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Assessing the Effectiveness of Natural Ester Fluid Retrofilling in Mitigating the Deposition of Copper Sulfide on Kraft Paper Insulation

Nur Izyan Husnina Zulkefli [a], Mohd Shahril Ahmad Khiar [a], Sharin Ab Ghani [a], Imran Sutan Chairul [a], & Mohd Affendi Che Musni [b]*

[a] *Electrical Asset Condition Monitoring Research Group, Faculty of Electrical Technology and Engineering, Universiti Teknikal Malaysia Melaka (UTeM), Melaka, Malaysia*
[b] *Power Transcoteq & Services Sdn. Bhd., 429, Jalan Nilai 7/17, Kawasan Industri Nilai 7, 71800 Nilai, Negeri Sembilan, Malaysia*

**Corresponding author: mohd.shahril@utem.edu.my*

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ABSTRACT

Numerous methods have been implemented to overcome the issues related to sulfur corrosion in power transformers. The common method involves the use of metal passivators. However, metal passivators are nitrogen-based and sulfur-based, and they are non-biodegradable and toxic. Therefore, in this study, the effectiveness of natural ester fluid retrofilling in mitigating the deposition of copper sulfide on kraft paper insulation was investigated. Comparison was made with regards to accelerated thermal aging of two retrofilling fluids (mineral oil and natural ester fluid derived from soybean (FR3)). The higher value in surface resistivity of paper insulation aged in FR3 fluid (1.67×10^{11} Ω/sq) as compared to mineral oil (1.62×10^{11} Ω/sq) reflects the ability of this retrofilling fluid in inhibiting paper conductivity due to sulfur corrosion. The tensile strength of paper insulation immersed in FR3 fluid slowly deteriorates (5.6 kN/m) as opposed to the paper insulation immersed in mineral oil (5.44 kN/m). The results obtained from scanning electron microscopy–energy dispersive X-ray spectroscopy analysis show that the amounts of sulfur and copper deposited on paper insulation remain relatively invariant for the paper insulation sample immersed in the oil after retrofilling with FR3. The decrease in the amount of sulfur in insulating oil from 1.854 cps/ma (retrofilling with mineral oil) to 0.760 cps/ma (retrofilling with FR3) obtained from X-ray fluorescence analysis revealed that retrofilling with FR3 fluid is the most effective technique in mitigating the deposition of copper sulfide on kraft paper insulation.

Keywords: Copper sulfide; mitigation method; natural ester; retrofilling; sulfur corrosion

INTRODUCTION

Multiple condition monitoring techniques have been introduced such as partial discharge sensors, dissolved gas analysis (DGA) sensors, and static winding resistance test (Ahmad Khiar 2012) to prevent transformer failures. Nowadays, there have been numerous reports of power transformer failures caused by sulfur corrosion since many years ago (Cong et al. 2020; Ahmad Khiar et al. 2022). Hence, numerous methods have been devised to mitigate sulfur corrosion. Oil retrofilling, passivation (Ahmad Khiar et al. 2018), and oil reclamation are the most commonly used methods. Although these methods may halt the spread of corrosive sulfur, they cannot prevent the deposition of copper sulfide onto the paper insulation.

Adsorption clay, also known as fuller's earth, is used in oil reclamation. Fuller's earth is a hydrated magnesium and aluminum silicate adsorbent with a distinct crystalline structure. After being heated to a certain temperature, this clay can have a surface area of up to 13 ha/kg. Fuller's earth readily adsorbs most of the polar contaminants found in used insulating oil, which prolongs the lifetime of power transformers. When a high vacuum degasser system is used in conjunction with fine particulate filters (pore size: 0.5 μm), almost all oxidation by-products can be removed, and the oil can be brought back to its original, new oil specifications.

Corrosive sulfur and metals are also extracted during the reclamation process. Reclaimed oil is often more stable than new oil because the natural inhibitors consumed in the oxidation process have been replaced with a synthetic antioxidant, typically 2,6 di-tert-butyl-4-methylphenol (also called di-tert-butyl-p-cresol (DBPC) and butylated hydroxytoluene (BHT)) (Gwynn 2017).

