ORIGINAL RESEARCH PAPER

DC breakdown characteristics and charges accumulation behaviour of thermally upgraded paper aged in natural ester

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Funding information
National Natural Science Foundation of China, Grant/Award Numbers: 52077015, 51707022; National Natural Science Foundation Innovation Research Group Project, Grant/Award Number: 51321063, 111 Project, Grant/Award Number: BP0820005

Abstract
Considering that the working temperature is much higher in high-voltage direct-current (HVDC) convert transformers than in conventional AC transformers, the combination of thermally upgraded paper and natural ester might be a better choice to withstand high temperature in long-term operation. This study compares the chemical structure, DC breakdown voltage, space charge and trap distribution of thermally upgraded paper aged in natural esters and mineral oil. The relationship between DC breakdown and accumulated charges was analyzed. Results show that the thermally upgraded paper aged in natural ester (NEI-TUP) has a higher volume resistivity and DC breakdown voltage than that aged in mineral oil (MOI-TUP). The NEI-TUP group has a larger deep trap density than the MOI-TUP group due to the chemical structure of natural ester. The larger deep trap density could contribute to the lower charge accumulation quantity and higher DC breakdown voltage. The thermally upgraded paper immersed in natural ester presents better insulation capability under thermal and DC electrical stress than that immersed in conventional mineral oil. This research indicated that the combination of thermally upgraded paper and natural ester might be a potential alternative for the mineral oil immersed paper in the future.

1 | INTRODUCTION

Transformers are the most valuable apparatuses in the power transmission grid. Oil-immersed paper insulation is widely used in power transformers due to its high reliability and economic benefit [1]. At present mineral oil and natural ester are two main kinds of insulating liquids used in large transformers. Mineral oil has been utilized for over 100 years due to its good dielectric properties, excellent heat dissipation performance, low freezing point and low viscosity. However, the drawbacks of mineral oil such as poor degradability, low flash point and non-renewability have become more evident with the rapid development of the society and electrical industry [2]. Under this background, many researchers have tried to find an alternative insulating oil which could meet the green and environmentally friendly requirements since 1990s. It now appears that natural ester is the answer due to its biodegradability, non-toxicity and renewability [3–6].

There are more than 2 million transformers filled with natural ester in operation in the world with voltage levels ranging from 10 to 420 kV and capacities ranging from 6.25 to 300 MVA [7]. However, it could be seen that the application of natural ester in transformers with higher voltage levels and in convert transformers within a high voltage direct current (HVDC) transmission system is limited. This could be partly attributed to the lack of experimental works, design criterion and the disadvantages of natural ester itself such as high viscosity and low oxidation stability. Nevertheless, it is believed that natural ester based transformers would play a leading role and that the future of application is fairly brilliant. To achieve this goal and further support the technology progress, there is still much research work that needs to be done.

The insulating performance of the oil-paper depends on the property of both the insulation oil and cellulose paper. For one thing, some research has investigated the comparison of the...
It was observed that natural ester showed higher time-dependent electrical conductivity in comparison with that of mineral oil due to its different molecular structure [9]. Hao et al. studied the DC breakdown and space charge characteristics of mineral oil impregnated kraft paper and natural ester impregnated kraft paper. It was concluded that the kraft paper impregnated in natural ester had a higher DC breakdown strength compared with that of the kraft paper impregnated in mineral oil. In addition, the total space charge amount in natural ester impregnated kraft paper was less than that of the mineral oil impregnated kraft paper [10]. Sivaramalakshmi et al. studied the AC and DC breakdown change of kraft paper impregnated with natural ester with different thermal ageing stage, and concluded that because of decomposition of oil, the breakdown voltage and flash point get decreased meanwhile viscosity gets increased. Then the AC breakdown voltage of oil-impregnated paper insulation gets degraded because of the decline in quality after ageing. Thermal stress leads to the significant degradation process of oil and paper insulation [11]. For another thing, some research focused on promoting the performance of cellulose paper. Sukswat et al. studied the deterioration of impregnated kraft papers immersed in mineral oil and natural ester; their study showed that the amount of furan compounds detected from the FR3 samples is higher than those in the mineral oil samples under same temperature [12]. Besides, the manufacture of thermally upgraded paper was a success. Because of the higher thermo-stability, thermally upgraded paper has been widely used in HVDC transformers, and it has been studied by some researchers [13, 14]. However, most of the studies have mainly focused on its thermal properties and very few on its electrical properties [13, 14].

