

Degradation of Thermally Aged Insulation Paper in Mixed Polyol Ester and Used Mineral Oil

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Abstract—This study investigates the mechanical, chemical, and microstructural changes of thermally aged transformer insulation paper in polyol ester and its mixtures with residual used mineral oil. The mixture was intended to simulate retrofilling conditions where complete removal of aged mineral oil from transformers cannot be ideally achieved. The proportions of used mineral oil in the mixtures are 0, 2.5, 5.0, and 7.5% by volume. Kraft paper, cut into approximately 45 cm × 20 cm, was wrapped around 9 g of copper conductor, immersed in about 1000 ml of oil mixture to imitate real transformer conditions. Samples were aged in an oven at 140 °C for 14 and 28 days. The results demonstrate that the chemical and physical integrity of the insulation paper, with tensile strength, FTIR, and SEM analyses consistently indicating increasing cellulose degradation, oxidation, and fiber damage at higher used mineral oil concentrations and longer aging durations. In addition to assessing degradation, the study highlights that hydrolysis of polyol ester generates long-chain fatty acids capable of esterifying cellulose. This reaction contributes to improved paper insulation's thermal stability by slowing depolymerization and limiting further fiber damage. Conversely, the presence of used mineral oil accelerates cellulose deterioration.

Keywords: FTIR Spectroscopy, Kraft paper, Polyol ester, Retro-filled transformer, SEM characterization, Tensile strength.

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1. Introduction

Transformer insulation systems play a critical role in ensuring the reliability and longevity of power transformers. The combined performance of insulating oil and cellulose-based paper insulation determines the overall dielectric and thermal endurance of the equipment [1], [2]. Over time, exposure to high operating temperatures and oxidative environments leads to the degradation of both oil and solid insulation, which in turn reduces transformer efficiency and increases the risk of failure [3], [4]. Traditionally, mineral oil has been used as the primary insulating liquid due to its favorable dielectric properties, low cost, and established performance record. However, its poor biodegradability and low fire resistance have prompted growing interest in ester-based alternatives [5], [6].

Ester based insulating oils, including Polyol esters, have emerged as promising fluids with superior thermal stability, oxidation resistance, and environmental compatibility compared to mineral oils [7], [8], [9]. They offer enhanced moisture tolerance, allowing higher water solubility without compromising dielectric strength, which effectively extends the service life of the paper insulation [5], [10]. Moreover, their high flash and fire points provide improved operational safety, especially in indoor and urban installations [7], [11], [12]. These advantages have made retrofilling with ester-based insulating liquid a preferred option for extending the service life of existing mineral-oil-filled transformers while improving sustainability [13], [14].

Despite these benefits, one of the practical challenges in retrofilling is the incomplete removal of aged mineral oil from the transformer's internal components. Even after thorough draining and flushing, a small fraction of the used mineral oil often remains absorbed in the cellulose insulation or trapped in the system's crevices [5], [15], [16]. This residual oil can mix with the newly introduced ester oil, potentially altering the chemical interactions within the insulation system. Several studies have highlighted that mineral oil residues may induce chemical incompatibility, accelerate oxidation, and deteriorate cellulose integrity under thermal stress. Consequently, understanding the effects of such residual contamination on the mechanical and chemical behavior of insulation materials becomes crucial for evaluating the reliability of retrofilled transformers [5], [17].

Recent research has investigated various aspects of mixed oil–paper systems, including the thermochemical interactions between mineral oil and esters, the kinetics of cellulose degradation under mixed oil conditions, and the changes in dielectric and mechanical properties during accelerated aging [18], [19], [20]. FTIR spectroscopy has been widely applied to monitor oxidation products and the loss of hydroxyl groups in aged insulation papers [21], while Scanning Electron Microscopy (SEM) provides microstructural evidence of fiber damage, void formation, and surface cracking [22]. Mechanical testing, such as tensile strength measurements, further quantifies the degree of cellulose chain scission and loss of structural cohesion [19], [23], [24]. However, limited studies have focused on the degradation effects of small residual fractions of used mineral oil in polyol ester on insulation papers, particularly in simulating transformer oil retrofilling process, under accelerated thermal aging conditions.

