Study on Thermal Ageing Characteristics of Silicone Rubber and Pressboard in Mineral Oil

M.Sc. Smitha Karambar, Prof. Dr.-Ing. Stefan Tenbohlen
University of Stuttgart, Institute of Power Transmission and High Voltage Technology (IEH), Stuttgart, Germany
smitha.karambar@ieh.uni-stuttgart.de

Abstract

Silicone rubbers are widely used as outdoor insulators due to their excellent electrical, mechanical and thermal properties. Investigations have been carried out to determine the influence of mineral oil on silicone rubber. This research aims to investigate the thermal ageing characteristics of different silicone rubbers and pressboard in mineral oil (NYTRO LYRA X) and their influence on dielectric properties of mineral oil. Properties of three different types of silicone rubbers, namely, insulative silicone rubber, conductive silicone rubber, silicone rubber with conductive and insulative layers have been investigated in this research. The solid insulation materials are immersed in mineral oil and are thermally aged at 130° C in airtight glass bottles for 360 hours. Samples of mineral oil without any solid insulation materials are also prepared and are aged under similar conditions. After thermal ageing, mineral oil and solid insulation samples are investigated for their dielectric properties. Mineral oil samples are investigated for their moisture content, dissipation factor, breakdown voltage and dissolved gases. Whereas, solid insulation samples are tested for their moisture content. In this paper, the measurement results for different samples are compared and analyzed. The experimental results demonstrate that the dielectric properties of mineral oil largely depends on the type of solid insulation material used during the ageing test.

1 Introduction

Silicone rubbers are widely used as insulators due to their excellent electrical, thermal and mechanical properties [1]. In [2] the influence of mineral oil on the behaviour of room temperature vulcanized (RTV) silicone rubber is investigated by studying its hydrophobicity, functional groups and Gas Chromatography. However, numerous researches that have been carried out, mainly concentrate on the influence of mineral oil on the behaviour of silicone rubber [2] [3]. In this paper, the opposite is proposed, where the influence of silicone rubber on the behaviour of mineral oil is investigated.

Mineral oil is used as insulation material in different electrical devices because of its favourable dielectric and thermal properties, as well as a good oxidation stability. The dielectric properties of mineral oil are greatly influenced by its moisture content [4], therefore the study of the influence of silicone rubber on the moisture content of mineral oil is of interest. In this research, the moisture content in aged mineral oil is measured using Karl Fischer titrator.

In an electrical equipment like a transformer, the life span is inversely proportional to the moisture content in its solid insulation (paper/pressboard) [5]. Hence, it is important to measure the moisture content within the insulation itself. The moisture content in a solid insulation is measured using the Karl Fischer headspace module and analyzed.

The breakdown voltage of oil is significantly affected by the presence of moisture and other impurities. The dielectric strength of oil is inversely proportional to the moisture and impurity content [6]. In this research, the effect of silicone ageing in mineral oil on the breakdown strength of oil is determined according to IEC 60516.

Another important factor to be considered while investigating insulating oil is its dissipation factor or tan δ, as it is sensitive to the insulating ability and the ageing degree [7] of mineral oil. Dissipation factor is measured for aged oil at room temperature and at 90° C.

Dissolved Gas Analysis (DGA) is widely used to determine the ageing status and incipient faults in oil filled electrical equipment. In this research, DGA is performed on thermally aged mineral oil to compare the gases dissolved in mineral oil containing silicone rubber to that in mineral oil containing pressboard (PB) material. Siemens Sichromat Gas Chromatograph is used in this research to perform DGA and is measured according to the methods prescribed in ASTM D 3612.

2 Sample description and thermal ageing

2.1 Samples considered

Five types of samples are considered in this investigation. Type 1 is composed of mineral oil with insulative transparent silicone rubber having a dielectric constant of 2.8. Type 2 consists of black conductive silicone rubber and mineral oil. Type 3 consists of mineral oil with conductive and insulative silicone rubber. Type 4 is composed of mineral oil and pressboard. Type 5 is pure mineral oil without any

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solid insulation. For each type of solid insulation and mineral oil combination, three samples of each type are prepared and are named Sample 1, Sample 2 and Sample 3.