Nowadays, the development of HVDC transmission is blossoming, especially in China. The working temperature for HVDC converter transformers is relatively higher than that of the conventional AC transformer. Natural ester prevails among the thermal decomposition stability and breakdown strength over mineral oil [6]. From this perspective, natural ester may be more suitable for HVDC apparatus. Considering that the combination of natural ester and thermally upgraded paper has the potential to be applied, the study into the dielectric behaviour of natural ester impregnated thermally upgraded paper is beneficial. In addition, space charge problem becomes more and more severe with the increasing of voltage levels under DC stress. The hazard of space charge accumulation mainly includes electric field distortion which could further trigger degradation and insulation failure. However, rare research focused on the breakdown and charge injection behaviour of the natural ester impregnated thermally upgraded paper.

| Property       | Parameter          | Parameter          | Parameter          |
|----------------|--------------------|--------------------|--------------------|
| Resistivity (Ω-m, 20 °C) | 1.01               | 0.303              |
| Moisture content (ppm)   | <10                | <20                |
| Acid value (mg KOH/g)     | 0.007              | 0.016              |

**TABLE 2** Main parameters for thermally upgraded paper

| Property          | Parameter |
|-------------------|-----------|
| Thickness (μm)    | 160       |
| Resistivity (Ω-m at 20°C) | 1.5     |
| Moisture content (%) | <1       |

This article studies the DC breakdown and space charge characteristics of the aged thermally upgraded paper impregnated in natural ester by comparison with those of the aged thermally upgraded paper impregnated in mineral oil. A thermal ageing experiment was conducted to prepare aged paper samples. Firstly, The DP value of the samples was tested, and the corresponding microstructure and chemical components were analyzed through a scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FT-IR), respectively. Secondly, the DC resistivity of the samples was measured and the DC breakdown tests were conducted with and without pre-stressing. Space charge measurement of the samples was also performed using pulsed electro-acoustic (PEA) method. Finally, the relationship between the charge accumulation behaviour, trap distribution, and the DC breakdown strength was discussed.

### 2 | EXPERIMENTAL ARRANGEMENTS

#### 2.1 | Sample preparation

The thermally upgraded paper was produced by Weidmann Company with a thickness of 0.16 mm and it was cut into a 5 × 5 cm² square sheet. KI-50 X mineral oil and soybean natural ester were used in the experiments. The parameters for both oils are shown in Table 1, and the parameters of the thermally upgraded paper are shown in Table 2.

The thermally upgraded papers were dried in a vacuum box at 60°C for 24 h. Then, the treated paper was immersed in the degassed and filtered mineral oil and natural ester in ground-glass stopper flasks. The weight ratio of oil and paper was around 10:1. These flasks were put into a vacuum box at 60°C for 24 h to make sure of sufficient immersion. Then, the accelerated thermal ageing experiment was performed at 130°C. A previous study showed that the degree of polymerization value (DP) of the mineral oil impregnated kraft paper dropped to below 200 after 50 days of ageing at 130°C [15]. In these experiments, samples were collected at 0, 25, and 55 days, respectively.
Prepared aged samples were then used for the various tests. In order to minimize the influence of humidity from the environment, after the moisture content test, samples would go through the DC breakdown test, volume resistivity test and space charge characteristic measurement within 36 h. In between the tests, all samples were kept in sealed vacuum bags for storage. In the following text, MOI-TUP refers to the mineral oil impregnated thermally upgraded paper and NEI-TUP refers to the natural ester impregnated thermally upgraded paper, respectively.

2.2 Characteristic tests

Scanning electron microscope (SEM) analysis was conducted by Multiview 2000 (Nanonics Imaging Ltd. Company). As for the electric property test, the volume resistivity of the aged samples was tested according to ASTM D257-07 at 25°C. The DC breakdown voltage test was conducted according to the IEC 60243 standard. A pair of plate-plate electrodes with a radius of 12.5 mm was chosen. The increasing rate of voltage was at 1 kV/s until breakdown. For the pre-stressing breakdown test, the samples were pre-stressed at the DC electrical field of 15 kV/mm for 5 min, followed by the increment of the homopolarity DC voltage at 1 kV/s until breakdown.

