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FTIR AND FDS ASSESSMENT OF MINERAL OIL UNDER LOW ELECTRICAL DISCHARGE

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Abstract

Transformers are crucial elements in the transmission and distribution of electrical energy. The importance of diagnosing these equipments are two-fold: (1) the necessity of service reliability and (2) the likelihood to avoid economic and environmental concerns. Under service conditions, the electrical and thermal stresses or chemical contaminants may degrade the insulation oil inside the transformer and cause incipient failures or reduce its service life. Partial discharges well recognized to be among the most common stresses that can lead to slow but steady degradation of insulating oil in transformers. The present work aims at understanding the influence of low energy electrical discharge on mineral oil based on two spectroscopic methods: FTIR spectroscopy and Frequency Domain Spectroscopy (FDS). An electrical fault has been created by continuous discharge of 10 kV on the surface of various oil samples according to the ASTM D6180. From the FDS results, it was found that the amount of charge carriers and moisture increased with the aging time elapsed that influences the conduction phenomena and in turn, increases the dissipation factor. These results are confirmed by the FTIR results, which show that the intensity of the peak absorbance of the C–H and C-C functional group decreased with aging. The application of these two methods may help monitoring the condition of oil. A combined FTIR and FDS measurements highlighted the correlations between modifications in electrical properties and changes in the chemical structure of the oil under electrical accelerated ageing.

Keywords: mineral oil, diagnostic, low electrical discharge, infrared spectroscopy, dielectric spectroscopy

1. INTRODUCTION

In service condition, the insulating oil in a transformer undergoes a slow but steady degradation under combined thermal, electrical and chemical stresses. This in turn affects the physicochemical properties. Consequently, the dielectric response, including the electrical conductivity along with the dielectric dissipation factor and permittivity, which are some of the important parameters to monitor the safe operation of the transformer, is affected. These properties are also important parameters describing the liquid's function as an insulant [1][2]. Any significant increase in the conductivity and dielectric dissipation factor may indicate that the oil is no longer able to perform its vital function [2].

In this paper, a combination of two spectroscopy methods is explored for assessing the condition of a mineral oil: FTIR spectroscopy for chemical characterization and a Frequency Domain Spectroscopy (FDS) for assessing the electrical properties. The influence of the electrical aging on the electrical and chemical properties of transformer oil were investigated, and the spectroscopic measurements performed to understand the aging process and diagnosing the transformer condition.

New oil samples were submitted to electrical discharge according to the ASTM Test D6180 [4]. After that, these samples were characterized by FTIR and FDS measurement techniques.

The results obtained were analyzed and the physical mechanisms behind both spectra were interpreted.

2. EXPERIMENTAL PROCEDURE

2.1. Preparation of the samples

A mineral oil LUMINOL-TM from Petro-Canada was used in this experiment. The procedure of oil degasification and dehumidification was performed as follows. The oil samples were stored in a beaker and placed in a KIMAX Desiccators with Detachable Stopcock Valve. To avoid moisture absorption from the samples, a quantity of silica gels were placed in the desiccators. The preparation

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guarantees, very low water content (less than 5 ppm for transformer oil).

The test arrangement setup consists in a Merellbased test cell type, as specified in the ASTM Test Method D6180 (Figure 1). The HV electrode consists in a cylindrical copper electrode of 15 mm (0.6 inches) in diameter and 10 mm long sealed in a 500 ml Erlenmeyer glass. The electrode was fixed in the center of the discharge cell with a 1 inch gap above the oil surface. The pressure inside the test cell was reduced down to 1Torr (133 Pa). After this degasification, a 10 kV high-voltage discharge was generated above the oil sample during 5 h; 12h, 24h, 50h and 75h [2-4].



Fig. 1. Overview of the test method for stability of oil under electrical discharges according to the ASTM D6180[4]. (1) Erlenmeyer; (2) foam of oil after voltage application; (3) glassware containing salted water; (4)high-voltage electrode; (5) lowvoltage electrodes connected to ground

2.2. Frequency Domain Spectroscopy (FDS)

The Insulation Diagnostic Analyzer IDA200 was used to assess the dielectric proprieties of the fluid samples over a wide range of temperature using the liquid test cell type 2903 for liquid insulation manufactured by Tettex [3]. During measurements, the test cell's temperature was fixed at 90 °C in a controlled oven. The frequency range of the measurements was set from 10-3 MHz to 1 kHz, and the oil was sampled for analysis after 5, 12, 24, 50 and 75 h under electrical discharge.

2.3. FTIR Spectroscopy

The FTIR is a chemical technique used to assess material's functional groups [5]. It is therefore for monitoring changes suitable in the physicochemical properties of the materials before and after ageing. The investigations were carried out with a Nicolet Protege TM 460 ESP, FTIR spectrometer. The infrared spectra were collected in the transmission mode over the wave ranges varying from 4000-500 cm¹ with an optical resolution of 1.0 cm⁻¹, using 32 scan repetitions with a resolution of 4cm⁻¹.The FTIR spectra enables monitoring the chemical and physical structural changes of the transformer oils after electrical discharge for different discharge application durations.