However, oil reclamation eventually creates elemental sulfur in the insulating oil. (Lewand 2002) found that after being aged for 19 h at 140 °C, there were trace amounts of elemental sulfur (1 ppm) on the copper surface. In addition, elemental sulfur can also react with silver at low temperatures. Silver corrosion penetrates through the paper insulation and creates a low-resistance path between the windings. Hence, according to the DIN 51353 standard, the insulating oil becomes corrosive after oil reclamation (Amaro 2015).

Passivation is one of the methods frequently used to reduce the corrosive effects of sulfur on copper conductors. Passivation with additives such as Irgamet 39 and 1,2,3-benzotriazole (BTA) have been used to shield the surface of copper conductors from corrosive sulfur formation. However, this method cannot eliminate the presence of corrosive sulfur compounds and it is not environmentally-safe. The addition of metal passivator into the insulating oil causes the passivator to react with copper, forming a complex layer on the copper surface. This layer inhibits sulfur from reacting with copper, which slows down the formation of copper sulfide (Amimoto et al. 2009). The complex layer degrades over time by heating and oxygen; however, it can be restored through the interaction between copper and the passivator in the insulating oil. The passivator in the insulating oil is consumed throughout the on-going reconstruction of the complex layer. Once the passivator in the oil is used up, the suppressive effect vanishes.

Another concern with the addition of metal passivators (e.g., Irgamet 39 and BTA) into insulating oils is that this method releases stray gasses as the oils age over time (Ren et al. 2015). The addition of passivator can effectively inhibit further progression of sulfur corrosion. Nevertheless, this method will likely lead to misinterpretation of DGA results due to the presence of stray gasses. The stray gasses such as hydrogen (H_2), carbon monoxide (CO), and carbon dioxide (CO_2) in the insulating oil are generated from residual oxygen contained in the insulating oil. In addition, the presence of metal passivators encourages rapid production of CO and H_2 since the passivators promote the catalytic activity of copper when acting as copper sulfide passivator films (Höhlein 2006; Winkler 2007; Perrier, Marugan and Beroual 2012; Atanasova-Höhlein 2015; Herath et al. 2020; Gao, Zeng and Zhang 2021).

In the 1980s, insulating oils contaminated with polychlorinated bipenyls (PCBs) were subjected to retrofilling, which was successful in removing 90–95 % of the impurities (Winkler 2007). However, retrofilling mineral oil is not a viable option during periodic maintenance due to the high cost of the insulating oil and its negative effect on the surrounding environment (Facciotti et al. 2014; Dixit et al. 2022). Rapp et al. (Rapp et al. 2008) showed that when the mineral oil was replaced with natural ester fluid, the damaging effects of sulfur corrosion were reduced by around 76 %. This forms the motivation of this study and therefore, the effectiveness of natural ester fluid retrofilling in mitigating the deposition of copper sulfide on kraft paper insulation is investigated.

METHODOLOGY

PRE-PROCESSING OF THE INSULATING OIL SAMPLES

The insulating oils used for this study were mineral oil (Nytro Libra) and natural ester fluid derived from soybean (FR3). The insulating oils must first be filtered to eliminate sludge or other contaminants such as water. The filtration process will promote the dielectric strength of the insulating oils and therefore, in this study, both insulating oils were filtered using a vacuum filtration assembly fitted with a 47-mm nylon membrane filter with a pore size of 0.2 µm.

Next, the insulating oils were treated using nitrogen gas. A constant flow of pure nitrogen (99.5 %) was bubbled across the surface of the insulating oil samples. This procedure took ~30 min for each sample and was performed three times. It shall be noted that the oxygen used by the insulating oils is replaced by nitrogen, which reduces the water content of the insulating oil samples (Vahidi et al. 2017).

After nitrogen treatment, the water content of insulating oil samples was tested to ensure that the water content in oil samples were below the limit prescribed in the ASTM D3487 (mineral oil: < 25 mg/kg) and ASTD D6871 standard (natural ester: < 200 mg/kg). This procedure is crucial to avoid any misinterpretation of the results because high water content values may contribute to a significant change, particularly in tensile strength and surface resistivity values. The Karl Fischer titration method, which was designed in accordance with the ASTM D1533 standard, was used to measure the amount of water present in the insulating oil samples. Metrohm 899 Karl Fischer coulometer was used for this purpose.

