Investigating the effects of corrosion parameters on the surface resistivity of transformer's insulating paper using a two-level factorial design

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ABSTRACT

The integrity of the insulation in oil-filled power transformers, shunt reactors, and high voltage bushings can be affected when copper dissolves in the insulating oil and then deposits onto the paper insulation. The presence of dissolved copper in the oil increases dielectric losses, while copper deposition significantly improves the conductivity of the paper insulation. Various factors, including temperature, oxygen, sulfur groups, passivators, and ageing time, have been found to contribute to the acceleration of corrosion activity in transformer insulating oils. Unfortunately, there is a lack of extensive research focused on systematically analysing and measuring the impact of corrosionrelated factors on the dissolution of copper in transformer insulating oils and the deposition of copper onto solid insulation surfaces (Kraft paper). Therefore, this study aims to thoroughly examine the effects of corrosion factors on copper and sulfur deposition on Kraft paper insulation when it is submerged in transformer mineral oil (TMO). Using a two-level (2k) factorial design, we investigated three crucial factors: i) oil temperature, ii) elemental sulfur concentration, and iii) ageing time. It is worth mentioning that the results obtained from the two-level factorial design indicate that the surface resistivity is primarily affected by the temperature of the oil. This factor alone explains a significant 38.68% of the observed variation. In order to improve predictability, a regression model was created to estimate the surface resistivity of TMO-impregnated paper insulation. This model takes into account factors such as oil temperature, elemental sulfur concentration, and ageing time.

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1. INTRODUCTION

Power transformers are an essential element of power systems. Sulfides in transformer insulating oils can deteriorate the efficacy of paper insulation during transformer operation, thereby compromising the safety and stability of transformers. Transformer corrosion has become more likely in recent years due to the increased presence of sulfur. To prevent and reduce the damage caused by sulfur corrosion on transformers [1], [2], scholars have conducted extensive research worldwide on the mechanism of corrosive sulfide and its effects

on transformers along with the factors influencing sulfur corrosion [3]. The main types of failure in power transformers, according to the International Council on Large Electric Systems (CIGRE) Technical Report (Working Group A2.40), are interturn failures and failure of the insulation system, which make up 64% of all failures. Copper sulfide can arise from transformer mineral oil (TMO). The natural sulfur content of TMO falls within a range of 0.001–0.500%. Sulfur was intentionally incorporated into the TMO as an antioxidant, namely dibenzyl disulfide (DBDS), to enhance the thermal stability of the TMO [4]–[9]. This study explores the impact of oil temperature, elemental sulfur concentration, and ageing time on the corrosion activity of TMO, using elemental sulfur (S8) as the focus. It should be mentioned that among the sulfur group, S8 is the most reactive [10]–[14]. Sulfur can be found in a wide range of materials, not just limited to TMOs. Sulfur compounds are present in various transformer components, including gaskets, copper windings, paper insulation, and certain water-based adhesives. Sulfur can enter the transformer through unintended methods, such as using hoses that are not compatible [7]. At temperatures ranging from 80 to 150 °C, copper corrosion and the formation of copper sulfide can occur due to the decomposition of DBDS into benzyl mercaptan or DBDS copper complex. Once it diffuses through the oil-paper insulation and installs itself on the copper conductors, the intermediate compounds in the paper insulation absorb the copper sulfide [6], [15]–[21].

In this study, the effects of three factors on the corrosion activity of TMO were quantified: i) the temperature of the oil, ii) the concentration of elemental sulfur, and iii) the ageing time. A two-level factorial design was employed to methodically structure the experiment and measure the impacts of the factors. A response model was developed using analysis of variance (ANOVA). The optimal combination of parameters to minimize the surface resistivity of the paper insulation impregnated with TMO–S8 mixture was determined by quantifying the impacts of each element [10], [17], [22], [23]. The obtained results underwent validation through ANOVA. Following this, a regression model was constructed to anticipate the surface resistivity of paper insulation impregnated with a TMO–S8 mixture, correlating it with corrosion factors such as oil temperature, elemental sulfur concentration, and aging time. The model's suitability was then confirmed using ANOVA.

