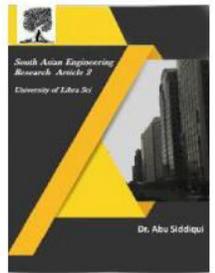




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PERMITTIVITY, THERMAL CONDUCTIVITY AND THERMAL STABILITY OF POLY NANOCOMPOSITES

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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.

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.

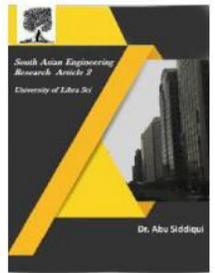
1.1 Chemical properties

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



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impurities increase the conductivity and cause a poorer dissipation factor and breakdown 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.

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

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 W·m ⁻¹ ·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:

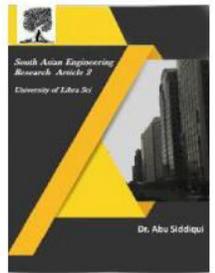
- (1) Preparation of catalysts for CNT
- (2) Preparation of CNT
- (3) Purification of CNT

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 hexa molybdate 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



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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)
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		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

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.

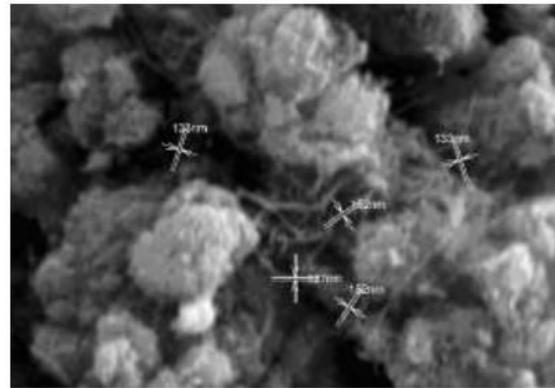


Figure 1 SEM analysis for carbon nanotubes at 10 kV, 10 μm

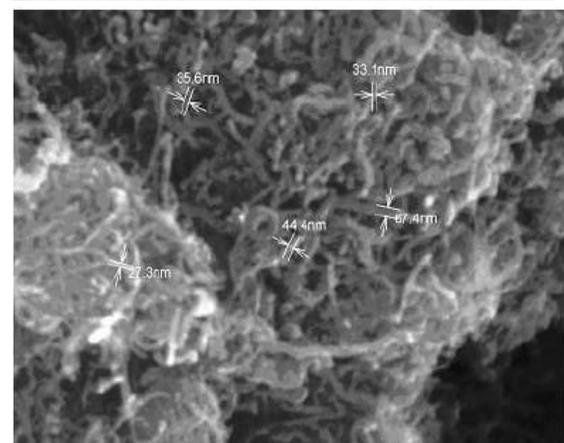


Figure 2 SEM analysis for carbon nanotubes at 25 kV, 1 μm



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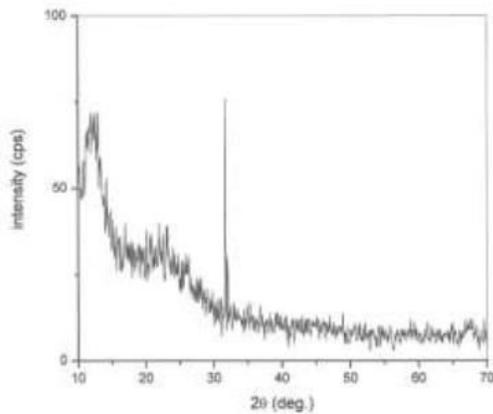
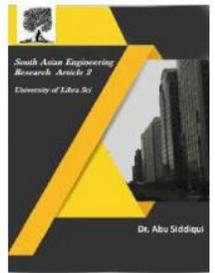


Figure 3 XRD pattern for carbon nanotubes

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 1000kV 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 was 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.

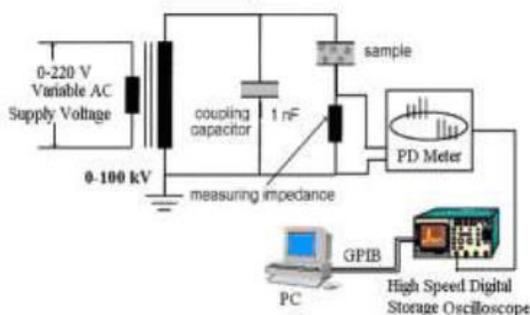


Figure 4 Circuit arrangements for Partial Discharge Measurement

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

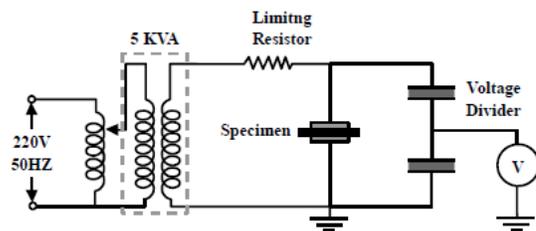


Figure 5 Circuit arrangements for AC Breakdown voltage test

The breakdown test was conducted with alternating voltage, which should be increased from zero to the breakdown value 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

