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Comparative Evaluation of Treated and Untreated Methyl Ester for Mitigating Sulphur Corrosion in Retrofilled Power Transformers

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Abstract

Sulphur-induced corrosion is a major degradation mechanism in oil-immersed power transformers, where reactive sulphur compounds form copper sulphide deposits that accelerate the deterioration of paper insulation and reduce transformer reliability. Although ester-based retrofilling has been widely adopted as a mitigation strategy, the use of edible vegetable oils raises concerns regarding sustainability and competition with food resources. Therefore, this study investigated the potential of methyl ester as a sustainable retrofilling fluid for transformer insulation systems. Prior to application, the methyl ester was treated using Fuller's earth bleaching and synthetic silicate adsorption to improve its chemical quality. The treated methyl ester was thermally aged at 104 °C with sulphur-contaminated mineral oil, copper strips, and kraft paper insulation under controlled laboratory conditions. The performance of the insulation system was assessed through acidity analysis, tensile strength testing, and surface resistivity measurements. The results demonstrated that the treatment process substantially reduced the acidity of the methyl ester and improved its suitability for retrofilling transformer applications. Following retrofilling, the paper insulation retrofilled with treated methyl ester exhibited higher tensile strength and surface resistivity values than both the sulphur-aged mineral oil and untreated methyl ester conditions. Overall, the findings demonstrate that treated methyl ester possesses promising potential as a sustainable retrofilling oils for mitigating sulphur-related insulation degradation and improving the long-term reliability of power transformers.

Keywords: Methyl Ester; Paper Insulation; Power Transformers; Retrofilling, Sulphur Corrosion.

1. Introduction

Corrosion is broadly defined as the degradation of a material, usually a metals, that results from a chemical or electrochemical interactions with its surrounding environment [1], [2] .In the context of transformers, corrosive sulphur refers to the elemental sulphur (S_8) and thermally unstable sulphur-containing compounds present in insulating oils, which can react with metallic components such as copper and silver [3]. Corrosive sulphurs may originate from various sources, including crude oil, electrical apparatus, pyrolysis processes, and hydrogenation reactions [4], [5].

The corrosive sulphur compounds react with copper conductors, lead to the formation of copper sulphide compounds and initiate the corrosion process. As oxidation progresses, the oil may become more viscous, lose its insulating capability, and experience overall deterioration [1], [2]. To reduce oxidation, antioxidants are added to mineral oil to extend their service life [6], [7], [8]. Antioxidants are commonly added either during oil production or as part of transformer maintenance practices [9] These antioxidants help slow down oxidation reactions by limiting the harmful effects of oxygen on the insulating oil. Sulphides and disulphides are commonly used as antioxidant additives in transformer oils. One of the most widely used disulphides is dibenzyl disulphide (DBDS) [10], [11], [12], [13] .

Numerous mitigation strategies have been proposed to address sulphur corrosion in power transformers. Retrofilling constitutes a strategic approach to transformer maintenance and mitigation [14], [15]. The retrofilling process typically entails the removal of existing oil, followed by the flushing of residual mineral oil and contaminants with compatible fluids from the transformer. This process aims to reinstate dielectric strength, enhance cooling efficiency, and reduce the presence of remaining reactive sulphur or oxidation by-products. Replacing aged or contaminated oil through retrofilling can lead to a notable decrease in the deposition of copper sulphide on windings, while also decelerating the degradation of paper insulation. Retrofilling serves to simultaneously revitalize the insulation system, augmenting the operational safety of the transformer, and improving its environmental impact. This process ultimately contributes to the prolongation of the transformer's operational lifespan [16], [17], [18], [19].

Retrofill fluids can be classified into two primary categories: natural ester and synthetic ester. Natural ester fluids are generally originated from vegetable-based sources, including soybean, rapeseed, and palm oil, which undergo refinement for use as electrical insulation [20], [21]. These fluids offer several advantages over conventional mineral oil, including biodegradability, higher fire safety due to their elevated flash points, and improved environmental compatibility [22], [23].

