

Dielectric and Thermal Characterization of Insulating Organic Varnish Used in Electrical Machines Having Phenolic Chemical Base Filled With Carbon Nanotubes

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Abstract: In recent days, a lot of attention was being drawn towards the polymer nanocomposites for use in electrical applications due to encouraging results obtained for their dielectric properties. Polymer nanocomposites were commonly defined as a combination of polymer matrix and additives that have at least one dimension in the nanometer range scale. Carbon nanotubes were of a special interest as the possible organic component in such a composite coating. The carbon atoms were arranged in a hexagonal network and then rolled up to form a seamless cylinder which measures several nanometers across, but can be thousands of nanometers long. There were many different types, but the two main categories are single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs), which are made from multiple layers of graphite. Carbon nanotubes were an example of a nanostructure varying in size from 1-100 nanometers (the scale of atoms and molecules). Nano composites were one of the fastest growing fields in nanotechnology. Extensive literature survey has been done on the nanocomposites, synthesis and preparation of nano filler. The following objectives were set based on the literature survey and understanding the technology.

- Complete study of Organic varnish and CNT
- Chemical properties
- Electrical properties
- Thermal properties
- Mechanical properties
- Synthesis and characterization of carbon nanotubes
- Preparation of polymer nanocomposites
- Study of characteristics of the nanocomposite insulation

Dimensioning an insulation system requires exact knowledge of the type, magnitude and duration of the electric stress while simultaneously considering the ambient conditions. But, on the other hand, properties of the insulating materials in question must also be known, so that in addition to the proper material, the optimum, e.g. the most economical design of the insulation system must be chosen.

Keywords: Varnish, CNT, SEM, X-ray diffraction, TGA

1. INTRODUCTION

The important properties of insulating materials were physical, chemical, thermal, electrical, mechanical and optical

properties. These properties were necessary for the selection of insulation materials.

1.1 Chemical properties

When impurities diffuse into insulating materials, this can cause chemical change. Only inorganic materials such as glass and densely fired ceramic materials are practically impermeable. In synthetic organic materials diffusion can take place within the molecular framework of the polymer. The diffusion velocity depends upon the material structure and the affinity of the base material. For example, all organic insulating materials absorb moisture by diffusion. The dielectric and electrical properties deteriorate as a result. Dissolved salts produced by hydrolysis or from impurities increase the conductivity and cause a poorer dissipation factor and break-down field strength [1]. The high dielectric constant of water modifies the dielectric constant of the material and causes a change in the voltage distribution during alternating voltage stressing [2]. In addition, the absorbed water can lead to changes in dimension and corrosion of the electrodes. Insulating materials for outdoor application should have poorly wettable surfaces so that closed water paths, which reduce the strength, are avoided. Inorganic materials show resistance to alkalis and acids [3]. Organic materials are attacked by strongly oxidizing acids, by alkalis as well as by hydrocarbons specific to the materials. In outdoor application of insulating materials, wet pollution layers can be decomposed by the electric stress and the heat so generated to form aggressive chemicals: these with the additional influence of light, oxygen, ozone, heat and UV radiation attack the insulating material.

1.2 Electrical properties

1.2.1 Breakdown field strength

The breakdown field strength is an extraordinary important material property for dimensioning an insulation system. Although it does not represent a constant specific to the material, what is more, it depends more or less strongly upon several influencing parameters as radius of curvature and surface finish of the electrodes, layer thickness, type of voltage, stress duration, pressure, temperature, frequency and humidity [4]. For solid insulating materials, certain criteria are available from measurement of the breakdown voltage or the breakdown field strength on plate-like samples in a homogeneous or weakly inhomogeneous field [5]. A standardized testing arrangement for the determination of the breakdown field strength of plates or foils up to sample thickness of 3mm. in order to prevent gliding discharges along the surface of the insulating plate, the entire arrangement is embedded in an insulating liquid with an appropriate dielectric constant. The breakdown test is conducted with an alternating voltage, which should be increased from zero to the breakdown value within 10-20 s. The median value of the breakdown voltage is determined from five samples; if any value lies more than 15% of the median value, 5 additional samples must be tested and the median value then determined from 10 samples. The breakdown field strength can be evaluated from the breakdown voltage and the smallest electrode spacing.

1.2.2 Insulation resistance

Practical insulation systems frequently comprise many dielectrics which are stressed in parallel. Thus, for example, the insulation resistance of a support insulator consists of the combination in parallel of surface resistance and volume resistance. While the volume resistance, commonly expressed as specific resistance in Ω cm, is often independent of surrounding medium, the surface resistance is appreciably influenced by ambient conditions such as pressure,

temperature, humidity, dust etc. An arrangement for the measurement of the volume resistance of plate-type insulating material samples is discussed. In this arrangement, the live electrode, which also supports the plate-type sample, is arranged opposite a measuring electrode. The volume resistance is calculated from the direct voltage applied (100 V or 1000 V) and the current taken by the measuring electrode. A guard arranged concentrically around the measuring electrode with a 1 mm gap prevents erroneous measurements caused by surface currents. Special testing arrangements are available for tube-shaped insulating material samples, for insulating compounds which can be melted and for liquid insulating materials [6]. Common insulating materials exhibit a specific volume resistance of 10^{12} to 10^{13} Ω cm, whereas superior materials can reach resistance values up to 10^{17} Ω cm or even higher. To measure the surface resistance metallic knife-edges are used, set up at a gap spacing of 1 cm over a length of 10 cm on the surface of the insulating material under test and direct voltage is applied. From the voltage and current, the surface resistance, expressed in ohms, is determined.

1.2.3 Tracking strength

When an insulation system was electrically stressed a current which was determined by the surface resistance flows on its surface and was referred to as leakage or creepage current. It was easy to understand that the ambient conditions temperature, pressure, humidity and pollution essentially determine the magnitude of this leakage current, i.e. no, or only limited, deterioration of the surface properties shall occur. Leakage currents result in thermal, and due to the by-products, also chemical stressing of the surface. The visible effects of overstressing are tracks resulting from material decomposition; these can appear in the form of a conducting path making further electric stressing of the material impossible, or as erosion, without leaving a conducting track behind [7]. Although the insulating properties are adversely affected by erosion, e.g. by the ease of dust deposition, yet further electric stressability was not precluded. Erosion occurs either in plates or as pits. Tracking is not restricted to insulating surfaces of outdoor arrangements but can also occur, under unfavorable circumstances, indoor applications or even in the interior of equipment. It is influenced for instance by the material properties, by the form and finish of the electrodes and surfaces, and also by the external conditions. Due to merging of several localized tracks, a complete flashover can be facilitated or initiated. Testing of insulating materials for their tracking strength is undertaken according to prescribed methods. In the method, VDE electrodes are placed on insulating material sample of at least 3 mm thickness and 380 V alternating voltages as applied to the electrode arrangement as per IEC publication 112 (1979). A pipette provides one drop of the testing solution of prescribed conductivity every 30 s; this wets the surface of the insulating material between the electrodes and causes leakage currents. As test result either the number of drops up to the time of automatic switch-off of the test circuit is evaluated, or the greatest pitting depth is measured.