This study aims to address this gap by experimentally investigating the influence of residual used mineral oil in polyol ester on the degradation behavior of kraft insulation paper under thermal aging. The experiment involved tensile strength testing, Fourier Transform Infrared (FTIR) spectroscopy, and Scanning Electron Microscopy (SEM) characterization to evaluate the mechanical, chemical, and morphological changes in the insulation paper. The independent variables are the concentration of used mineral oil (0%, 2.5%, 5.0%, and 7.5% v/v) and the accelerated thermal aging duration (14 and 28 days at 140°C). The dependent variables to quantify the mechanical, chemical, and morphological degradation parameters are tensile strength (MPa), FTIR functional group changes (O-H, C-H, and C=O intensities), and SEM fiber integrity.

2. Method

2.1 Specimen

The material investigated in this study was kraft insulation paper, commonly used in transformer windings. The paper was immersed in polyol ester and mineral oil mixtures. The paper specimens were cut into rectangular sheets with dimensions of approximately 45 cm in length and 20 cm in width before immersion. Each sample set contained 10 g of kraft paper, and it was wrapped around 9 g of copper conductor, maintaining a ratio of 10 g of paper per 1000 mL of oil mixture. These specimens were used to simulate the paper–oil ratio of a power transformer, and they were used under accelerated thermal aging conditions.

2.2 Preparation and Conditioning of Paper Insulation

The conditioning process aimed to simulate the thermal aging of kraft insulation paper in different oil environments, representing retrofilled transformer conditions. Polyol ester oil was used as mixtures containing 0%, 2.5%, 5%, and 7.5% used mineral oil by volume. These compositions reflect residual mineral oil portions which typically remain in transformer's tank after a retrofilling process. Each oil mixture was prepared in a 5000 mL glass beaker with a total oil volume of 2000 mL and homogenized using a magnetic stirrer at 250 rpm for one hour.

After mixing, the oil was transferred into 1000 mL thermal bottles containing the prepared kraft paper and copper conductors (Fig. 1). The bottles were sealed and then placed in a laboratory oven and heated at 140°C for 14 and 28 days to induce accelerated thermal aging. The temperature of 140°C was

chosen based on transformer hotspot conditions known to accelerate insulation degradation [1]. After aging, the kraft paper samples were carefully removed from the oil, cleaned of residual fluid, and conditioned in a desiccator to maintain stable moisture content prior to testing. All samples were prepared and aged under identical conditions to ensure consistency.

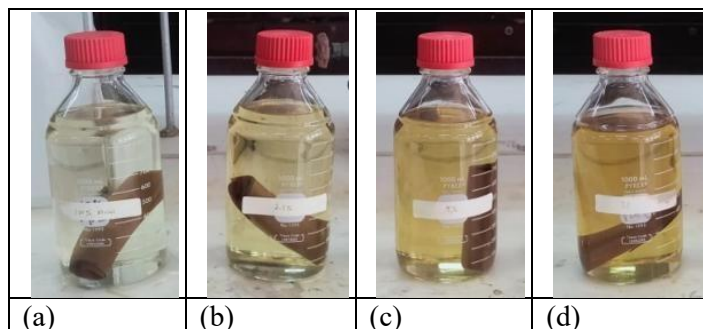


Figure 1. Thermal resistance bottles containing kraft paper immersed in oil mixture with different used mineral oil proportions; (a) 0%, (b) 2,5%, (c) 5%, and (d) 7.5%.

2.3 Tensile Strength Test

Mechanical degradation of kraft paper was evaluated by tensile strength test (Fig. 2). The measurement was conducted using a COM-TEN Testing Machine, 95T Series 5K (Fig. 2.a), in accordance with the ASTM D638 Type V standard. The thermally aged paper was conditioned at room temperature, cut into standardized Type V specimens (Figs. 2.b-f), and then it was mounted carefully between the machine grips for testing. The specimen dimensions and test speed were adjusted following ASTM specifications. During the test, the load and elongation were continuously recorded until specimen failure occurred. The stress–strain data obtained were used to calculate the tensile strength of each sample. Each testing condition was performed with 3 sample repetitions to ensure repeatability.

