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Effects of Degraded Optical Fiber Sheaths on Thermal Aging Characteristics of Transformer Oil

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Abstract: With the development of fiber optic sensing technology, optical fiber sensors have been widely used in online monitoring of power transformers. To investigate the influence of aging fiber sheaths on transformer oil, two kinds of special optical fibers with thermoplastic polyester elastomer (TPEE) and poly tetra fluoroethylene (PTFE) as sheaths underwent thermally accelerated aging in transformer oil at 130 °C. The volume resistivity, dielectric dissipation factor (DDF), and breakdown voltage of the oil were measured to indicate insulation strength. The water content and acid value of the oil were measured and fitted to predict the aging tends. The thermal aging characteristics of the oil were quantitatively compared and results showed two kinds of optical fibers could exacerbate all the physical and chemical parameters of oil, and the TPEE sheath had a more significant impact on the oil. The reasons contributing to such phenomenon were analyzed using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Both TPEE and PTFE were depolymerized in high-temperature transformer oils, producing water and small molecule hydrocarbon groups. The accumulation of small hydrocarbon groups promoted positive feedback of pyrolysis in the oil. The free hydrogen produced by oil pyrolysis increased the acidity of the oil, which in turn increased the solubility of the water produced by sheath depolymerization. The chain depolymerization of TPEE was more severe than that of PTFE, further exacerbating the deterioration of TPEE-containing oil.

Keywords: transformer; optical fiber; copolymer; thermal aging properties

1. Introduction

The power transformer is one of the most important electrical pieces of equipment in power systems. In recent years, due to the unique advantages of optical fiber sensors such as high immunity to electromagnetic interference (EMI), strong corrosion resistance and low unit cost [1–3], optical fiber sensors are widely used in online monitoring to improve the reliability of power transformers. However, when the optical fiber laid along transformer windings is running at high temperatures for a long time, the oil insulation system of the transformer is affected by the degraded optical fiber sheath [4]. The development of better fiber sheaths and assessment of thermal aging characteristics of the oil containing fiber have become the focus of worldwide research.

Oil-paper insulation is an important part of internal insulation in oil-immersed power transformers [5–10]. As the dielectric constant of the insulating oil is much lower than that of the paper, the field strength of the oil is higher than that of the paper in this insulating scenario [11]. Thus, partial discharge in the oil can occur easily [12,13]. The influence of thermal aging on the electrical properties of the oil and the corresponding mechanisms is a concern which can impact the operational stability of transformers. In the process of thermal aging, the main reason for the deterioration of insulating oil is the pyrolysis reaction. Thermal aging experiments have been performed by many researchers over the past decades to study the aging mechanisms of oil and variations of aging parameters [14–18]. However, most studies have focused on the effects of insulating paper, copper, water, and oxygen on the oil. Few results are available for fiber-containing oil.

Optical fiber sensors in transformers, based on the fiber Bragg grating (FBG), fluorescence, the Brillouin scattering principle, and Raman scattering principle, are used to measure the partial discharge, temperature, and winding deformation [19–23]. Ordinary optical fiber sheaths, such as polyethylene, low-smoke zero-halogen, and polyvinyl chloride have successively been used in the field of communications [24]. However, they cannot operate at high temperatures in transformers. Special optical fibers which have polyimide coating and heat resistant outer sheaths are the most ideal built-in optical fiber sensors used in transformers. Two typical commercial sheaths are thermoplastic polyester elastomer (TPEE) and poly tetra fluoroethylene (PTFE) [25]. These two special optical fiber variants are being used in the insulation of wires and cables that are exposed to highly corrosive environments across the word [26].

Some previous studies have been carried out to test the thermal aging properties of TPEE and PTFE in air. Only a few reports have focused on changes in the properties of oil-polymeric materials during the thermal aging process. In 2017, Shuguo Gao [27] analyzed the thermal aging stability of fluorescent fiber sheaths of cross-linked polyolefins, polyurethanes, and polyvinyl chlorides at different temperatures in oil. The experimental results show that the cross-linked polyolefin and polyurethane sheaths are decomposed, and the performance of the polyvinyl chloride (PVC) sheath had little change. However, the influence of TPEE and PTFE sheaths on aging characteristics of oil and related mechanisms have not been fully addressed.