1.2.4 Arcing resistance

Flashovers along the surfaces of insulating materials, with a subsequent power-arc, are indeed very rare, but basically unavoidable faults in technical insulation systems. Insulating materials exposed to the influence of the arc must therefore experience no, or only minimal, variations in their electrical and mechanical properties, viz. be arc resistant. Due to the

high arc temperature and as a consequence of incomplete burning of the insulating material, conducting tracks can remain which no longer permit further electric stressing. To determine the arcing resistance, carbon electrodes supplied with 220 V direct voltages are set up on the insulating plate. With an arc struck on the surface of the insulating, the electrodes are moved apart at a velocity of 1 mm/s up to a maximum separation of 20 mm. Six levels of arcing resistance, L1 to L6, defined according to the destruction caused by the arc, are used to judge the materials.

1.2.5 Dielectric constant and dissipation factor

For practical insulating materials, apart from the deformation polarization (electronic, ionic, lattice polarization), the orientation polarization is of particular significance since many insulating materials have permanent dipoles in their molecular structure. This is major cause of polarization losses and is responsible for the frequency dependence of ϵ_r and $\tan\delta$, which is important for technical applications. The different relaxation times result in frequency limits beyond which the respective mechanisms no longer exist, because the corresponding dipole movement does not occur. This is why the dielectric constant must also decrease. The occurrence of polarization losses, as well as the increase of losses due to ionic conduction are recognized from the curve $\tan \delta = f(\nu)$. Measurement of $\tan \delta$ and the determination of ϵ_r were done using bridge circuits.

1.3 Thermal properties

In equipment and installations for the supply of electricity, heat is generated by ohmic losses in conductors, through dielectric losses in insulating materials and through magnetization and eddy-current losses in the iron [8]. Since, by comparison with metals, insulating materials have only a very low thermal stability, the permissible temperature rise of the insulating material often restricts the use of the equipment. Knowledge of the thermal properties of the insulating materials is, therefore, important for the construction and design of equipment and setups.

1.3.1 Specific heat

Due to the inertia of thermal transport, an insulating material must be in a position to absorb short-duration thermal pulses; caused by rapid load variations, via its thermal capacitance by an increase in temperature.

$$\Delta T = W / c * m \quad (1)$$

Where

c is the specific heat of insulating material,

m is the mass and

W is the supplied energy.

1.3.2 Heat transport

During continuous stressing under static operating conditions, the heat generated as a result of loss must be dissipated through the surroundings. Transport mechanisms are thermal conduction, convection and radiation. In thermal conduction, the thermal current flowing between flat plates was expressed by

$$P = A * \lambda * (T_1 - T_2) / s \quad (2)$$

Where

A is the area of the plate,

s is the plate thickness and

(T1- T2) is the temperature difference.

The proportionality factor λ is the thermal conductivity which can be assumed to be assumed constant in the technically interesting temperature range. For rapid removal of loss heat from electrical equipment good thermal conductivity is desirable. This requirement is satisfied best by crystalline insulating materials because the regular arrangement of atomic movement. In contrast, amorphous materials have distinctly poorer thermal conduction.

1.3.4 Linear thermal expansion

Insulating materials are construction materials which are frequently employed in contact with metals. On account of the larger thermal expansion of organic insulating materials, the danger of mechanical overstressing exists and this could result in the development of cracks or electrode arrangement. For inorganic insulating materials, the linear thermal expansion is lower than for metals; so an improvement is effected by filling organic materials with inorganic substances. Partly crystalline materials very often have a greater thermal expansion than amorphous materials.

1.3.5 Thermal stability

An important property of insulating materials is the shape retention on heating. There are two methods to determine this. The warm shape retention according to Martens is determined for a standard testing rod of 10 x 15 mm² cross-section and 120 mm length, which is stressed uniformly over the entire length with a bending stress of 500 N/cm². At the same time the temperature of the surrounding air is increased at the rate of 50°C/h. The temperature at which the rod reaches a specific bending denotes the warm shape retention according to Martens. For thermoplastic materials the Vicat method is also applied. The Vicat temperature is that temperature at which a blunt needle of 1 mm² cross-section under a force of 10 N or 50 N penetrates to a depth of 1 ± 0.1 mm into the insulating material. A high value for shape retention under heat is a decisive advantage of inorganic insulating materials over the inorganic kind.

1.4 Mechanical properties

The mechanical requirements are a consequence of the fact that most of the insulating materials are simultaneously construction materials with load-bearing properties. Depending upon the application, important properties are: Tensile strength, Bending strength, pressure strength, Bursting-pressure withstand strength, Modulus of elasticity, Hardness and Impact resistance [9]. The choice of an insulating material for a particular purpose often makes it necessary to find a compromise between the diverse requirements and the properties which satisfy them only incompletely. In addition, cost is also a major consideration for the insulation.

1.5 Properties of the Organic Varnish

The important electrical, chemical, physical and thermal properties of the organic varnish were given in Table 1.

Table 1 Properties of the organic varnish

Properties	Quantity
Density	1430 kg/m ³
Young's modulus	3200 MPa
Tensile strength	75-90 MPa
Elongation	4-8 kJ/m
Glass temperature	>400 °C
Vicat softening point	220 °C

Thermal conductivity	0.52 W/(m·K)
Coefficient of thermal expansion	5.5×10^{-5} /K
Specific heat capacity	1.15 kJ/(kg·K)
Water absorption	0.32
Dielectric constant at 1 MHz	3.5

		grams	ate grams		
A	0.16:1:16	2.0995	0.8062	9.09	25
B	0.32:1:16	4.199	0.8062	9.09	25
C	0.32:2:16	4.199	1.6124	9.09	25
D	0.48:2:16	6.2985	1.6124	9.09	50

1.6 Properties of Carbon Nanotubes

Carbon nanotubes have several unique properties. Carbon nanotubes are among the strongest and stiffest materials yet produced owing to their extremely high strength-to-weight ratio. It has unique electrical, thermal and optical properties which make them highly conductive to electricity and heat. It also has a distinctive flexible and kinetic property. The different properties of the carbon nanotubes were given in Table 2.

Table 2 Properties of carbon nanotubes

Properties	Quantity
Tensile strength	63 Gpa
Density	1.3 to 1.4 g·cm ⁻³
Specific strength	48,000 kN·m·kg ⁻¹
Current density	4×10^9 A/cm ²
Temperature stability in air	750 °C
Temperature stability in vacuum	2800 °C
Thermal conductivity	$385 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$

2. SYNTHESIS AND CHARACTERIZATION OF CARBON NANOTUBES

2.1 Synthesis of Carbon Nanotubes

It was observed that most of the research work was carried out with single metal catalysts and a very little amount of research work was carried out on the synthesis of carbon nanotubes using bimetallic catalysts [10]. Hence it was decided to take up research on synthesis of carbon nanotubes using bimetallic catalyst. The synthesis of CNT involves three stages:

- (1) Preparation of catalysts for CNT
- (2) Preparation of CNT
- (3) Purification of CNT

2.1.1 Preparation of Catalysts for carbon nanotubes

Catalysts with different Mo: Fe: Al₂O₃ molar ratios were prepared by adding calculated amount of Ammonium hexamolybdate tetra hydrate and ferrous sulphate heptahydrate into a suspension of alumina powder in methanol. The solvent was evaporated under nitrogen gas flowing in a water bath and the resultant material was heated to 100 – 200° C for 3 hours. The fine powders were then calcined for 1 hour at 500° C and then heat treated with Argon gas flowing for 30 min at 820° C and reground before loading into CVD apparatus. The different concentrations of catalysts prepared were illustrated in the Table 3.