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

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	7.09×10^8	1.36×10^9	-	-	-	-
1	6.21×10^8	1.16×10^9	-	12.41	-	14.7

% wt of carbon Nano tubes	10^8	10^9				
3	5.15×10^8	1.01×10^9	-	27.36	-	25.73
5	12.66×10^8	2.62×10^9	77.71	-	92.64	-

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

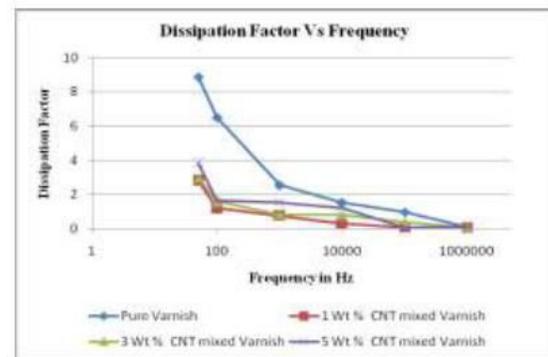


Figure 6 Comparison of Dissipation Factor Vs Frequency for various samples at 90° C

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

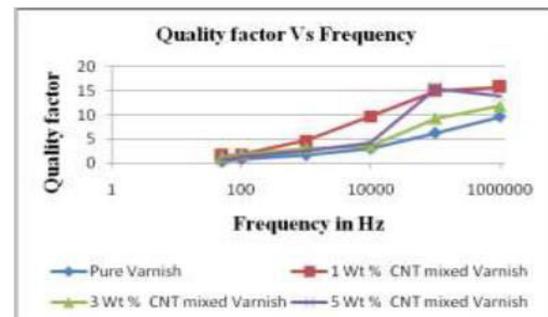


Figure 7 Comparison of Quality factor Vs Frequency for various samples at 90° C



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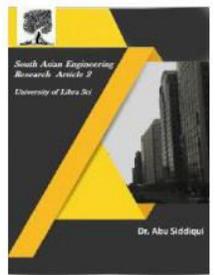


Table 10 Dielectric Constant

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

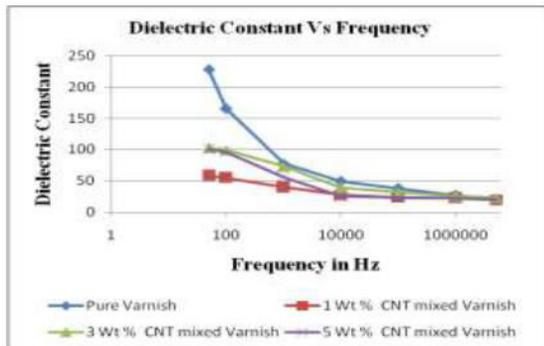


Figure 8 Comparison of Constant Vs Frequency for various samples at 90° C

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

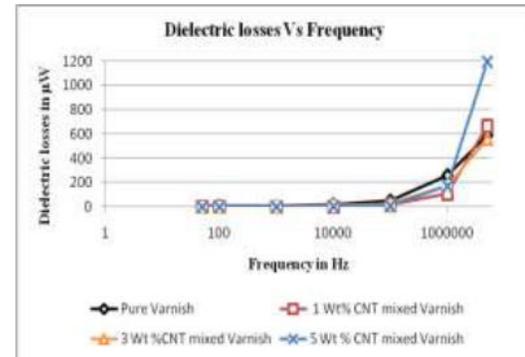


Figure 9 Comparison of Dielectric losses Vs Frequency for various samples at 90° C

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	4.82×10^{-6}	1.67×10^{-12}	9.61×10^{-14}	2.28×10^{-13}
100	4.9×10^{-9}	4.5×10^{-12}	2.81×10^{-13}	5.81×10^{-3}
1000	6.2×10^{-6}	1.22×10^{-10}	3.63×10^{-11}	1.77×10^{-10}
10000	1.57×10^{-8}	1.38×10^{-8}	8.94×10^{-9}	1.62×10^{-8}
100000	3.8×10^{-8}	1.44×10^{-9}	1.22×10^{-6}	1.17×10^{-6}
1000000	1.58×10^{-4}	8.66×10^{-10}	5.86×10^{-4}	9.81×10^{-4}
5000000	4.6×10^{-4}	5.2×10^{-11}	57.91×10^{-3}	103.32×10^{-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

