



OXIDATION STABILITY ENHANCEMENT OF NATURAL ESTER INSULATION OIL: OPTIMIZING THE ANTIOXIDANTS MIXTURES BY TWO-LEVEL FACTORIAL DESIGN

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ABSTRACT

Natural ester insulation (NEI) oil is known as an alternative solution for mineral-based insulation (MI) oil used in power transformer applications. Majority of NEI oil properties are reported to have better performance than MI oil. However, NEI oil still has limited properties especially in oxidation stability, pour point, viscosity and lightning impulse event. Recent studies have shown that the addition of selected mixed antioxidants into NEI oil has improved the oxidation stability properties. However, previous researchers only implemented one-factor-at-a-time (OFAT) method as their experimental design approach and might overlooked any possibility of mixed antioxidants at optimum ratios that will acquire more preferable result. Furthermore, a wide number of test runs are necessary to examine the effect of these antioxidant mixtures on the performance of NEI oil. Therefore, in this study, two-level (2^k) factorial design of experiments is used to define the optimum concentration of propyl gallate (PG) and citric acid (CA) antioxidants which will amplify the oxidation stability of the oil. Oxidation induction time (OIT) is used to assess the oxidation stability of NEI oil according to ASTM E1858. The results show that the optimum concentration of PG and CA that yields the highest OIT of NEI oils is 0.25 and 0.25 wt.%, respectively. A regression model is also established to estimate the OIT of NEI oil as a function of PG and CA concentrations.

Keywords: insulation oil, natural ester, antioxidants, optimization, design of experiments, oxidation stability.

INTRODUCTION

Insulation oil plays three vital roles in liquid-immersed transformers which are (1) cooling medium; (2) protect against electrical faults; and (3) transformer health condition agent. In general, mineral-based insulation (MI) oil is used in liquid-immersed transformers for almost a century. MI oil is still dominated the insulation oil application due widely available in the market and moreover, they are inexpensive and they possess good thermal and electrical insulation properties [1], [2]. However, much attempt is being made nowadays to replace MI oil with products that is eco-friendly due to environmental concerns linked with these oils [3], [4]. The accessibility of natural esters derived from vegetable oils in recent years has made them a favorable alternative to replace conventional mineral insulation oils for power or distribution transformers. Numerous studies are being carried out to define the practicability of natural ester insulation (NEI) oils for both power and distribution transformers [5]-[7]. This is definitely unsurprising since NEI oils are highly biodegradable. Additionally, NEI oils have higher fire and flash points plus with good dielectric properties. NEI oils can also benefit by extending the lifespan of transformers due to the superior hydrophobic properties compared to mineral insulation oils [8], [9].

Nevertheless, NEI oils are not without drawbacks. Researchers are now focused on improving the properties of NEI oils for use in transformers. In order to improve the oxidation stability of NEI oils, antioxidants

are added into the oils to prevent the oils from further oxidation. Wilhelm *et al.* [10], [11] and Xu *et al.* [12] also added this antioxidant into NEI oils at a concentration of 0.3 wt.%. Abdelmalik *et al.* [13] formulated an improved version of NEI oil, whereby palm kernel oil alkyl ester (PKOAE) was mixed with 3 wt.% tertiary butyl hydroxyquinone (TBHQ). In addition, Raymon *et al.* [14] and Thanigaiselvan *et al.* [15] also used various combinations of antioxidants in order to enhance the properties of NEI oils. However, it shall be noted that the studies were only focused on evaluating the properties of NEI oils such as the breakdown voltage, viscosity, fire point and flash point whereby the oils were mixed with antioxidants using the following mass ratios in grams: 0.5:0.5, 1:1, 1.25:1.25 and 2.5:2.5. Meanwhile, Hao *et al.* [16] and Liao *et al.* [17] found an excellent oxidation stability of the insulation oil by using a combination of DPBC and high purity alkylation- α -naphthylamine (HPAN) antioxidants. These studies are concurring with the recommendations specified in the Cigre WG D1.30 report [18], which highlights on improving the oxidation stability of MI and NEI oils by means of the normal practice of lubricant technology. According to the report, one should consider adding more than two antioxidants into the insulation oils in order to exploit their favorable properties.

