Thermal Aging of Mineral Oil-Paper Composite Insulation for High Voltage Transformer

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Abstract: High voltage transformer is one of the most important equipments in an electric power system. High voltage insulation plays important role in determining transformer performance and greatly determines the lifetime. In general, paper and oil composite is used as main insulations in a high voltage transformer. Due to thermal aging, the insulation performances of the insulation may be degraded. The degradation mechanisms are important to be clarified. This paper reports the experimental results on the effects of thermal aging on the properties of paper-oil composite insulation. The samples used were thermally upgraded kraft paper and mineral oil. They are widely used in high voltage transformers. Samples were conditioned to the same initial conditions through the heating at a temperature of 100 °C for 24 hours. Mineral oil samples with volume of 800 ml and 6 gram insulating kraft paper were put in hermetical bottles. The paper-oil ratio reflects the typical ratio of oil and kraft paper inside a real transformer. The bottles containing oil and paper samples were subjected to thermal aging at 120°C and 150°C in a controllable oven for a period up to 4 weeks. Dielectric properties of the oil such as breakdown voltage, resistivity, water content were measured after and before aging. Dissolved gas analysis (DGA) was used to identify the gasses released in the paper-oil composite insulation as the results of thermal aging process. The morphological aging of the kraft paper was investigated using SEM (scanning electron microscopy) while chemical element change was investigated using EDS (energy dispersive spectroscopy) with accelerated voltage of 0.3-30 kV. The results showed that the dielectric properties will decrease with the increasing duration and temperature of aging. From these experiments obtained CO gases, produced from thermal degradation of paper cellulose chain. H_2 and C_2H_2 gases are not generated in this experiment. The result was consistent since H_2 usually generated under partial discharges while C_2H_2 released under very high temperature such as partial combustion, electric arc which were not available in the experiment. Some hydrocarbon gas such as CH_4, C_2H_4 and C_2H_6 were detected. CO gas was obtained as the result thermal aging of paper insulation through oxidation process. The EDS analysis showed that during aging the C element increased while O element decreased. EDS data of krafft paper aged at 120°C for a duration of 4 weeks (672 hours) taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 kV showed that the aged paper consists of 3 elements, C , O and K elements with mass percentage of 75.23 % of C at energy of 0.277 kV, O of 21.69 % observed at energy of 0.525 kV and new element of K with mass percentage of 3.08 observed at energy of 3.312 kV. It is clearly observed that C increased from 58.83 % to 75.23 %. This is due to the migration from the ester into the paper on the other hand oxygen reduced from 41.17 % to 21.69 % because the oxygen from the paper reacted with oil in an oxidation and release CO gas as confirmed by DGA (dissolved gas analysis). Similar behavior was observed for aging at 150°C.

Keywords: Mineral oil, thermal aging, kraft paper, dielectric properties, dissolved gas analysis, SEM, EDX

1. Introduction

It is common that paper and mineral oil are being used as main insulation in a high voltage transformer. Paper insulation in the transformer is generally recognized as the determining

Received: August 19th, 2016. Accepted: December 23rd, 2016
DOI: 10.15676/ijeei.2016.8.4.9

factor of the transformer lifetime[1-5]. Cellulose is an organic compound with the formula n, a polysaccharide consisting of a linear chain of several hundred to many thousands of β linked D-glucose units. The chemical formula of cellulose that compose a paper is $(C_6H_{10}O_5)_n$ [6] Typically it has density of 1.5 g/cm³. The chemical structure is shown in figure 1.

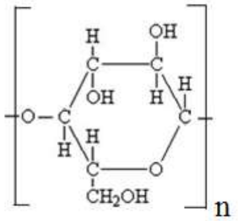


Figure 1. Chemical structure of cellulose

Mineral oil is a complex substances of hydrocarbon. Its chemical formula consists of alkenes (parraffine), cyclic alkenes (napthene), and aromatics. The chemical structure is shown in figure 2.