Table 3 Different catalyst concentrations

S. No	Molar ratio of Mo:Fe:Al ₂ O ₃	Weight of Ammonium hexamolybdate	Weight of ferrous sulph	weight of alumina grams	Volume of methanol (ml)

2.1.2 Preparation of carbon nanotubes

The experimental setup consists of a horizontal reaction furnace, Quartz tube, PID controller, Flow meters, Control valves, Gas sources and Thermocouple. CVD apparatus consists of a horizontal reaction furnace with a heating capacity of up to 1500° C. The length of the furnace is 1m and has an inner diameter of 10cm. The quartz tube used to carry out the deposition has a length of 1.2m with outer diameter of 8cm and inner diameter of 7.5cm. The quartz tube is placed inside the horizontal reaction furnace. The heating coils go around the quartz tube and heat it uniformly [11]. The quartz tube is fixed on either side of furnace with supports. A proportional integral differential (PID) controller is provided in the furnace to control the temperature, time and heating rate of the reaction. The PID programming and current control is done manually. The controller displays the set and current temperature and the current is automatically adjusted by the controller to maintain the set temperature [12]. The controller also compensates the heat loss due to the gas flow. Three cylinders of pure Nitrogen (N₂), Acetylene (C₂H₂) and Argon (Ar) were connected to the quartz tube through control valve and flow meter. The flow meter shows the rate of carbon source and nitrogen, which enters the reaction tube. Control valves are used to control the flow rate of gases passed in to the furnace. Rubber hoses are used to connect the cylinders with flow meter and valves [13]. Approximately 200mg of catalyst powder is spread uniformly into the quartz boat (Figure 3.4) and was placed in the central region of a horizontal 100 cm long quartz tube furnace having an inner diameter of 4.5cm. The furnace was flushed with argon gas while it was heated at a rate of 5° C/min till it attained 800° C. After the attainment of the desired temperature, H₂ gas was introduced into the furnace at a flow rate of 100 cc/min for 60 minutes so as to generate active metallic(Fe, Mo) or bimetallic (Fe-Mo) nano particles on Alumina support. Subsequently, C₂H₂ gas in different quantities varying from 10 to 50 cc/min was also introduced into the furnace for 1 hour maintaining the flow of H₂ gas constant. After that it is allowed to cool at a rate of 5° C/min. and removed from the quartz tube for purification.

2.1.3 Purification of Carbon Nanotubes

After synthesis the raw products were treated with a solution of KOH (1M) at 80° C in order to remove the alumina support and molybdenum will be evaporated at this temperature. The resulting material is washed several times with distilled water and then treated with a solution of HCL (1M) in order to remove the remaining iron particles. It was then washed with distilled water before being dried at 200° C.

2.2 Characterization of Carbon Nanotubes

The particle size of CNT was augmented by the scanning electron microscope and x ray diffraction.

2.2.1 Scanning Electron Microscope

The scanning electron microscope (SEM) was a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample

and detecting signals from the interaction of the incident electrons with the sample's surface. The SEM was capable of producing high-resolution images of a sample surface in its primary use mode, secondary electron imaging. Due to the manner in which this image was created, SEM images have great depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. Here the image was built up by using an electron probe of very small diameter which scans the specimen surface in parallel straight lines. When the electron probe scans the specimen, secondary electrons were emitted and these were collected, the current was amplified and the image was formed on the screen. The magnification was about 300,000. The SEM analysis was done for the carbon nanotubes at 10 kV, 10 μm and 25 kV, 1 μm . The result was given in Figure 1 and 2. SEM result was used to augment the particle size of carbon nanotubes.

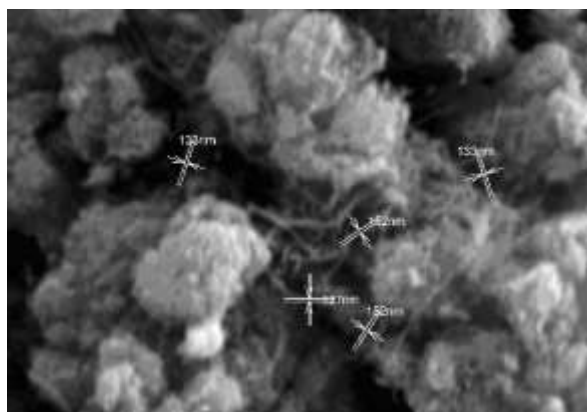


Figure 1 SEM analysis for carbon nanotubes at 10 kV, 10 μm

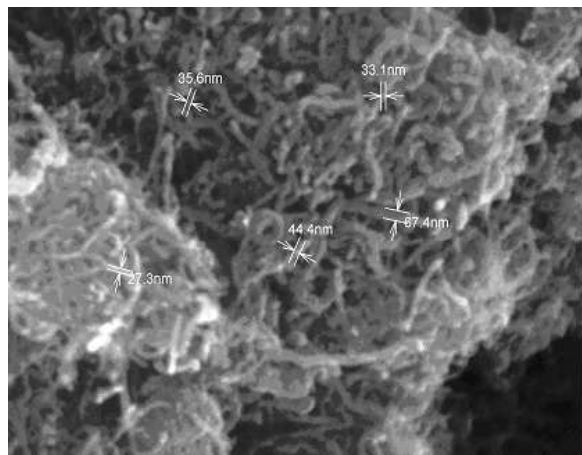


Figure 2 SEM analysis for carbon nanotubes at 25 kV, 1 μm

2.2.2 X-ray diffraction

X-ray diffraction (XRD) is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured materials. The X-ray diffraction pattern of a pure substance is like a fingerprint of the substance. The powder diffraction method is thus ideally suited for characterization and identification of polycrystalline phases. The XRD result for the carbon nanotubes was shown in Figure 3. The micrometer size of the carbon particles is found using XRD. From the graph, it is clear that the value of 2θ should lie between 30° to 40° .

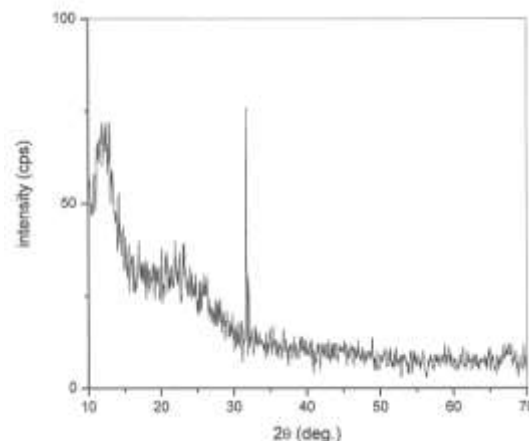


Figure 3 XRD pattern for carbon nanotubes

2.3 Sample Preparation

The process of converting liquid state organic varnish into solid state sample was called as curing.