3. RESULTS AND DISCUSSION

3.1. FDS Analysis of the insulating liquids

Figure 2 shows the dielectric responses of the permittivity versus frequency for mineral oil at different discharge application durations. Recall that the relative permittivity is oil's chemical composition dependant [6].



Fig. 2. Real part of the complex permittivity of the oil samples. The discharge application duration acted as a parameter

From Figure 2, it can be noticed that the real part of the permittivity of the transformer oil is almost not affected by ageing and is around 1, in the frequency ranges from 0.1 Hz to 1000 Hz. This is probably due to the fact that the composition of carbon group does not vary considerably to affect the value of the real permittivity [6]. It can be concluded that the relative permittivity of insulating oil is almost not affected by the electrical aging process at industrial frequencies. The same results were reported in other studies [7] [8]. Figure 2, shows an increase in the real part of the complex permittivity at low frequency. This may be traced to the space charge polarization processes [9, 10].

With long discharge application durations, more charge carriers are generated which results in an increase in the conductivity of the oil. With more charge carriers, the space charge polarization increases with concomitant increase in the real part of the complex permittivity [7, 9].

Out of the Figure3, it can be seen that the imaginary part of the complex permittivity decreases with frequency over the whole range with a slope of approximately at -1. It indicates a near-constant conductivity of the mineral oil's ionic conduction process and this is indicative of Maxwell-Wagner interfacial polarization [7, 9, 11].

Figure4 shows the variation in the loss factor (Tan δ) versus frequency. At different duration of electrical aging, the loss factor decreases with frequency, and increases with aging elapsed time. The applied electric field may lead to the dissociation of impurities and the consequent

formation of mobile ions. Recall that at lower frequencies, the loss factor is dominated by conduction of charged particles [12].



Fig. 3. The imaginary part of the complex permittivity of the electrically-stressed aged oil



Fig. 4. The loss tangent Tanδ of the electrically-stressed aged oil

In service conditions, very few stable molecules may accumulate enough energy for electronic transitions during the elastic collisions caused by thermal agitation at the origin of the homolytical breakdown of weak valence bonds responsible for free radicals generation. Free radicals may randomly interact through reduction-oxidation reactions and build up charge carriers, which consequently increase the power factor of an insulating oil [13].

Figure 5 shows the conductivity of the oil samples at different electrical ageing duration. It can be seen that the conductivity increases with time, and remains constant at low frequency.

Figure 6 indicates moisture generation with electrical discharge elapsed time. The conductivity of an insulating material is known to be affected by moisture content and charge carriers. Recall that oil's ageing byproducts are generally polar in nature and may therefore affect conductivity as well as loss factor [14].



Fig. 5. The real part of Conductivity $\sigma'(\Omega^{-1}cm^{-1})$ of the electrically aged oils



Fig. 6. Change in moisture of mineral oil caused by the low electrical discharging

The conductivity of mineral oil increases with aging as more charge carriers are generated due to physical and chemical degradation.

More investigations onto the chemical composition are needed to assess ageing by-product in mineral oil, especially after electrical stress. Several studies [15-17] have focused on the analysis of the behavior of the transformer insulating oil during aging by infrared spectroscopy, based on the fact that, the deterioration of the properties of insulating materials, over time is evidenced by changes in the chemical structure.

3.2. FTIR spectroscopy results

The change in structure of the mineral oil during electrical ageing was assessed by using Fourier transform infrared (FTIR) spectroscopy.

Transformer oil basically consists in a mixture of various naphthenic, paraffinic and aromatic molecules. It contains cyclo-alkane (CN), alkane (CP) and aromatic hydrocarbons (CA). During the electrical aging process, changes must have occurred in a carbon group component affecting the performance of the transformer oil [6]. Table 1 shows the wave number and its functional group for virgin oil [18].

Wave number (cm ⁻¹)	Functional group
2921	C-H (Alkane & Stretching)
2853.3	C-H (Alkane & Stretching)
1458.23	C-H (Methylene & Bending)
1376.44	O-H (Alkohol & Bending)
722.31	C-C (Methylene & Skeletal
	vibration)

Table 1. FTIR analysis for virgin oil [18]

Figure 7 shows the FTIR spectrum of aged oil samples, with the electrical discharge duration acting as parameter.



Fig. 7. The FTIR absorbance spectra in the range of 400 cm-1- 4000 cm-1ofmineral oil samples. The electrical discharge application duration acted as a parameter

The low electrical discharge energies led to the breaking of carbon-hydrogen and carbon-carbon bonds. The physicochemical structure of insulation oil is greatly affected causing deterioration with electrical discharge time elapsed. Generally, the aging and degradation of the insulating oil is associated to oxidation, moisture and dissolved contaminants from the solid materials [19].