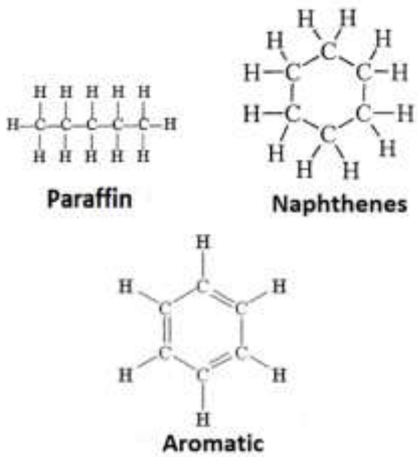


Figure 2. Chemical structure of mineral transformer oil

Mineral oil is the oil insulation used in transformers since long time. During the last century, power transformers have generally been filled with mineral oil, which has good compatibility with the cellulosic insulating paper; good physical and electrical properties, not only as a cooling fluid but also as an electrical insulator; and suitable properties such as good electrical arc quenching and mainly availability, low cost, and long history. Failure of oil insulation may cause the transformer failure leading to the interruption of power delivery. Thermal ageing can change the dielectric properties of insulation oil[7-10]. In this paper, the experimental results on the thermal aging of kraft paper-mineral oil composite insulation is presented. The change of dielectric properties of mineral oil due to the thermal aging is discussed. The generated gases during the thermal aging are analysed using DGA (dissolved gas analysis) method[11-13]. The morphological aging of the kraft paper was investigated using SEM (scanning electron microscopy) while chemical element change was investigated using EDS (energy dispersive spectroscopy)[14-16].

2. Experimental setup

A. Sample

The sample used in the experiment were thermally upgraded kraft insulating paper and mineral oil. Insulating paper is inserted into the bottle with insulating oil.

Mineral oil samples with volume of 800 ml and 6 gram insulating kraft paper were put in hermetical bottles. The paper-oil ratio reflects the typical ratio of oil and kraft paper inside a real transformer. The bottles containing oil and paper samples were subjected to thermal aging at 120^{o}C and 150^{o}C in a controllable oven for a period up to 4 weeks. The selected temperature is in accordance with Mc Shane [17], while the temperature of 120°C is a maximum hotspot temperature according to the IEEE[18]. Prior to aging, the sample is heated at a temperature of 100^{o}C for 24 hours in order to obtain the same initial conditions. The sample name and treatments are shown in table 1.

Table 1. Sample and treatment

Sample	Aging
M. T0	Initial State
M. T1. 120	120^{o}C for 336 hours
M. T1. 150	150^{o}C for 336 hours
M. T2. 120	120^{o}C for 672 hours
M. T2. 150	150^{o}C for 672 hours

Figure 3. Photographs of typical sample before thermal aging

B. Measurement of Dielectric Properties

B.1. Breakdown voltage

Breakdown voltage testing is done by using LD60 model of Liquid Dielectric Test made in Phenix Technologies. Standard used in breakdown voltage testing is IEC 60156 [19]. According to the used standard, breakdown voltage has been defined as the magnitude of the voltage when electrical breakdown occurs between the two ball electrodes within 2.5 mm at a rate of voltage rise of 2000 V/s. The used testing cells are both of the ball electrodes. The size of the testing cell is 10 x 8 x 8 cm and made from acrylic material. Liquid insulation samples which their breakdown voltage will be tested, is inserted into the testing cell. LD60 model of Liquid Dielectric Test uses the input of 220V, 50 Hz.

B.2. Resistivity

The testing is intended to determine the resistivity of the type of tested samples. The equipment used in this test is a High Resistance Meter. The test is carried out by using tube electrode which is part of Tettex Instruments and also used in Tan δ testing. Type resistivity testing is done with reference to the standard of IEC 60247 named "Insulating liquids - Measurement of dielectric constant, dielectric dissipation factor (tan δ) and dc resistivity". [20]

C. Dissolved Gas Analysis

Analysis of the type and concentration of gas contained in the oil sample is done with Gas Chromatograph HP 6890. The procedure to extract the gas from the oil sample refers to the IEEE and ASTM standards[21,22]. Oil samples were placed in a vial. Vials contain the sample is then placed in the sample container. An automated sample traction device, Automatic Liquid Sampler HP 7649, works to take the vial containing the sample one by one from the container to be analyzed. Vial is shaken by an automatic shaker to extract the gas trapped in the oil. Gas accumulated in the top of the vial is then collected with a needle that also works automatically.

D. SEM and EDX analysis

In order to understand the effects of thermal aging on the properties of kraft paper in natural ester several measurements are conducted. They are visual observation, scanning electron microscopy (SEM) and EDS measurement. Morphology of paper was observed using SEM JEOL JSM 6610 series with accelerated voltage of 0.3-30 kV as shown in figure 4. The equipment has a built in EDS which enables to identify the chemical elements in the paper.

Figure 4. SEM JEOL JSM 6610

3. Experimental Result and Analysis

A. Visual appearance

Figure shows visual appearances of oil before aging (a), after aging of 336 hours at 120°C (b) after aging of 672 hours at 120°C (c) after aging of 336 hours at 150°C (d) and after aging of 672 hours at 150°C.

(a)	(b)	(c)	(d)	(e)

Figure 5. Pictures of oil samples before and after thermal aging at different temperature and aging time

Figure 5 shows that the color of the oil become darker with aging time and the aging temperature. By using the color scale standard, the color scale of the oil before and after aging are shown in table 2.