In this study, propyl gallate (PG) and citric acid (CA) antioxidants are added into NEI oil in order to improve the oxidation stability properties. The oxidation



stability of NEI oil is assessed through oxidation induction time (OIT). The OIT test is a best way to measure the thermal stabilization of a material under oxygen influence by using Differential Scanning Calorimetry (DSC). This method also linked to the oxidation stability characterization of a material [12], [16], [19]. The PG and CA antioxidants are chosen for this study since they have been proven to improve the oxidation stability of NEI oil and bio-lubricants [14], [15], [19]. However, the optimum concentrations of these antioxidants which will maximize the oxidation stability of the NEI oil is not known and therefore, they are determined using two-level factorial design of experiments. This technique is suitable for multi-factor experiments and it is capable of determining the optimum combination of factors with a fewer number of samples. In addition, this technique is inexpensive and less time-consuming [20]–[24]. The results obtained from this technique are then verified using analysis of variance (ANOVA). A regression model is then developed in order to predict the OIT of the NEI oil as a function of the PG and CA concentrations.

METHODOLOGY

a) Sample preparation

In this research work, NEI oil used is originated from rapeseed-based oil. Meanwhile, propyl gallate (PG) and citric acid (CA) antioxidants were purchased from Sigma-Aldrich. Before proceed for the samples preparing, water content of the NEI oil is quantified and must be less than 200ppm according to ASTM D6871 [25]. Vacuum oven is used for the water content treatment with a temperature setting of 70°C in 48 hours. After treatment, the NEI oil samples were prepared by mixing PG and CA antioxidants with NEI oil within a concentration range of 0.05 to 0.25 wt.%. The concentration of each antioxidant was chosen such that, so the total concentration of both antioxidants in the mixtures was in range of 0.3 to 0.4 wt.% [12], [26]. Precautions need to be takes, since higher concentrations of antioxidants may increase the conductivity of NEI oil [27]. High conductivity may increase the chances of oil to breakdown early which reduce the function as insulation oil. The oil samples were uniformly dispersed using a magnetic stirrer integrated with a hot plate at a stirring speed of 750 rpm. The temperature of the hot plate was set according to the melting point of each antioxidant [14], [15], [28].

b) Oxidation induction time (OIT) test

The oxidation induction time (OIT) was measured using Perkin Elmer Jade DSC (Figure-1) according to the ASTM E1858 standard test method [29]. Figure-2 shows the OIT response of hydrocarbon oil. The temperature changes setting are set to 40°C/min and isothermal test temperature range is set between 170°C and 210°C. The oxygen flow rate is 50 ml/min and specimen mass range is within 3.00mg to 3.30mg. Three samples are prepared for every number of test run to provide an average value for each OIT test.

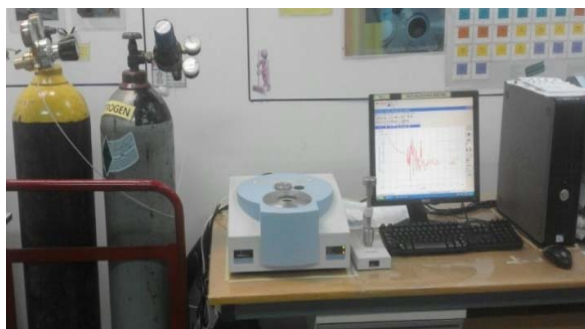


Figure-1. Perkin Elmer Jade DSC equipment for OIT test.

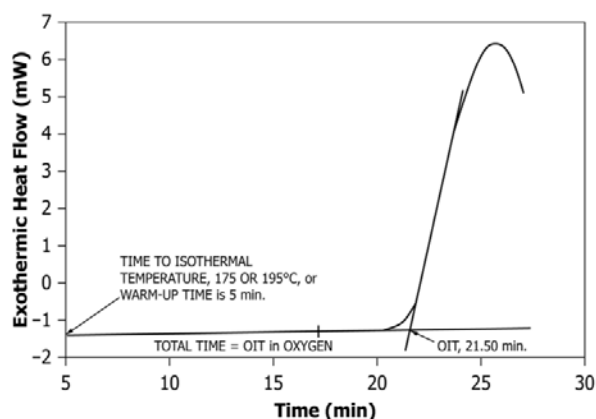


Figure-2. Example of hydrocarbon oil oxidation under OIT test (OIT is determined as a time which intersection occur between base line and tangential line of the rising exotherm) [29].