As shown in figure 7, there is a significant difference between FTIR spectra of samples. This confirms that low electrical discharge influences the chemical structure of transformer oil. The peaks are observed at 722.2 cm-1 and 741.5 cm⁻¹ (Figure 8) assigned to (C-H) out of the plane stretching of the saturated carbon-carbon bonds [20].

The peaks between 1300 cm⁻¹- 1500 cm⁻¹ are presented in Figure 9. As shown in this figure, the peak positions for 1459.8 and 1376.9 cm⁻¹, are attributable to (C-H) bounding vibrations that result in the similar trend as the main –CH2 and –CH3 vibrations. Figure 10 shows some peaks at 2921.6 and 2852.2 cm⁻¹ wavelenghts. These peaks are assigned to (C-H) stretching of the saturated carboncarbon bonds and attributed to –CH2 vibrations followed by a small peak at 2954.4 cm⁻¹related to –CH3 vibrations [15, 21].



Fig. 8. FTIR absorbance spectra in the range of 700 cm⁻¹- 800 cm⁻¹ of mineral oil during electrical aging



Fig 9. FTIR absorbance spectra in the range of 1300 cm⁻¹ -1500 cm⁻¹ of mineral oil during electrical aging



Fig. 10. FTIR absorbance spectra in the range of 2700cm⁻¹- 3100 cm⁻¹ofmineral oil during electrical aging

Figure 8 shows an increase in the intensity of the absorbance peaks approximately around 720 and 742cm⁻¹. This rise appears more intensive after 24

hours of electrical discharge, which indicate an increase in the alkane C-H bending groups.

From Figures 9 and 10, it can be observed that the intensity of the absorbance peaks decreases at, 1459.8, 2852.2, 2921.6, 2954.4 wavelengths. The decrease in the absorption intensity of CH2(2921.6 cm⁻¹, 2852.2cm⁻¹, 1459.8cm⁻¹) and CH3 (2954.4 cm⁻¹) in the aged oil samples can be traced to the breakage of some of the C-C and C-H bonds in the mineral oil [22] due to a slow deterioration process under the electrical discharge energy. In this case, fault gases can be then produced.

Table 2 shows the energies required for breaking the molecular bonds of hydrocarbons [23]

hydrocarbons	Bond energies(kJ/mol)
Aliphatic saturated	315 - 380
Aliphatic	255 - 960
unsaturated	
Aromatic	230 - 480
Aliphatic saturated	395 - 440
Aliphatic	320 - 560
unsaturated	
Cyclic	310 - 485
Aromatic	270 - 480
	hydrocarbons Aliphatic saturated Aliphatic unsaturated Aromatic Aliphatic saturated Aliphatic unsaturated Cyclic Aromatic

Under electrical fault, the scission of some of the C-H and C-C bonds may occur, since the C-H bonds are weak. Low-energy faults may therefore break these bonds to form active hydrogen atoms and hydrocarbon fragments. These fragments may then recombine to form fault gases such as hydrogen (H-H), methane (CH₃-H), ethane (CH₃-CH₃), ethylene (CH₂ = CH₂) or acetylene (CH=CH) [24].

4. CONCLUSION

In this contribution, the electrical and chemical properties of mineral insulating oil were investigated experimentally under low electrical discharge. Based on the results, it can be concluded that there is a correlation between changes in functional groups and the increase in the loss factor and conductivity within the mineral oil. This can be explained by the increase in the polar product. The frequency domain spectroscopy's results show that the electrical discharge with time elapsed, produce charge carriers and moisture in mineral oil and influence the conduction phenomena. As a result, the dissipation factor increases. The FTIR spectra confirmed that the aging changes in the investigated mineral oil are mainly caused by the scission breaking of some of the C-H and C-C bonds leading to the formation of fault gases such as hydrogen, methane, ethane, ethylene or acetylene that are dissolved in the oil.

The FTIR measurements provided the information on different functional groups existing in the sample molecules and also the gases released during the electrical aging process. The results indicate that various gases and moisture particles such as CH4, C2H6, C2H4, and H2O are dissolved, as a result of electrical discharge in the oil.

The correlation between these two methods proves the high impact of the dissolved decay products in the aging of mineral oil as presented in the theory. The FTIR method may possibly be used for diagnostic and monitoring of mineral oil comparing the optical spectra of aged transformer oil with reference one. By means of an evaluation of all the obtained results, it can be confirmed that FTIR can directly monitor the degradation features from the spectral data analyses. The root cause of oil's properties degradation such as moisture, oxidation and other dissolved decay, can be correlated with the dielectric properties.

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