Table 2. Color scale of oil samples before and after aging

Sample	Picture #	Color scale
M. T0	A	2.0
M. T1. 120	b	2.3
M. T2. 120	c	2.8
M. T1. 150	d	3.5
M. T2. 150	e	4.3

B. Breakdown Voltage

Breakdown voltage illustrates the dialectric strenght of insulation in a very high electrical voltage. The higher value of breakdown voltage could bring the better dielectric strenght of insulation material.

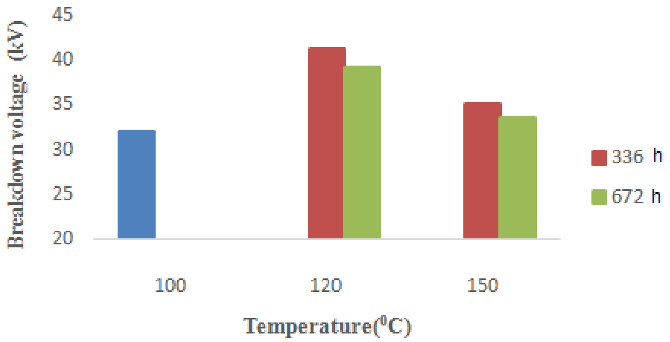


Figure 6. Dependence of Breakdown Voltage on temperature for aging time of 336 and 672 hours

Figure 6 shown breakdown voltage of mineral oil sample before and after aging at 120°C and 150°C for duration of 336 and 672 hours. The blue bar indicates the breakdown voltage of initial sample after heated at 100oC for 24 hours. The figure clearly shows that breakdown voltage increases after heating the samples. The phenomena was due to the reduction of water content which was from 64 ppm to 43 ppm after 336 hours. As the time elapsed the breakdown voltage reduced again. This was due to the degradation of the oil as well as paper as indicated by the darker color of the aged oils.

C. Resistivity

The test results are given in the following graph.

Figure 7 shows the resistivity of mineral oil as a function of aging temperature. Graph presents the decrease in resistivity value of oil with the increasing aging temperature. Because if the larger temperature given, then greater the energy received by the oil. The condition causes more solid contaminants arise in the form of carbon that is conductive. Thus increasing the conductivity of the insulation on oil continuously will lower the value of resistivity. Such conditions can be seen in the graph, the sample M.T2.150. Samples that are heated 150°C and duration of 4 weeks, a lot of the formation of contaminant particles that can increase the conductivity of the insulation of the oil samples. And over time will lower the resistivity of the type of oil. This condition proves that the greater the temperature and duration of aging by, can lower the resistivity value of oil type.

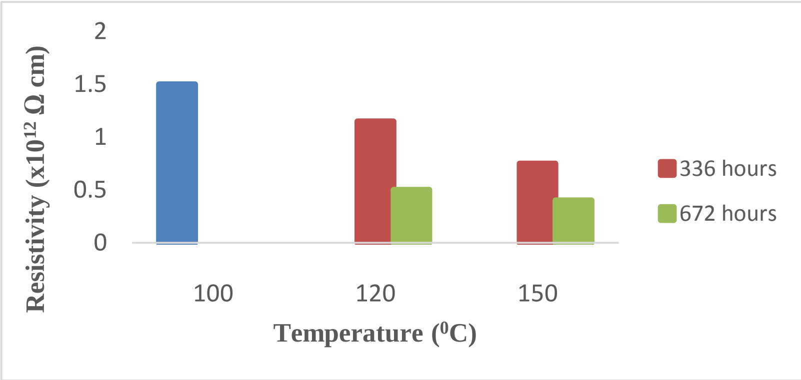


Figure 7. Dependence of Resistivity on temperature for aging time of 336 and 672 hours

D. Dissolved Gas Analysis

Table 2 shows the concentration of gases in the ester oil samplesThey are several gases obtained, those are H_2 (Hydrogen), CH_4 (Methane), C_2H_2 (Acetylene), C_2H_4 (ethylene), C_2H_6 (ethane), and CO (Carbon Monoxide). In this paper, we collect the concentration all of the gases and choose the most significant gases to interpret the condition of ester oil after ageing process.

From the table 3 it is clear that CO appeared in all of the samples. At high-temperature cellulose molecules are decomposed and evolve carbon oxides (CO_2 and CO). High level of dissolved carbon oxides in oil indicates the thermal degradation of cellulose insulation in the system. Hence, it is possible to assess the level of solid insulation degradation using amount of CO2 and CO concentration in oil.

In this experiment, the amount gas concentration H_2 and C_2H_2 were little detected due to different experimental conditions. When high energy of electric defects such as partial discharge or arcs is concentrated on insulating oil, H_2 and C_2H_2 which the molecular weights are relatively small are generated in large amount because the degree of degradation of insulating oil is high.