2.3.1 Types of curing

There were two different types of curing. They were

- (1) Thermal curing - The oligomer which was having a repeat unit (1 to 10) was converted into polymer by using thermal curing.
- (2) Radical initiator curing - In this method, curing agents such as amines, anhydrates and acids are used as curing agents.

2.3.2 Curing of organic varnish

The curing method used for organic varnish having phenolic base was radical initiator curing. In this process DDM was used as curing agent. The procedure used in this curing process was as follows:

80% of enamel and 20% of epoxy resin was taken.

DDM was taken in proportion to epoxy resin. For 1gm of resin 0.27 gm of DDM was added.

DDM was melted at $60-80^\circ\text{C}$ for 10 minutes.

Organic varnish having phenolic base, resin and melted DDM was mixed in a beaker.

The mixture was poured into the die which was coated by a Teflon sheet.

The die was heated at 120°C for 2 hours and 130°C for 3 hours.

The die was cooled and the solid sample was taken.

2.3.3 Mixing of the organic varnish with carbon nanotubes

The enamel and the synthesized carbon nanotubes were mixed with the help of an ultrasonic vibrator. Rotary or linear motion was used in Ultrasonic vibrators to provide consistent vibration for laboratory and industrial shakers, screeners and classifiers, and bins and hoppers. They were powered by an electric motor, pneumatic pistons, or hydraulic fluid. The varnish and the carbon nanotubes taken in 1 wt%, 3 wt% and 5 wt% were completely mixed by the ultrasonic vibrations produced by the ultrasonic vibrators. Ultrasonic vibrations would refer to sound waves that have a frequency higher than human hearing. The vibration themselves would be the physical vibration of the molecules in the medium in which

the sound is traveling. Frequency Range= 20 kHz to 60 kHz (that would be above 20 kHz, or 20,000 cycles per second).

2.4 Curing of the Carbon Nanotubes Filled Enamel

The carbon nanotubes filled enamel so far obtained was in the liquid state. For the various analysis (DSA, TGA and breakdown strength measurements) solid form of the carbon nanotubes filled enamel would be very much effective. So the liquid state carbon nanotubes filled enamel was converted to solid state carbon nanotubes filled enamel. The process of converting the liquid state carbon nanotubes filled enamel into solid state is called as curing. Radical initiator curing method was used and DDM was used as a curing agent.

3. DIELECTRIC ANALYSIS

The various dielectric properties of the carbon nanotubes filled organic varnish such as breakdown electric strength, surface resistance, dielectric loss factor, quality factor, phase angle, heat generated, dielectric loss and dielectric conductivity were found.

3.1 Partial Discharge Measurement

Partial discharges are in general a consequence of local electrical stress concentrations in the insulation or on the surface of the insulation. Generally such discharges appear as pulses of duration of much less than 1s. The partial discharge includes a wide group of discharge phenomena such as internal discharges occurring in voids or cavities within solid or liquid dielectrics, surface discharges appearing at the boundary of different insulation materials, corona discharges occurring in gaseous dielectrics in the presence of inhomogeneous fields and continuous impact of discharges in solid dielectrics forming discharge channels (treeing). Every discharge event causes a deterioration of the material by the energy impact of high energy electrons or accelerated ions, causing chemical transformations of many types. The number of discharge events during a closed interval of time is strongly dependent on the kind of voltage applied and will be largest for a.c. voltages. The actual deterioration is dependent upon the material used. It was still the aim of many investigations to relate partial discharges to the life time of specified materials. The detection and measurement of discharges is based on the exchange of energy taking place during the discharge. These exchanges are manifested as electrical pulse currents, dielectric losses, e.m. radiation, sound, increased gas pressure and chemical reactions.

Partial discharge (PD) current pulses are very sharp pulses having a width of the order of nanoseconds. The IEC 60270 PD detectors are approximated the measured PD pulses as an impulse (less than 500kHz bandwidth). Auto transformer takes an input of 220 V, 50Hz, single phase and gives an output varies from 0 to 220V, 50Hz, single phase. The rating of high voltage transformer is 220/100kV, 50Hz, single phase. By varying the input from 0 to 220V the output varies from 0 to 100kV in proportion. Core made of iron type. The coupling capacitor shall be mounted insulated or other measures shall be taken to ensure that ground foil can be connected to the insulated terminal of the measuring impedance AKVD. Discharge free coupling capacitor is used in this setup. The coupling capacitor should preferably be high ($C_c=1000\text{pF}$). AKVD is the measuring impedance, an inductor in parallel with a capacitor. The measuring impedance unit (Z_m) was located physically close to the high

voltage coupling circuit and serves two main purposes: It attenuates the test voltage present on the high voltage coupling circuit to the safe value for the measurement of the partial discharge signals. It matches the amplifier to the high voltage coupling circuit in insuring a flat frequency response across the full measurement band width. The measuring impedance unit (Z_m) should be configured in such a way as to permit test voltage level monitoring and to observe the phase relationship between the test voltage and the partial discharge pulses; this technique helps to identify the nature of the discharges. Agilent oscilloscope 54621A is used for the display of the waveforms. The DSO is connected to the DTM at the backend. The oscilloscope displays the phase position of the partial discharge pulses with respect to the AC test voltage. The phase position of the partial discharge pulses with respect to the AC test voltage allows conclusions about the location of the partial discharges in the test object.

The arrangement of electrodes and experimental set up used for partial discharge measurements is shown in the Figure 4.

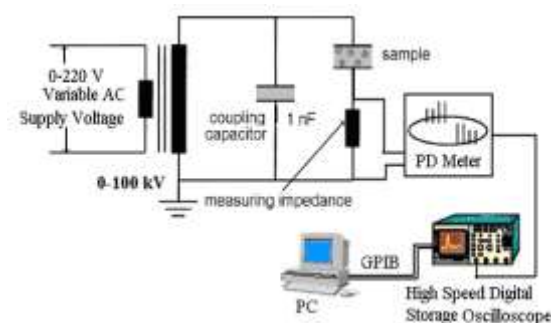


Figure 4 Circuit arrangements for Partial Discharge Measurement

Sample thickness : 3 mm

Diameter of upper electrode : 25mm

Diameter of lower electrode : 75mm

The entire arrangement in an insulating liquid with an appropriate dielectric constant (Ex: Insulating oil).

Before starting the experiment on the test specimen PD meter should be calibrated. The calibrator comprises a pulse generator in series with small capacitor of known value. The generator and the capacitor may be placed in the small box or may be connected together via a properly terminated coaxial cable of sufficient length to permit a calibration from the control room. The pulse generator should either have a known calibrated output level or its output level should be monitored. A suitable output level adjustment in the form of a calibrated potentiometer or a calibrated step attenuator should be provided. The following three methods specify the test procedure for PD measurements, the first verifies that test object is free from significant partial discharges up to a specified test voltages; second is used to determine the discharge inception and extinction voltages; and the third is used for measurement of discharge level at a voltage or voltages in range between the inception voltage and the maximum dielectric voltage. A voltage, well below the specified discharge free voltage, is applied to the test object, gradually increased to the specified voltage, and maintained for the specified time; there after it is decreased and switched off. The object is considered to have passed the test if the discharges do not exceed a specified level. A voltage well below the inception value is applied to the test object and gradually increased until discharge exceeds a specified level.