However, the widespread application of natural esters is constrained by the competition for vegetable oil resources with the food industry and domestic consumption. In addition, the effectiveness of treated natural ester compared with untreated natural ester in mitigating sulfur corrosion in retrofilled power transformers remains insufficiently understood. Therefore, this study comparatively evaluates the performance of treated and untreated methyl ester as retrofilling fluids for mitigating sulfur corrosion, with the objective of improving transformer insulation reliability and extending service life.

2. Methodology

The methodology adopted in this study consisted of six main stages, namely: (1) treatment of methyl ester, (2) pre-processing of insulating oil samples, (3) preparation of corrosive mineral oil samples, (4) preparation of insulating paper and copper samples, (5) accelerated thermal ageing and retrofilling experiments, and (6) assessment of the insulating paper performance. The overall experimental workflow is illustrated in **Figure 1**.

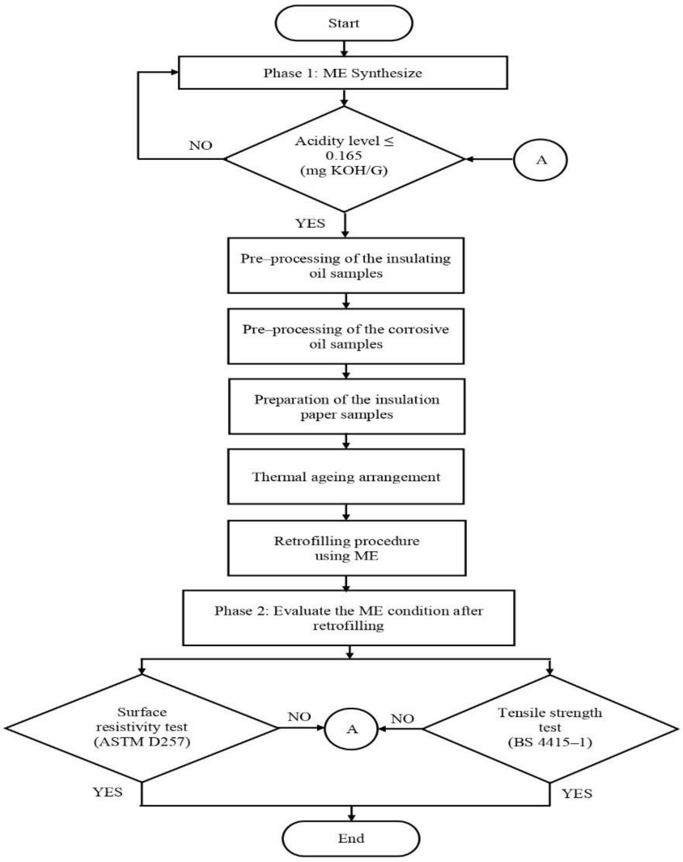

Figure 1. Flowchart depicting the methodology applied in this study

2.1 Preparation and Treatment of Methyl Ester

This treatment comprised two bleaching cycles using Fuller's earth absorbent clay in accordance with IEEE Std C57.147 (2018). During the first cycle, Fuller's earth equivalent to 10 wt.% of the methyl ester sample was pre-heated in a laboratory oven at 120 °C for 20 minutes. The pre-heated Fuller's earth was then added directly to the methyl ester sample. The mixture was immediately placed on a hotplate magnetic stirrer at 750 rpm and 80 °C for 1 hour to promote effective contact between the adsorbent and the methyl ester [16]. The samples were covered and left undisturbed under ambient laboratory conditions for 24 hours to allow the methyl ester to clarify by settling out the adsorbent, before the second bleaching cycle was performed under identical operating conditions.