The reason why C2H6 gas were generated in this experimental results considered to be the molecular bonding structures of natural ester fluid. Natural ester fluid is composed of unsaturated fatty acid structures which are compositely composed of double bonds and single bonds of carbon. When these structures are heated, the single bonds (C-C) that have weak binding force are degraded first to generate hydrocarbon. The hydrocarbon and water weaken the double bonds (C=C), so that the double bonds are decomposed to single bonds.

Table 3. Gases obtained from DGA

Samples	Gas Concentration (ppm)					
	H_2	CH_4	C_2H_2	C_2H_4	C_2H_6	CO
M. T0	0	0	0	0	0	0
M. T1. 120	0	68,52	0	11,16	73,43	92,35
M. T1. 150	0	21,74	0	29,17	45,40	94,10
M. T2. 120	0	41,22	0	11,83	39,95	110,30
M. T2. 150	0	267,39	0	33,45	293,14	438,15

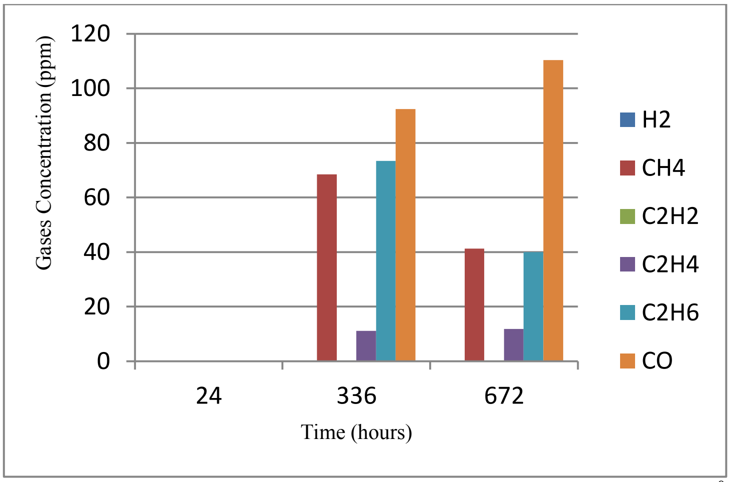


Figure 8. Dependence of Gases Concentration on aging time at 120 ^{0}C

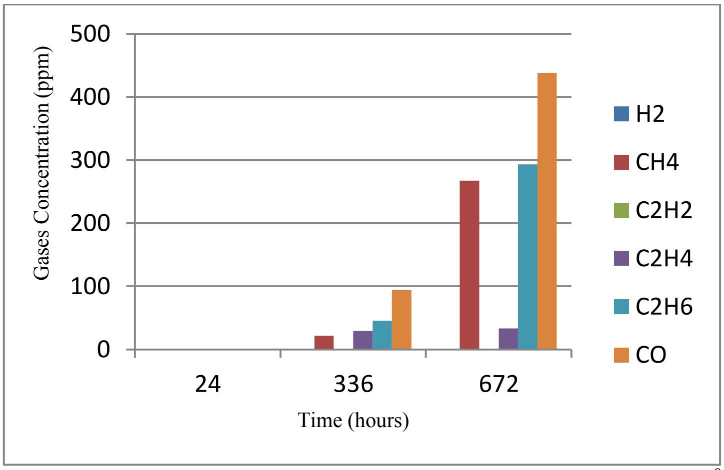


Figure 9. Dependence of Gases Concentration on aging time at 150 ^{0}C

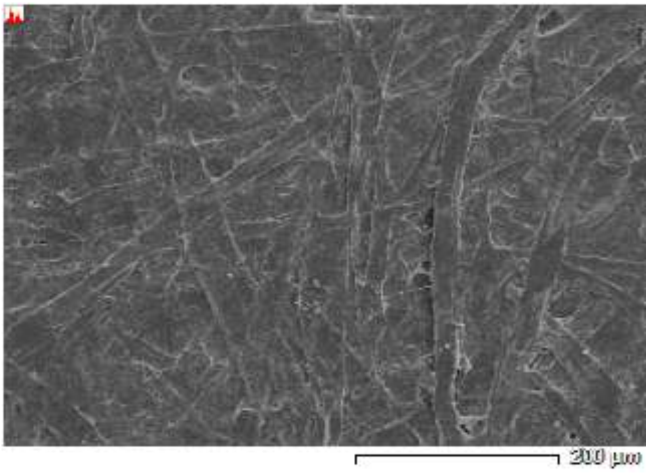
From the graph above, we can see an increase in the concentration of CO. This indicates the occurrence of thermal degradation of the paper cellulose chains [9]. The addition temperature and duration of aging will increase the concentration of CO. Effect of thermal aging can produce CH_4, C_2H_4 and C_2H_6. The trend of gases concentration increases with increasing temperature aging. Because the gases formed by thermal degradation of paper cellulose chains[23,24].