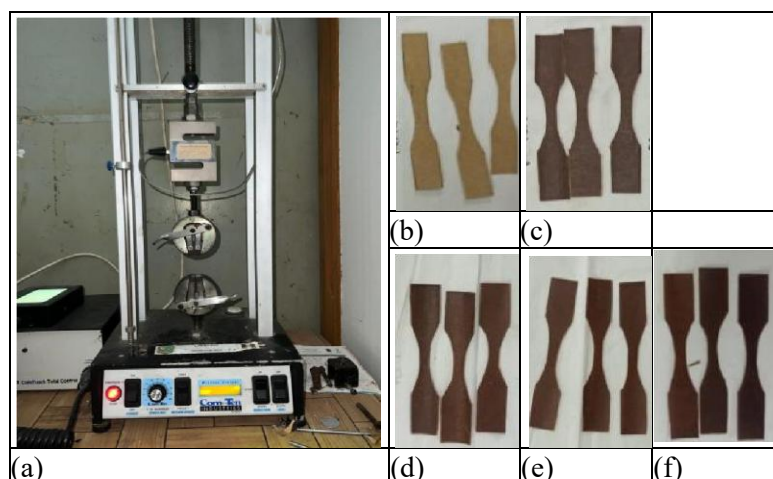


Figure 2. Tensile Strength test for kraft paper; (a) The COM-TEN 95T Series 5K universal testing machine, (b) new paper (c) paper aged in 0.0% mineral oil, (d) paper aged in 2.5% mineral oil, (e) aged in 5.0% mineral oil, and (f) paper aged in 7.5% mineral oil

2.4 Fourier Transform Infrared (FTIR) Spectroscopy

Chemical structure changes in the insulation paper were analyzed using a Shimadzu FTIR spectrometer (Fig. 3). Samples (3×3 cm) from unaged, 14-days, and 28-days conditions were placed on a cleaned FTIR disk and scanned across the infrared spectrum. Spectral data were saved in both Excel and graphical formats and compared to identify variations in absorption peaks associated with the change in functional group of cellulose. The FTIR spectra were recorded in the wavenumber range of

4000–400 cm^{-1} with a resolution of 4 cm^{-1} and an average of 32 scans per spectrum.



Figure 3. FTIR spectrometer used in the experiment

2.5 Scanning Electron Microscopy (SEM) Characterization

Surface morphology was examined using a scanning electron microscope, as shown in Fig. 4. Paper samples aged for 28 days were cut into 5×5 cm sections, mounted on clean specimen holders, and observed at 1000 $\times$ magnification. SEM micrographs were captured at an accelerating voltage of 15 kV using a secondary electron (SE) detector. Prior to imaging, the paper samples were sputter-coated with a thin layer of gold (Au) to prevent charging. The micrographs were analyzed to evaluate fiber separation, surface roughness, and other structural deterioration associated with thermal aging.



Figure 4. SEM characterization

3. Results and Discussion

3.1 Results

The experimental results were obtained from three main characterization techniques: tensile strength testing, Fourier Transform Infrared (FTIR) spectroscopy, and Scanning Electron Microscopy (SEM). These tests were performed to evaluate the effects of residual used mineral oil in the polyol ester under thermal aging conditions, replicating a retrofilling process in which complete removal of aged oil is not feasible.

3.1.1 Tensile Strength

The tensile strength test results on new kraft paper and those aged in polyol ester and mineral oil mixtures at different compositions are shown in Fig. 5. Remaining percentage of tensile strength of insulation paper under thermal stress are summarized in Table 2. The new insulation paper exhibited the highest tensile strength, indicating a robust hydrogen-bonded cellulose fiber network. After thermal aging, a notable decline in tensile strength was observed for all samples, due to both, with increasing used mineral oil content and aging duration. The strength reduction was more pronounced with increasing concentrations of used mineral oil in the ester mixture. The results are in line with those reported in literature [19].

