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Effects of Oil Temperature, Elemental Sulfur Concentration, and Aging Time on the Corrosion Activity of Transformer Mineral Oil using Two-Level Factorial Design

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ABSTRACT

The insulation of oil-filled power transformers, shunt reactors, and high voltage bushings may be affected by copper dissolution in the insulating oil and copper deposition on the paper insulation. Dissolved copper increases dielectric losses in the oil while copper deposition can significantly increase the conductivity of the paper insulation. The corrosion activity of transformer insulating oils has been shown to be accelerated by multiple factors such as temperature, oxygen, sulfur groups, passivators, and aging time. To date, there is a lack of studies that systematically design and quantify the effects of corrosion factors on the copper dissolution of transformer insulating oils or copper deposition on the surface of solid insulation (Kraft paper). Therefore, in this study, the effects of corrosion factors on the copper and sulfur deposition on the paper insulation immersed in transformer mineral oil (TMO) were investigated using two-level (2^k) factorial design. The factors investigated in this study were: (i) oil temperature, (ii) elemental sulfur concentration, and (iii) aging time. Based on the results of the two-level factorial design, it is found that the oil temperature has the most significant effect on the surface resistivity, with a percentage contribution of 38.68%. A regression model was also developed in this study, and it is found that the model is adequate to predict the surface resistivity as a function of oil temperature, elemental sulfur concentration, and aging time, where the coefficient of determination (R^2) and p-value of the regression model are 0.9694 and 0.0070, respectively.

Keywords:
Surface resistivity; design of experiment; factorial design; experiment parameters; mineral insulating oil; transformer; kraft paper

1. Introduction

Power transformers are a critical component of power systems. The presence of sulfides in transformer insulating oils during transformer operation can degrade the performance of the paper insulation and potentially ruin the safety and stability of transformers. In recent years, the likelihood of transformer corrosion due to the presence of sulfur has increased. Scholars have conducted

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extensive research worldwide on the mechanism of corrosive sulfur including the factors that cause the occurrence of sulfur corrosion, the effects of sulfur corrosion on transformer failures, and techniques to inhibit the progression of sulfur corrosion in power transformers [1-4].

According to the International Council on Large Electric Systems (CIGRE) Technical Report (Working Group A2.40), inter-turn failures and failure of the insulation system are the primary types of failure, accounting for 64 % of power transformer failures [5,6]. One of the sources of copper sulfide formation is the transformer mineral oil (TMO). It shall be noted that the permissible natural sulfur content in TMO should be less than 5 mg/kg [5]. It was discovered that sulfur was purposefully added into the TMO as an antioxidant (such as dibenzyl disulfide (DBDS)) in order to improve the thermal stability of the TMO [6-11]. DBDS is classified as thermally unstable sulfur compounds and may produce any reactive sulfur compounds (such as elemental sulfur (S8)) once the chemical bonding is broken. In this study, S8 was used to investigate the effects of oil temperature, elemental sulfur concentration, and aging time on the corrosion activity of TMO. It is worth nothing that S8 is the most reactive sulfur in the sulfur group [12-16].

Sulfur is found in a variety of materials, and it is not limited to TMOs. Sulfur compounds can also be found in other transformer components such as gaskets, copper windings, paper insulation, and some water-based adhesives. The ingress of sulfur into the transformer can also occur through unintentional means such as by the use of incompatible hoses [9]. The decomposition of DBDS into benzyl mercaptan or DBDS copper complex can cause copper corrosion and the formation of copper sulfide at temperatures ranging from 80°C to 150°C. The copper sulfide deposits onto the copper conductors and diffuse through the oil–paper insulation, where it is absorbed by the intermediate compounds in the paper insulation [8,17-23].