PREPARATION OF CORROSIVE OIL SAMPLES

To create a corrosive environment, elemental sulfur was added into the non-corrosive mineral oil samples to achieve the desired concentration (5 ppm). The solutions were stirred for ~1 h using a hot plate magnetic stirrer to ensure that the yellow elemental sulfur dissolved thoroughly in the mineral oil samples. Based on the melting point of elemental sulfur, the hot plate magnetic stirrer was set at a temperature of 119 ± 2 °C. The glass beakers were covered with aluminum foil during the mixing process to prevent the ingress of contaminants into the mixtures. The mixtures were left to cool at room temperature for ~20 min.

PRE-PROCESSING OF SOLID INSULATION

The dimensions of the kraft paper used were 0.07 mm × 70 mm (thickness × width). The kraft paper was cut to achieve a length of ~250 mm since for tensile strength measurements, the paper insulation needs to have a length of at least 250 mm according to the BS 4415-1:1992 standard. Following this, ~10 g of kraft paper was measured and prepared for each mineral oil sample. The weight of the kraft paper was measured using a digital weighing scale. The weight of the kraft paper before and after the drying process was recorded. The kraft paper was dried for 12 h at 105 °C in a ventilated oven according to the procedure outlined in the BS EN 60641-2:2004 standard.

Copper strips (area: 4900 mm^2) were polished with sandpaper and then cleaned with acetone. This will reduce the excessive oxide layers formed on the surface of the copper strips. In order to prevent contamination or oxidation of the copper strips, the strips must be prepared on the same day as the accelerated thermal aging process.

EXPERIMENTAL SET-UP

The corrosive mineral oil with copper strips wrapped in kraft paper were placed in the thermal aging chamber. Three aging conditions were considered in this study. First, the corrosive mineral oil was thermally aged at 95 ± 1 °C for 48 h. Second, the corrosive mineral oil was thermally aged 95±1 °C for 48 h and after retrofilling with FR3 fluid, the oil was thermally aged for another 48 h. Third, the corrosive mineral oil was thermally aged 95 ± 1 °C for 48 h and after retrofilling with mineral oil, the oil was thermally aged for another 48 h.

For retrofilling, the thermally aged mineral oil was flushed out from the chamber into amber bottles. The chamber was then rinsed with warm FR3 fluid or mineral oil (60–65 °C) to remove residual aged mineral oil as much as possible.

Next, the chamber was allowed at least 2 h of drip-down time to allow the chamber to drain fully. The treated FR3 fluid or mineral oil was heated at 60 °C to reduce its viscosity. The heating chamber was filled with the heated FR3 fluid or mineral oil through the designated retrofilling chamber. Following this, the heating chamber was filled with nitrogen to remove oxygen. The thermal aging conditions after retrofilling were the same as those before the retrofilling process.

Table 1 shows the aging conditions for this study. Figure 1 shows the experimental setup used for accelerated thermal aging.

TABLE 1. Thermal aging condition throughout the experiment

Aging condition	Orientation (h)
Thermally aged corrosive mineral oil for 48 h	48
Thermally aged corrosive mineral oil for 48 h + thermally aged oil after retrofilling with FR3 fluid for 48 h	96
Thermally aged corrosive mineral oil for 48 h + thermally aged oil after retrofilling with mineral oil for 48 h	96

FIGURE 1. Experimental setup used for accelerated thermal ageing

RESULTS AND DISCUSSION

TENSILE STRENGTH

The tensile strength of the paper insulation represents its mechanical strength. This property can be determined by obtaining the maximum mechanical stress of the paper insulation when it is stretched. Figure 2 shows the average tensile strength of the paper insulation samples under different aging conditions. The tensile strength of the new kraft paper is 8.4 kN/m (initial value. Figure 3 presents the evidence that the paper insulation has rapidly deteriorated under thermal stress as the aging period is increased. The FR3 fluid has been shown to have greater thermal stability than mineral oil, which makes it less prone to degradation due to aging. This can be clearly seen from the percentage degradation of tensile strength for both samples which were aged for 96 h (retrofilling with mineral oil and retrofilling with FR3 fluid compared with before retrofilling (48 h. It can be deduced that thermal stress plays an important role in deteriorating the mechanical strength of the paper insulation.