Space charge characteristics measurement was carried out using the pulsed electro-acoustic method (PEA method). The PEA platform used in the test shown in Figure 1 had an aluminium plate as its bottom electrode and the top electrode as a semiconducting polymer film. It has a pulse amplitude ranging from 0 to 1 kV while the pulse width can be adjusted from 0 to 10 ns with a frequency of 50 Hz. The space charge sensitivity is 0.2 C/m³. In the measurement process, the pulse amplitude was 500 V and the pulse width was selected to be 4 ns. Space charge distribution was measured at a DC electric field of 15 kV/mm and 25 kV/mm, respectively, using the PEA method at 15°C. A negative DC voltage was applied.

3 RESULTS AND ANALYSIS

3.1 SEM analysis

The microstructure change for the samples after the aging process is shown in Figure 2. By comparing Figures 2(a) and 2(c), it could be found that the thermal ageing process caused more voids within the MOI-TUP, but the phenomenon of crack formation cellulose fibre structure was not very evident. It was also observed from Figures 2(b) and 2(c) that fewer changes took place in the NEI-TUP's fibre structure. This indicated that the deterioration influence of the thermal ageing process on NEI-TUP was less serious than that on MOI-TUP.

| Aging days | MOI-TUP (Ω/m) | NEI-TUP (Ω/m) |
|------------|---------------|---------------|
| 0          | $1.04 \times 10^{12}$ | $3.39 \times 10^{12}$ |
| 25         | $2.75 \times 10^{12}$ | $4.51 \times 10^{12}$ |
| 55         | $3.29 \times 10^{12}$ | $4.46 \times 10^{12}$ |

3.2 DC volume resistivity

The DC volume resistivity of the samples aged for different days is shown in Table 3. The NEI-TUP samples showed higher DC volume resistivity than MOI-TUP samples at the same ageing time. With the increase in ageing time, the resistivity of MOI-TUP had increased from $1.04 \times 10^{12}$ to $3.29 \times 10^{12}$ Ω/m, while the figures for NEI-TUP group had increased from $3.39 \times 10^{12}$ to $4.46 \times 10^{12}$ Ω/m. The volume resistivity of the oil-impregnated paper depends on several factors, for example, DP value, acid value and moisture content [16]. Higher DP values mean more cellulose molecules and less immersed oil per unit volume, leading to higher volume resistivity.
Small-molecule acids were produced during the ageing process, so the acid values for both MOI-TUP and NEI-TUP were increasing with the increase of ageing time as shown in Table 4. The production of these acids resulted in a decrease in the resistivity. Besides, the moisture contents for all the samples are shown in Table 5. It was found that the moisture content for both groups of samples decreased with the increase of the ageing time, which could bring about a potential increase in resistivity. In this case, it could be inferred from the increase in the resistivity values of samples with the increase of ageing time that the decline in moisture content was the dominant factor which influenced the resistivity.

3.3 DC breakdown field strength

The testing result of DC breakdown field strength without pre-stressing of the aged samples is shown in Figure 3(a,c). Normally the values of breakdown field strength for the oil-impregnated paper were decreased with the increase of ageing time. It was observed that the average values of DC breakdown field strength of the NEI-TUP were higher than those of the MOI-TUP given the same ageing time. To be specific, the average value of DC breakdown field strength for new NEI-TUP was 16.76% higher than that of new MOI-TUP, and the figures for NEI-TUP samples aged for 25 days and 55 days were 12.49% and 15.03% higher than that of MOI-TUP, respectively. Figure 3(b,d) shows the result of the DC breakdown test with pre-stressing. It was found that the figures were on a marginal increase with the increase of ageing time. This variation trend was contrary in comparison with that of the breakdown test without pre-stressing. The average values of DC breakdown field strength with pre-stressing for NEI-TUP were still higher than those of MOI-TUP. To be specific, the average value of the breakdown field strength for the new NEI-TUP was 13.48% higher than that of MOI-TUP. The figures for NEI-TUP aged for 25 days and 55 days were 6.99% and 8.33% higher than the figures for MOI-TUP. The degradation resulting from thermal stress was gradually developed during the thermal ageing process. It is noteworthy that the pre-stressing DC breakdown values were not in decrease but in increase. This phenomenon was related to the space charge characteristics.

