CHAPTER 5

DETERIORATION OF SOLID INSULATION FOR
THERMAL DEGRADATION OF TRANSFORMER OIL

5.1 INTRODUCTION

It is necessary to ensure the proper functioning of transformer during operating conditions. Condition monitoring of transformer helps us to assess the remaining life time of the power transformer. One of the monitoring methodologies is to estimate the strength of the solid insulation systems for thermal degradation of transformer oil. Rui-Jin Liao et al (2008) presented a methodology to analyze the aging mechanism of insulation paper for several weeks using Atomic force microscope, Scanning electron microscope and X-Ray diffraction techniques. Jiaming Yan et al (2011) observed the surface products of oil-impregnated paper during damage process caused by partial discharge. Taro Hino et al (1972) analyzed the deterioration of the oil-immersed papers by the presence of CO, CO₂ and H₂O with the help of gas chromatograph techniques. Shi-qang et al (2010) presented an accelerated thermal aging test on oil immersed pressboard for 120, 250 and 400 hours. They interpreted the Degree of Polymerization and structure through SEM images. In this work an effort has been made to investigate the degradation of solid insulation (Kraft paper-20 hours, Press board-100 hours) for thermal degradation of transformer oil. The main reason for aging of solid insulation in power transformer is influenced by thermal, electromechanical and chemical stresses produced due to over loaded operating conditions. It is found that thermal stress leads to the major degradation process of oil and there by deterioration of paper insulation. So
the brittleness and durability of the insulation paper is reduced. As a result the expected life of the transformer is reduced. Aging of liquid dielectrics is analyzed by breakdown voltage, acidity, flash point, dielectric dissipation factor and interfacial tension. Gases dissolved in the oil are monitored with the help of diagnostic techniques like DGA. The ageing of insulation paper is analyzed with the degree of polymerization, furan content analysis and tensile strength etc. In order to exactly determine aging degree of solid insulation suitable techniques like Optical microscope, Atomic force microscope, Scanning electron microscope, Nuclear magnetic resonance, X-ray diffraction technique and Fourier transform-Infrared spectroscopy methods are used.

In the present study Atomic force microscope, Scanning electron microscope and X-ray diffraction techniques were used to determine the aging degree of Kraft paper and pressboard immersed in mineral oil.

5.2 EXPERIMENTAL SETUP

A synthetic based mineral oil with the density of 0.89 g/cm$^3$ at 29°C, maximum and water content of 15 ppm was used in this test. The Kraft paper and pressboard made of cellulose fiber content was used for accelerated thermal aging test. The Kraft paper and pressboard satisfies IEC 60641-2 standard. The test samples were cut to the dimension of 120mm X 80mm and the thickness of the samples were about 0.5mm for Kraft paper and 1mm for the pressboard which is shown in Figure 5.1.
Figure 5.1 Test Samples

(a) New Kraft Paper (b) Mineral Oil Aged Kraft Paper at 90°C for 20 hours (c) New Pressboard (d) Mineral Oil Aged Pressboard at 90°C for 100 hours.

The arrangement of solid dielectrics, mineral oil and outlet valve of the test chamber is shown in Figure 5.2.

Figure 5.2 Design of Test cell
Initially the test samples were dried to reduce their moisture content. The whole assembly of the test sample is immersed with the mineral oil in the test cell and temperature of the test cell in maintained at 90°C. The Kraft paper and Pressboard are heated up to 20 hours and 100 hours respectively. Aging degree of Kraft paper and pressboard was analyzed using AFM, SEM and XRD Techniques

5.3 RESULTS AND INFERENCE

5.3.1 AFM Analysis

AFM instrument was used to analyze the surface of the samples. The AFM studies were conducted by using Park systems Korea model XE70. The detail of instrument is provided in Appendix. The scanning probe modes which scan across surface of the test samples and a repulsive force between the tip and the samples were recorded relative to spatial variations and then converted into analogue images which are shown below. The Figure 5.3 shows surface image of the unaged Kraft paper. The scanning probe range is about 5µm x 5µm. The colored surface image of the AFM provides the changes in the surface that is from brown to white indicate the lowest to the highest position. The meshed structure of cellulose fibers were in regular order.

Figure 5.3 Image of new Kraft Paper Surface
The Figure 5.4 shows the cropped image of unaged Kraft paper. The meshed structure of cellulose fibers were in the range of 300 x 300 nm. The meshed structure of the cellulose fibers were viewed clearly and found no changes in the structure. The value of the molecular units in the unaged Kraft paper is 0-100 nm.