The test voltage at this discharge limit is recorded. The voltage is then increased by 10% and there after reduced to a value at which the discharge ceases or become less than a specified level. The voltage corresponding to this limit is recorded. For some insulation systems, the extinction and inception values may be influenced by the length of the time that the test voltage is maintained above the inception level. The PDG pulse generator is connected in parallel with the test object and normally operated on position "500pC". The effective PD of the PDG simulates a known partial discharge intensity of the object. With the measuring range selector 'E' of the DTM set on the respective position, the display is also set to 500pC with the potentiometer 'G' "CORR". For a defined test arrangement and with identical test objects, the correction factor has to be determined only once and holds good for all the measurements in the setup. The time domain data was collected after a stabilization period of 5 minutes. 25 cycles of data has taken with each discharge source. The period of 25 sampling with 2M data points was 500ms. The period of one sampling with 80k data points was 20ms. The discharge signal is captured on along with the superimposed sinusoidal signal for 25 cycles for the five types of discharges considered. The Partial discharge inception and extinction voltage values of the organic varnish and the organic varnish filled with the different (1 wt %, 3 wt% and 5 wt%) proportions of carbon nanotubes filled organic varnish and it was shown in the Table 4.

Table 4 Partial discharge inception and extinction voltage of the organic varnish and the organic varnish with different (1 wt %, 3 wt% and 5 wt%) proportions of carbon nanotubes

% wt of carbon Nano tubes	Inception voltage (kV)	p C	Extinction voltage (kV)	p C	% Decrease in Inception voltage	% Decrease in Extinction voltage
0	4.74	55	4.10	1.3	-	-
1	3.27	64	2.19	1.4	31.01	46.58
3	4.01	65	3.53	1.2	15.40	13.90
5	4.21	66	3.63	1.3	11.18	11.46

3.2 Breakdown voltage and electric strength

The breakdown voltage of an insulation system is that of the voltage with a certain time dependence for which the dielectric either temporarily or permanently loses its insulating property by the way of a discharge process. The intensity of the electric field at which breakdown occurs was called as dielectric strength or breakdown strength. Breakdown strength = Breakdown voltage / Thickness of the dielectric. It was measured in kV/mm or kV/cm. The electric breakdown strength of insulating material depends on a variety of parameters, such as pressure, temperature, humidity, field configurations, nature of applied voltage, imperfections in dielectric materials, material of electrodes, and surface conditions of electrodes, layer thickness, types of voltage, stress duration and frequency. The breakdown voltage shows an increasing dependence on the nature and smoothness of the electrode material. The breakdown strength reduces considerably due to the presence of impurities. The

breakdown field strength is an extraordinary important material property for dimensioning an insulation system.

For solid insulating materials, certain criteria are available from measurement of the breakdown voltage or the breakdown field strength on plate-like samples in a homogeneous or weakly inhomogeneous field. A standardized testing arrangement for the determination of the breakdown field strength of plates or foils up to sample thickness of 3mm as per IEC 60243 – 1 was as shown in Figure 5. AC breakdown tests were carried out at power frequencies (50 -60 Hz) using a step-up transformer supplied from a variable sinusoidal low voltage source. If tests were performed at different frequencies, the low voltage source may be a power amplifier controller by a low frequency generator. However, that in such circumstances the maximum test frequency should be considerably below the value at which dielectric losses within the material may become significant to precipitate failure due to thermal instability. The rate of voltage increase may be controlled by the use of a motor driven auto transformer. Alternatively an induction regulator may be employed. A controlled voltage rate of increase was necessary for the performance of the short-time and slow rate of rise breakdown tests. A step by step test would require only a manual voltage control. It was so popular before constant rate of rise sets became commonplace. A current limiting resistor was used to limit the current at breakdown. Its value should not be too large, since it may give rise to an unduly large voltage drop with the higher currents preceding breakdown. A value of 106 Ω / kV appears to be convenient in most cases.

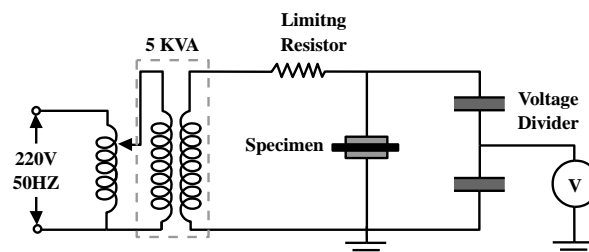


Figure 5 Circuit arrangements for AC Breakdown voltage test

The breakdown test was conducted with alternating voltage, which should be increased from zero to the breakdown voltage within 10-20 s. The median value of the breakdown voltage was determined from five samples; if any value lies more than 15% of the median value, 5 additional samples must be tested and the median value then determined from 10 samples. The breakdown field strength can be evaluated from the breakdown voltage and the smallest electrode spacing. The AC breakdown voltage values of pure organic varnish and the organic varnish filled with the different (1 wt %, 3 wt% and 5 wt %) proportions of carbon nanotubes filled organic varnish was shown in the Table 5.

Table 5 AC breakdown voltage values of pure organic varnish and the organic varnish filled with different (1 wt %, 3 wt% and 5 wt %) proportions of carbon nanotubes

% wt of carbon Nano tubes	Breakdown Voltage (kV)
0	7.7
1	5.54
3	6.22
5	6.55

The breakdown strength values of pure organic varnish and the organic varnish filled with the different (1 wt %, 3 wt% and 5 wt%) proportions of carbon nanotubes filled enamel was shown in the Table 6.

Table 6 Breakdown strength of pure organic varnish and the organic varnish filled with different (1 wt %, 3 wt% and 5 wt%) proportions of carbon nanotubes

% wt of carbon Nano tubes	Breakdown strength (kV/mm)	% Decrease
0	2.56	-
1	1.85	27.73
3	2.07	19.14
5	2.18	14.84

3.3 Surface resistance measurement

Practical insulation systems frequently comprise many dielectrics which were stressed in parallel. Thus, for example, the insulation resistance of a support insulator consists of the combination in parallel of surface resistance and volume resistance. While the volume resistance, commonly expressed as specific resistance in Ω cm, was often independent of surrounding medium, the surface resistance was appreciably influenced by ambient conditions such as pressure, temperature, humidity, dust etc. An arrangement for the measurement of the volume resistance of plate-type insulating material samples was discussed. In this arrangement, the live electrode, which also supports the plate-type sample, was arranged opposite a measuring electrode. The volume resistance was calculated from the direct voltage applied (100 V or 1000 V) and the current taken by the measuring electrode. A guard arranged concentrically around the measuring electrode with a 1 mm gap prevents erroneous measurements caused by surface currents. Special testing arrangements were available for tube-shaped insulating material samples, for insulating compounds which can be melted and for liquid insulating materials. Common insulating materials exhibit a specific volume resistance of 1012 to 1013 Ω cm, whereas superior materials can reach resistance values up to 1017 Ω cm or even higher. To measure the surface resistance metallic knife-edges are used, set up at a gap spacing of 1 cm over a length of 10 cm on the surface of the insulating material under test and direct voltage is applied. From the voltage and current, the surface resistance, expressed in ohms, is determined. The surface resistance and surface resistivity of the organic varnish and the organic varnish filled with different (1 wt %, 3 wt% and 5 wt%) proportions of carbon nanotubes filled organic varnish and was presented in the Table 7.