2. METHOD

2.1. Sample preparation

In this investigation, transformer mineral oil (TMO) was selected and it underwent initial treatment, involving filtration followed by nitrogen bubbling. A nylon membrane filtration paper with a pore size of 0.2 µm facilitated the filtration process. Subsequently, the treated TMO was blended with elemental sulfur (S8) in a 1-L beaker as per the predefined test run combinations derived from the two-level factorial design, where each run represented a distinct combination of oil temperature, elemental sulfur concentration, and aging time. Concurrently, preparations were made by crafting bare copper strips (5×5 cm) and copper strips enveloped with Kraft paper (7×7 cm). Before the mixing phase, elemental sulfur was precisely weighed using an electronic balance to achieve the desired concentration of 5 and 20 ppm, resulting in respective weights of 0.0048 g and 0.0179 g. The mixing process ensued with the addition of elemental sulfur into the treated TMO, followed by stirring on a hot plate magnetic stirrer until complete dissolution of the sulfur at 119 °C and 230 rpm. The resulting TMO-S8 mixture was then insulated with aluminum foil and allowed to cool to ambient temperature. Upon cooling, the mixture was transferred into two separate 500-mL bottles, each designated for a distinct aging period. Subsequently, a set of copper strips, both bare and wrapped with Kraft paper, were inserted into one of the bottles. Replication of the entire procedure was conducted for varying oil temperatures and elemental sulfur concentrations. Additionally, TMO-S8 samples were prepared based on the factorial design matrix obtained from the preliminary screening, with the inclusion of fuller's earth and synthetic silicate adsorbent.

2.2. Surface resistivity

The surface resistivity was designated as the output response for all 13 samples, which were prepared in accordance with the two-level factorial design matrix. Measurement of the surface resistivity adhered to the ASTM D257 standard test method, employing a portable resistance meter (Model: 272A, Monroe Electronics Inc, California) [24], [25]. The electrode assembly consists of two concentric circular electrodes made of conductive elastomeric material, positioned on the insulating base. The electrodes are sized so that when placed on the material being examined, ten squares of the material line up between them. Activating the instrument applies voltage to the outer ring, causing current flow in the material to be measured by the inner electrode. This technique defines the resistivity properties of the material. The instrument's internal circuitry processes the signal to provide a direct measurement of the material's surface resistivity in ohms per square (Ω /sq) according to ASTM D257, paragraph 3.5. In the resistance to ground mode, the voltage applied to the outer electrode is diverted to ground via a test lead provided with the instrument. Subsequently, the current traversing

between the ground and the inner sensing electrode is monitored, and this value is converted into a direct readout of the resistance along the intervening path in ohms (Ω).

2.3. Design of experiments

The significance of individual factors, namely oil temperature, elemental sulfur concentration, and aging time, on the surface resistivity of paper insulation impregnated with the TMO–S8 mixture was investigated utilizing a two-level factorial design. Design-Expert version 10.0.8.0 software (Stat-Ease, Inc., USA) was employed for this purpose. In Table 1, the low, medium, and high levels of these factors were represented by coded values of -1, 0, and +1, respectively. A two-level factorial design matrix, augmented by five center points, facilitated the screening of these factors, resulting in a total of 13 test runs. Subsequent to the surface resistivity tests conducted based on this design matrix, the effects of oil temperature, elemental sulfur concentration, and aging time (designated as Factors A, B, and C, respectively) were evaluated using ANOVA. Following the ANOVA analysis, three-dimensional response surface plots were generated to ascertain the optimal combination of factors for maximizing and minimizing the surface resistivity of paper insulation impregnated with the TMO–S8 mixture.

Table 1. Two-level factorial design matrix for three independent variables obtained from design-expert software

Test run no.	Variable code				
	A: Oil temperature (°C)	B: Elemental sulfur concentration (ppm)	C: Aging time (days)		
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 (-1)	12.5 (0)	3 (0)		
6	100 (-1)	5 (-1)	5 (+1)		
7	120 (-1)	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

During this phase, comprehensive analysis of data from each test run was conducted, aiming to estimate the optimum points. The outcomes derived from the screening process, a regression model was formulated to forecast the surface resistivity in relation to oil temperature, elemental sulfur concentration, and aging time. Here, the surface resistivity served as the response variable, while the oil temperature, elemental sulfur concentration, and aging time functioned as independent variables. ANOVA was employed to ascertain the statistical significance of the regression model. The regression equation is given by (1).