Further comparison of retrofilling with two types of insulating oils (FR3 fluid and mineral oil reveals that the tensile strength of paper insulation immersed in FR3 fluid slowly deteriorates (33.30 % of tensile strength degradation as opposed to the paper insulation immersed in mineral oil (35.23 % of tensile strength degradation). When the kraft paper is immersed in mineral oil contaminated with corrosive sulfur compounds, the degradation process occurs more rapidly due to the presence of acidic compounds that accelerate the hydrolysis reaction. This can further degrade the tensile strength of the paper insulation immersed in mineral oil compared with that immersed in FR3 fluid. The degradation of paper insulation due to the presence of corrosive sulfur compounds can be explained by the hydrolysis reaction that occurs when the β-1,4 glycosidic bonds in the cellulose chain break down (McShane et al. 2003; Sari et al. 2018). This reaction leads to structural degradation of the paper insulation, which in turn, results in a reduction in tensile strength. The breakage of these bonds weakens the intermolecular forces that hold the cellulose fibers together, leading to the formation of voids and cracks within the paper insulation. As a result, the paper insulation becomes more brittle and prone to tearing, resulting in a decrease in tensile strength. This suggests that the use of natural ester fluids such as FR3 fluid as a retrofilling material may be a solution to mitigate the effect of sulfur corrosion.

SURFACE RESISTIVITY MEASUREMENTS

A resistivity meter was used to measure the surface resistivity of the paper insulation. Figure 4 shows the average surface resistivity of the paper insulation samples, which decreases in proportion to the aging time. This is due to the degradation of cellulose fibers within the paper insulation caused by high-temperature exposure and the presence of corrosive sulfur compounds, which can lead to the formation of voids and cracks within the paper insulation. Consequently, the paper insulation becomes more conductive, which means that electrical current can flow more easily across the bulk of the paper insulation. This increases the risk of electrical breakdowns. Even though both paper insulation samples were aged for 96 h, it can be clearly seen that the paper insulation immersed in the oil after retrofilling with FR3 fluid has higher surface resistivity compared with that after retrofilling with mineral oil. This implies that the sulfur compounds in the mineral oil have altered the characteristics of the paper insulation. In addition, the higher surface resistivity of the paper insulation immersed in the oil after retrofilling with FR3 fluid is likely due to the excellent oxidation stability and thermal stability of the FR3 fluid, which may aid in inhibiting the degradation of cellulose fibers against corrosive sulfur compounds. Since conductivity is the reciprocal of resistivity, this means that paper insulation thermally aged in FR3 fluid has low conductivity. This suggests that retrofilling with FR3 fluid can preserve the insulation property of the paper insulation. This is an important consideration for transformers because good insulation is necessary to prevent electrical breakdowns and ensure safe and reliable operation of transformers. Retrofilling with FR3 fluid may be a viable strategy to extend the lifespan of transformers and other electrical equipment.

SEM-EDX SPECTROSCOPY

SEM–EDX spectroscopy was used to quantitatively analyze the elements present on the surface of the kraft paper due to sulfur corrosion. The instrument used in this investigation was COXEM EM-30AX scanning electron microscope and the EDX accelerating voltage was adjusted to 20 kV. Once electrons are fired at a sample by the EDX's primary beam, distinctive X-rays are generated. Energy measurements of the X-rays are used to create elemental spectra from the material. The inner shell electron will be ejected if the energy of the electron beam is higher than the binding energy of the electrons. When the electrons from the outer shell enter the empty inner shell, they release an X-ray to equalize the energy level.

This means that the energy of the X-ray is equivalent to the difference in energy between the two shells. Owing to its unique electrical configuration, each atomic element interacts with the beam electrons in a distinct way, causing them to generate X-rays at different energies. The elements in a sample can be identified by their characteristic X-ray energies, and their concentrations can be quantified by measuring the intensities of their characteristic X-ray peaks (Gao et al. 2020).