In this paper, transformer oil was aged with TPEE and PTFE in parallel to investigate the influence of the sheaths of optical fibers on the thermal aging characteristics of oil. Firstly, accelerated thermal aging tests were performed at 130 °C. At the same time, pure oil without any optical fibers was set as a reference sample. Secondly, the volume resistivity, dielectric dissipation factor (DDF), and breakdown voltage of oil were measured to indicate insulation strength. Changes in the chemical properties of fiber-containing oil samples, such as water content and acidity, were fitted to predict the aging tend. Finally, Fourier transform infrared spectroscopy (FTIR) was used to analyze the change in oil molecule structures before and after aging. Scanning electron microscopy (SEM) was used to observe the microstructure of the sheath and aging mechanisms were also investigated.

2. Experimental Methods

2.1. Preparation of Test Material

The test materials were Karamay #25 naphthenic transformer oil (China National Petroleum Corporation, Xinjiang, China) and two types of special optical fibers. The entire fiber was made of silica and had a polyimide coating and heat resistance on the outer sheaths. The two types of outer sheaths were PTFE and TPEE. The specific pre-treatment processes were as follows.

(1) The two types of special optical fibers were placed in a stainless steel tube with an inner diameter of 2.5 mm for drying and de-aerated at 80 °C/50 Pa for 48 h.

(2) The weight ratio of oil and special optical fibers was 20:1, the dried 9.25 g sample of optical fiber and degassed 185 g sample of new transformer oil were fully immersed for 24 hours under conditions of 40 °C/50 Pa.

(3) According to the ratio of 0.05 cm^2 copper to 1 g oil, the copper foil was added [28].

(4) The oil samples of the experimental groups were set with PTFE or TPEE fiber, and pure oil without any optical fiber was set as a reference sample.

(5) The prepared samples were placed in a vacuum aging tank at 130 °C. This tank was evacuated, then nitrogen was added and repeated three times, and finally maintained at standard atmospheric pressure.

(6) Three groups of oil samples were set up with nine aging steel bottles in each group. The total number of samples was 270. We checked the oil samples every three days. Five kinds of aging characteristics in insulation were measured including water content, acid value, DDF, volume resistivity, breakdown voltage, and infrared spectroscopy of oil.

2.2. Test Platform

2.2.1. Electric Performance of Oil Test

The DDF ($\tan\delta$) and volume resistivity (ρ) of the oil reflected its electrical properties. The $\tan\delta$ and ρ of the oil were measured according to IEC 61620 (International Electrotechnical Commission, Geneva, Switzerland). The tester model was LST-121 (Zhong Hang Ding Li, Beijing, China), and test electrode gap was 2 mm. Test voltages included 2000 V of AC and 500 V of DC at 90 °C.

The dielectric strength of the insulating oil was one of the indicators of oil performance. The breakdown voltage of the oil was measured according to IEC 156. Tests were carried out as follows.

(1) The disk electrodes selected had a diameter of 25 mm and the distance between them was 2.5 mm. The test temperature was 20 °C.

(2) Before voltage was applied, initial oil was put into the electrode systems. The oil surface was 5 cm above the electrodes and the oil was allowed to stand for 30 minutes.

(3) Voltages were then applied on the electrodes with a voltage rate increase of 1 kV/s until breakdown of the oil. Samples were then held for 0.5 h.

(4) The oil was changed and the tests repeated.

2.2.2. Acid Values and Water Content Test

Water and acid in the oil can weaken the electrical properties of the insulation system causing degradation of the insulation material. Therefore, the water and acid in the oil are strictly regulated in the actual production of electricity [29]. In this paper, the water content of the initial oil sample was less than 15 mg/L. The Karl Fischer titration method was used to test the water content of various oil samples. The tester model was JWS-1(Xuan Wei, Baoding, China).

Macromolecular acid was produced by the decomposition of insulating oil in heat aging. The detection of acid values in the transformer can assess oil aging. The acid values of the oil sample at different heat aging periods were measured using a JKCS-3 (Jin Ke Hui, Baoding, China) automatic tester according to IEC 62021-1 (International Electrotechnical Commission, Geneva, Switzerland).