Following the bleaching process, the methyl ester underwent treatment with a synthetic silicate adsorbent. Synthetic silicate equivalent to 20 wt.% of the sample subsequently stirred with the methyl ester using a magnetic stirrer with a speed of 750 rpm while the temperature was maintained at 70 °C for 1 hour [24]. The treatment was conducted with the objective of reducing the acidity value towards the recommended limit specified in IEEE Std C57.147 (2018).

2.2 Pre-processing of the Insulating Prior to Ageing

The insulating oil samples, comprising both mineral oil and methyl ester, underwent a pre-processing stage for contaminant removal, which involved filtration followed by nitrogen gas bubbling treatment. This step was necessary because filtration eliminated suspended particles, while nitrogen purging minimized dissolved oxygen. The filtration setup consisted of a 1000 mL ground-joint flask fitted with a tubulated cap assembly and a sand-core filter head. A 47 mm nylon membrane filter with a pore size of 0.2 µm was placed on top of the tubulated cap and secured together with a glass funnel using a spring clamp. Filtered oil samples were then treated with nitrogen gas in a 500 mL impinger bottle connected to a gas cylinder. Nitrogen gas with a purity of 99.5% was continuously bubbled through the oil sample for approximately 30 minutes per cycle, and the treatment was repeated three times for each sample. After the nitrogen treatment, the moisture content of each oil sample was measured to verify compliance with the limits specified in ASTM D1533. Only samples meeting the required moisture specification were used for subsequent experimental procedures.

2.3 Pre-processing of the Corrosive Oil Samples

The corrosive oil samples were prepared by introducing S_8 into pre-processed non-corrosive mineral oil using a dilution-by-weight method expressed in parts per million (ppm). The required amount of S_8 was calculated based on the target sulphur concentration of 7 ppm using equation as in (1).

$$\text{Quantity of } S_8 \text{ (ppm)} = \frac{\text{grams of solute}}{\text{grams of solution}} \times 1{,}000{,}000 \qquad (1)$$

S_8 with a purity of 99.99% was added to 500 mL of pre-processed mineral oil to obtain the desired sulphur concentration. The mixture was subsequently heated and stirred using a hot-plate magnetic stirrer for approximately 1 hour at 119 ± 2 °C.

2.4 Preparation of the Insulating Paper Samples

The insulation paper used in this study had dimensions of 0.07 mm × 70 mm (thickness × width). The paper was cut into strips with an approximate length of 250 mm to satisfy the minimum specimen length requirement for TS testing in accordance with BS 4415-1 (1992). The insulating paper samples were dried in a ventilated oven at 105 °C for 12 hours following the procedure specified in BS EN 60641-2 (2004). After drying, the paper samples were reweighed and compared with their initial mass. In addition to the insulating paper, copper strips with a surface area of 4900 mm² were prepared for the accelerated thermal ageing experiment. Prior to use, the copper surfaces were polished using P1000-grade sandpaper and then subsequently cleaned with acetone. To minimise contamination and further oxidation before testing, the copper strips were prepared on the same day as the accelerated thermal ageing experiment.

2.5 Experimental Set-up

The thermal ageing conditions employed throughout the experiment are summarized in **Table 1**. The experiment was designed to evaluate the degradation behaviour of the oil-paper insulation system under elevated temperature conditions and to assess the effect of retrofilling with alternative insulating fluids after an initial ageing period. Upon completion of the initial ageing period, the aged corrosive mineral oil was completely drained from the specimen bottles through a retrofilling process. The bottles were rinsed with warm retrofilling fluid at 60–65 °C and then left undisturbed for at least 2 hours to drain completely. After retrofilling, nitrogen gas was introduced into the samples, which were then returned to the ventilated oven for a second thermal ageing period at 104 ± 1 °C. At the end of this stage, the samples were removed and prepared for subsequent electrical and physical performance assessments.