The formation of copper sulfide in insulating oils was due to the chemical reaction between corrosive sulfur compounds and copper surfaces. Copper sulfide may be deposited or penetrated through paper insulation which in turn lead to turn-to-turn transformer breakdowns. Due to this fact, only sulfur and copper were quantified and the results reported are based on the percent by weight (wt%).

The mean and standard deviation of sulfur and copper deposited on each paper insulation sample were determined, as shown in Figure 5. The results from this study indicate that the amount of copper sulfide deposition increases rapidly for the paper insulation immersed in the oil after retrofilling with mineral oil as the aging time is increased. This suggests that corrosive sulfur compounds are present in the mineral oil, leading to degradation of the kraft paper insulation. However, both the amounts of sulfur and copper remain relatively invariant for the paper insulation sample immersed in the oil after retrofilling with FR3 fluid. This indicates that the degradation of corrosive sulfur does not occur because it is already inhibited after retrofilling with FR3 fluid takes place.

XRF SPECTROSCOPY

XRF spectroscopy was used to detect sulfur in the insulating oils investigated in this study. By studying the characteristic secondary (fluorescent) X-rays emitted by a material in response to being bombarded with high-energy X-rays, XRF provides a nondestructive method for determining the composition of a substance. A high-energy incident X-ray can eject an electron from a low energy level. A higher-energy electron relaxes into the lower energy level and emits a secondary X-ray characteristic of the atom and energy levels between which the process occurs to fill the hole (Holt et al. 2013; Rehman et al. 2017). Before being poured into vials, each insulating oil sample was shaken. Firstly, the calibration curves for sulfur were generated to determine the limit of detection Before being poured into vials, each insulating oil sample was shaken. Firstly, the calibration curves for sulfur were generated to determine the limit of detection (LoD) and limit of quantitation (LoQ) (Brouwer 2010)

Figure 6 shows the average intensity of sulfur for the three aging conditions. Continuous aging for 48 h has the highest intensity (17.530 cps/ma) at a peak energy of 2.33 keV. However, after retrofilling with mineral oil and FR3 fluid, the intensity drops to 1.854 and 0.760 cps/ma, respectively. The results of the XRF analysis indicate that the rate of sulfur corrosion increases with the aging time, which is consistent with the findings from the other analytical methods used in this study. The decrease in the amount of sulfur after retrofilling with FR3 fluid is a positive outcome and suggests that the retrofilling with FR3 fluid is effective in reducing the formation of copper sulfide and mitigating the effects of sulfur corrosion on transformers. The use of XRF spectroscopy in this study provides valuable information on the concentration of sulfur in the insulating oils and supports the conclusion that retrofilling with FR3 fluid can be an effective strategy for reducing the impact of sulfur corrosion on transformer performance.