SEM and EDX Analysis

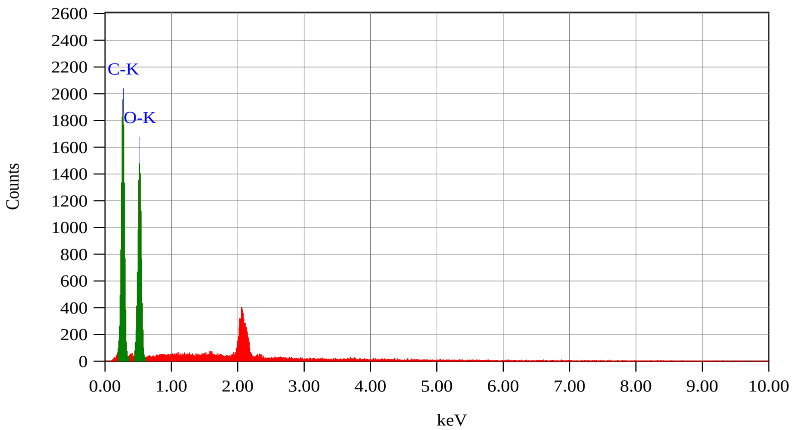
SEM and EDS results of a new paper

Figure 10 (a) shows the SEM picture of a new krafft paper taken using accelerated voltage of 20 kV and magnification of 200 x. It is seen that there are reasonable pore in the paper. Figure 10 (b) shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-10 keV. From the spectrum it is seen that the new paper is mainly consists of C and O elements with mass percentage of 58.83 % of C at energy of 0.277 keV and O of 41.17 % observed at energy of 0.525 keV. The results are consistent with the facts that Paper is composed of Cellulose which is an organic compound with the formula n, a polysaccharide consisting of a linear chain of several hundred to many thousands of β linked

D-glucose units. The chemical formula of cellulose is ($C_6H_{10}O_5$). Hydrogen was not detected because The H 1s electrons are valence electrons and as such participate in chemical bonding. Any signal from hydrogen would overlap with signals from excitation of valence electrons from other surface atoms. It is generally not possible to distinguish between H 1s valence electrons and valence electrons of other elements. Therefore, H 1s valence electrons are not useful in elemental identification using EDS method[25].



(a)



Fitting Coefficient : 0.6497

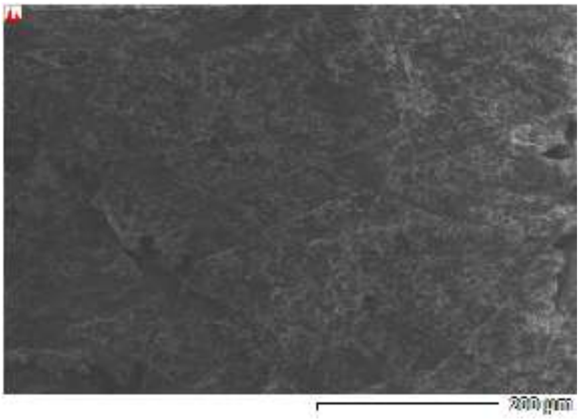
Element	(keV)	Mass%
C K	0.277	58.83
O K	0.525	41.17
Total		100.00

(b)

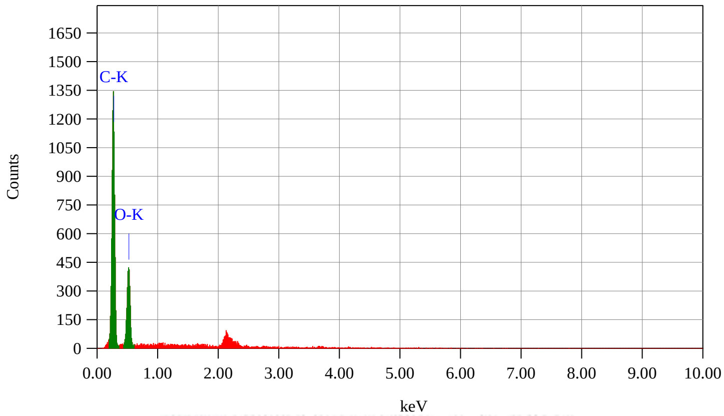
Figure 10. (a) SEM and (b) EDS spectra for new paper

SEM and EDS results of paper aged at 120 deg for 336 hours in mineral oil

Figure 11(a) shows the SEM picture of a krafft paper aged at 120^{o}C for a duration of 2 weeks (336 hours) taken using accelerated voltage of 10 kV and magnification of 200 x. From the figure it is seen that there are reasonable pore in the paper. Figure 11 (b) shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-10 keV. From the spectrum it is seen that the aged paper consists of 2 elements, C and O which are similar to those of new paper. However, they have different mass percentages. They are 72.3 % of C at energy of 0.277 keV and O of 27.7 % observed at energy of 0.525 keV. It is clearly observed that C increased from 58.83 % to 72.3 %. On the other hand oxygen reduced from 41.17 % to 27.7 %. This is due to the migration from the oil into the paper and the reduction of oxygen in the paper because of reaction with oil in an thermal agitated oxidation and release CO gas as confirmed by DGA (dissolved gas analysis) . As shown in the table 2, 92,35 ppm CO gas was released.