Table 1. Accelerated Thermal Ageing Tests Conditions

Ageing condition at 104 ± 1 °C	Total ageing time (hours)
Thermally aged pure MO 662 h	662
Thermally aged corrosive MO 331 h	331
Thermally aged corrosive MO 662 h	662
Thermally aged corrosive MO (331 h) + Retrofilled with pure MO (331 h)	662
Thermally aged corrosive MO (331 h) + Retrofilled with untreated ME (331 h)	662
Thermally aged corrosive MO (331 h) + Retrofilled with treated ME (331 h)	662

3. Results and Discussions

In this study, methyl ester samples were analysed to evaluate the effectiveness of the bleaching and synthetic silicate treatments in reducing acidity. In

Figure 2, the untreated methyl ester sample exhibited an acid value of 0.515 mg KOH/g, indicating the presence of free fatty acids and oxidation by-products commonly associated with methyl ester-based insulating liquids [24]. This value considerably exceeds the permissible acidity limit of 0.165 mg KOH/g specified for new natural ester-based insulating liquids therefore suggesting that further treatment is necessary before the insulating oil can be considered suitable for transformer applications.

Following the first bleaching cycle, the acid value decreased slightly to 0.4748 mg KOH/g, corresponding to a reduction of approximately 7.8% compared to the untreated methyl ester sample. The relatively modest reduction indicates that a single bleaching cycle was insufficient to remove a significant proportion of acidic compounds from the oil. In the first cycle of bleaching process, adsorption consumed first by larger polar molecules due to their strong affinity toward the active surface [24]. Hence, only a limited number of acidic compounds is removed during the initial cycle.

This suggests that the adsorbent surface was occupied by high polar contaminants such as oxidation products, trace metals, and suspended impurities rather than free fatty acids.

A more substantial improvement was observed after the second bleaching cycle, where the acid value decreased to 0.2667 mg KOH/g with the result representing an overall reduction of approximately 48.2% where the major impurities had been removed from the initial untreated methyl ester sample during the first cycle. The enhanced performance suggests that repeated bleaching cycles increase the contact between the adsorbent material and acidic constituents, thereby improving contaminant removal efficiency [16]. This finding is consistent with previous studies reporting that multiple adsorption cycles can effectively reduce free fatty acid content in methyl ester insulating fluids.

Figure 2 presents the lowest acid value was achieved by the treated methyl ester insulating oil samples after the adsorbent treatment stage exhibited an acid value of reaching 0.165 mg KOH/g.