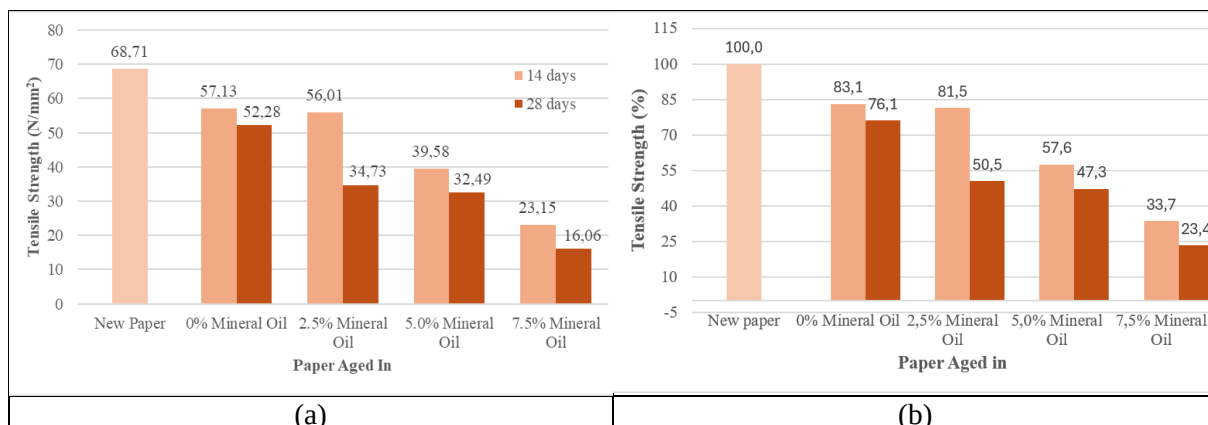


Figure 5. Tensile strength of insulation paper before and after being aged in polyol ester and mixed oils at different percentages of mineral oil; (a) In N/mm², (b) In Percent.

3.1.2 FTIR Spectral Analysis

FTIR spectroscopy was employed to examine molecular structure changes and chemical degradation in the cellulose fibers. FTIR spectra of new and aged Kraft papers for 14 and 28 days in oil mixture with different percentages of used-mineral oil are shown in Fig. 6. As seen in Fig. 6.a, the FTIR spectrum of new insulation paper shows characteristic cellulose absorption peaks: O–H stretching (3700–3000 cm⁻¹), C–H stretching (3000–2700 cm⁻¹), and C–O stretching (1500–900 cm⁻¹) [22].

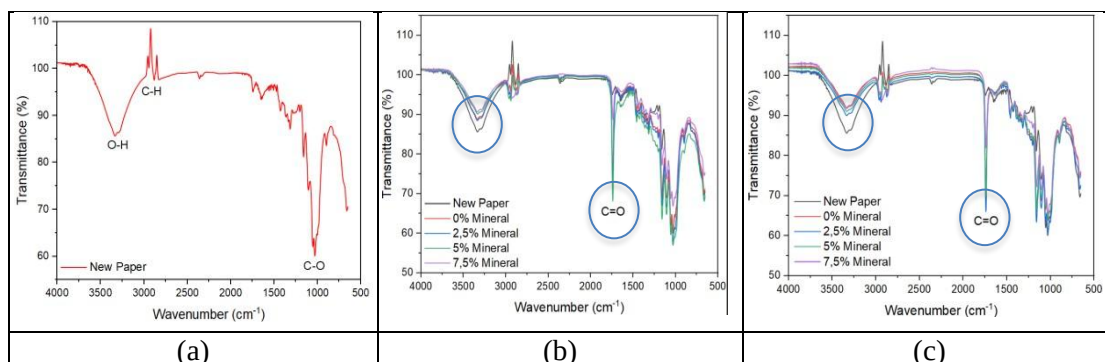


Figure 6. FTIR spectra of insulation paper; (a) new paper (b) paper in different oil mixtures aged for 14 days, (c) paper in different oil mixtures aged for 28 days.

In Figs. 6 (b and c), the FTIR spectra after thermal aging reveal significant structural changes. The O–H absorption band decreased with increasing aging duration and with higher used mineral oil content, indicating the reduction of hydrogen bonding due to cellulose depolymerization. The C–H stretching near 2922 cm⁻¹ increased, suggesting the accumulation of hydrocarbon residues. A new absorption band appeared in the 1800–1700 cm⁻¹ range, corresponding to C=O stretching vibrations, which indicates the presence of esters group [19], [25]. The intensity of this carbonyl band increased with aging time but decreased as the used mineral oil content increased. The C–O region (1500–900 cm⁻¹) showed minor variations, remaining largely consistent with cellulose’s primary molecular framework.

3.1.3 SEM Microstructural

SEM micrographs provide visual evidence of fiber morphology and degradation mechanisms, as shown in Fig. 7. In Fig. 7.a, the new insulation paper displays tightly packed and intact cellulose fibers, with no apparent surface damage. Fig. 7.b shows that after aging in pure polyol ester, micro-voids begin to form due to partial breaking of hydrogen bonds. Fig. 7.c (2.5% used mineral oil mixture) shows a partial recovery of fiber density, suggesting that small traces of mineral oil may assist in fiber

impregnation. However, small pinholes remain visible, indicating incomplete restoration. In Fig. 7.d (5% mineral oil mixture), the fibers appear less distinct and small cracks emerge, indicating incompatibility and progressive degradation. Fig. 7.e (7.5% mixture) shows severe fiber distortion, microcracks, and irregular structure—signs of advanced thermal and oxidative degradation.