c) Design of experiments

Two-level (2^k) factorial design of experiments was applied by using the Design Expert software version 10.0 (Stat-Ease, Inc., Minneapolis, USA) with the purpose of screen a significance effect between main and interaction of antioxidants towards enhancement of oxidation induction time (OIT) in NEI oil. Next, the optimization process was carried out using a response surface model which can define the optimum concentrations of PG and CA that will maximize the OIT of the NEI oil.

d) Screening process

The 2^2 factorial design with two independent variables and three replications at the center points was used to screen the factors which will have a significant effect on the oxidation induction time (OIT) of the NEI oil. Table 1 show the level of the PG and CA variables, whereby low, medium and high is indicated by -1, 0 and +1, respectively. The 2^2 factorial design matrix used to screen the factors is shown in Table-2, and it can be seen that there are seven test runs. The OIT tests were carried out according to the 2^2 factorial design matrix. The effects of the PG and CA antioxidants (factor 1 and factor 2, respectively) on the OIT of the NEI oil were determined using a half-normal graph and effect list.

**Table-1.** Level of variables.

Factor 1 Propyl gallate (PG) A: PG (wt. %) Type: Numeric	Factor 2 Citric acid (CA) B: CA (wt. %) Type: Numeric
0.05 (-1)	0.05 (-1)
0.15 (0)	0.15 (0)
0.25 (+1)	0.25 (+1)

Table-2. Factorial design matrix used to screen the factors.

Experiment (Test run)	Variable code	
	A: PG (wt.%)	B: CA (wt.%)
1	-1	+1
2	+1	-1
3	0	0
4	0	0
5	+1	+1
6	0	0
7	-1	-1

e) Optimization process

A regression model was then established to estimate the oxidation induction time (OIT) of NEI oil as a function of the PG and CA concentrations. Analysis of variance (ANOVA) was used to explain the adequacy and statistical significance of the regression model. The results obtained from the 2² factorial design of experiments were used to fit the regression model. The regression model is composed of a list of coefficients multiplied by its associated factor levels. The regression model is given by the (1):

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_{12}x_1x_2 \quad (1)$$

According to (1), β_n represents the coefficient associated with factor n , whereas x_1 and x_2 denote the factors of the model. The product x_1x_2 represents the interaction between the individual factors. β_0 is the intercept of the model whereas β_1x_1 and β_2x_2 represent the individual effects of x_1 and x_2 . $\beta_{12}x_1x_2$ represents the two-factor interaction between x_1 and x_2 . The regression analysis was done in coded units and the coefficients were based on these coded units. ANOVA was then used to determine the sum of squares (SS), mean squares (MS), F -value, p -value, coefficient of determination (R^2) and correlation coefficient ($|R|$). Response surface plot was used to determine optimum concentrations of PG and CA antioxidants which will maximize the OIT of the NEI oil.

RESULTS AND DISCUSSIONS**a) Oxidation induction time (OIT) test results**

The oxidation induction time (OIT) values for all seven test runs are presented in Table-3.

Table-3. Oxidation induction time for each test run.

Experiment (Test run)	Variable code		Average oxidation induction time (minutes)
	A: PG (wt.%)	B: CA (wt.%)	
1	-1	+1	30
2	-1	-1	25
3	0	0	28
4	+1	+1	32
5	0	0	29
6	0	0	29
7	+1	-1	28

b) Results of the screening process

Table-4 and Figure-3 shows the effect list and half-normal plot, respectively, which are obtained from the 2² factorial design of experiments. It can be observed from Figure-3 that factor B (citric acid) and factor A (propyl gallate) are positioned at a distance far away from the straight line. Otherwise, interaction AB is positioned at closer to the straight line. This indicates that factors A and B are all significant model terms. Meanwhile, interaction AB shows no significant model terms. The effect list which shows the sum of squares and percentage contribution for all model terms is shown in Table-4. The results indicate that factor B is the most significant factor with a percentage contribution of 73.83% while the sum of squares for this factor is 20.25. Meantime, factor A has a contribution of 22.79%, which clearly shows that this factor is the second contributors among all factors. The sum of squares for factor A is 6.25. In contrast, interaction AB has a contribution of 0.91% while its sum of squares is 0.25. Based on the results, it is evident that factor B (citric acid) and factor A (propyl gallate) have a significantly higher contribution on the oxidation induction time (OIT) of NEI oil compared to interaction AB. It can be considered that no interaction between factors is occurred in this test.

Table-4. Effect list obtained from the 2² factorial design of experiments.