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2, \tag{1}$$

In the regression (1), where *y* represents the response variable (predicted variable), x_1 and x_2 denote the independent variables (factors), and $x_1 x_2$ signifies the interaction between factors x_1 and x_2 . β_1 and β_2 signify the coefficients associated with factors x_1 and x_2 , respectively, while β_{12} represents the coefficient associated with the interaction $x_1 x_2$. Furthermore, $\beta_1 x_1$ and $\beta_2 x_2$ denote the effects of factors x_1 and x_2 , and β_0 represents the intercept of the regression model. Regression analysis was conducted based on equation (1), wherein the sum of squares (SS), mean squares (MS), F-value, p-value, coefficient of determination (R²), and correlation coefficient (|R|) were determined using ANOVA. Additionally, response surface plots were generated to identify the combination of oil temperature, elemental sulfur concentration, and aging time that would optimize the surface resistivity of paper insulation impregnated with the TMO–S8 mixture.

3. **RESULTS AND DISCUSSION**

3.1. Surface resistivity

In this investigation, the two-level factorial design incorporated three factors: oil temperature, elemental sulfur concentration, and aging time. The recorded surface resistivity values signify the resistance of the paper insulation subsequent to the aging process. Table 2 outlines the mean resistance values corresponding to each TMO–S8 mixture across the 13 test runs.

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Table 2. Mean surface resistivity for each TMO–S8 mixture						
Test run		Mean surface				
no.	A: Oil temperature (°C)	B: Elemental sulfur concentration (ppm)	C: Aging time (days)	resistivity (Ω/sq)		
1	100 (-1)	20 (+1)	1 (-1)	0.283×1010		
2	140 (+1)	20 (+1)	5 (+1)	1.39×10^{10}		
3	120 (0)	12.5 (0)	3 (0)	1.20×10^{10}		
4	140 (+1)	20 (+1)	1 (-1)	2.60×10^{10}		
5	120 (-1)	12.5 (0)	3 (0)	1.40×10^{10}		
6	100 (-1)	5 (-1)	5 (+1)	2.30×1010		
7	120 (-1)	12.5 (0)	3 (0)	1.40×10^{10}		
8	140 (+1)	5 (-1)	1 (-1)	2.70×10^{10}		
9	140 (+1)	5 (-1)	5 (+1)	5.37×1010		
10	120(0)	12.5 (0)	3 (0)	1.50×10^{10}		
11	100 (-1)	20 (+1)	5 (+1)	1.16×10^{10}		
12	100 (-1)	5 (-1)	1 (-1)	0.43×10 ¹⁰		
13	120(0)	12.5 (0)	3 (0)	2.20×10^{10}		

3.2. Half-normal probability plot

Figure 1 depicts the half-normal probability plot, while Table 3 presents the effects list obtained from the two-level factorial design. Analysis of Figure 1 reveals that Factor A (oil temperature), Factor B (elemental sulfur concentration), and Factor C (aging time) exhibit notable distances from the straight line. Similarly, Interactions AB, ABC, and AC also display considerable distances from the straight line, albeit less pronounced than that of Factor A. This observation suggests the significance of Factors A, B, and C, as well as Interactions AB, ABC, and AC, as model terms. Table 3 provides insights into the sum of squares (SS) and percentage contributions for all model terms. Factor A emerges as the most significant factor, with a percentage contribution of 38.68% and an SS of 7.7756. In contrast, Factor C exhibits the lowest contribution among all factors, with a percentage contributions of 11.00% and an SS of 2.2124. Factor B and Interactions AB, AC, and ABC demonstrate percentage contributions of 17.91%, 4.85%, 1.03%, and 5.18%, respectively, with corresponding SS values of 3.6006, 0.9751, 0.2070, and 1.0419. These results underscore the substantial contribution of Factor A (oil temperature) to surface resistivity in comparison to Factors B and C, as well as Interactions AB, AC, and ABC. However, the contributions of other factors remain noteworthy, indicating their importance in the analysis.



Figure 1. 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 (%)
А	1.9718	7.7756	38.68
В	-1.3418	3.6006	17.91
С	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

Table 4 presents the ANOVA results. Additionally, a regression model was formulated as per (1) and displayed in (2). This model represents the surface resistivity as a function of the oil temperature (variable: x1; unit: $^{\circ}$ C), elemental sulfur concentration (variable: x2; unit: ppm), and aging time (variable: x3; unit: days).