3.4 Space charge characteristic

The threshold voltages for charge injection of all the samples were determined and the result is shown in Figure 4. Y-axis represented the corresponding voltage values of the test signal in the PEA system. This test showed the I–V curves of the samples in essence. When charge injection occurred, the slope of I–V curves would increase dramatically, thus the threshold
voltage could be determined. It was observed that the threshold voltage of charge injection for MOI-TUP was 0.5 kV (corresponding to the electric field of 3.125 kV/mm), while the charge injection threshold voltage for NEI-TUP was 0.7 kV (4.375 kV/mm). Results showed that thermal ageing did not influence the threshold voltage for both groups of samples.

During the charge injection threshold voltage test, it is found that a 5 kV/mm field strength can ensure the presence of space charge. In order to get a clear test result, 15 kV/mm field strength (three times higher than 5 kV/mm) was chosen as the external field strength applied. In order to study the influence of increasing field strength with the consideration of safety, 25 kV/mm field strength was chosen as the next applied external field strength. The space charge characteristics for MOI-TUP samples with different ageing days under the DC electrical field strength of 15 kV/mm are shown in Figure 5(a,c,e). Homo-charge injection was observed at the interface of both electrodes for all the samples. With the time of applied voltage increasing, the negative charge carriers injected into the bulk of samples from the cathode. For the unaged samples, the charge densities at both electrodes increased in the beginning and then decreased ending with the charge density reached a steady state at around 30 C/m³. For the samples aged for 25 days, the charge densities at anode and cathode reached a steady state around 30 and 60 C/m³, respectively. The steady-state charge densities at both electrodes for the sample aged for 55 days were only about 10 C/m³. The space charge distribution for NEI-TUP samples with different ageing days at a DC electric field of 15 kV/mm are shown in Figure 6(b,d,f). It was observed that for NEI-TUP samples aged for different days, the steady-state charge densities on the anode were in the range of 15–20 C/m³ and the figures for cathode were in the range of 20–40 C/m³, respectively.

The space charge characteristics for MOI-TUP and NEI-TUP with different ageing days for 25 kV/mm are shown in Figures 6(a,c,e) and 6(b,d,f), respectively. It could be seen that the steady-state charge densities at the interface of both electrodes increased and the charges moved more quickly at higher applied DC electric field strength. Similar charge injection phenomena were also observed near the electrodes.

The total charge amount \( Q \) in the oil-impregnated insulation paper at a steady state was calculated based on Equation (1) [17]. In this equation, \( J \) stands for the area of the sample, \( l \) stands for the thickness of the sample, \( q(x) \) represents the charge density at position \( x \), and \( 0 \leq x \leq l \). At a steady state, the total charge amount for MOI-TUP and NEI-TUP with different ageing days...
under different DC electric field strengths is shown in Figure 7.

\[ Q = S \int_0^T |q(x)| \, dx. \]  (1)

The increment of the applied DC field strength significantly increased the total charge amount for new MOI-TUP samples, while rise amplitude for new NEI-TUP samples was much lower. With the increase of the ageing time, the total charge amount of the 55-day aged MOI-TUP decreased, especially at a higher applied field strength. At 15 kV/mm, thermal ageing seemed to have no influence on the charge amount of NEI-TUP samples. At 25 kV/mm, the space charge amount of the NEI-TUP samples decreased at first and then increased with the increase of ageing time. It could be inferred from the above comparison that the space charge amount of MOI-TUP samples was more sensitive to the applied electric field and ageing time.