2.2 Samples preparation

The ratio of weights of solid insulation materials to mineral oil considered in this research is 1:15. Three portions (weighing 50 g) of each solid insulation material are pre-dried in a vacuum oven at 110º C for 24 h without the application of vacuum. After 24 hours, vacuum is applied and materials are further dried at 110º C for another 48 h. Solid insulation materials are weighed after the drying process and the moisture loss in each sample is determined using the following formula

$$\text{Moisture loss}=\frac{(W_{in}\text{-}W_{out})}{W_{in}}\cdot 100 \quad (1)$$

Where, W_{in} = Weight before drying (g)

W_{out} = Weight after drying (g)

2.3 Oil impregnation process and thermal ageing

Oil impregnation is carried out at 90° C for 30 minutes under vacuum. 15 airtight glass containers are pre-treated to avoid any moisture content and are filled with 850 ml of dry mineral oil (moisture content of < 5 ppm). Each portion of the solid insulation material is transferred to a separate airtight glass container, containing dry mineral oil.

Figure 1 Oil impregnation of solid insulation materials

The vacuum oven is heated to 90° C, the glass containers are placed in the vacuum oven and vacuum is applied for 30 minutes as shown in Figure 1. This process ensures a minimum atmospheric interference during the impregnation and ageing process. Type 5 sample containing mineral oil without any solid insulation is also allowed to undergo this process to avoid any atmospheric interference on the ageing process. After the completion of the vacuum impregnation process, the glass bottles containing impregnated solid insulation materials are sealed immediately and are placed in the ageing oven. The samples are then thermally aged at 130º C for 360 hours.

3 Experimental analysis

3.1 Determination of absolute moisture content in mineral oil

Absolute moisture content is measured using Coulometric Karl Fischer Titrator Aqua 40 from ECH GmbH. A Karl Fischer (KF) titrator without any diaphragm is used in this research. A common reagent coulomat AG is used and the amount of moisture content is obtained by measuring the coulombs of electricity required to generate the necessary iodine to react with the moisture. The moisture content of the mineral oil is obtained by directly injecting a predefined amount of oil into the measuring cell containing the anode and the cathode. The titrator adjusts the measuring current automatically and continuously, based on the amount of moisture present in the oil. The amount of moisture in the oil is measured in terms of ppm (parts per million) i.e. 1 µg of moisture in 1 g of oil.

3.2 Determination of moisture content in solid insulation

The moisture content in solid insulation is measured using the headspace module of the Karl Fischer Titrator. The moisture determination process is similar to that of oil, as explained in Section 3.1. However, instead of injecting oil directly into the measuring cell, the solid insulation is heated to 150° C in the headspace oven of the KF titrator. Moisture released due to the heating of solid insulation material is passed through the measuring cell. This moisture reacts with the coulomat in the measuring cell and the amount of moisture content is determined by measuring the coulombs of electricity required to release the iodine that reacts with the moisture. In order to obtain a reliable moisture content value, it is ensured that the solid insulation weighs at least 100 mg for each measurement. The amount of moisture in the solid insulation is determined by the ratio of weight of moisture in solid insulation to the weight of the measured solid insulation in %.

3.3 Dissipation factor (tan δ) measurement

Dissipation factor is normally used as an indicator for mineral oil ageing. Dissipation factor of aged oil is measured using a DIRANA measurement equipment from Omicron GmbH using the FDS method for frequencies ranging from 1 Hz to 100 Hz. The measuring cell consists of two flat electrodes and a guard ring. The high voltage electrode is a circular flat electrode with a radius of 50 mm and the ground electrode is composed of a circular flat electrode of 30 mm radius. The ground electrode is surrounded by a circular guard ring of 20 mm width with a 1 mm gap between them. The measuring cell is filled with the mineral oil under measurement and the distance between electrodes is set to 2 mm. A voltage of 200 V is applied. The capacitances are measured and their ratio is determined in order to measure the tan δ of the oil.

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3.4 Breakdown voltage measurement of mineral oil

The breakdown voltage of aged mineral oil samples is determined according to IEC 60156. The experimental construction is shown in Figure 2. It consists of a glass vessel having two mushroom shaped electrodes with a gap of 2.5 mm between them. A magnetic stirrer is placed inside the vessel to ensure a uniform distribution of the particles, if these are present in the oil due to ageing. Initially, the oil in the glass vessel is stirred for 5 min and then the voltage is applied at a rate of 2 kV/s until the breakdown is achieved. The measurements are repeated six times in intervals of 2 min between them. For each measurement, the mean value and standard deviation are obtained. The same process is repeated three times for each type of oil.