2.2.3. Infrared Spectroscopy of Oil

Spectra were obtained using a Nicolet Model 6700 research-grade fourier transform infrared spectrometer (Thermo Scientific, Shanghai, China). The number of scans in the experiment was set at 32 with a resolution of 4 cm^{-1} and a wave number range of 4000–500 cm^{-1}. The final result is shown as absorbance Abs. Air background was scanned prior to each measurement.

2.2.4. Optical Fiber Microscopic Morphology

The secondary electron resolution of SEM (Phenom, Shanghai, China) was 1.5 nm, the backscattered electron resolution was 3.0 nm, and the magnification was 20 to 200,000 times. During the aging, polymer insulation can generate microscopic defects. The microstructures of the optical fiber sheaths in different thermal aging stages were observed.

3. Results and Discussion

3.1. Electric Performance of Oil

The DDF ($\tan\delta$) is closely related to the structure and properties of insulating materials. It is a macroscopic physical quantity which comprehensively reflects the polarization behavior of the dielectric. Figure 1 shows that the DDF of oil increases during aging. After 24 d of aging, the DDFs of the PTFE and TPEE-coated optical fibers were 2.5 and 4.5 times that of pure oil, respectively.

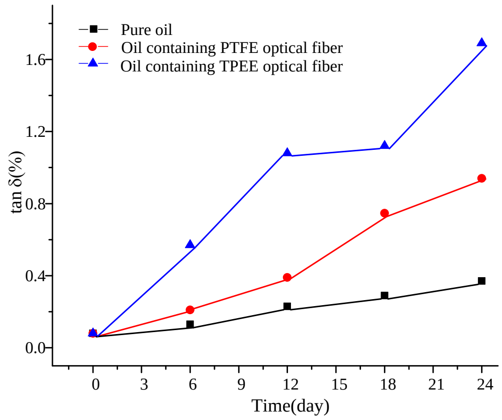


Figure 1. The dielectric dissipation factor (DDF) of oil. PTFE is poly tetra fluoroethylene; TPEE is thermoplastic polyester elastomer.

Volume resistivity (ρ) is a quantitative physical description of dielectric conductivity. For the oil, the volume resistivity partly reflects the degree of contamination and aging. As shown in Figure 2, volume resistivity decreases significantly during aging. The decreasing trend of oil samples containing optical fibers is steepest in the early stage of aging and is moderate as time progresses. The volume resistivity of the two kinds of oil samples containing fibers does not change significantly during thermal aging which were both 33.1% of pure oil.

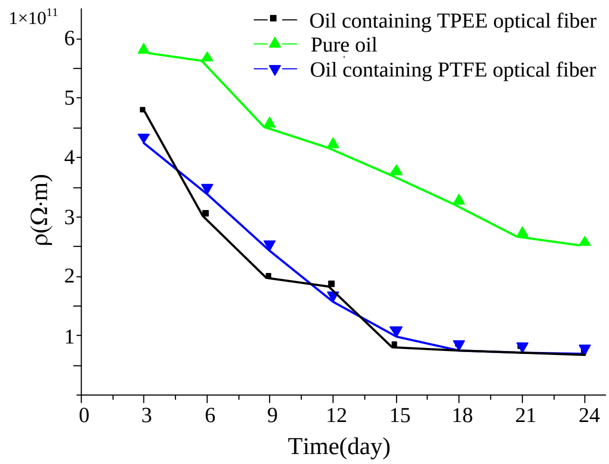


Figure 2. Volume resistivity of oil.

According to the test method of the electrical parameters, the oil sample was subjected to a dielectric strength test. For each set of insulating oil samples, six breakdown voltage tests were performed. The arithmetic mean value was calculated to obtain the corresponding average breakdown voltage (U). The results are shown in Figure 3.

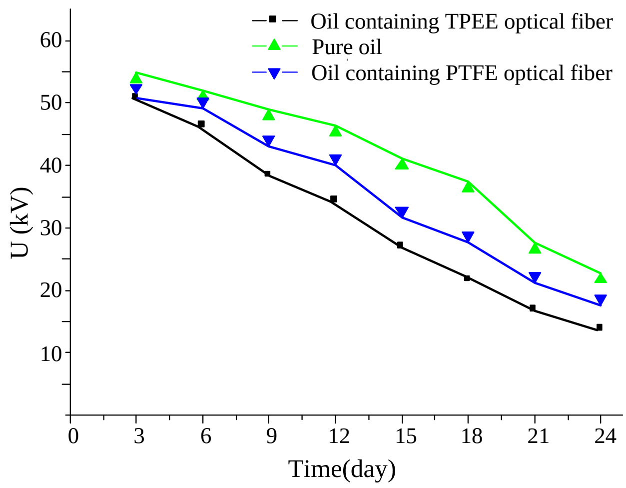


Figure 3. Breakdown voltage of oil.