4 | DISCUSSIONS

4.1 | Thermal ageing and trap level change

The trap levels and distribution densities of the new and aged samples were also tested using the method of thermal stimulated depolarization current (Novocontrol Concept 80). The trap levels for the new and aged samples were measured and calculated based on [18]. The equation to calculate trap level and trap density is given below in Equation (2),

\[ f_0(E_m)N_i(E_m) = \frac{2d}{el^2}J(T)/A(E_m), \]  (2)

where \( f_0 \) is the initial occupancy of a trap level and it is a constant; \( E_m \) is the energy level; \( N_i \) is the distribution function of the trap, which is spatially uniform deep into the sample with a distance \( \xi \); \( d \) is the thickness of the sample; \( e \) is the electronic charge quantity; \( J(T) \) is the measured thermally stimulated current; \( A \) is a function of \( E_m \). It was assumed that all the traps were initially filled and that \( f_0 = 1 \). The trap levels for the MOI-TUP and the NEI-TUP are shown in Figure 8.

The highest trap level for the new MOI-TUP group was 1.123 eV with a density of \( 3.43 \times 10^{15} \text{ m}^{-3} \text{ eV} \). After thermal ageing, the trap density increased to \( 1.39 \times 10^{16} \text{ m}^{-3} \text{ eV} \). No change was observed in the highest trap level. However, the highest trap level for the new NEI-TUP group was 1.141 eV with a density of \( 2.64 \times 10^{17} \text{ m}^{-3} \text{ eV} \). After thermal ageing, the trap density increased to \( 4.02 \times 10^{17} \text{ m}^{-3} \text{ eV} \) with no change in trap level also. The trap density for the new NEI-TUP was 75 times more than that of the new MOI-TUP and the trap density for the aged NEI-TUP was 25 times more than that of the MOI-TUP.

The thermally upgraded paper was mainly made of cellulose fibre. The main degradation mechanism involved oxidation and hydrolysis in the process of thermal ageing, which led to the decomposition of the cellulose chain. The ageing byproducts were mainly acidic products, organic compounds such as ethanoic aldehyde and furfural [19]. As shown in Figure 10, the mineral oil was made of fossil oil, and it consisted of hydrocarbon compounds with various bonds [6]. These molecule structures could be divided into paraffinic, naphthenic, aromatic and olefin bounds. Natural ester molecules are called triglycerides, originating from the chemical linkage of three fatty acids to one.

**FIGURE 7** Total charge quantity for MOI-TUP and NEI-TUP under different field strength with different ageing days

**FIGURE 8** Trap level for MOI-TUP and NEI-TUP with different ageing days. (a) MOI-TUP, (b) NEI-TUP
4.2 | Space charge accumulation and DC breakdown

After thermal ageing, the volume resistivity for MOI-TUP increased by 216%, and the trap density for MOI-TUP increased by 305% with thermal ageing. The volume resistivity and the trap density for NEI-TUP were increased by 32% and 52%, respectively. As observed in [22], the increase in deep trap density has reduced the carrier mobility, enhanced the height of the injection barrier, and reduced the accumulation of the space charge. Therefore, despite the deterioration of the material structure, the effect of the deep traps on the increase of resistivity was significant. As shown in Figure 9, the trap density for NEI-TUP was much higher than that of MOI-TUP. The resistivity for the new NEI-TUP was 225% higher than that of the new MOI-TUP. The resistivity for the aged NEI-TUP was 35% higher than that of MOI-TUP.

With the Schottky injection theory [23], the injected charge carriers can be described by Equation (3). In Equation (3), $J$ is the charge quantity injected into the sample, $A$ is the Richardson constant, $T$ is the temperature with units of K, $\varphi_B$ is the injection barrier energy level with units of eV, $k_B$ is Boltzmann constant, $\epsilon$ is the electron’s charge quantity, $E$ is the applied field strength, and $\varepsilon$ is the relative dielectric constant.

$$J = AT^2 \exp\left(-\frac{\varphi_B}{k_B T}\right) \exp\left(\frac{1}{k_B T} \sqrt{\frac{\epsilon^3 E}{4\pi \varepsilon_0 \varepsilon}}\right).$$

From Equation (3), it is concluded that the higher field strength increases $E$ resulting in an increase in the quantity of injected charge carriers. Therefore, for both the MOI-TUP and the NEI-TUP, the increase of the electrical field strength has increased the steady-state charge quantity for all ageing stages. The increase of the deep trap density has enhanced the injection barrier level ($\varphi_B$) and reduced the space charge injected into the sample [23]. The NEI-TUP had a much higher deep trap density and volume resistivity than the MOI-TUP. The higher deep trap density and volume resistivity had reduced the charge carriers injected into the sample. Therefore, the overall charge quantity at the steady state for the NEI-TUP was less than that of the MOI-TUP.