 $y = 2.03 + 0.9859x_1 - 0.6709x_2 + 0.5259x_3 - 0.3491x_1x_2 - 0.1609x_1x_3 - 0.3609x_1x_2x_3$ (2)

The mean and standard deviation of the surface resistivity, derived from the regression equation, are determined to be 1.84×10^{10} and $0.3847 \times 10^{10} \Omega$ /sq, respectively. Detailed statistical metrics, including 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 are presented in Table 4 based on the analysis of variance (ANOVA). According to Khiar *et al.* [22] and Facciotti *et al.* [23], a p-value equal to or less than 0.05 signifies the statistical significance of the model or its respective terms. Notably, the overall regression model demonstrates significance, with a p-value of 0.0070. Moreover, Factor A (oil temperature), Factor B (elemental sulfur concentration), Factor C (aging time), and Interactions BC and ABC are identified as significant model terms, as their respective p-values fall below 0.05 (0.0019, 0.0079, 0.0181, 0.0110, and 0.0568, respectively). Conversely, 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, surpassing the threshold of 0.05. Consequently, these model terms are deemed statistically insignificant.

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 sulfur 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				

Based on the ANOVA findings, it is evident that Interactions AB (oil temperature and elemental sulfur concentration) and AC (oil temperature and aging time) are not likely to exert a substantial impact on surface resistivity when considered individually as parameters in the mixing process for TMO–S8 mixtures. Conversely, Interaction BC (elemental sulfur concentration and aging time) exhibits a noteworthy effect, particularly when combined with Factor A (temperature), leading to a significant enhancement in surface resistivity. Furthermore, the developed regression model is deemed adequate, as indicated by the high coefficient of determination ($R^2 = 0.9694$), signifying that the model elucidates 96.94% of the total variation in surface resistivity attributable to fluctuations in the independent variables (i.e., oil temperature, elemental sulfur concentration, and aging time). Additionally, the overall regression model demonstrates significance, with a p-value of 0.0070, which is below the conventional threshold of 0.05, underscoring its validity in surface resistivity.

Figure 2 illustrates three-dimensional response surface plots, each representing the variation of surface resistivity in paper insulation impregnated with the TMO–S8 mixture as the corrosion factors (namely, oil temperature, elemental sulfur concentration, and aging time) are manipulated. These plots provide an intuitive visualization of the interaction among these factors influencing the surface resistivity of sulfur deposition on the paper insulation. Upon inspection of Figure 2, it becomes evident that the optimal combination of corrosion factors resulting in minimized surface resistivity ($0.283 \times 10^{10} \Omega/sq$) for sulfur deposition on the paper insulation entails: i) oil temperature set at 100 °C, ii) elemental sulfur concentration maintained at 20 ppm, and iii) aging time of 5 days.

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In essence, the interaction between elemental sulfur concentration and aging time (Interaction BC) emerges as the most influential factor affecting the surface resistivity of sulfur deposition on the paper insulation. As depicted in Figure 2(a), the surface resistivity peaks at $5.37 \times 10^{10} \Omega/\text{sq}$ for the longest aging time (5 days) coupled with an oil temperature of 140°C and an elemental sulfur concentration of 5 ppm. This outcome aligns with expectations, as higher temperatures tend to foster reactions between sulfur and the paper insulation, leading to passivation and consequently elevating surface resistivity. Conversely, Figure 2(b) reveals that the surface resistivity reaches its value ($0.283 \times 10^{10} \Omega/\text{sq}$) for the shortest aging time (1 day) in conjunction with an oil temperature of 100° C and an elemental sulfur concentration of 20 ppm.



Figure 2. Three-dimensional response surface plots used to determine the (a) highest and (b) lowest surface resistivity values based on the corrosion factors

4. CONCLUSION

This study demonstrates the utility of the two-level factorial design in discerning the primary contributors to surface resistivity in paper insulation impregnated with TMO–S8 mixtures, namely oil temperature, elemental sulfur concentration, and aging time. A notable advantage of this design is its ability to identify significant factors with high precision, thereby minimizing the number of required test runs. This efficiency not only saves time but also reduces costs compared to traditional experimental methods. Analysis

reveals that oil temperature exerts the most significant influence on surface resistivity, contributing 38.68% to the observed variation. Moreover, response surface plots elucidate that the optimal combination of corrosion factors—oil temperature of 100°C, elemental sulfur concentration of 20 ppm, and aging time of 1 day—yields the lowest surface resistivity. Furthermore, a regression model was developed, exhibiting adequacy in predicting surface resistivity based on oil temperature, elemental sulfur concentration, and aging time. The high coefficient of determination ($R^2 = 0.9694$) and the statistically significant p-value (0.0070) underscore the robustness of the model in explaining 96.94% of the variability in surface resistivity.

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