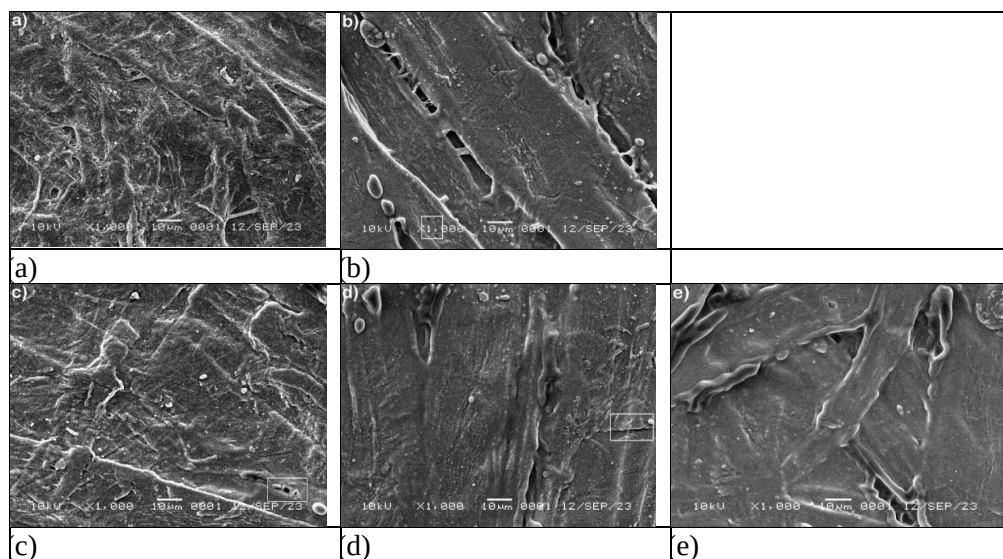


Figure 7. SEM micrographs of insulation paper: (a) new; (b) aged in pure polyol ester; (c) aged in 2.5% used mineral oil; (d) aged in 5% used mineral oil; (e) aged in 7.5% used mineral oil.

3.2 Discussion

The mechanical, chemical, and microstructural analyses collectively demonstrate the degradation progress of cellulose insulation during thermal aging in mixed polyol ester and used mineral oil. The decrease in tensile strength (Fig. 5) correlates directly with the FTIR evidence of O–H group depletion (Fig. 8) as well as the SEM-observed fiber cracking and micro-void formation (Fig. 7). Specifically, the tensile strength decreased from 68.71 N/mm² for new paper to 52.28 N/mm² after 28 days of aging in pure polyol ester and further declined to only 16.06 N/mm² when aged in polyol ester containing 7.5% residual mineral oil, corresponding to a retention of merely 23.4% of the original strength. Consistently, the minimum transmittance in the O–H region (~3300 cm⁻¹) increased from approximately 85.5% for new paper to about 92.0–93.0% after 28 days of aging, indicating a reduction in hydroxyl groups associated with cellulose hydrogen bonding [19], [26], [27]. In contrast, the minimum transmittance in the C=O region (~1740 cm⁻¹) decreased from approximately 95.0% for new paper to 69.5% in pure polyol ester and 82.0% in the 7.5% mineral oil mixture, indicating the formation of carbonyl-containing products through transesterification and oxidation reactions (Fig. 9) [19], [28]. The stronger C=O band observed in pure polyol ester suggests a greater extent of transesterification, which may contribute to reducing cellulose depolymerization [25], [29]. This protective effect becomes less pronounced as the residual mineral oil content increases.

The reduction in O–H absorption band confirms the breakdown of inter-fiber hydrogen bonds, a key factor in mechanical weakening [26], [27]. SEM results visually support these chemical changes, showing the loss of fiber compactness and the formation of cracks as aging progresses [10], [20]. The presence of C=O absorption band indicates the occurrence of transesterification which consists of the substitution of the reactive OH groups of glucose molecules with fatty acids resulted from hydrolysis of esters molecules [25], [28]. It should be noted that the degradation process of esters under thermal aging mainly due to hydrolysis and oxidation, whereas mineral oil predominantly aged through the oxidations [26], [28]. Interestingly, the 2.5% used mineral oil mixture exhibited partial microstructural recovery in SEM, likely due to impregnation that reduces air gaps [8].