The relationship between the space charge accumulation and the DC breakdown behaviour is shown in Figure 10. Under external DC field, charges (electrons and holes) are injected into the sample and migrate to the opposite electrode side. Due to the presence of physical and chemical defects of the sample, traps with different energy levels are distributed in it. During the migration process, charges can be entrapped forming space charge, while some entrapped charges can get sufficient energy from the external field and de-trapped. The accumulation of space charge can distort the local field, and cause a breakdown in the most serious area of the sample. With the same ageing time, the average DC breakdown field strength for the MOI-TUP and the NEI-TUP with pre-stressing was higher than that without pre-stressing. With the increase of the ageing time, the difference in the DC breakdown field strength became more significant. The difference was the result of the combined influence of the variation in material structure, resistivity and trap density. The deep traps could increase the DC breakdown field strength for polymers by reducing the charge injection and the mobility of the charge carriers [19]. The DC voltage pre-pressing process allowed the trap to play a better role. In general, the pre-stressing caused a homo charge injection in the sample. The injected space charge reduced the
local field strength near the electrode and increased the DC breakdown field strength. In addition to the increase of resistivity, after thermal ageing, the total charge quantity of the MOI-TUP decreased and alleviated the local electrical field distortion, which could also contribute to the increase of the DC breakdown field strength.

4.3 Natural ester’s influence on TUP

NEI-TUP showed higher resistivity compared with MOI-TUP at any thermal ageing status even with the fact that the NEI-TUP group showed a higher moisture content at every stage in the thermal ageing process. Impregnating in natural ester, the thermally upgraded paper had an increase in DC breakdown strength compared with the MOI-TUP group. Moreover, with the thermal ageing process, the decrease of moisture content gave a further increase for NEI-TUP’s resistivity and kept its dielectric strength to counteract a potential dielectric strength loss from the decrease of DP value. According to [20, 21], natural ester is hydrophilic, and it will absorb the moisture from the thermally upgraded paper during the thermal ageing process, which further alleviates the damage of the moisture to the microstructure and the decrease of the degree of polymerization during the thermal ageing process. At 0 and 25 days, NEI-TUP had better performance to reduce the accumulation of space charge. However, at the end of the experiment, it had a higher space charge quantity compared with MOI-TUP. The higher accumulation of space charge compared with the MOI-TUP group can be the result of two factors: an increase of deep trap density which can cause more charges to be entrapped and an increase of resistivity which can increase the difficulty for charges to migrate in samples.

5 CONCLUSIONS

From the experimental results above, the following conclusions were drawn:

The NEI-TUP group had a higher DC volume resistivity than the MOI-TUP group for every ageing status. The DC breakdown field strength for NEI-TUP was about 15% higher than the MOI-TUP in the process of thermal ageing. After thermal ageing for 55 days, the volume resistivity for the MOI-TUP group decreased and alleviated the local electrical field distortion, which could also contribute to the increase of the DC breakdown field strength.

The NEI-TUP group had a higher deep trap density than the MOI-TUP group. This contributed to its higher volume resistivity, less charge accumulation quantity, and higher DC breakdown field strength. The increase of the deep trap density caused the DC breakdown field strength enhancement with the thermal ageing status.

Natural ester showed an overall better electrical and thermal performance compared with mineral oil when impregnated with thermally upgraded paper during the thermal ageing process. However, because of the poor fluidity of natural ester, its heat dissipation performance is not as good as that of mineral insulating oil. Therefore, when it is applied to the transformer, the working temperature of the transformer may be higher. Whether natural ester at a higher temperature can guarantee its better electrical performance needs further research.

ACKNOWLEDGEMENTS

This work was supported by the National Natural Science Foundation of China (52077015), the National Natural Science Foundation of China (51707022), the National Natural Science Foundation Innovation Research Group Project (51321063) and the 111 Project (BP0820005).

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How to cite this article: Zou Runhao, et al. DC breakdown characteristics and charges accumulation behaviour of thermally upgraded paper aged in natural ester. IET Sci. Meas. Technol. 1–9 (2021).
https://doi.org/10.1049/smt2.12070