In this study, three factors were used to quantify their effects on the corrosion activity of TMO: (i) oil temperature (℃), (ii) elemental sulfur concentration (ppm), and (iii) aging time (days). Previously, these three factors were commonly used to individually investigate their effect on the progression of sulfur corrosion using one-factor-at-a-time (OFAT) method. These three factors were identified as the main contributors that accelerate the progression of sulfur corrosion mechanism in power transformers. Due to this reason, this study utilized three factors at one time to determine the individual significance of each element associate with sulfur corrosion related problems. The two-level (2k) factorial design was used to systematically design the experiment and quantify the effects of the factors. A response model was developed with the aid of analysis of variance (ANOVA). The effects of the factors were quantified to determine the best combination of factors that would minimize the surface resistivity of the paper insulation impregnated with TMO–S8 mixture [12,19,24-27]. The results obtained were verified using ANOVA. Subsequently, a regression model was developed to predict the surface resistivity of the paper insulation impregnated with TMO–S8 mixture as a function of the corrosion factors (oil temperature, elemental sulfur concentration, and aging time), and the adequacy of the model was verified using ANOVA.

2. Methodology

This study utilized a systematic experimental design technique, utilizing design of experiment software, to examine the influence of three factors on the sulfur corrosion activities. The factors were selected based on their significant effect to this research study. The experiment design software enabled a creation of comprehensive collection of tests. The experimental procedures were automatically generated by the Design-Expert version 10 program (Stat-Ease, Inc., USA). In addition, 13 experiments were created based on the given factors. The output response is based on the values obtained from the surface resistivity measurements. The acquired data will then be subjected to the

analysis of variance (ANOVA) for further study. The robustness and validity of the experimental design underline the technique, establishing a strong basis for deriving significant findings from the study.

2.1 Sample Preparation

In this study, TMO was chosen as the transformer insulating oil for the experiment since it has been widely used as a dielectric liquid and coolant in electrical equipment for many years. The TMO was first treated, where the TMO was filtered (Figure 1) and then subjected to nitrogen bubbling treatment (Figure 2). A nylon membrane filtration paper with a pore size of 0.2 µm was used for the filtration. The treated TMO was then mixed with S8 in a 1-L beaker according to the list of test runs obtained from the two-level factorial design, where each test run consisted of a different combination of oil temperature, elemental sulfur concentration, and aging time.

Fig. 1. Filtration process of transformer mineral oil

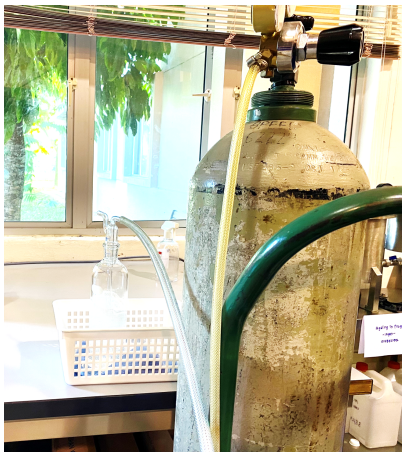

Fig. 2. Nitrogen bubbling process conducted following filtration

Before the mixing process, the elemental sulfur was weighed using an electronic balance based on the desired elemental sulfur concentration. The elemental sulfur weighed 0.0048 and 0.0179 g for an elemental sulfur concentration of 5 and 20 ppm, respectively. Meanwhile, a bare copper strip (length × width: 5 cm × 5 cm) and a copper strip wrapped with Kraft paper (length × width: 7 cm × 7 cm) were prepared. Next, the elemental sulfur was added into the treated TMO and the TMO–S8 mixture was stirred using a hot plate magnetic stirrer until the elemental sulfur was completely dissolved at a temperature of 119°C and stirring speed of 230 rpm. The TMO–S8 mixture was then wrapped with aluminum foil and left to cool to room temperature. After the TMO–S8 mixture had cooled, the mixture was transferred from the beaker to two 500-mL bottles, where each bottle was intended for a different aging time. The whole procedure was repeated for other oil temperatures and elemental sulfur concentrations. Following this, a bare copper strip and a copper strip wrapped with Kraft paper was inserted into the other bottle. The TMO–S8 samples were prepared based on the two-level factorial design matrix obtained from the screening process with the addition of fuller's earth and synthetic silicate adsorbent.