![Figure 5.4 Typical cropped image of New Kraft Paper](image)

The Figure 5.5 shows the surface images of the mineral oil aged Kraft paper for 20 hours. The meshed structures of cellulose fibers were disordered.

![Figure 5.5 Image of mineral oil aged Kraft Paper at 90° C for 20 Hours](image)
The Figure 5.6 shows the enlarged AFM image of the mineral oil aged Kraft paper for 20 hours. The meshed structure of the cellulose fibers were viewed clearly and found drastic changes in the structure. The value of molecular units in the mineral oil aged Kraft paper is reduced to 0-10 nm range.

![AFM Image](image)

**Figure 5.6  Cropped Image of mineral oil aged Kraft Paper at 90°C for 20 Hours**

From this we can infer that surface of Kraft paper is highly deteriorated with thermal degradation of mineral oil and finally leading to decrease of insulation strength of the Kraft paper.

The Figure 5.7 shows surface images of the unaged pressboard. The scanning probe range is about 5µm x 5µm. The colored surface image of the AFM provides the changes in the surface that is from brown to white indicate the lowest to the highest position. The meshed structure of cellulose fibers were in regular order.
The Figure 5.8 shows the enlarged image of the unaged pressboard. The meshed structure of the cellulose fibers were viewed clearly and found no changes in the structure. The value of the molecular units in the unaged pressboard is 0-200 nm range.

The Figure 5.9 shows the surface AFM images of the mineral oil aged pressboard for 100 hours. The meshed structure of cellulose fibers were in disordered form.
Figure 5.9 Image of mineral oil aged Pressboard at 90°C for 100 Hours

The Figure 5.10 shows the enlarged image of the mineral oil aged pressboard for 100 hours. The meshed structure of the cellulose fibers were viewed clearly and found drastic changes in the structure. The value of molecular units in the mineral oil aged pressboard is reduced to 10-30 nm range.

Figure 5.10 Cropped image of mineral oil aged Pressboard of AFM

From this we shall infer that surface of pressboard is highly deteriorated with thermal degradation of mineral oil and leads to decrease of insulation strength of the pressboard.
5.3.2 Structural Analysis with SEM

Scanning electron microscope was used in order to study the microstructural analysis of the aged Kraft paper and pressboard samples. The SEM studies were conducted by using HITACHI model S-3000H.

![Typical SEM image of New Kraft Paper](image)

Closely tied fibers 19.59 µm

**Figure 5.11 Typical SEM image of New Kraft Paper**

The detail of instrument is provided in Appendix. The Figure 5.11 shows the SEM image of fresh Kraft paper at x200 magnifications. The SEM image indicates that cellulose fibers were tied together and closely packed. It is found that average width of the cellulose fibers is 19.59 µm. The SEM image indicates that the cellulose fibers form the chain structures and no bond breaking in structure.

![SEM image of mineral oil aged Kraft Paper for 20 Hours](image)

Structures disordered 19.35 µm

**Figure 5.12 SEM image of mineral oil aged Kraft Paper for 20 Hours**
The Figure 5.12 shows the SEM image of mineral oil aged Kraft paper for 20 hours with x200 magnifications. The image indicates the cellulose fibers were found lack in order of arrangement. The sides of the walls in the structure of the cellulose fibers are destroyed. The average width of the cellulose fibers is decreased to 19.35 µm.

![Image of mineral oil aged Kraft paper]

**Figure 5.12 SEM image of Mineral Oil aged Kraft paper**

The Figure 5.13 shows the SEM microscope of fresh pressboard at x200 magnifications. The SEM image indicates the cellulose fibers were tied together and closely packed. The average width of the cellulose fibers is 19.05 µm. The micrograph indicates that the cellulose fibers form the chain structures and no bond breaking in them.

![Image of fresh pressboard]

**Figure 5.13 SEM image of New Pressboard**

The Figure 5.14 shows the SEM images of mineral oil aged pressboard with x200 magnifications. The image indicates the cellulose fibers...
were found lack in order of arrangement. The sides of the walls in the structure of the cellulose fibers are destroyed. The average width of the cellulose fibers is decreased to 15.65 µm. The image indicates that the chain structures of the cellulose fibers destroyed severely and leading to the rupturing of the pressboard.

5.3.3 X-ray Diffraction Method (XRD)

X-ray diffraction technique is an analytical technique used for identification of crystalline material. The detail of instrument is provided in Appendix. X-rays of known wavelength are passed through the sample to identify the crystal structure. XRD studies were conducted using Panalytical model ‘X’ per PRO. The wave nature is diffracted by crystal to give unique pattern of peaks of ‘reflections’ at differing angles and of differing intensity. The X-ray detector moves around the sample and measures the intensity of these peaks and positions of these peaks.