Figure 2 Breakdown Voltage (BDV) Determination equipment

3.5 Dissolved gas analysis (DGA) of mineral oil

Dissolved gas analysis is performed using a Siemens Gas Chromatograph Sichromat 2-8 which is connected to a headspace sampler Hewlett Packard HP 7694. Oil samples are taken using syringes of 30 ml and placed in an Argon filled glove box. Argon gas is circulated for an hour to avoid atmospheric interference. Vials of 20 ml are filled with about 10 ml of oil and their lids are closed within 45 seconds. These vials are then transferred to the headspace sampler of the Gas Chromatograph (GC) for further analysis. Each vial is stirred for 30 min and is heated to 70° C, in order to achieve an equilibrium between the dissolved gases in the oil and in the headspace. After completing this process, the gas samples are injected into the GC for analysis. The GC separates the gas samples into its components and their concentration is determined either by the Flame Ionization Detector (FID) or the Thermal Conductivity Detector (TCD). FID is mainly used to determine the concentration of combustible gases. However, carbon monoxide (CO) and carbon dioxide (CO_2) are non-combustible gases and hence are converted to methane using a methanizer, and is measured using FID. The following gases are measured using the FID: methane (CH_4), carbon monoxide (CO), carbon dioxide (CO_2), acetylene (C_2H_2), ethene (C_2H_4), ethane (C_2H_6), propene (C_3H_6) and propane (C_3H_8). The concentration of oxygen (O_2), nitrogen (N_2) and hydrogen (H_2) cannot be determined using FID, therefore the TCD method is used. In TCD, the concentration of a gas is determined by comparing its thermal conductivity with the one of a carrier gas, Argon is used in this case.

The results obtained from this experiment provide the gas concentration within the headspace. The gas concentration in a liquid can be determined using the Ostwald's constant (K factor), as described in the standard ASTM D 2779.

4 Results and Analysis

4.1 Moisture loss based on weight loss

The weight of a solid insulation before drying and after drying is measured and the moisture loss in percentage is determined.

Table 1: Moisture loss based on the weight loss

Serial Number	*Name*	*Moisture loss in %*
1	Type 1	0.345
2	Type 2	0.502
3	Type 3	0.282
4	Type 4	5.757

Silicone insulators (Type 1, Type 2 and Type 3) exhibit a smaller moisture loss compared to the PB material (Type 4), as shown in Table 1. Silicone materials are hydrophobic, hence they do not absorb moisture under atmospheric conditions and therefore no significant change in their weight is observed. Whereas, pressboard absorbs and retains moisture content within, hence the drying process resulted in a relatively higher moisture loss.

4.2 Moisture content in aged mineral oil determined

The Moisture content in aged mineral oil is determined using the Karl Fischer Titration method as explained in Section 3.1. Figure 3 shows the average of absolute moisture content in aged mineral oil. The moisture content in the samples containing PB material displayed the lowest moisture content in oil. Whereas, the sample without any solid insulation (i.e. Type 5) displayed the highest moisture content, as the moisture produced due to the ageing process is completely retained within the oil itself. As shown in Figure 3, Sample 2 of Type 5 exhibited a much lower moisture content, because the sealing gasket between the bottle and the lid had not loosened during ageing process. However, for the other samples, the sealing gasket had loosened dur-

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ing the ageing process, producing a higher moisture content. During the ageing process of Type 4 samples, initially the moisture content in oil is low and as ageing progresses, moisture migrates from paper/PB towards the oil due to the low surface combining power. However, moisture is also produced due to the thermal decomposition of PB insulation. The migration of the moisture continues from PB to oil and vice versa until saturation is attained. PB is a hygroscopic material, which absorbs moisture under equilibrium conditions, whereas silicone being a hydrophobic material does not absorb much moisture content within.