2.2 Surface Resistivity

The surface resistivity was chosen as the output response for all 13 samples prepared based on the two-level factorial design matrix. The surface resistivity was measured using a portable resistance

meter (Model: 272A, Monroe Electronics Inc, California) according to the ASTM D257 standard test method. Two concentric circular electrodes made of conductive elastomeric material were located on the insulating base of the electrode assembly. The dimensions of these rings are such that when the assembly is placed on the flat surface of the material to be tested, ten squares of the material will lie between the electrodes. When the power of the instrument is switched on, a voltage is impressed on the outer ring and the current flow in the interposed material is detected by the inner electrode, which defines the resistivity characteristics of the material. The internal circuitry of the instrument interprets this signal and provides a direct readout of the surface resistivity of the material under test in ohms per square (Ω/sq) (ASTM D257, para. 3.5). In the resistance to ground mode, the test voltage is removed from the outer electrode and is applied to the ground via a test lead supplied with the instrument. The current flowing between the ground and inner sensing electrode is detected and the value is converted into a direct readout of the resistance of the intervening path in ohms (Ω).

2.3 Design of Experiments

Design-Expert version 10 program (Stat-Ease, Inc., USA) was used to obtain 13 tests run (shown in Table 1) based on the two-level factorial design matrix for three independent variables (8 tests) combined with central composite design (5 tests). These techniques allow a thorough investigation on the impact of each factor (i.e., oil temperature, elemental sulfur concentration, and aging time) on the surface resistivity values as the response variables. In general, two-level full factorial design was commonly used to methodically evaluate any potential combinations of levels for multiple factors. This technique enabled operators to capture any potential interactions and primary impacts resulting from the dynamic interplay of various elements. On the other hand, the central composite design enables the operators to effectively investigate the response surface within the designated experimental range. The central composite design involved conducting experiments at certain points that corresponded to low (-1), middle (0), and high (+1) levels for every factor.

Table 1
Two-level factorial design matrix for three independent variables obtained from Design-Expert software

Test run no.	Variables					
	A: Oil temperature (°C)	Coded variable	B: Elemental sulfur concentration (ppm)	Coded variable	C: Aging time (days)	Coded variable
1	100	(−1)	20	(+1)	1	(−1)
2	140	(+1)	20	(+1)	5	(+1)
3	120	(0)	12.5	(0)	3	(0)
4	140	(+1)	20	(+1)	1	(−1)
5	120	(0)	12.5	(0)	3	(0)
6	100	(−1)	5	(−1)	5	(+1)
7	120	(0)	12.5	(0)	3	(0)
8	140	(+1)	5	(−1)	1	(−1)
9	140	(+1)	5	(−1)	5	(+1)
10	120	(0)	12.5	(0)	3	(0)
11	100	(−1)	20	(+1)	5	(+1)
12	100	(−1)	5	(−1)	1	(−1)
13	120	(0)	12.5	(0)	3	(0)

2.4 Screening Process

This phase involves analyzing all the data from each test run and estimating the maximum and minimum points. Based on the results obtained from the screening process, a regression model was developed from the design experiment software to predict the surface resistivity as a function of the oil temperature, elemental sulfur concentration, and aging time. Here, the surface resistivity is the response variable while the oil temperature, elemental sulfur concentration, and aging time are the independent variables. ANOVA was used to determine the statistical significance of the regression model. The regression equation is given as:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \varepsilon_i, \quad (1)$$

where y represents the response variable (predicted variable), x_1 and x_2 represent the independent variables (factors), x_1x_2 represent the interaction between factors x_1 and x_2, β_1 and β_2 represent the coefficients associated with Factors x_1 and x_2, β_{12} represents the coefficient associated with interaction x_1 x_2, β_1x_1 and β_2x_2 represent the effects of factors x_1 and x_2, and β_0 represents the intercept of the regression model. Regression analysis was carried out based on Eq. (1). The sum of squares (SS), mean squares (MS), F-value, p-value, coefficient of determination (R^2), and correlation coefficient (|R|) were determined using ANOVA. Response surface plots were plotted to determine the combination of oil temperature, elemental sulfur concentration, and aging time.

3. Results

3.1 Surface Resistivity

In this study, three factors (oil temperature, elemental sulfur concentration, and aging time) were considered for the two-level factorial design. The surface resistivity values recorded represents the resistivity of the paper insulation after ageing process. The mean resistance values for each TMO–S8 mixture for the 13 test runs are presented in Table 2.