Table 7 Surface resistance and surface resistivity of the organic varnish and the organic varnish filled with different (1 wt %, 3 wt% and 5 wt %) proportions of carbon nanotubes

% wt of carbon Nano tubes	R_s (Ω)	ρ_s (Ω cm)	% Increase in R_s	% Decrease in R_s	% Increase in ρ_s	% Decrease in ρ_s
0	7.09 x 10 ⁸	1.36 x 10 ⁹	-	-	-	-
1	6.21 x 10 ⁸	1.16 x 10 ⁹	-	12.41	-	14.7

3	5.15 x 10 ⁸	1.01 x 10 ⁹	-	27.36	-	25.73
5	12.6 x 10 ⁸	2.62 x 10 ⁹	77.71	-	92.64	-

3.4 Calculation of relative permittivity, dielectric conductivity and dielectric losses

Dielectric spectroscopy was used to find the loss factor, quality factor and phase angle of the dielectric material. From these factors, dielectric constant, dielectric conductivity, dielectric loss and heat generated under AC fields were found. Dielectric spectroscopy [LCR HITESTER 3532-50] also known as Electrochemical Impedance Spectroscopy, measures the dielectric properties of a medium as a function of frequency. It is based on the interaction of an external field with the electric dipole moment of the sample, often expressed by permittivity. It is also an experimental method of characterizing electrochemical systems. This technique measures the impedance of a system over a range of frequencies, and therefore the frequency response of the system, including the energy storage and dissipation properties, is revealed. Often, data obtained by EIS is expressed graphically in a Bode plot or a Nyquist plot. Real and Imaginary part of Relative Permittivity of the organic varnish was calculated from the following equations which was used to calculate the dielectric loss.

$$\text{Loss factor } \tan \delta = \frac{\epsilon_r''}{\epsilon_r'} \quad (3)$$

$$\text{Real part of relative permittivity } \epsilon_r' = \left(\frac{t * C_p}{A * \epsilon_0} \right) \quad (4)$$

Imaginary part of relative permittivity

$$\epsilon_r'' = \left(\frac{t}{\omega * R_p * A * \epsilon_0} \right) \quad (5)$$

$$\text{Dielectric conductivity } \sigma = \omega * \epsilon'' \quad (6)$$

$$\text{Dielectric losses } P = 2\pi * f * C * V^2 * \tan \delta \quad (7)$$

$$\text{The heat generated under AC field } W_{ac} = E^2 * f * \epsilon_r * \tan \delta / 1.8 \times 10^{12} \text{ W / cm}^3 \quad (8)$$

Where

f is the frequency in Hz,

δ is the loss angle of the dielectric material and E is the applied electric field.

C_p is the equivalent parallel capacitance [F]

R_p is the equivalent parallel Resistance [Ω]

D is the dissipation factor (measured value)

t_m is the average thickness [m]

A is the guarded electrode's surface area [m²]

D is the guarded electrode's diameter [m]

ϵ_0 is the permittivity of free space = 8.854 x 10⁻¹² [F/m]

V is the applied voltage [V]

$\tan \delta$ is the dielectric loss tangent

The different dielectric properties like dissipation factor, quality factor and phase angle were found with the help of the Dielectric Spectroscopy. Dielectric constant, dielectric losses, dielectric conductivity and heat generated under AC fields were analyzed for various frequencies and the values were shown in Table 8 to 14.

Table 8 Dissipation Factor

Frequency in HZ	Pure Varnish	1 Wt % CNT mixed Varnish	3 Wt % CNT mixed Varnish	5 Wt % CNT mixed Varnish
50	8.865	2.793	2.98	3.83
100	6.496	1.194	1.590	1.663
1000	2.537	0.74	0.831	1.52
10000	1.508	0.29	0.8	1.229
100000	0.97	0.072	0.37	0.063
1000000	0.121	0.03	0.07	0.093
5000000	0.0627	0.003	0.063	0.028

Figure 6 shows Comparison of Dissipation Factor Vs Frequency for various samples at 90° C

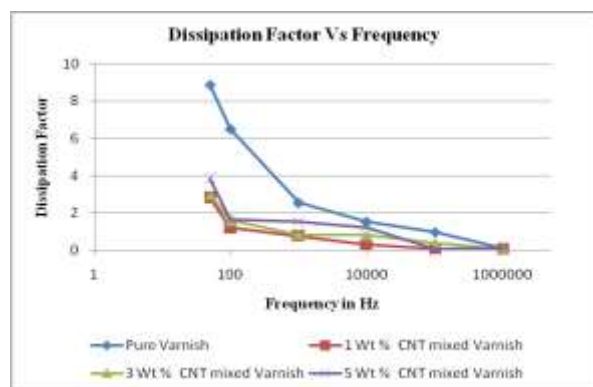


Figure 6 Comparison of Dissipation Factor Vs Frequency for various samples at 90° C

Table 9 Quality Factor

Frequency in HZ	Pure Varnish	1 Wt % CNT mixed Varnish	3 Wt % CNT mixed Varnish	5 Wt % CNT mixed Varnish
50	0.3	1.6	1.2	0.72
100	0.8	1.8	1.9	1.31
1000	1.65	4.65	3.1	2.6
10000	2.97	9.72	3.58	4.2
100000	6.26	14.97	9.3	15.43
1000000	9.64	15.8	11.9	13.84
5000000	18.95	10.5	15.72	9.8

Figure 7 shows Comparison of Quality factor Vs Frequency for various samples at 90° C

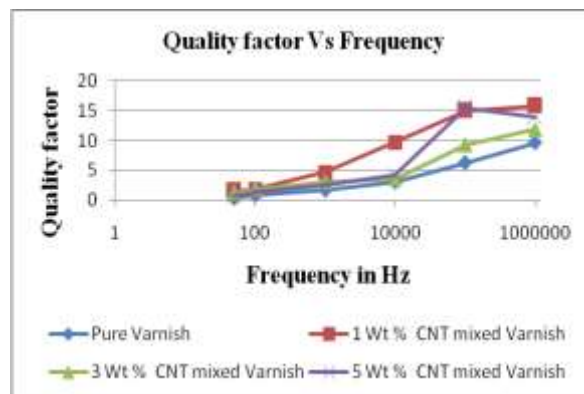


Figure 7 Comparison of Quality factor Vs Frequency for various samples at 90° C

Table 10 Dielectric Constant

Frequency in HZ	Pure Varnish	1 Wt % CNT mixed Varnish	3 Wt % CNT mixed Varnish	5 Wt % CNT mixed Varnish
50	227.35	58.37	102.9	100.37
100	165.47	54.85	99.24	96.41
1000	78.054	39.66	73.26	56.3
10000	49.51	27.66	39.29	25.36
100000	38.43	23.81	32.03	23.23
1000000	26.96	22.5	25.95	22.23
5000000	20.39	19.81	23.33	20.16