It can be seen from Figure 3 that the breakdown voltages of all three oil types show a gradual decline along with increasing heat aging time. However, there is a difference in the dielectric strength of the fiber-containing oil compared to the pure oil. The breakdown voltage of the fiber-containing oil is lower than pure oil, and this phenomenon is most obvious in the later stages. At the end of thermal aging (24 days), compared with pure oil, the breakdown voltages of the PTFE- and TPEE-containing oil decreased by 14.3% and 33.1%, respectively. The influence of the optical fiber sheath on the dielectric strength of the oil can be classified as follows: in the initial stage of thermal aging, the impact of the sheath on the breakdown voltage of the insulating oil is similar to that of the pure oil sample; in the middle and end stages, compared with the pure oil sample, the sheath greatly affects the breakdown voltage of oil.

3.2. Water Content and Acid Value of Oil

As shown in Figure 4, the water content of the oil samples continuously increases during thermal aging. At the early aging stage, the water value of the oil containing the TPEE fiber was lower than that of the oil containing the PTFE fiber. After seven days of aging, the water value of the oil containing the TPEE fiber increases rapidly, exceeding that of the other oil. At 24 days of aging, the water of the two kinds of oil containing fibers showed an exponential growth trend with the oil sample containing the TPEE fiber growing faster.

According to the fitting relationships between water contents and aging time shown in Table 1, good exponential relationships were observed. The R-squared values of the two exponential relations are all over 0.92.

Table 1. Fitting relations between the water contents and aging time.

Oil Category	Fitting Equation $W = A + B \times \exp(C \times t)$	R^2
oil containing TPEE fiber	$W = -309.612 + 324.164 \times \exp(0.0037 \times t)$	0.98
oil containing PTFE fiber	$W = -140.324 + 157.927 \times \exp(0.0054 \times t)$	0.95

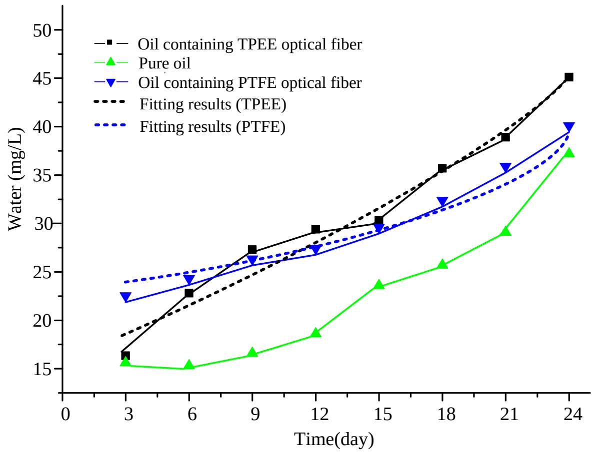


Figure 4. Water content of oil.

The acid value results are shown in Figure 5. In the early stages of aging, the acid values of all types of oil samples are approximately equal. As aging progresses, the acid value of each oil sample increases regularly; however, the acid value of oil containing TPEE fiber increases rapidly after 15 days and exceeds that of oil containing the PTFE fiber at 18 days. At the end of aging, the acid value curves of the TPEE-containing oil samples tend to show an exponential growth trend. However, the acid value curves of the PTFE-containing oil samples tend to show much slower growth. According to the fitting relationship between acid values and aging time shown in Table 2, it was observed that the acid value curves of the TPEE-containing oil samples tend to show an exponential growth trend, however, the acid value curves of the PTFE-containing oil samples are slower.