Table 2
Mean surface resistivity for each TMO-S8 mixture

Test run no.	Variable code						Mean surface resistivity (Ω/sq) ($x10^{11}$)	Standard deviation (Ω)
	A: Oil temperature (°C)	Coded variable	B: Elemental sulfur concentration (ppm)	Coded variable	C: Aging time (days)	Coded variable		
1	100	(−1)	20	(+1)	1	(−1)	0.28	0.003
2	140	(+1)	20	(+1)	5	(+1)	1.39	0.070
3	120	(0)	12.5	(0)	3	(0)	1.20	0.070
4	140	(+1)	20	(+1)	1	(−1)	2.60	0.021
5	120	(0)	12.5	(0)	3	(0)	1.40	0.042
6	100	(−1)	5	(−1)	5	(+1)	2.30	0.035
7	120	(0)	12.5	(0)	3	(0)	1.40	0.006
8	140	(+1)	5	(−1)	1	(−1)	2.70	0.046
9	140	(+1)	5	(−1)	5	(+1)	5.37	0.185
10	120	(0)	12.5	(0)	3	(0)	1.50	0.027
11	100	(−1)	20	(+1)	5	(+1)	1.16	0.040
12	100	(−1)	5	(−1)	1	(−1)	0.43	0.015
13	120	(0)	12.5	(0)	3	(0)	2.20	0.031

3.2 Half-Normal Probability Plot

Figure 3 and Table 3 show the half-normal probability plot and effects list, respectively, obtained from the two-level factorial design. It can be observed from Figure 3 that Factor A (oil temperature), Factor B (elemental sulfur concentration), and Factor C (aging time) are located at a distance far away from the straight line. Likewise, Interaction AB, Interaction ABC, Factor C, Interaction AC, and Factor B are also located at a distance far away from the straight line, though the distance is not as marked as that for Factor A. This indicates that Factors A, B, and C, as well as Interactions AB, ABC, and AC, are significant model terms. The effects list, which shows the sum of squares (SS) and percentage contribution for all model terms, is shown in Table 3.

The results indicate that Factor A is the most significant factor, with a percentage contribution of 38.68%, while the SS for this factor is 7.7756. In contrast, Factor C only has a percentage contribution of 11.00%, which clearly shows that this factor has the lowest contribution among all factors. The SS for Factor C is 2.2124. Finally, Factor B and Interactions AB, AC, and ABC have a percentage contribution of 17.91, 4.85, 1.03, and 5.18%, respectively, and the corresponding SS values are 3.6006, 0.9751, 0.2070, and 1.0419, respectively. Based on the results, it is evident that Factor A (oil temperature) has a significantly higher contribution to the surface resistivity compared with Factors B and C, and Interactions of AB, AC and ABC. Nonetheless, the contribution of other factors is still considered important.

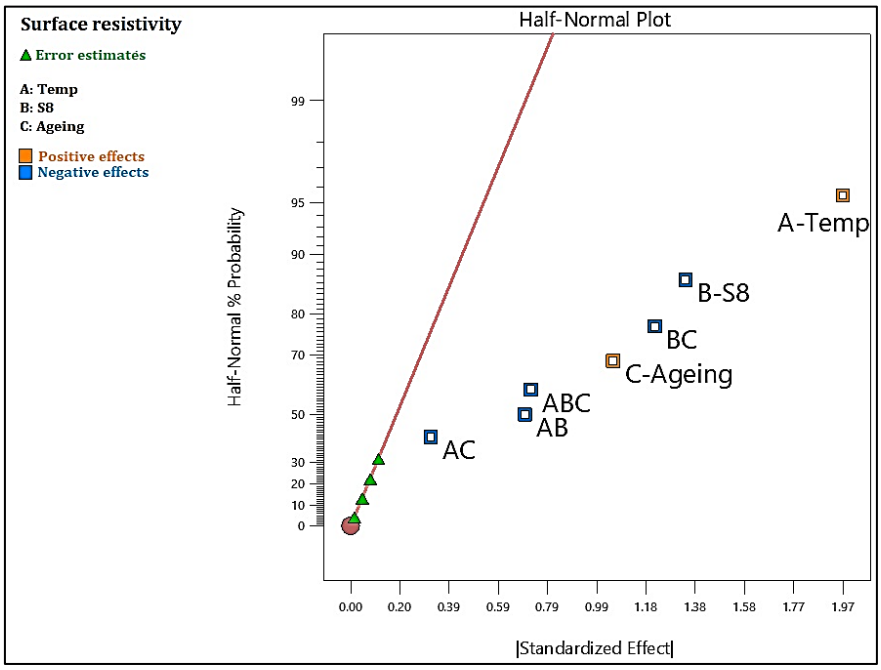

Fig. 3. Half-normal plot probability generated from the two-level factorial design