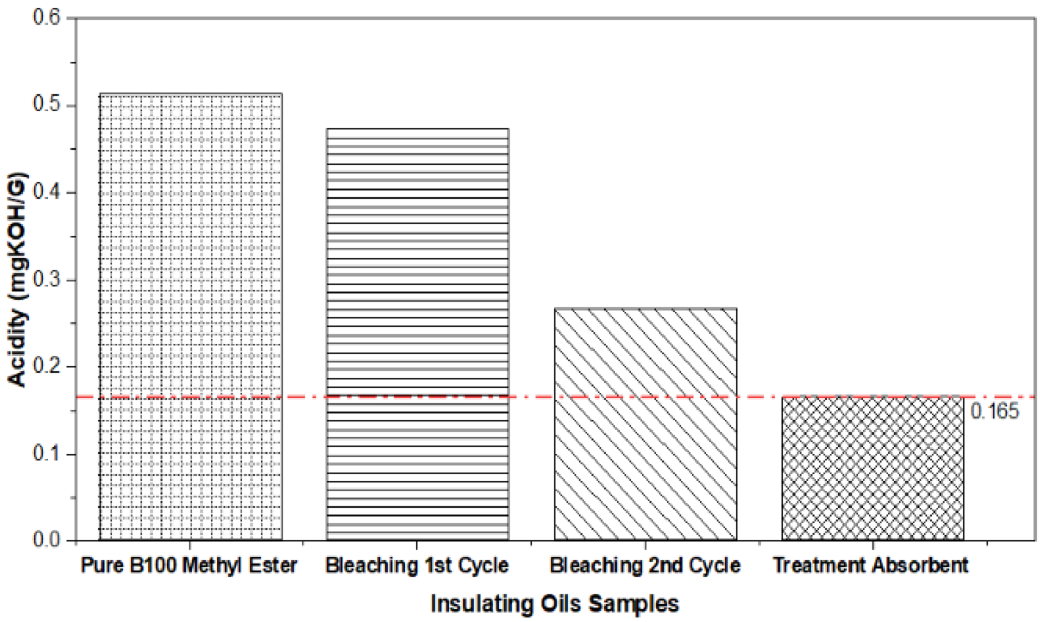

Figure 2. Acidity levels for different forms of insulating oils

This value considerably achieves the permissible acidity limit of 0.165 mg KOH/g specified for new natural ester-based insulating liquids to be considered suitable for transformer applications. The significant decrease indicates that the adsorbent material provided additional adsorption sites capable of removing residual acidic species that remained after the bleaching process. The result confirms that combining bleaching and adsorbent treatment offers a more effective purification approach than bleaching alone.

Figure 3 presents the average surface resistivity values obtained for insulating paper samples exposed to different insulating oils samples. The paper aged in (pure MO 662 h) exhibited the highest surface resistivity of 8.12×10^9 Ω/sq. This result indicates that the insulating paper maintained good dielectric properties, with minimal conductive deposits and limited degradation of the cellulose insulation. The insulating paper aged in (corrosive MO 331 h) showed a slightly lower surface resistivity of 7.97×10^9 Ω/sq, representing a reduction of approximately 1.8% compared to the sulphur-free sample.

However, when the ageing duration was extended to 662 hours, the surface resistivity decreased substantially to 5.99×10^9 Ω/sq, corresponding to a reduction of approximately 26.2%.

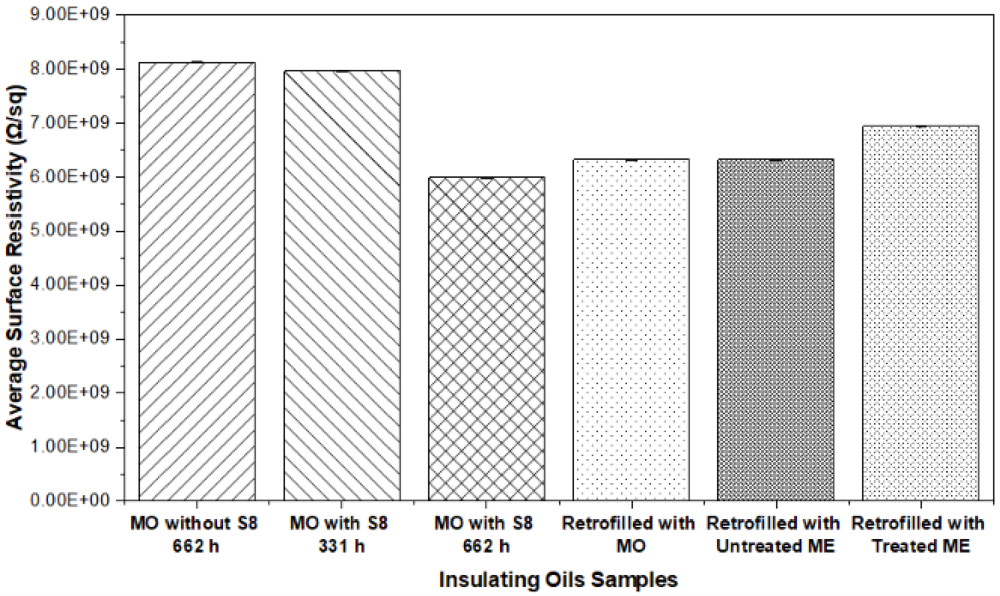

Figure 3. Surface resistivity of insulation paper for each oil sample

This significant decline indicates progressive deterioration of the insulation system due to prolonged exposure to sulphur contamination. The reduction in surface resistivity may be attributed to the formation of conductive copper sulphide deposits and oxidation by-products on the paper surface, which promote leakage current conduction and reduce the dielectric performance of the insulation. This observation suggests that some irreversible degradation had already occurred during the sulphur ageing process prior to retrofilling.