(a)



Fitting Coefficient : 0.7588

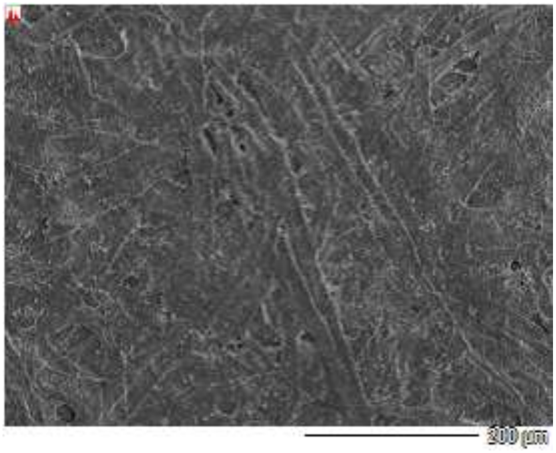
Element	(keV)	Mass%
C K	0.277	72.30
O K	0.525	27.70
Total		100.00

(b)

Figure 11. (a) SEM and (b) EDS spectra for paper in mineral oil aged at 120^{o}C for 336 hours

SEM and EDS results of paper aged at 120 deg for 672 hours in mineral oil

Figure 12 (a) shows the SEM picture of a krafft paper aged at 120^{o}C for a duration of 4 weeks (672 hours) taken using accelerated voltage of 10 kV and magnification of 200 x.



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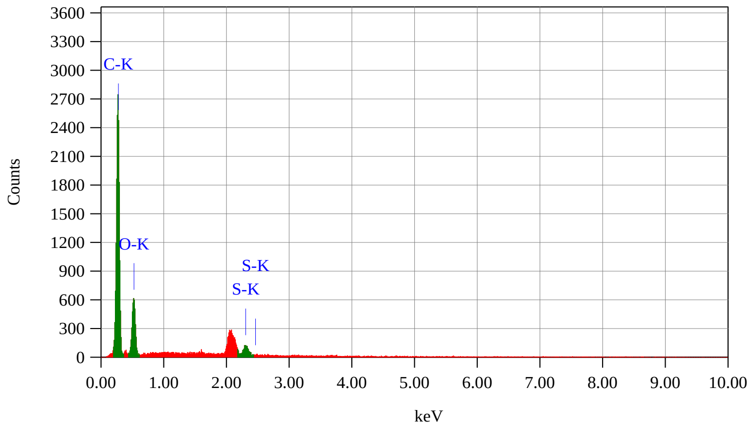


Fitting Coefficient : 0.7636

Element	(keV)	Mass%
C K	0.277	78.14
O K	0.525	19.75
S K	2.307	2.10
Total		100.00

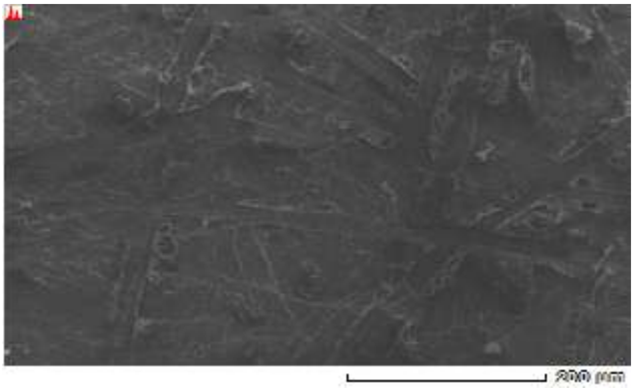
(b)

Figure 12. (a) SEM and (b) EDS spectra for paper in mineral oil aged at 120°C for 672 hours