Model term	Standardized effects	Sum of squares (SS)	Percentage contribution (%)
A	2.50	6.25	22.79
B	4.50	20.25	73.83
AB	-0.50	0.25	0.91

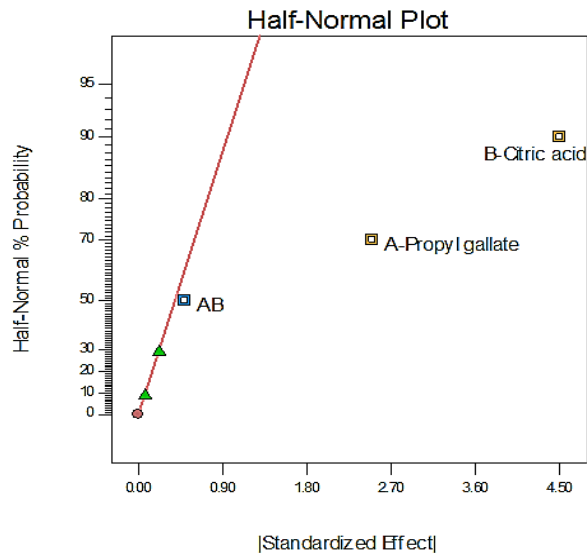


Figure-3. Half-normal plot obtained from the 2^2 factorial design of experiments.

c) Results of the optimization process

The optimization process is carried out using response surface plot to determine the optimum concentrations of PG and CA which will yield the highest oxidation induction time (OIT) for the rapeseed-based NEI oil. A regression model is developed according to (1), as shown in (2) which indicates the OIT of the NEI oil as a function of the propyl gallate concentration (variable: x_1 ; unit: wt.%) and citric acid concentration (variable: x_2 ; unit: wt.%).

$$y = 28.71 + 1.25x_1 + 2.25x_2 - 0.25x_1x_2 \quad (2)$$

The OIT for each experimental point predicted using (2) is summarized in Table-5, along with the observed values obtained from experiments. The mean and standard deviation of the OIT obtained from the regression equation is found to be 28.71 minutes and 0.48, respectively. Table-6 shows the sum of squares (SS), degrees of freedom (df), mean squares (MS), F -value, p -value, coefficient of determination (R^2) and correlation coefficient ($|R|$) for the regression model terms determined using ANOVA. According to Mohamed *et al.* [21] and Aman *et al.* [30], a p -value that is less than or equal to 0.05 indicates that the model (or model term) is statistically significant. The results show that the overall regression model is significant since the p -value is 0.0066. Factor A (propyl gallate) and factor B (citric acid) are significant model terms since the p -value is less than 0.05 (0.0134 and 0.0025 for factor A and factor B, respectively). In contrast, the p -value for interaction AB is 0.3703 (which is more than 0.05) and therefore, this model term is not significant.

Based on the ANOVA results, it can be found that interaction AB will not have a pronounced effect on the OIT of NEI oil. It can also be realized that the regression model developed in this study is adequate since the R^2 value is 0.9753, which indicates the model explains almost 98% of the total variation of the OIT is due to variation of the independent variables (*i.e.* PG and CA concentrations). In addition, the p -value of the overall regression model is 0.0066 (which is less than 0.05), indicating that the model is significant. The correlation coefficient $|R|$ shows there is a correlation between the observed and predicted OIT values. In general, if $|R|$ is closer to 1, this indicates that there is a strong correlation between the observed and predicted values. The $|R|$ value of the regression model is found to be 0.98, which shows that there is a strong correlation between the predicted OIT values and those obtained from experiments. Figure-4 is the response surface plot which shows the variation of OIT of NEI oil when the concentrations of PG and CA are varied. It can be seen from Figure-4 that OIT of NEI oil increases gradually as the CA concentration is increased from 0.05 to 0.25 wt.% while the PG concentration is increased from 0.05 to 0.25 wt.%. The maximum OIT with average of 32 minutes is attained when the PG concentration and CA concentration is 0.25 and 0.25 wt.%, respectively. Both of these antioxidants are needed since each antioxidant serves a different purpose - PG is a radical scavenger whereas CA acts as a synergist. In other words, CA is a hydro-peroxide scavenger. The enhancement of OIT responses between fresh NEI oil and optimized NEI oil are shown in Figure-5 and Figure-6. About 80% of oxidation stability improvement is produced by using the optimum combination of PG and CA antioxidants in this rapeseed-based NEI oil.

