDPO	Refined, Bleached And Deodorized Palm Oil
RSM	Response Surface Methodology
TBHQ	Tert-butylhydroquinone
TS	Tensile Strength
TUP	Thermally upgraded paper

LIST OF SYMBOLS

X_1, X_2, X_3	Constant
C	Aging rate
t	Aging time
TS_0	Initial tensile strength

CHAPTER 1

INTRODUCTION

1.1 Research background

Most of the transformers in the power system network are using insulating oil which is commonly known as transformer oil (Azis *et al.*, 2014). Transformer oil serves two main purposes; as electrical insulator and as a coolant. Cooling is attained when the oil absorbs the heat from a winding coil and exchanges with the surroundings while circulating through the cooling ducts (Martin *et al.*, 2014).

Transformer oils have conventionally been manufactured from petroleum or mineral oil and have been used in a transformer since before 1890s (Vishal *et al.*, 2011). Since each transformer is likely to be used for more than 40 years, the transformer oil is expected to be very stable for that long service life (Behera and Murugan, 2013). Excellent electrical and cooling properties possessed by mineral based transformer oils are the major factor of its dominancy in global consumption. However, since mineral based oil is normally obtained from fractional distillation of crude oil, the main concern is its flammability and corrosive nature. The flash and fire points are relatively low (below 155°C and 165°C) which constitute high risk for fire and explosion (Cargill, 2012).

Polychlorinated biphenyls (PCBs) and silicone oil have also been extensively used as transformer oil due to their excellent electrical insulating properties which are comparable to mineral oil. The only disadvantages of PCBs and silicone oil are their highly toxicity and lack of biodegradability, respectively (Vishal *et al.*, 2011). Any spillage or leakage of the fluids will lead to serious environmental problems. Starting from 1990s, natural ester oils such as vegetable oils have attracted much attention as alternative transformer oil (Hosier *et al.*, 2014; Martin *et al.*, 2014; Xu *et al.*, 2014).

A considerable amount of literature has been published to study the potential of natural esters as transformer oil. Sunflower, rapeseed, linseed, soybean, cotton, safflower, corn and olive seeds are among the vegetable oils that have been tested for transformer oil (Sanchez *et al.*, 2014). Although palm oil have been widely used as a feedstock for biofuel production, very few studies have been focused on its usage as insulating fluids or transformer oil (Basu *et al.*, 1994). Natural esters are fairly good insulator, having higher flash and fire point than mineral oil and fully biodegradable. Despite commercial success of these vegetable oils, there has been limited use of it especially in free breathing transformers. The presence of unsaturated bonds in its molecule makes the natural esters highly susceptible to degradation. In addition, the viscosity of natural esters is normally high which is not so favorable to be used as transformer oil. Hence, in this study, the chemical modification on the natural ester

has been done in order to improve its properties to comply with the requirement of the transformer oil.

The polyol esters, which are made by reacting esters with polyhydric alcohols, have a unique feature that can overcome the oxidation problem by vegetable oils. Various polyol esters derived from different carbon chain lengths, degrees of unsaturation and types of alcohols are commercially produced for a broad range of applications. The choice of suitable fatty acid or methyl esters and alcohol is of particular importance to give the desired properties for transformer oil. Within the realm of polyol esters, esters of neopentyl alcohols such as neopentyl glycol (NPG), trimethylolpropane and pentaerythritol which were characterized by higher oxidative and thermal stabilities have been found to be very useful in transformer application (Gryglewicz *et al.*, 2013; Qiu and Brown, 2013b; Aziz *et al.*, 2014).

Apart from having excellent thermal and oxidative stabilities, the viscosity of transformer oil at operation temperatures is another principle factor for determining whether circulation of the oil will be adequate for heat dissipation. It is understood that low oil viscosity attributed to better cooling efficiency by having high circulation speed (Jiao, 2010). IEC 61099 specifies that the viscosity (at 40°C) of any synthetic organic esters that is to be used in transformers must be below 35 cSt (International Electrotechnical Commission, 2010) The reported viscosity of NPG, trimethylolpropane and pentaerythritol esters were in the range of 24 to 27 cSt, 39.7 to 49.7 cSt and 68 cSt, respectively (Yunus *et al.*, 2003; Qiu and Brown, 2013b; Aziz *et al.*, 2014). It appears from the aforementioned investigations that NPG esters have the highest potential to be used as transformer oil.

The aim of this study was to explore the potential of palm oil-based NPG diesters as transformer oil. There have been several studies in the literature reported about synthesis of NPG esters (Basu *et al.*, 1994; Bongardt *et al.*, 2000; Gryglewicz *et al.*, 2003; Padmaja *et al.*, 2012; Inayama *et al.*, 2013; Qiu and Brown, 2013a). Most of the synthesis methods implied range of temperature of 180 to 200°C and pressure of 5 mbar to 1 atm. The reaction time taken to yield at least 80% of NPG esters was 5 to 20 hours which was quite long. Most of the authors used excess of methyl ester in their reaction.