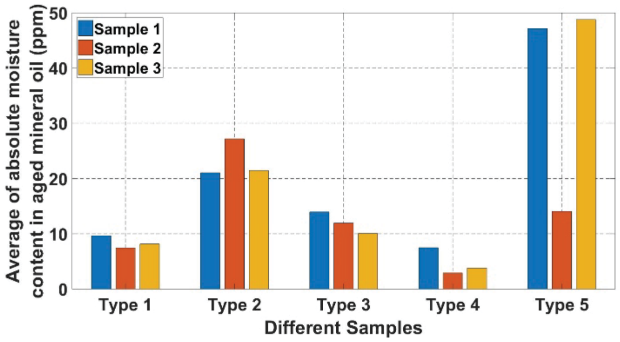

Figure 3 Average absolute moisture content in aged mineral oil measured using Karl Fischer Titrator

4.3 Moisture content in aged solid insulation

The Moisture content in solid insulation materials after thermal ageing is obtained according to the process described in Section 3.2. In order to be able to understand the moisture migration between mineral oil and solid insulation, it is necessary to determine the moisture content in solid insulation after the ageing process. Moisture in silicone rubbers is lesser than in PB, as shown in Figure 4. The silicone rubbers under study retained their hydrophobic behaviour in spite of their ageing.

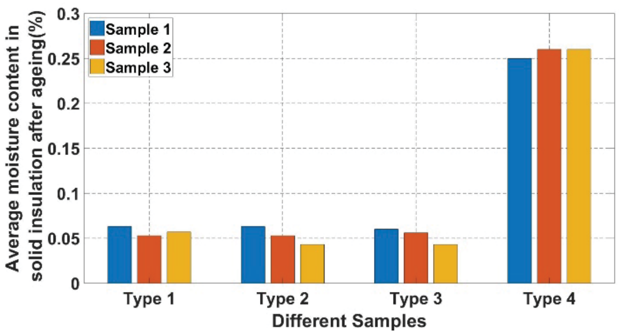

Figure 4 Average moisture content in aged solid insulation using Karl Fischer Titrator Headspace method

4.4 Dissipation factor of aged mineral oil

The dissipation factor of aged mineral oil is measured according to the procedure described in Section 3.3. Table 2 shows the dissipation factor of aged oil at 50 Hz measured at 20° C and 90° C.

Table 2: Dissipation factor of aged mineral oil at 20 °C and 90° C

Serial Number	*Sample name*	*Dissipation factor (tan δ) at 20 °C*	*Dissipation factor (tan δ) at 90 °C*
1	Type 1	0.061	0.382
2	Type 2	0.062	0.347
3	Type 3	0.052	0.414
4	Type 4	0.044	0.304
5	Type 5	0.058	0.341

From Table 2, it can be observed that the dissipation factor is directly proportional to the temperature. This can be noticed from the dissipation factors obtained at 20° C and at 90° C. The dissipation factor is mainly influenced by the moisture content in the samples and the presence of particles that were produced during the ageing process. The dissipation factor of mineral oil with the PB shows the lowest value, whereas mineral oil with silicone rubbers exhibited the highest dissipation factor for both temperatures.

4.5 Breakdown voltage of aged mineral oil

The Breakdown voltage of aged mineral oil is measured as explained in Section 3.4. Figure 5 shows the mean and standard deviation values of breakdown voltage obtained for various mineral oil samples. From the results, it is noticed that the mineral oil without any solid insulation (Type 5) exhibited the lowest breakdown voltage value. Whereas, Type 1, Type 3 and Type 4 samples exhibited relatively higher breakdown voltages. The breakdown voltage is influenced mostly by the impurities in the mineral oil and its moisture content. The low breakdown voltage of Type 5 samples are due to their high moisture content. The mineral oil containing Type 1, Type 3 and Type 4 samples exhibited a higher breakdown voltage because of their relatively lower moisture content in oil.