Following retrofilling with treated methyl ester, the surface resistivity increased to 6.95 × 10^9 Ω/sq. This represents an improvement of approximately 16.0% compared with the (corrosive MO 662 h). In contrast, the paper retrofilled with untreated methyl ester and mineral oil exhibited lower surface resistivity values of 6.32 × 10^9 Ω/sq and 6.16 × 10^9 Ω/sq, respectively. The improvement in surface resistivity following retrofilling with treated methyl ester suggests that the treated fluid provided a cleaner dielectric environment for the aged insulation system. The higher surface resistivity obtained with treated methyl ester indicates that the treatment process enhanced the electrical insulating performance of the retrofilling fluid. The superior performance of treated methyl ester may be attributed to the reduction of acidic compounds and polar contaminants through bleaching and synthetic silicate treatment may have reduced the concentration of charge carriers within the insulating fluid [14], [15]. Lower concentrations of ionic and polar species limit electrical conduction suppress the formation of conductive pathways on the paper surface, thereby contributing to higher surface resistivity values [25]. The observed improvement demonstrates the potential of treated methyl ester as a retrofilling fluid for mitigating electrical insulation degradation associated with sulphur contamination. The findings indicate that the treatment process enhanced the ability of the methyl ester to maintain the dielectric characteristics of aged paper insulation, thereby supporting its application in transformer retrofilling strategies.

Figure 4 presents the average tensile strength values obtained for insulating paper samples subjected to different insulating oil conditions. The insulating paper aged in (pure MO 662 h) exhibited the highest tensile strength of 4.7671 kN/m. This result indicates that the cellulose structure remained relatively intact, with limited degradation of the paper fibres during the ageing period. A slight reduction in tensile strength was observed for the paper aged in (corrosive MO 331 h), where the average tensile strength decreased to 4.52972 kN/m. The relatively small decrease suggests that the initial stages of sulphur exposure had only a limited effect on the physical condition of the cellulose insulation.

When the ageing duration was extended for (corrosive MO 662 h), the tensile strength decreased substantially to 3.62923 kN/m, representing a reduction of approximately 23.9% compared with the sulphur-free aged sample. This significant decline indicates severe degradation of the cellulose insulation following prolonged exposure to corrosive sulphur. Although corrosive sulphur primarily attacks copper conductors, the resulting copper sulphide formation in prolonged thermal ageing may indirectly accelerate the deterioration of cellulose insulation. Sulphur-related reactions generate additional degradation products and promote a more chemically aggressive ageing environment within the oil-paper insulation system. Furthermore, copper ions released during corrosion may act as catalysts for oxidation reactions, accelerating cellulose degradation and contributing to the observed reduction in tensile strength [25]. The loss of tensile strength may be attributed to the combined effects of thermal ageing, oxidation, and sulphur-induced degradation, which accelerate the breakdown of cellulose polymer chains and weaken the paper structure [26].

The reduction in tensile strength is closely associated with the depolymerization of cellulose molecules within the insulation paper. Within supported by thermal ageing, the glycosidic bonds linking glucose units in cellulose gradually break, reducing the degree of polymerization (DP) of the kraft paper [27]. As the average chain length decreases, the cellulose fibre network becomes physically weaker and less capable of resisting tensile loading [28]. Following retrofilling with treated methyl ester, the tensile strength increased to 4.16868 kN/m, compared to 3.79231 kN/m for untreated methyl ester and 3.71013 kN/m for mineral oil, respectively. The treated methyl ester therefore produced an improvement in tensile strength of approximately 9.9% compared to untreated methyl ester, and 12.4% relative to retrofilled mineral oil. These results indicate that retrofilling with treated methyl ester was more effective in preserving the physical condition of the insulating paper than untreated methyl ester. The higher tensile strength retained by the retrofilled paper suggests that the treatment process contributed to the preservation of the cellulose fibre network by reducing acidic contaminants, and improving moisture management.