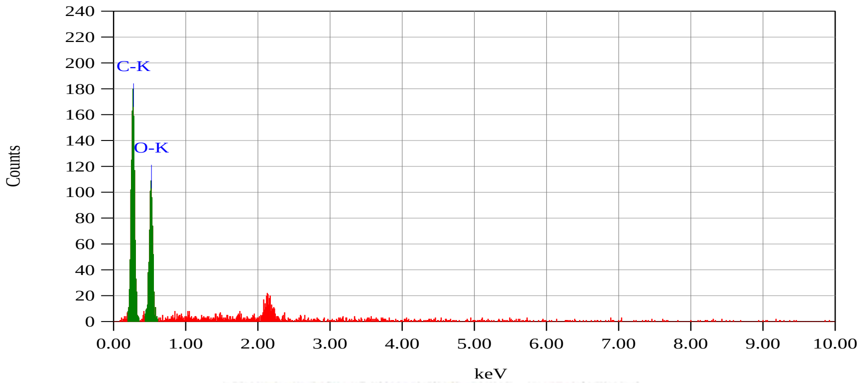
It is seen that there are reasonable pore in the paper. Figure 12 (b) shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 kV. From the spectrum it is seen that the aged paper consists of 3 elements, C , O and S elements with mass percentage of 78.14 % of C at energy of 0.277 kV, O of 19.75 % observed at energy of 0.525 kV and new element of S with mass percentage of 2.1 % observed at energy of 2.307 kV. It is clearly observed that C increased from 58.83 % to 78.14 %. This is due to the migration from the oil into the paper and the reduction of oxygen in the paper because of reaction with oil in an thermal agitated oxidation and release CO gas as confirmed by DGA (dissolved gas analysis). As shown in the table 2, 110.3 ppm CO gas was released under this aging condition. The appearance of new species S is due to the migration of S from the mineral oil. It is well known that mineral oils is made from a crude oil which contains plenty sulphur elements. During the refining process, most of these elements are removed, however, small amount of the sulphurs are still remaining. Sulphur is accepted to be the most corrosive element in transformer oil[26-30].

SEM and EDS results of paper aged at 150 °C deg for 336 hours in mineral oil

Figure 13 (a) shows the SEM picture of a krafft paper aged at 150°C for a duration of 336 hours.



(a)



Fitting Coefficient : 0.7203

Element	(keV)	Mass%
C K	0.277	63.01
O K	0.525	36.99
Total		100.00

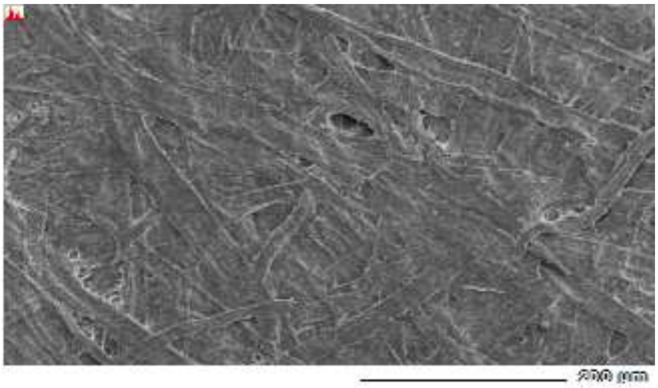
(b)

Figure 13. (a) SEM and (b) EDS spectra for paper in mineral oil aged at 150°C for 336 hours

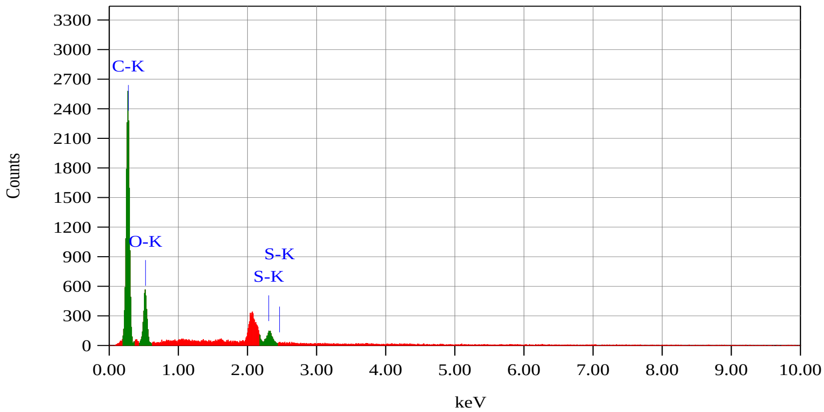
Under aging temperature of 150oC up to 336 hours, the detected elements in the paper are C and O with composition of 63.01 % of C which is detected at 0.277 keV and O detected at 0.525 keV. However, they have different mass percentages. The data showed that mass percentage of C increased from 58.83 % at initial condition to 63.01 % after aging at 150°C for 336 hours. On the other hand oxygen reduced from 41.17 % to 36.99 %. This is due to the migration from the oil into the paper and at the same time oxygen elements in the paper were consumed in an oxidation reaction with oil in a thermal agitated oxidation and release CO gas as confirmed by DGA (dissolved gas analysis) . This process released CO gas with concentration of 94,10 as shown in the table 2. No S element was detected at this stage.

SEM and EDS results of paper aged at 150 °C deg for 672 hours in mineral oil

Figure 14 (a) shows the SEM picture of a krafft paper aged at 150°C for a duration of 672 hours.