The structure and number of atoms present in the structure are the deciding factors of the crystalline material of the test samples. Relative intensity of the material is analyzed with help of the ordered peak (sharp) and disordered peak (smooth). In addition to the relative intensity the mean size of the crystalline domains can cause changes in the crystalline material of the test samples and it’s calculated with the Scherer formula (Rui-Jin Liao 2008). Figure 5.15 shows diffraction pattern of the unaged Kraft paper. The relative intensity of the unaged sample was 35.04%. The size of the crystalline domains is 3.084Å0.
Figure 5.15 XRD pattern of new Kraft paper

The Figure 5.16 shows diffraction pattern of the mineral oil aged Kraft paper for 20 hours. The relative intensity of the mineral oil aged paper was 14.65% and the mean size of the crystalline domains is 1.518Å. Hence from the above result it’s proved that the intensity of the Kraft paper is reducing with time.

Figure 5.16  XRD pattern of Mineral Oil Aged Kraft Paper at 90 °C for 20 Hours
The Figure 5.17 shows the diffraction pattern of the unaged pressboard. The relative intensity of the unaged sample was 23.44%. The size of the crystalline domains is 2.161Å.

![Figure 5.17 XRD pattern of New Pressboard](image)

Figure 5.17 XRD pattern of New Pressboard

The Figure 5.18 shows diffraction pattern of the mineral oil aged pressboard for 100 hours. The relative intensity of the mineral oil aged paper was 10.54% and the mean size of the crystalline domains is 1.994Å.

![Figure 5.18 XRD pattern of Mineral Oil Aged pressboard at 90°C for 100 Hours](image)

Figure 5.18 XRD pattern of Mineral Oil Aged pressboard at 90°C for 100 Hours
Hence from the above obtained results it’s inferred that the relative intensity of the pressboard is reducing with increase in time. Various % values of relative intensity and crystal sizes for aged and un-aged samples are listed in Table 5.1.

Table 5.1 Relative intensity and the crystal size of Kraft paper and Pressboard

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Samples</th>
<th>Relative Intensity (%</th>
<th>Crystal size (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Un-aged Kraft paper</td>
<td>35.04</td>
<td>3.084</td>
</tr>
<tr>
<td>2</td>
<td>Aged Kraft paper for 20 hours</td>
<td>14.65</td>
<td>1.518</td>
</tr>
<tr>
<td>3</td>
<td>Un-aged pressboard</td>
<td>23.44</td>
<td>2.161</td>
</tr>
<tr>
<td>4</td>
<td>Aged pressboard for 100 hours</td>
<td>10.54</td>
<td>1.994</td>
</tr>
</tbody>
</table>

The Figures 5.19 and 5.20 show that aging in hours of Kraft paper and pressboard decreases the relative intensity.

Figure 5.19 Variations in Relative Intensity of Kraft Paper with Increase in Aging of Hours
The Scherer equation can be written as:

$$D_{hkl} = \frac{K\lambda}{\beta \cos \theta}$$  \hspace{1cm} (5.1)

- $D_{hkl}$ – Crystallite size
- $K$ – Scherrer constant (0.94)
- $\lambda$ – X-ray wavelength;
- $\beta$ – Full-width at half-maximum of the reflection surface $hkl$ measured in $2\theta$ ($\theta$ is the corresponding Bragg angle)

5.4 DISCUSSION

- Deterioration of solid insulation for thermal ageing of transformer oil has been investigated.

- Pressboard has higher density and tensile strength when compared to Kraft paper and pressboard can withstand more stress condition. The Kraft paper could withstand continuous heating for 20 hours, after that it was difficult to remove the paper from chamber, hence 20 hours was chosen.
- Pressboard due to its higher tensile strength was able to withstand heating up to 100 hours, after it was difficult to collect samples.

- Result reveals that structure of molecular units and arrangements of cellulose fibres has been disordered for hours of heating.

- Estimating the number of molecular units in insulation paper will help the user to identify the level of degradation of solid insulation paper.

### 5.5 CONCLUSION

One of the critical parameter which determines the life of power transformer is strength of solid insulation. Insulation strength of Kraft paper and Pressboard is mainly determined by thermal degradation of transformer oil. Detoriation level of solid insulation systems is examined using AFM, SEM and XRD Techniques. Exact monitoring of solid insulation systems surely paves way for optimized performance of power transformers. The next chapter deals with enhancement of critical characteristics of transformer oil using nano particles.