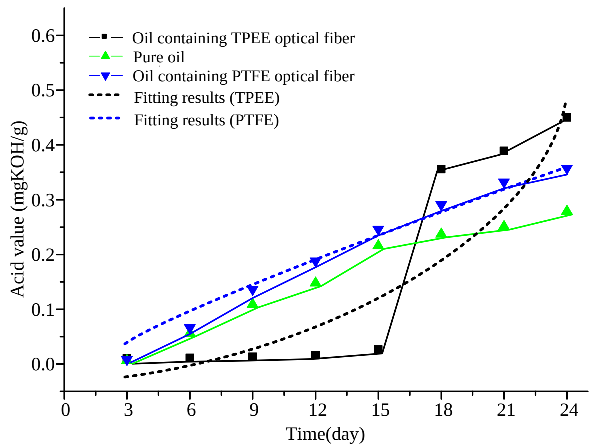


Figure 5. Acid of oil curve.

Table 2. Fitting relations between the acid values and aging time.

Oil Category	Fitting Equation $V = A+B \times \exp(C \times t)$	R^2
oil containing TPEE fiber	$V = -0.069 + 0.039 \times \exp(0.1113 \times t)$	0.93
oil containing PTFE fiber	$V = 2.553 - 2.577 \times \exp(-0.0067 \times t)$	0.98

3.3. Test Analysis

Through comparison of the main thermal aging characteristics of the oil in different aging stages, it was found that all types of built-in optical fibers could exacerbate the oil characteristics and affect several of them. In thermal aging, the main reason for the deterioration of insulating oil is the pyrolysis reaction, which generates organic acids and unstable small molecule hydrocarbon radicals. The hydrocarbon in acid is strongly hydrophilic group, which can increase the saturated solubility of water in oil [30]. Therefore, the water and acid values of all kinds of oil samples increase. The mineral oil degradation process can be expressed as follows.

$$RH + e^* \rightarrow R^* + H^*$$

e^* is the energy acting on the mineral oil molecule RH, R^* and H^* are hydrocarbon radicals and hydrogen radicals, respectively.

At the same time, the sheath of fiber is composed of organic polymeric materials. Thermal aging can cause depolymerization. Figure 6a illustrates the structure of the polyether TPEE used in this study, which consists of a highly crystalline hard segment of polybutylene terephthalate (PBT) and an oligomer soft segment of butanediol. For TPEE, as the hydrogen at the chain-end of the hydrocarbyl group is more active than the hydrogen at the β-linked carbon atom of the macromolecular ester group, the degradation activation energy at the chain-end is less than at interchain. When the test is below 200 °C, the degradation at the chain ends is dominant. The degradation at the chain ends produce a large amount of carboxyl groups (Figure 6b) and it undergoes a polycondensation reaction with a hydrocarbon group at the TPEE macro terminal (Figure 6c). The water produced diffuses into the insulating oil.

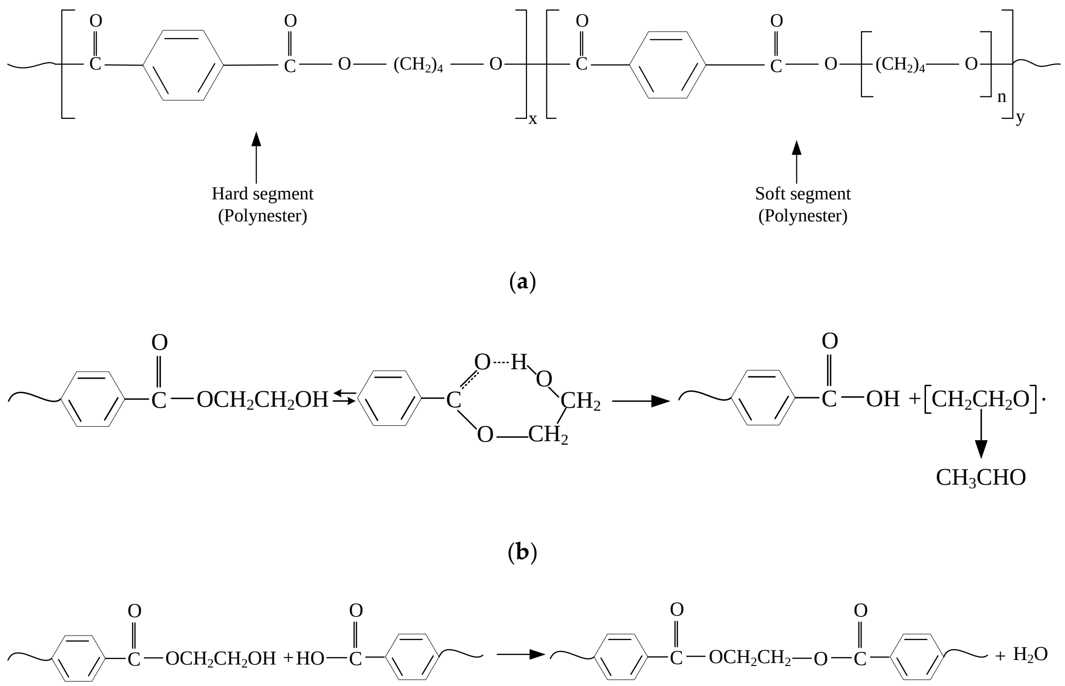
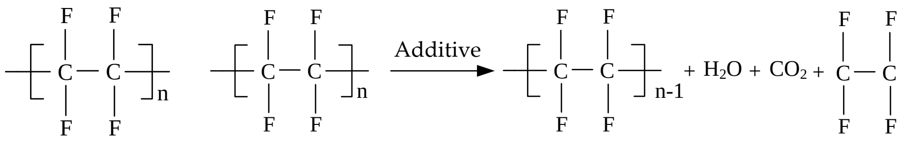