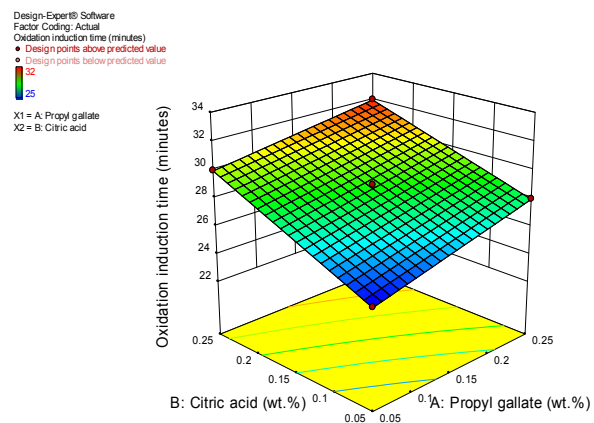


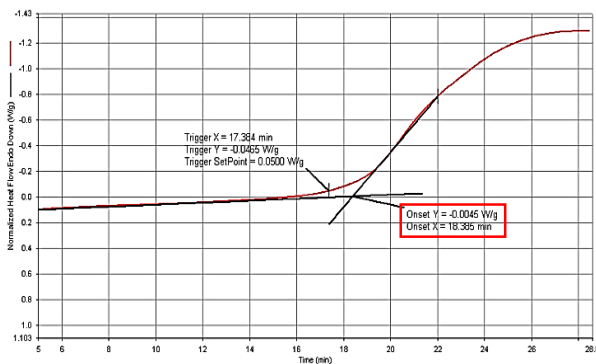
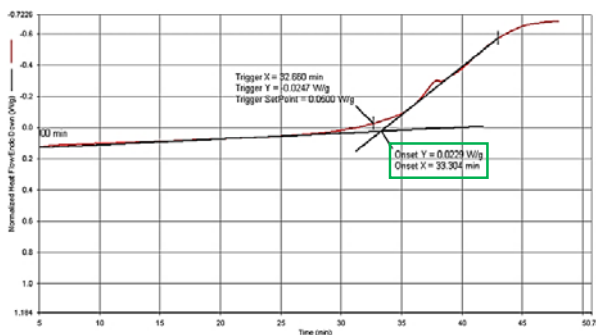
Figure-4. Response surface plot which shows the effect of the propyl gallate and citric acid concentrations on the oxidation induction time.

**Table-5.** Observed and predicted oxidation induction time.

Experiment (Test run)	Observed value, y (minutes)	Predicted value, y' (minutes)	Residual y-y'
1	30.00	30.00	0.00
2	25.00	25.00	0.00
3	28.00	28.00	0.00
4	32.00	32.00	0.00
5	29.00	28.67	0.33
6	29.00	28.67	0.33
7	28.00	28.00	0.00

Table-6. ANOVA results for the regression model with factorial response surface fitting.

Source	SS	df	MS	F-value	p-value	R ²
Overall model	26.75	3	8.92	39.42	0.0066	0.9753
A-Propyl gallate	6.25	1	6.25	27.63	0.0134	
B-Citric acid	20.25	1	20.25	89.53	0.0025	
AB	0.25	1	0.25	1.11	0.3703	
Residual	0.68	3	0.23			
Lack of fit	0.012	1	0.012	0.036	0.8675	
Pure error	0.67	2	0.33			
Total correlation	27.43	6				

**Figure-5.** OIT response of fresh NEI oil (OIT is 18 minutes).**Figure-6.** OIT response of optimized NEI oil (OIT is 33 minutes).

CONCLUSIONS

From this study, it is confirmed that two-level factorial design of experiments is suitable technique to determine the optimum concentrations of propyl gallate and citric acid antioxidants which will increase the oxidation induction time (OIT) of rapeseed-based NEI oil. The results obtained from the 2² factorial designs reveal that citric acid has the most pronounced effect on the OIT of NEI oil, with a percentage contribution of 73.83%. The response surface plot generated reveals that the optimum concentration of propyl gallate and citric acid which will yield the highest OIT is 0.25 and 0.25 wt.%, respectively. A regression model is also developed in this study, and it is found that the model is adequate to predict the maximum OIT of the NEI oil as a function of propyl gallate and citric acid concentrations, whereby the R² and p-value of the model is 0.9753 and 0.0066, respectively.

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