Central Power Research Institute

Vol. 15(2)/122-129, July-December 2019
ISSN (Print) : 0973-0338
DOI : 10.33686/pwj.v15i2.150407

Power Research

Development of Epoxy with Nano and Micro Fillers for Core Insulation of Composite Insulators

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Abstract

Epoxy composites are extensively used in different applications in high voltage engineering. However, the use of these composites for outdoor insulators has been challenging due to complexities arising out of the conditions of operation of the insulators. Pollution performance of composite insulators is influenced by the nature of the pollution existing, in addition to the combination of stresses acting on the insulators. The use of nano and micro combination of fillers in the epoxy composites is an ideal choice for achieving a proper balance of electrical, thermal and mechanical properties. This paper considers the merits of using combination of micro and nano fillers in an epoxy matrix to demonstrate that such insulation systems would provide better alternatives to the conventional epoxy systems for use as central rod in composite insulation systems for severe contaminated conditions.

Keywords: Epoxy Composites, Nano and Micro Fillers, Outdoor Insulators

1. Introduction

The demand for electrical power has been consistently increasing due to rapid urbanization and industrialization. In order to meet the ever-increasing demand of power, the transmission voltage levels have been increased to 1200 kV AC and ± 800 V DC. With increasing transmission system voltage, the demand for reliable and stable insulation systems has been very much challenging. The insulating materials are expected to work under higher operating stresses including electrical, mechanical and thermal stresses under hostile environmental conditions. Therefore, there is a constant demand for improving the quality of insulating materials to meet the challenging needs of the composite insulators which are used in transmission systems. The use of conventional polymer composites seems to be inadequate for present day requirements and therefore nano composites have been considered as possible alternatives to conventional polymer composite insulators[1–5]. Since the use of one or combination of more inorganic fillers resulted in improvement of certain properties at the cost of other properties, it has become extremely difficult to arrive at a suitable combination which can yield optimum electrical, thermal and mechanical characteristics. Hence, the concept of hybrid composites consisting of a micro and nano filler or different combinations of micro and nano fillers are being studied to develop a new generation of insulating material which will be suitable for the demands of the present hostile environmental conditions. In this paper a brief review of fillers and filler combinations and their benefits have been undertaken. Experimental results are presented and discussed to highlight the advantages of hybrid polymer composites for high voltage outdoor insulation applications.

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2. Inorganic Fillers and their Benefits

2.1 Use of Nanofillers

Inorganic fillers with sizes of the order of few nanometers to tens of nanometers are considered under this broad category. The nano fillers considered in this investigation are alumina and silica. Silica is generally observed to improve the thermal properties and flexural modulus of the composites. But the disadvantage of this filler is that the dynamic mechanical properties are somewhat reduced due to their presence. Alumina fillers are useful for minimizing the impact damage of the composites. Generally, the fillers are used in very low concentrations, not exceeding 10 wt. % for optimal benefits, but there are instances where very high percentage of filler is used for meeting specific requirements[6–12].

2.2 Use of Micron Sized Fillers

The inorganic fillers are of the size of few m to tens of m. The micron sized fillers considered in this investigation are Alumina Trihydrate, Calcium Carbonate and Magnesium Oxide. The details of the fillers used are shown in Table 1. The wt. % of the ECR glass fiber used was 75 % and that of the epoxy was varied from 15 to 20 % and the combination of nano and micro fillers was balanced by corresponding reduction in the weight percentage of the base epoxy. Some variations in the percentages of the ECR glass fibers, epoxy and the fillers were made for balancing the combination to achieve optimum electrical, thermal and mechanical properties[13–16]. The details of the filler, their weight percentages along with their benefits are shown in Table 1. The thermal properties of the composites are not discussed in this paper. However, satisfactory improvements in thermal characteristics like thermal conductivity and coefficient of thermal expansion were achieved in the composites discussed.

In this paper fillers and filler combinations and their benefits are briefly reviewed. Experimental results have been presented and discussed to highlight the advantages of hybrid composites for high voltage outdoor insulators.

Among the advantages of some of the organic fillers used which is shown in Table 1, the benefits of the individual fillers are the main criterion for combining the nano and micron sized fillers. The range of their weight percentages indicated in the table is also maintained in the hybrid composites studied. The data of Table 1 is based on literature reports and the benefits that accrue are very specific to the base matrix, the filler properties and the method of fabrication employed and hence some differences are expected in the ultimate properties of the hybrid composites.