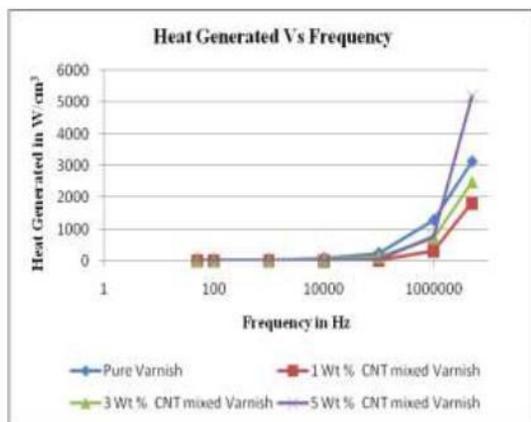


Figure 10 Comparison of Heat Generated Vs Frequency for various samples at 90° C

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.

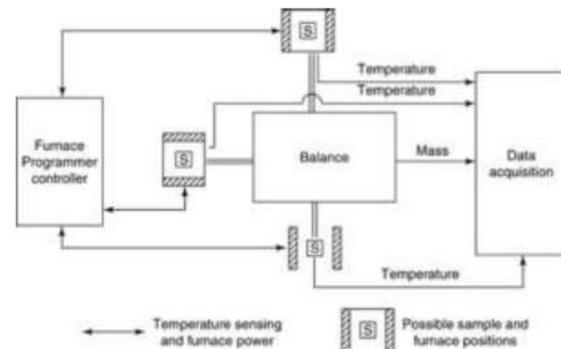


Figure 11 Typical arrangements for the components of a TGA instrument



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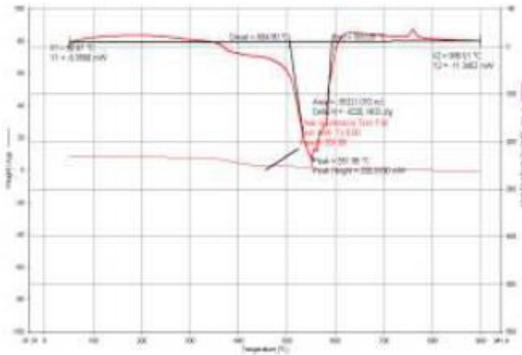
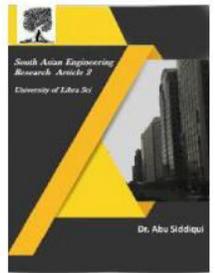


Figure 12 TG Analysis of the pure organic varnish

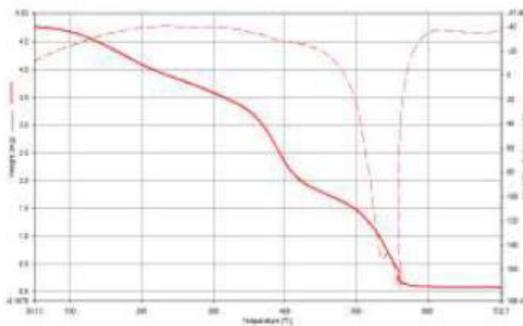


Figure 13 TG Analysis of the organic varnish mixed with 1 wt% of carbon nanotubes

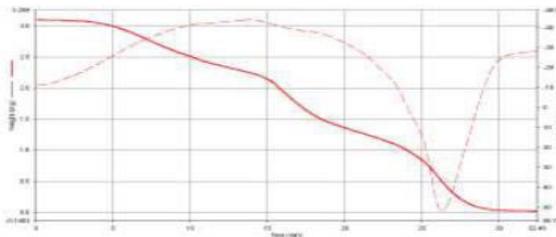


Figure 14 TG Analysis of the organic varnish mixed with 3 wt% of carbon nanotubes



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

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

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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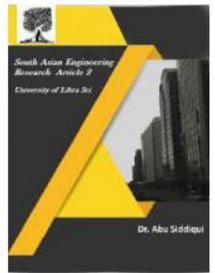


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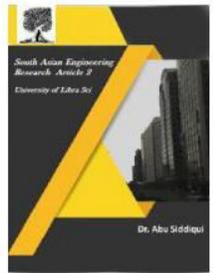


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