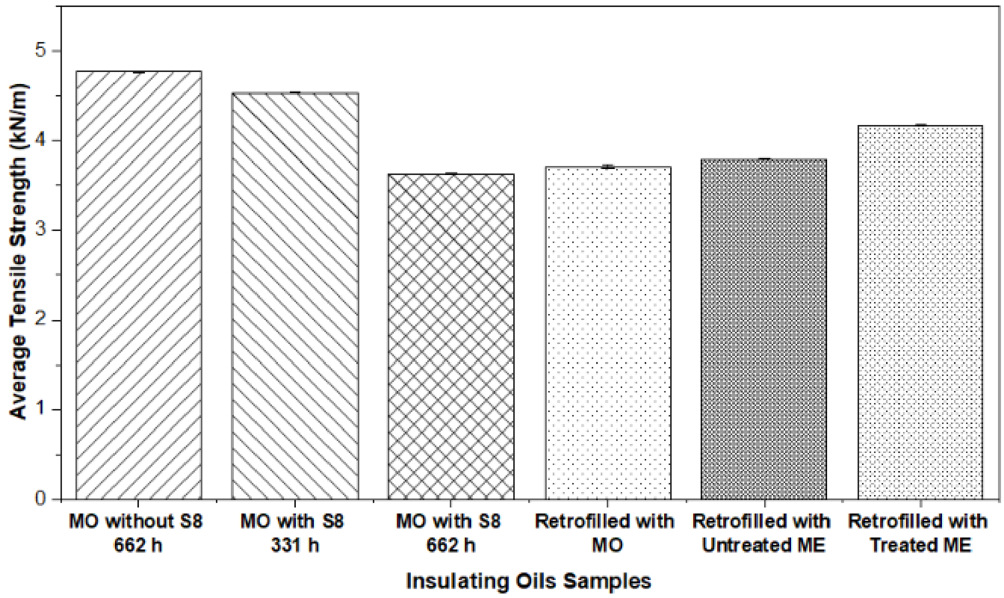

Figure 4. Tensile strength of insulation paper for each oil sample

In addition, methyl ester fluids are known to possess a higher affinity for moisture than mineral oil, which promotes the transfer of moisture from the cellulose insulation into the liquid phase [24]. Since hydrolysis is one of the dominant ageing mechanisms responsible for cellulose chain cleavage, the reduction of moisture within the paper can slow depolymerization and preserve the physical strength of the insulation [29]. This process can reduce hydrolytic degradation within the paper and help preserve the remaining cellulose structure [30]. These findings indicate that treated methyl ester has the potential to mitigate long-term cellulose degradation and extend the serviceability of transformer insulation systems affected by sulphur corrosion.

4. Conclusion

Overall, the objectives of this study were successfully achieved. The following conclusions were drawn based on the key findings obtained throughout the investigation. Firstly, the bleaching and synthetic silicate treatment processes progressively reduced the acidity of the methyl ester, indicating that both treatments contributed to improving the chemical quality of the insulating fluid. Secondly, the effects of retrofilling with mineral oil, treated methyl ester, and untreated methyl ester on the surface resistivity and tensile strength of paper insulation subjected to sulphur corrosion were compared and analysed. The results showed that samples retrofilled with treated methyl ester consistently exhibited higher surface resistivity and tensile strength values than those retrofilled with untreated methyl ester. These findings indicate the ability of retrofilling fluid to preserve the condition of the aged insulation system. Furthermore, the results demonstrate that retrofilling with treated methyl ester can mitigate the deterioration of aged paper insulation and contribute to maintaining both its physical and electrical performance.

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