As transformer ages, the insulations degrade and will eventually cause insulation failure. The transformer oil must remain chemically, physically and electrically stable over an extended period of time to prevent future failures. Previous studies suggest that 2,6-di-tert-butyl-p-cresol (DBPC) is most suited for use as an antioxidant in transformer mineral oil and is highly efficient to double the useful life of a mineral oil (Mehanna *et al.*, 2014; Liu *et al.*, 2015). However, the effect of DBPC on polyol ester is scarcely reported and the effect is still unknown since NPG diester has completely different structure and composition than mineral oil.

Therefore, the formulation of NPG diester with certain additives, namely anti-oxidant and pour point depressant has been investigated.

The performance of transformer depends heavily on the performance of its insulation system (Gour *et al.*, 2012; Ziomek, 2012). The main components of insulation system consist of the transformer oil and insulating paper. Due to the complex shape of the transformer, paper is the only insulator that can be used because it is flexible. Moreover, paper can confiscate severe mechanical and electrical stresses that were caused by a conventional metallic housing (Srinivasrao and Roman, 2014). In an oil-paper insulation system, oil-impregnated paper is used to protect winding electrodes and pressboard is positioned around transformer winding (Lison *et al.*, 2014).

The understanding of paper insulation is vital to ensure prolong life of transformers since unlike the oil, the paper cannot be replaced or regenerated (Wilhelm *et al.*, 2011). Over time, the paper tends to degrade and loses its mechanical strength as well as its ability to insulate the windings (Lelekakis *et al.*, 2014). The causes of paper aging or degradation have been widely investigated and studies have found that high temperature and buildup of water, oxygen and acids as the main factors (Emsley *et al.*, 2000; Lelekakis *et al.*, 2014). To ensure that the transformers can operate without failure for a long time, the properties of the transformer oil and the paper insulation should be maintained at a specific level.

There are several published studies that demonstrated the aging of paper insulation in transformer oil (Mcshane *et al.*, 2001; Mcshane *et al.*, 2003; Martins, 2010; Wilhelm *et al.*, 2011; Azis, 2012; Martin *et al.*, 2014). Most of the studies have revealed different in aging rates between paper aged in mineral oil, natural esters and commercial transformer oil, EnviroTemp® FR3. Paper aged slower in natural ester and FR3 than did paper in conventional mineral oil especially at high temperature. However, for neopentyl polyol esters, the aging studies are still lacking. Accordingly, this study was undertaken to better understand the interaction of the paper insulation in the synthesized neopentyl glycol diesters. The present study explores the influence of temperature and time as primary aging factors.

1.2 Problem statement

Based on the issues highlighted in previous section, several problems with regards to synthesis of NPG diester and its implementation as transformer oil arose. The identified problems are:

1. The production of NPG diester from high oleic palm oil methyl ester (PME) has yet to be investigated. The previous studies related to the production of NPG diester required a very long reaction time and no process optimization has been done.
2. The physicochemical and electrical properties of NPG diester synthesized from high oleic PME have not been assessed and its formulation in transformer oil has not been studied.
3. No studies have examined the aging/degradation of NPG diester itself and its interaction with paper insulation.

1.3 Research objectives

The objectives of this research are:

1. To synthesize and optimize palm oil-based NPG diester
2. To characterize the physicochemical and electrical properties of the palm oil-based NPG diester and to formulate the ester with additives to enhance its properties as transformer oil
3. To study the degradation process of the synthesized ester through aging studies

1.4 Scope of work

The scope of the research comprises of:

1. Synthesis of NPG diesters by reacting high oleic palm oil methyl ester with neopentyl glycol and optimize operating parameters such as mole ratio, reaction temperature, amount of catalyst and operating pressure that can produce the highest yield of diester
2. The chemical, physical and dielectric properties such as viscosity, oxidative stability, breakdown voltage and power factor of the synthesized diester was evaluated according to standard procedures. NPG diester was then formulated with antioxidant and pour point depressant to meet the performance requirements for transformer oil

3. The laboratory aging test was developed over an extended period of time at different temperatures to study the effect of aging on moisture content, acidity, viscosity, breakdown voltage and tensile properties

1.5 Thesis layout

The thesis consists of five chapters. Chapter 1 is on the introduction, which highlights the background of the study, problem statement, objectives and scopes of work. Chapter 2 covers the literature reviews on the subject where extensive review and analysis are given to the reported works of various authors. The review provides the basis for the experimental and analysis sections of the thesis.

Chapter 3 is on the methodology of optimization in synthesis of neopentyl glycol diester, formulation and also aging studies. The analyses of oil and paper insulation using standards methods were also discussed. In chapter 4, the results of optimization, characterization, formulation and aging were presented and discussed. Chapter 5 presents the conclusions of the work and recommendations for future works.

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