Figure 6. TPEE depolymerization reaction. (**a**) Chemical structure of the TPEE; (**b**) thermal degradation at chain ends; and (c) polycondensation.

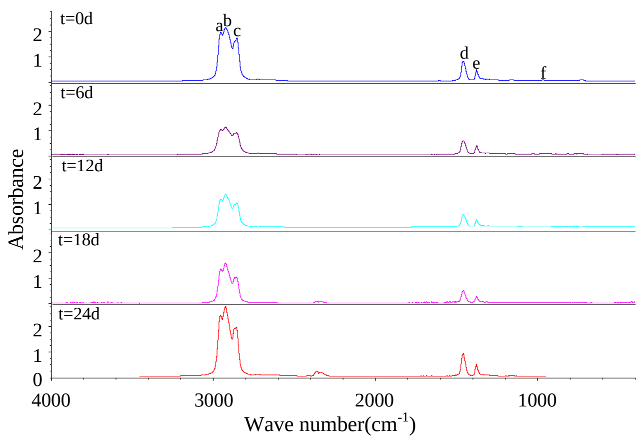
Moreover, PTFE (Figure 7a) undergoes mild depolymerization during thermal aging. Although the breakage of C-C and C-F in perfluorocarbons requires energy absorption of 346.94 kJ/mol and 484.88 kJ/mol respectively, the depolymerization of PTFE to generate TFE requires only 171.38 kJ/mol. Therefore, PTFE was mainly depolymerized into TFE in this test (Figure 7b). Due to the processing formula of PTFE, its pyrolysis product also contained a small amount of free water, amines and carbon dioxide.



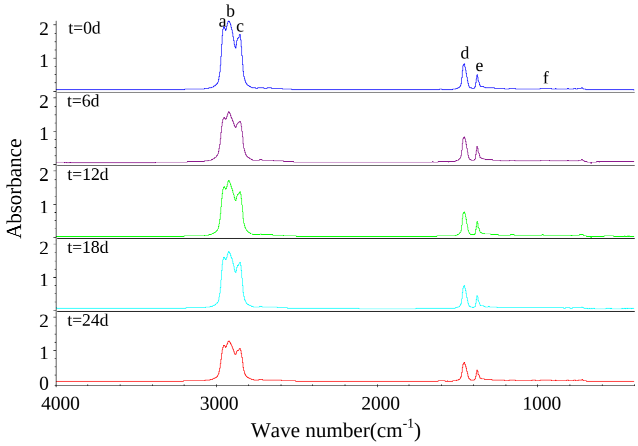
(**a**) (**b**)

Figure 7. PTFE depolymerization reaction. (**a**) Chemical structure of the PTFE; and (**b**) thermal degradation.

Based on the above analysis, it was shown that both TPEE and PTFE depolymerized in high-temperature transformer oils, producing water and small molecule hydrocarbon groups. The accumulation of small hydrocarbon groups promoted positive feedback of pyrolysis of the oil. The free hydrogen produced by oil pyrolysis increased the acidity of the oil, which in turn increased the solubility of the water produced by depolymerization of the sheath. However, different oil samples show a huge diversity in the characteristics of thermal aging, mainly due to the different degrees of aging for various fiber sheaths. To confirm the above inference, we tested the IR spectra of each oil sample. The results are shown in Figure 8 along with information of a–f absorption peaks shown in Table 3.