(a)



Fitting Coefficient : 0.7757

Element	(keV)	Mass%
C K	0.277	79.05
O K	0.525	18.46
S K	2.307	2.48
Total		100.00

(b)

Figure 14. (a) SEM and (b) EDS spectra for paper in mineral oil aged at 150°C for 672 hours.

Figure 14(a) shows the SEM picture of a krafft paper aged at 150°C for a duration of 4 weeks (672 hours) taken using accelerated voltage of 10 kV and magnification of 200 x. It is seen that pores are reasonably reduced due to oil migration into the paper pores and light degradation was already taken place. Figure 14 (b) shows the EDS spectrum taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 keV. It is clearly seen from the spectrum that under this aging condition, 3 elements are observed. C was observed at energy of 0.277 keV with mass percentage of 79.05, while O at 0.525 keV with percentage of 18.46 %. New element namely S was detected at energy of 2.307 keV with mass percentage of 2.48. As it was described for other aging condition the increase of C and the decrease of O was due to the migration of C into paper and at the same time the reduction of O from the paper in a oxidation process to form CO gas. The released gas was confirmed using DGA measurement with concentration of 438,15 ppm. The comparison of elements in new and aged kraft paper samples is shown in table 4.

Table 4. Comparison of elements in new and aged kraft paper samples

Element	Energy (keV)	New (%)	Aged at 120°C for 336 h (%)	Aged at 120°C for 672 h (%)	Aged at 150°C for 336 h (%)	Aged at 150°C for 672 h (%)
C	0.277	58.83	72.3	78.14	63.01	79.05
O	0.525	41.17	27.7	19.75	36.99	18.46
S	3.312			2.10		2.48

From the table it is seen that the thermal aging in ester-kraft paper composite insulation increased the C element due to oil permeation into the paper. However, the thermal aging reduced the O element of paper because of the usage of oxygen element from the paper in oxidation reaction to release CO which was confirmed by DGA. The small S element appeared after aging came from the mineral oil.

4. Conclusion

We have investigated the thermal aging on paper-mineral transformer oil composite insulation system at 120°C and 150°C with duration up to 672 hours. Dielectric properties of oil were investigated using IEEE and IEC standard. Gases generated during the aging were determined using DGA method. The morphological aging of the kraft paper was investigated using SEM (scanning electron microscopy) while chemical element change was investigated using EDS (energy dispersive spectroscopy) with accelerated voltage of 0.3-30 kV. The experimental results showed that the dielectric properties decreased with the increasing of duration and temperature of aging. Breakdown voltage increases after heating the samples. The phenomena was due to the reduction of water content which was from 64 ppm to 43 ppm after 336 hours. As the time elapsed the breakdown voltage reduced again due to the degradation of the oil as well as paper as indicated by the darker color of the aged oils. DGA date indicated that some hydrocarbon gases were detected in aged samples. They are H_2 (Hydrogen), CH_4 (Methane), C_2H_2 (Acetylene), C_2H_4 (ethylene), C_2H_6 (ethane), and CO (Carbon Monoxide). H_2 and C_2H_2 gases are not generated in this experiment. CO gas was obtained as the result thermal aging of paper insulation through oxidation process. The increase of temperature and the longer the aging period will increase the concentration of CO. The EDS analysis showed that during aging the C element increased while O element decreased. EDS data of krafft paper aged at 120°C for a duration of 4 weeks (672 hours) taken using JEOL 6510 at accelerated voltage of 10 kV with energy range of 0-20 keV showed that the aged paper consists of 3 elements, C, O and K elements with mass percentage of 75.23 % of C at energy of 0.277 keV, O of 21.69 % observed at energy of 0.525 keV and new element of K with mass percentage of 3.08 observed at energy of 3.312 keV. It is clearly observed that C increased from 58.83 % to 75.23 %. This is due to the migration from the ester into the paper on the other hand oxygen reduced from 41.17 % to 21.69 % because the oxygen from the paper reacted with oil in an oxidation and release CO gas as confirmed by DGA (dissolved gas analysis). Similar behavior was observed for aging at 150°C. EDX date taken from sample aged at 120°C with duration of 672 hours showed that C , O and S elements were detected with mass percentage of 78.14 % of C at energy of 0.277 keV, O of 19.75 % observed at energy of 0.525 keV and S with mass percentage of 2.1 % observed at energy of 2.307 keV. The increase of C element was due to the migration from the oil into the paper and the reduction of oxygen in the paper because of reaction with oil in an thermal agitated oxidation and release CO gas as confirmed by DGA (dissolved gas analysis). Similar behavior was detected for aging at 150°C and aging duration of 672 hour but with higher percentage of changes.

5. Acknowledgements

This study was financially supported by RISTEK DIKTI 2016

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