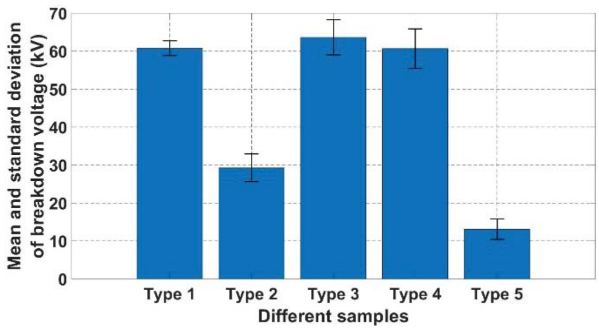

Figure 5 Mean and standard deviation of breakdown voltage

4.6 DGA Results

DGA is performed according to the procedure explained in Section 3.5. For three samples of each type of solid insulation materials considered, three vials containing mineral oil are prepared for each sample. From the obtained results, it can be noted that hydrogen (H_2), methane (CH_4), ethene (C_2H_4), acetylene (C_2H_2) and ethane (C_2H_6) exhibited their highest content in the oil containing PB material as shown in Figure 6. Silicone rubbers are known for their chemical stability and hence, the amount of gases dissolved in mineral oil samples aged with silicone rubber is lesser than the mineral oil samples containing PB. Propene and propane were produced in the mineral oil containing conductive silicone rubber and insulative silicone rubber respectively. Figure 7 shows the mean and standard deviation values of carbon dioxide gas content dissolved in aged mineral oil samples. Carbon dioxide is produced in mineral oil samples containing PB samples due to the overheating of cellulose, which results in its degradation. Therefore, a higher carbon dioxide content was noticed in mineral oil containing PB samples. As shown in Figure 7, carbon dioxide was also produced in Type 1, Type 2 and Type 3 samples. The presence of solid insulation increases the production of carbon dioxide due to overheating.

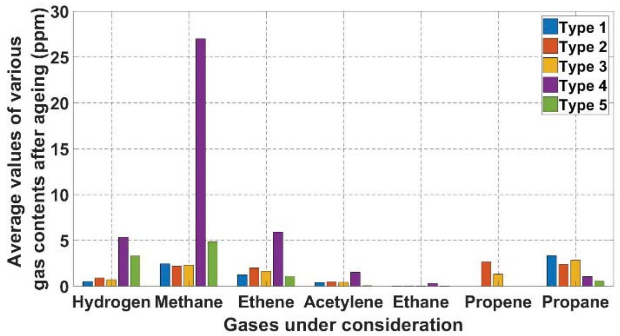

Figure 6 Average gas content in mineral oil after ageing

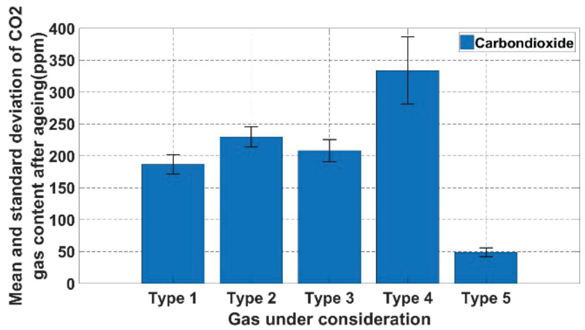

Figure 7 Mean and standard deviation of CO_2 content in aged mineral oil

5 Conclusion

In this research, the thermal ageing behaviour of different types of silicone rubbers was investigated. Silicone rubber samples were aged in mineral oil at 130° C for 360 h and their moisture content was studied. For the analysed samples, their moisture content, breakdown voltage, dissipation factor and gassing behaviour were measured.

Moisture content in mineral oil without any solid insulation exhibited the highest moisture content after the ageing process. Whereas, mineral oil with pressboard exhibited the lowest moisture content. Moisture in mineral oil was produced mainly due to the moisture ingress and oxidation of mineral oil. Pressboard material absorbed the moisture, because of its hygroscopic nature and hence, a lower moisture content in mineral oil was obtained.

The solid insulation materials analysed in this research were also measured for their moisture content. Pressboard exhibited a higher moisture content than silicone rubbers. This is also due to their respective hygroscopic and hydrophobic behaviours.

The measured breakdown voltages are directly proportional to the measured moisture content in aged mineral oil. Type 1, Type 3 and Type 4 samples exhibited higher breakdown voltages due to their lower moisture content. While, Type 5 exhibited the lowest breakdown voltage due to its higher moisture content.

Aged mineral oil containing pressboard samples exhibited the lowest dissipation factor. Whereas, mineral oil samples containing silicone rubber exhibited a higher dissipation factor due to the presence of moisture content and impurities.

Dissolved gas analysis of aged mineral oil samples were carried out. Silicone rubbers are known for their chemical stability and hence the mineral oil aged with silicone rubbers exhibited a lower gas content as compared to the mineral oil containing pressboard samples.

6 Acknowledgement

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7 Literature

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