(**a**)



(**b**)

Figure 8. The infrared spectrogram of oil sample containing optical fiber. (**a**) TPEE-containing oil; and (**b**) PTFE-containing oil.

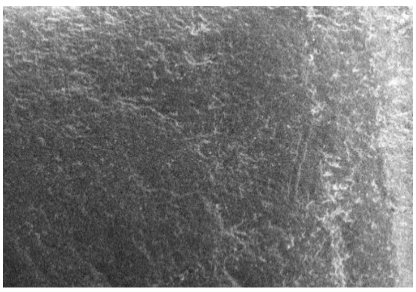
Table 3. Attribution of main IR absorption peaks of oil.

Absorption Peaks	Wave Number (cm^{-1})	Adscription
a	2952	Asymmetric stretching vibration of methyl C-H
b	2923	Asymmetric stretching vibration of methylene C-H
c	2868	Stretching vibration of methyl C-H
d	1461	Bending vibration of methylene C-H
e	1377	Bending vibration of methyl C-H
f	972	Bending vibration of carbon ring C-C

The IR spectra of oil with fibers are shown in Figure 8. The absorption peaks a ~f are main ingredient of new naphthenic transformer oil. Specific information of the absorption peaks is shown in Table 1. With increasing aging time, there was no change in the position of the main absorption peaks a ~f, because the molecular structure of the major elements C and H in the oil does not change. However, the intensity of the absorption peaks changes during heat aging. In the early stages of aging, the intensity of the absorption peaks in Figure 8a,b decreases. This indicates that the characteristic groups of fiber-containing oil suffer certain damage. In the middle of aging, the intensity of the absorption peaks in Figure 8a,b increases, indicating that both TPEE and PTFE were pyrolyzed and the macromolecular chains broken. Some of the aging products, like methyl and methylene, were dissolved in oil. Moreover, the rate of dissolution is greater than that of oil aging, resulting in intensity increases of the absorption peak. Free H+ and hydrocarbon groups promote further increases in water content and acid value in the oil. In the later period of aging, the intensity of the absorption peak in Figure 8a continues to increase, indicating that the TPEE in the high-temperature oil continues to age. In Figure 8b, the decrease in intensity of the absorption peak shows that the degradation of the oil predominates and the aging rate of the PTFE slows down.

To further verify the existence of different aging degrees in the optical fiber sheath, we used SEM at 200× magnification to observe two kinds of sheath at days 0 and 24 during aging. The result is shown in Figure 9.

Figure 9a,b show the thermal aging of the TPEE sheath at days 0 and 24. High-temperature oil causes corrosion and deterioration of the TPEE, resulting in many longitudinal cracks on the surface. The original sheet texture has been blurred. Figure 9c,d show the thermal aging of PTFE sheath at days 0 and 24. The longitudinal texture of the PTFE is still apparent, but the surface structure has become uneven due to corrosion. From Figure 9a–d, the observed physical changes in the optical fiber sheath reflect changes in the insulating features of the oil samples containing fiber sheaths.



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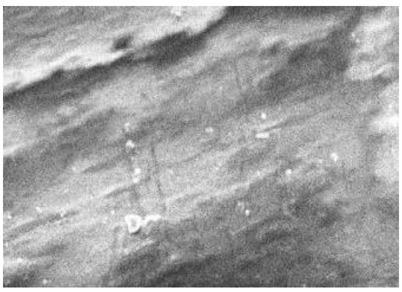

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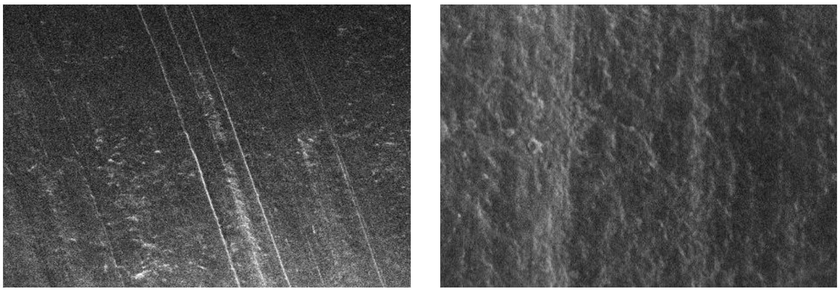
Figure 9. *Cont.*



(c) (d)

Figure 9. SEM image of fiber sheath. (**a**) 0 d TPEE; (**b**) 24 d TPEE; (**c**) 0 d PTFE; and (**d**) 24 d PTFE.