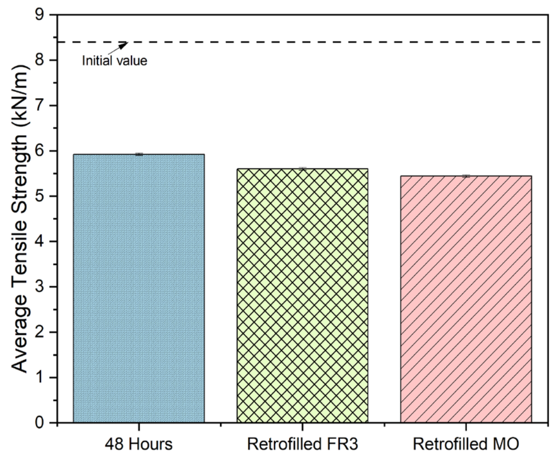

FIGURE 2. Average tensile strength

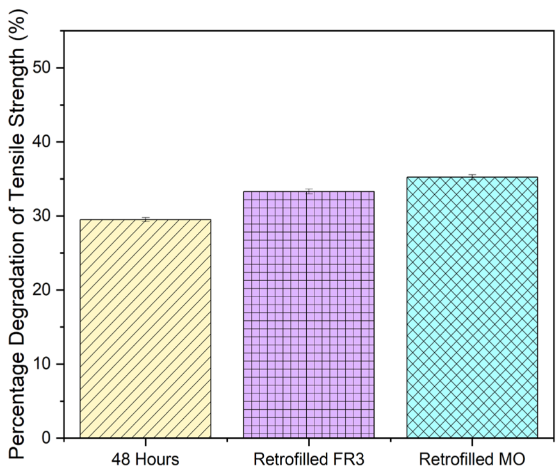

FIGURE 3. Percentage degradation of tensile strength

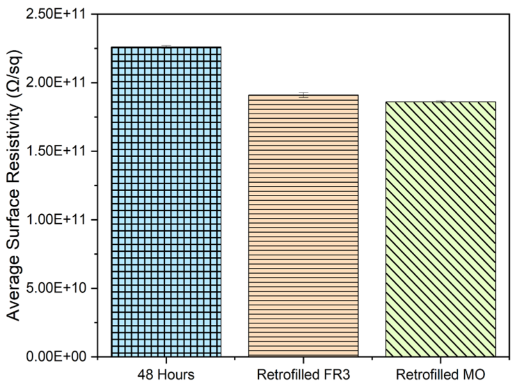

FIGURE 4. Average surface resistivity of the paper insulationsamples

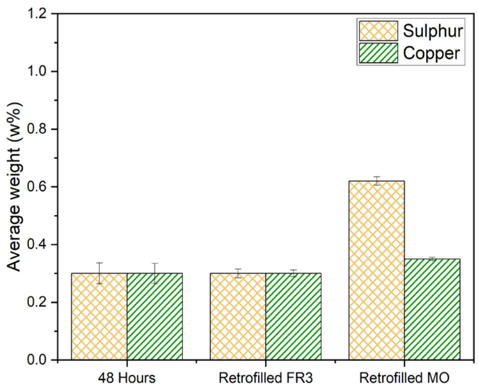

FIGURE 5. Mean percent by weight (wt %) of sulfur and copper on the paper insulation samples

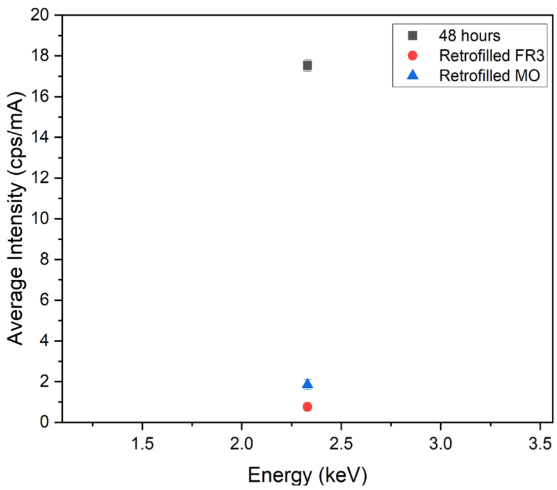

FIGURE 6. Average intensity of sulfur for the three aging conditions.

CONCLUSION

Based on the findings presented in the study, several conclusions can be drawn, as follows:

1. The formation of corrosive sulfur compounds such as copper sulfide in insulating oils will reduce the performance and longevity of power transformers.
2. Retrofilling with FR3 fluid (natural ester fluid), can mitigate the effects of sulfur corrosion by inhibiting the formation of copper sulfide and slowing down the degradation of the paper insulation.
3. The SEM–EDX spectroscopy results confirm that the copper sulfide deposition significantly decreases after retrofilling with FR3 fluid.
4. The XRF spectroscopy analysis shows that the intensity of sulfur decreases significantly after retrofilling with FR3 fluid, indicating that the amount of copper sulfide decreases as well.
5. In contrast, retrofilling with mineral oil does not provide the same level of protection against sulfur corrosion.

This study suggests that retrofilling with FR3 fluid can be a beneficial strategy for preserving the performance and longevity of transformers by reducing the effects of sulfur corrosion on the paper insulation and improving the properties of the insulating oil without causing detriment to the environment.

Application-wise, it is also essential to investigate the limitation of FR3 before this fluid are used as transformer retrofilling. The evaluation of several factors (i.e., load capacity, operating conditions, and compatibility with the existing insulation materials) should be taken into consideration, which is reserved for future work.

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DECLARATION OF COMPETING INTEREST

None

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