Table 3
Effects list obtained from the two-level factorial design

Model term	Standardized effects	Sum of squares (SS)	Percentage contribution (%)
A	1.9718	7.7756	38.68
B	-1.3418	3.6006	17.91
C	1.0516	2.2124	11.00
AB	-0.6983	0.9751	4.85
AC	-0.3218	0.2070	1.03
ABC	-0.7218	1.0419	5.18

3.3 ANOVA Results

The ANOVA results are tabulated in Table 4. A regression model was developed according to Eq. (1) and shown in Eq. (2), which shows the surface resistivity as a function of the oil temperature (variable: x_1 (°C)), elemental sulfur concentration (variable: x_2 (ppm)), and aging time (variable: x_3 (days)).

$$y = 2.03 + 0.9859x_1 - 0.6709x_2 + 0.5259x_2 - 0.3491x_1x_2 - 0.1609x_1x_3 - 0.3609x_1x_2x_3 \quad (2)$$

The mean and standard deviation of the surface resistivity obtained from the regression equation are found to be 1.841 and 0.0453 Ω/sq, respectively. Table 4 shows the sum of squares (SS), degrees of freedom (df), mean squares (MS), F-value, p-value and coefficient of determination (R^2) for the regression model terms determined from ANOVA. *P*-value of 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 0.0070. Factor A (oil temperature), Factor B (elemental sulfur concentration), Factor C (aging time), and Interactions BC and ABC are significant model terms since the p-values are less than 0.05 (0.0019, 0.0079, 0.0181, 0.0110, and 0.0568, respectively). In contrast, the p-values for Interactions AB (oil temperature and elemental sulfur concentration) and AC (oil temperature and aging time) are 0.0622 and 0.3024, respectively, which are more than 0.05, and thus, the model terms are not significant.

Table 4
ANOVA results for the regression model with factorial response surface fitting

Source	SS	Df	MS	*F*-value	*p*-value	R^2
Overall model	18.78	7	2.68	18.13	0.0070	0.9694
A-Oil temperature	7.78	1	7.78	52.54	0.0019	
B-Elemental sulphur concentration	3.60	1	3.60	24.33	0.0079	
C-Aging time	2.21	1	2.21	14.95	0.0181	
AB	0.98	1	2.97	6.59	0.0622	
AC	0.21	1	1.04	1.40	0.3024	
BC	2.97	1	0.27	20.06	0.0110	
ABC	1.04	1	1.04	7.04	0.0568	
Residual	1.33	5	0.27			
Lack of fit	0.74	1	0.74	4.97	0.0896	
Pure error	0.59	4	10.15			
Total correlation	20.11	12				

4. Conclusions

i. The results obtained from the two-level factorial design revealed that the oil temperature has the most pronounced effect on the surface resistivity of paper insulation impregnated with TMO–S8 mixtures, with a percentage contribution of 38.68%.
ii. The response surface plots showed that the best combination of sulfur corrosion factors that contributes to the lowest surface resistivity value are 100 ℃ (oil temperature), 20 ppm (elemental sulfur concentration), and 1 day (aging time), respectively.
iii. The developed regression model was adequate to predict the surface resistivity value as a function of oil temperature, elemental sulfur concentration, and aging time, where the coefficient of determination (R^2) and p-value of the regression model are 0.9694 and 0.0070, respectively.

iv. It is proven that the two-level factorial design is a useful technique to determine the most significance factors (oil temperature, elemental sulfur concentration, and aging time) that contribute to the lowest surface resistivity value of paper insulations that were impregnated with the TMO–S8 mixtures.

v. In future research, the acquired regression model might be use to perform further investigations incorporating other factors or a wider variety of variables in order to enhance the comprehension of these issues.

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