Figure 8 shows Comparison of Constant Vs Frequency for various samples at 90° C

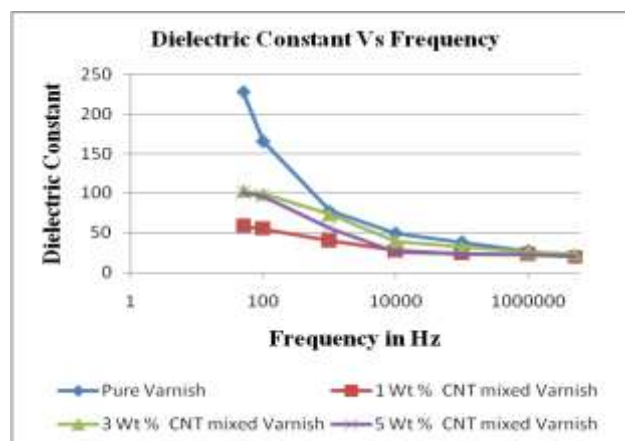


Figure 8 Comparison of Constant Vs Frequency for various samples at 90° C

Table 11 Dielectric losses (μW)

Frequency in HZ	Pure Varnish	1 Wt % CNT mixed Varnish	3 Wt % CNT mixed Varnish	5 Wt % CNT mixed Varnish
50	6.78	0.6	0.64	0.47
100	6.3	0.2	0.4	0.4
1000	8.5	0.97	1.53	1.9
10000	19.11	2.32	12.6	3.9

100000	49.01	18.22	25.53	12.2
1000000	259.76	107.79	178.24	172.26
5000000	589.63	667.35	557.68	1197.58

Figure 9 shows Comparison of Dielectric losses Vs Frequency for various samples at 90° C

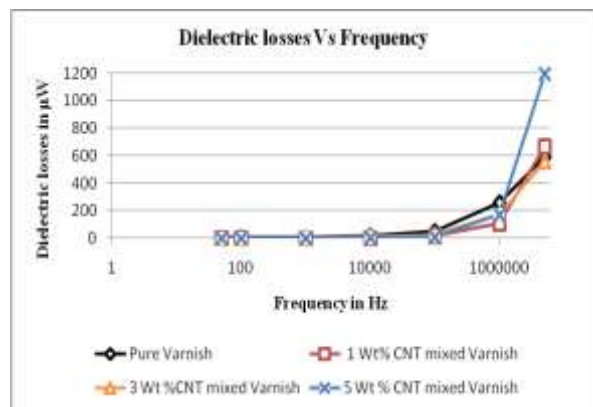


Figure 9 Comparison of Dielectric losses Vs Frequency for various samples at 90° C

Table 12 Dielectric conductivity (S)

Frequency in HZ	Pure Varnish	1 Wt % CNT mixed Varnish	3 Wt % CNT mixed Varnish	5 Wt % CNT mixed Varnish
50	4.82 x 10 ⁻⁶	1.67 x 10 ⁻¹²	9.61 x 10 ⁻¹⁴	2.28 x 10 ⁻¹³
100	4.9 x 10 ⁻⁹	4.5 x 10 ⁻¹²	2.81 x 10 ⁻¹³	5.81 x 10 ⁻³
1000	6.2 x 10 ⁻⁶	1.22 x 10 ⁻¹⁰	3.63 x 10 ⁻¹¹	1.77 x 10 ⁻¹⁰
10000	1.57 x 10 ⁻⁸	1.38 x 10 ⁻⁸	8.94 x 10 ⁻⁹	1.62 x 10 ⁻⁸
100000	3.8 x 10 ⁻⁸	1.44 x 10 ⁻⁹	1.22 x 10 ⁻⁶	1.17 x 10 ⁻⁶
1000000	1.58 x 10 ⁻⁴	8.66 x 10 ⁻¹⁰	5.86 x 10 ⁻⁴	9.81 x 10 ⁻⁴
5000000	4.6 x 10 ⁻⁴	5.2 x 10 ⁻¹¹	57.91 x 10 ⁻³	103.32 x 10 ⁻³

Table 13 Phase Angle

Frequency in HZ	Pure Varnish	1 Wt % CNT mixed Varnish	3 Wt % CNT mixed Varnish	5 Wt % CNT mixed Varnish
50	-7.05	-52.55	-44.62	-41.53
100	-10.21	-58.29	-58.44	-57.56
1000	-33.14	-75.66	-73.18	-63.25
10000	-63.36	-83.62	-69.78	-77.65
100000	-76.52	-86.91	-83.26	-86.32
1000000	-82.89	-87.38	-85.68	-85.63
5000000	-87.51	-94.76	-92.12	-81.29

Table 14 Heat Generated (W/cm³)

Frequency	Pure	1 Wt %	3 Wt %	5 Wt %
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in HZ	Varnish	CNT mixed Varnish	CNT mixed Varnish	CNT mixed Varnish
50	32.24	1.465	1.86	1.74
100	33.34	2.632	2.77	2.04
1000	43.61	2.23	9.13	8.9
10000	82.35	9.64	49.24	25.77
100000	242.31	36.9	137.76	53.38
1000000	1279.79	324.01	729.21	773.15
5000000	3128.76	1834.05	2488.81	5185.01

Figure 10 shows Comparison of Heat Generated Vs Frequency for various samples at 90° C

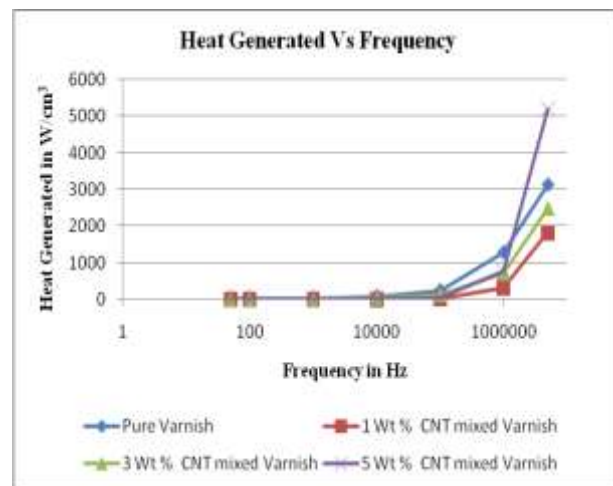


Figure 10 Comparison of Heat Generated Vs Frequency for various samples at 90° C

4. THERMAL ANALYSIS

A generally accepted definition of thermal analysis is “A group of techniques in which a physical property of a substance and/or its reaction products is measured as a function of temperature whilst the substance is subjected to controlled temperature program”. It is based upon the detection of changes in the heat content (enthalpy) or the specific heat of a sample with temperature (Skoog 2003). Such enthalpy changes may be detected by thermal analysis and related to the processes occurring in the sample. Thermal analysis can be done by different methods such as the measurement of heating curves, dynamic adiabatic calorimetry, differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermo gravimetry (TG), thermal mechanical analysis (TMA) and dynamic mechanical thermal analysis (DMTA). Thermo Gravimetric Analysis (TGA) is a simple analytical technique that measures the weight loss (or weight gain) of a material as a function of temperature. Thermo gravimetry (TG) was a material characterization technique, where the mass of a sample in a controlled atmosphere is recorded continuously as a function of temperature or time as the temperature of the sample is increased (usually linearly with time). In TG, the sample and reference are both maintained at the temperature predetermined by the program even during a thermal event in the sample. The typical arrangement for the components of a TGA instrument is revealed in Figure 11.