Depolymerization in paper insulation is reduced when glucose monomers undergo transesterification, since this modification inhibits the cleavage of esterified glucose units from the polymer backbone [25], [29]. However, this benefit diminishes at higher mineral oil concentrations, where increased oxidation leads to the increase in a non-esterified glucose chain which enhances the cellulose's molecule cleavage [26], [28]. Overall, these findings confirm that residual used mineral oil in polyol ester can accelerate cellulose degradation through oxidation and bond cleavage, resulting in reduced mechanical integrity [5], [28]. This emphasizes the critical role of proper retrofilling procedures and flushing effectiveness in maintaining insulation health.

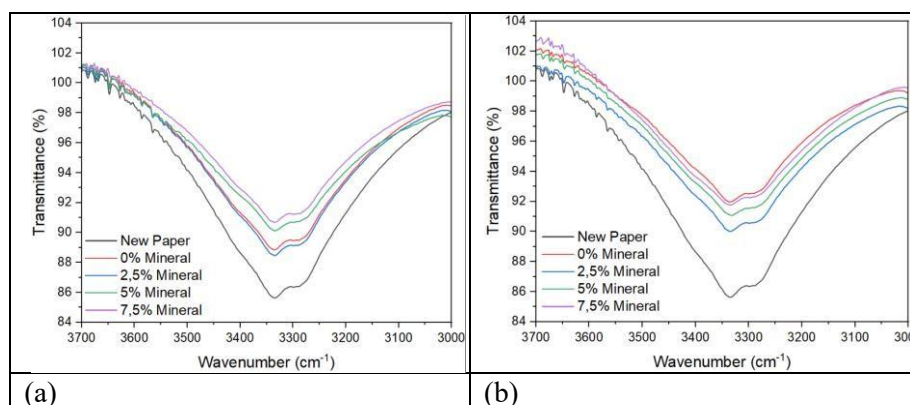


Figure 8. Enlarged view of O-H spectra of new insulation paper and paper aged in oil mixtures: (a) aged for 14 days; (b) aged for 28 days.

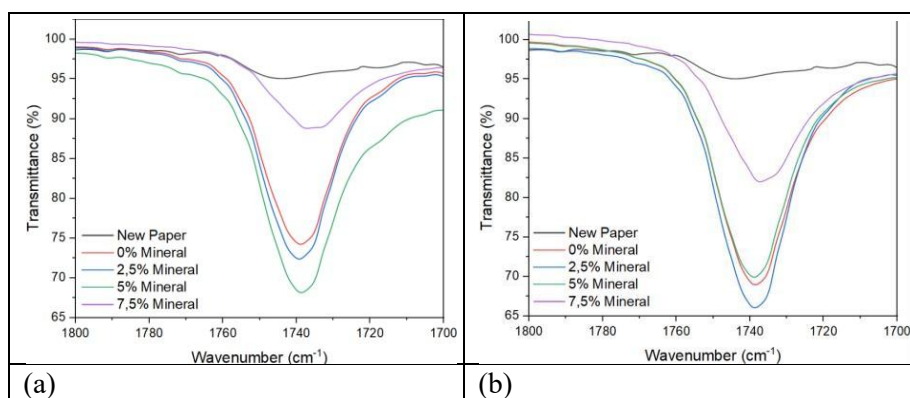


Figure 9. Enlarged view of C=O spectra of new insulation paper and paper aged in oil mixtures: (a) aged for 14 days; (b) aged for 28 days.

4. Conclusion

This study demonstrates that residual used mineral oil can significantly influence the aging performance of cellulose insulation in transformers retrofilled by polyol ester. Increasing mineral oil content resulted in weaker O–H and carbonyl-related FTIR absorption features, greater fiber damage observed by SEM, and lower tensile strength. These results indicate that residual mineral oil accelerated deterioration of the cellulose structure and a reduced beneficial effect of polyol ester under thermal aging conditions. Therefore, minimizing residual mineral oil through effective retrofilling and flushing procedures is essential for preserving the long-term performance of ester-based insulation systems.

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