Based on the above mechanistic analysis, it can be seen that the oil samples containing fibers were deeply aged in a sealed vacuum at 130 °C, whose water contents, acid values, and hydrocarbon groups accumulated during the thermal aging periods are higher than those of the pure oil. Therefore, the fiber-containing oil has worse electric performance.

Compared with the research of Gao Shuguo and colleagues [27], this paper uses a similar aging test method to analyze the aging process of fiber sheath in transformer oil. According to the literature [27], after 200 hours of heat aging test, the cross-linked polyolefin and polyurethane sheath decomposed to a certain extent in the transformer oil, and the infrared spectrum of the PVC sheath did not change much before and after heat aging. Therefore, the cross-linked polyolefin sheath has the worst heat aging resistance in the transformer oil, the polyurethane sheath is the second, and the PVC sheath has the best performance. Compared with the results of the heat aging test in this paper for 200 hours, neither TPEE nor PTFE had a significant effect on the physical and chemical properties of the transformer oil, that is, the stability of the two types of jackets is similar to that of the PVC sheath. If we want to compare the performance of polyvinyl chloride, TPEE and PTFE in long-term heat aging, we need to age the PVC for a longer period of time, and then compare and analyze it. Moreover, compared with simply arranging the aging of the fiber sheathing material in the transformer oil, this paper not only quantitatively analyzes the physicochemical properties of the fiber-containing oil sample, but also combines the SEM of the sheath material and the FTIR of the transformer oil from a microscopic point of view to analyze the effect of the aged fiber jacket on the oil sample.

4. Conclusions

The PTFE and TPEE fiber sheaths affect the thermal aging characteristics of transformer oil at 130 °C. From the above discussion, the conclusions from the work are as follows:

Two kinds of optical fibers can accelerate the aging of transformer oil and comprehensively affect the thermal aging characteristics of oil. Moreover, TPEE sheaths had a more serious impact on oil. Compared with PTFE, TPEE had a greater influence on the DDF of oil. It was 4.5 times and 2.5 times that of pure oil than DDF of TPEE fiber-containing oil and PTFE fiber-containing oil at the end of aging. The volume resistivity of both was similar which was about 33.1% that of the pure oil sample at the end of aging. The breakdown voltages of the PTFE- and TPEE-containing oil decreased by 14.3% and 33.1%, respectively. The water contents and acid values of both showed an exponential growth pattern during heat aging. The acid value curves of the TPEE-containing oil samples showed a growth trend at the end of aging; however, the acid value curves of the PTFE-containing oil samples tend to be gentler. Based on results of the oil FTIR and microscopic observations of the sheath, it was confirmed that the degree of depolymerization is larger than that of PTFE.

Furthermore, in the thermal aging, the main reason for the deterioration of the insulating oil is pyrolysis. At the same time, both TPEE and PTFE were depolymerized in high-temperature transformer oils, producing water and small molecule hydrocarbon groups. The accumulation of small hydrocarbon groups promotes positive feedback for pyrolysis of the oil. The free hydrogen produced by oil pyrolysis increases the acidity of the oil, which in turn increases the solubility of the water produced by sheath depolymerization. The chain depolymerization of the TPEE was more severe than that of PTFE, further exacerbating the deterioration of the TPEE-containing oil. Moreover, the content of charged particles in oil increases dramatically, which also increases the DDF and decreases the volume resistivity of the oil. Therefore, we recommend using a fiber with a jacket material of PTFE built into the transformer, and when the fiber is laid in the transformer, it is necessary to monitor the content of the micro water and acid value in the control oil.

Author Contributions: S.J. and Y.L. conceived and designed the experiments; S.J. and X.F. performed the experiments; Y.T. analyzed the data; S.J. contributed reagents/materials/analysis tools; S.J. wrote the paper.

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Conflicts of Interest: The authors declare no conflicts of interest.

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