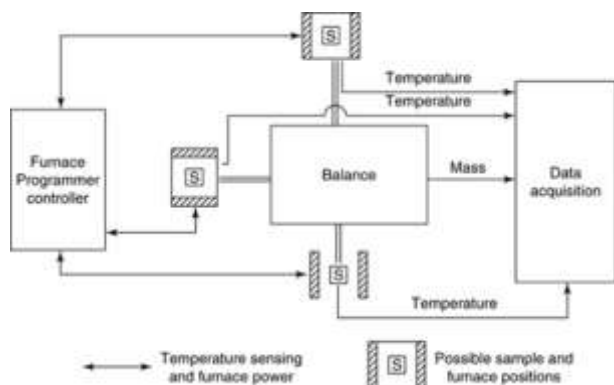


Figure 11 Typical arrangements for the components of a TGA instrument

In the Diamond TG/DTA 6000 Instrument system the sampling size can be analyzed from 0.1mg to 10g and the heating rate of 0.1-50°C/min for the temperature range from 50°C to 900°C and it maintain consistent heating rate and gas flow. This instrument measures sampling purity, reaction rate, identification, activation energy and heat of reactions. TG result of the organic varnish was shown in the Figure 12 and the 1 wt%, 3 wt% and 5 wt% of carbon nanotubes mixed with the organic varnish was shown in the Figure 13, 14 and 15. The test result was a graph of the TGA signal (actual weight loss or gain converted to percent weight loss) on the Y-axis plotted versus the sample temperature in °C on the X-axis. TG analysis was used to record and study the dependences of the mass of the sample on the ageing time or on a gradually increasing temperature and evaluate the heat of exothermic and endothermic reactions in the material. TG analysis was also used to determine the thermo elasticity and the thermo stability.

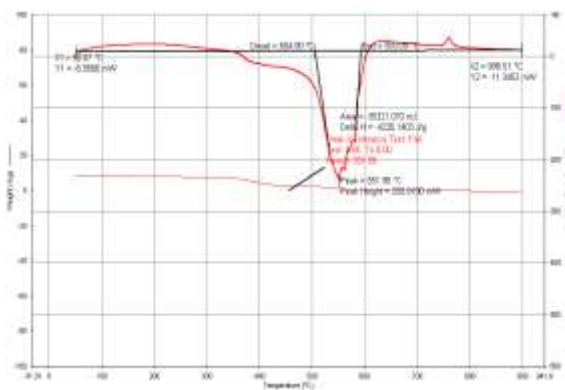


Figure 12 TG Analysis of the pure organic varnish

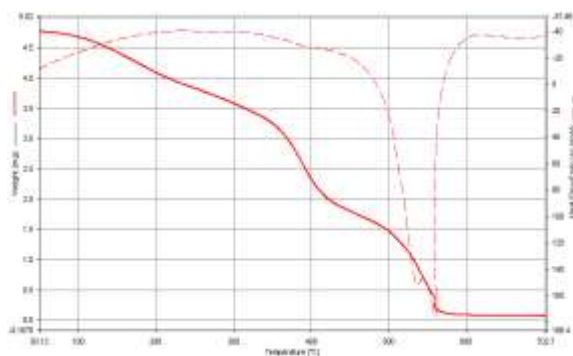


Figure 13 TG Analysis of the organic varnish mixed with 1 wt% of carbon nanotubes

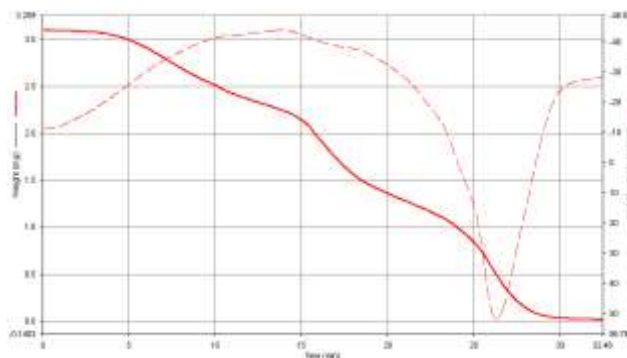


Figure 14 TG Analysis of the organic varnish mixed with 3 wt% of carbon nanotubes

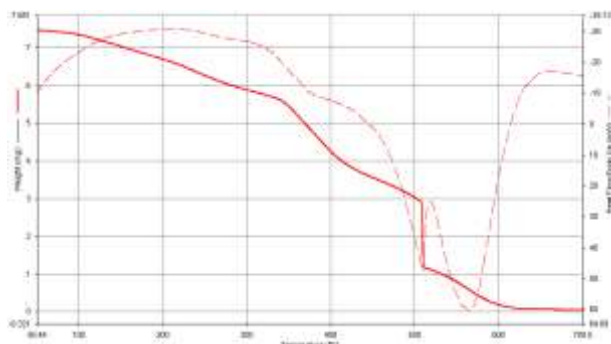


Figure 15 TG Analysis of the organic varnish mixed with 5 wt% of carbon nanotubes

From the above TG graphs, the onset temperature, the peak temperature and the end temperature of the organic varnish and the nano carbon filled enamel with various (1%, 3% and 5%) wt proportions were found and the readings were given in the Table 15.

Table 15 the onset temperature, the peak temperature and the end temperature of the organic varnish and the carbon nanotubes filled organic varnish with various (1%, 3% and 5%) wt proportions

Sample	On set temp (°C)	Peak temp(°C)	End temp (°C)	% Increase in On set temp	% Increase in Peak temp	% Increase in End temp
Pure	504.90	551.98	593.09	-	-	-
1%	517.76	569.59	595.16	2.54	3.19	0.34
3%	519.76	589.79	600.26	2.94	6.84	1.20
5%	531.26	595.63	609.30	5.22	7.90	2.73

5. CONCLUSION

It was observed from the table the PD inception voltage decreased with 32.49 %, 11.18 % and 9.07 % for 1 wt%, 3 wt% and 5 wt% carbon nanotubes filled enamel respectively from the PD inception

voltage of the enamel. Also the PD extinction voltage decreased in 44.15 %, 9.02 % and 9.02 % for 1 wt%, 3 wt% and 5 wt% carbon nanotubes filled enamel respectively from the PD extinction voltage of the enamel. Similarly it was also noted that the breakdown strength decreased by 25.39 %, 8.66 % and 7.48 % for 1 wt%, 3 wt% and 5 wt% carbon nanotubes filled enamel respectively from the breakdown strength of the enamel. From the thermal analysis the on set temperature was increased by 2.55 %, 2.94 % and 5.22 % for 1 wt%, 3 wt% and 5 wt% carbon nanotubes filled enamel respectively from the on set temperature of the enamel. Also it was observed that the peak temperature was increased by 3.19 %, 6.85 % and 7.91% for 1 wt%, 3 wt% and 5 wt% carbon nanotubes filled enamel respectively from the peak temperature of the enamel. Also it was noted that the peak temperature was increased by 0.35 %, 1.21 % and 2.73% for 1 wt%, 3 wt% and 5 wt% carbon nanotubes filled enamel respectively from the end temperature of the enamel.

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