Table 1. Benefits of filler and their weight percentages

Sl. No	Filler	wt % (range)	Benefits
1	Nano Silica	5-10	1. Increases the breakdown strength 2. Enhances surface and volume resistivity 3. Increases Glass transition temperature
2	Nano Alumina	2-5	1. Enhances volume resistivity 2. Increases breakdown strength 3. Enhances dielectric constant 4. Reduces dielectric loss 5. Increases dissipation factor
3	Aluminium trihydrate	5-20	1. Improves thermal conductivity 2. Improves permittivity
4	Calcium carbonate	10-20	1. Good dielectric properties 2. Improves thermal conductivity 3. Improves thermal stability. 4. Increases permittivity
5	Magnesium Oxide	5-10	1. Increases dielectric strength 2. Improves thermal conductivity 3. Reduces dissipation factor 4. Increases volume resistivity

3. Experimental Method

3.1 Materials

The details of the materials used in the fabrication of epoxy glass composites and their source along with the density are shown in Table 2.

3.2 Method of Fabrication

The Bisphenol-A epoxy resin [Araldite MY 740, Hardener HY 918, and Accelerator DY 062], surface functionalized ECR-glass long fiber (18 μm diameter), high purity grade of uncoated particles of nano-silica (SiO_2), with average

particle size 12 nm and nano-alumina (Al_2O_3) having an average size 12 nm were used in this investigation. Micro fillers namely Alumina Trihydrate ($Al_2O_3.3H_2O$), with average particle size of 2.6 μm, Calcium Carbonate ($CaCO_3$) of 5 mm average diameter or Magnesium Oxide (MgO) of 8 mm size was used in different combinations with nano fillers in the epoxy resin matrix to fabricate composites. The coupling agent used was 3-glycidyloxypropyl trimethoxysilane and the details of materials, fillers and reinforcement used, and their designations are shown in Table 3.

After breaking up of the agglomerates, the required amount of silane coupling agent was added to the mixture and maintained for 10 min for hydrolysis to occur. The mixture was then introduced in to the resin and sonication was carried out for 40 min. This mixture was subjected to vacuum 10^{-3} Torr at 85 °C to eliminate ethanol. Hardener (85 wt. %) and accelerator (2 wt. %) were added to the composite and mixed in a high shear mixture for 15 min at 3000 rpm. Using 75 to 78 wt. % of ECR-glass fibers as reinforcement, glass epoxy composites with different filler combinations were fabricated by either pultrusion technique or they were cast into laminates of different thickness.

Except the base glass epoxy composite, the other composites consisted of individual fillers or combination of micro and nano fillers. The discussions in this paper are confined to the four composites listed in Table 3 and the results of single nano or micro filler is not discussed due to limitations of space. The diameters of the rod manufactured using pultrusion method were 16 and 20 mm respectively.

Table 2. Details of filler and base epoxy used (Nano-nano sized filer; micro: micron sized filler)

Sl. No	Materials	Source	Density (g/cm^3)
1	Epoxy	Huntsman, USA	1.16
2	ECR glass fiber	Owens Corning, India	2.62
3	Alumina (Nano)	Sigma Aldrich	4.0
4	Silica (Nano)		2.6
5	$CaCO_3$ (Micro)		2.93
6	Mg O(Micro)		3.58
7	ATH (Micro)	Akrochem Corp.	2.42

3.3 Measurement Methods

The measurements were carried out under standard laboratory conditions with temperature of 25±5 C and humidity of 55±5 % RH. The electrical measurements were carried out in accordance with IEC 112, ASTM D2303, ASTM D149, ASTM D257, IEC 62217, IEC 61109 and IEC 60383-2 methods. The evaluation of electrical parameters was carried out either on 3-4 mm thick flat samples or on the 11 kV composite insulators which consisted of the central core rod made of the composites listed in Table 3 with silicone weather sheds. The results of individual material and the composite insulator are presented together for deciding the impact of the fillers in the composite materials.

Table 3. Details of composites and their identification

Sl. No	Composite	Description
1	GE	ECR glass fiber (78) reinforced epoxy (22)
2	GESAA	ECR glass fiber (78) reinforced epoxy (12) + nano-silica (2) + nano alumina (3) + micro ATH (5)
3	GESAC	ECR glass fiber (78) +epoxy (12) + nano silica (2) + nano alumina (3) + micro $CaCO_3$ (5)
4	GESAM	ECR glass fiber (78) + epoxy (12) + nano silica (2) +nano alumina (3) + micro MgO (5)

(Figures in parenthesis indicate the weight percentage of filler)

Contact angle measurements on the surface of insulating material are usually carried out to determine the surface properties of the materials like hydrophobic or hydrophilic nature and the surface energy. Hollmarc make contact angle apparatus was used for measurements. The samples were oriented so that the surface under consideration was horizontal. Water drop was carefully placed on the horizontal surface of the sample with the help of a needle of a hypodermic syringe. The drops were photographed with the help of high-resolution digital camera immediately after the placement of the water drop on sample. The data was then analyzed using software "ImageJ". The drop volume was controlled to be within 2μL and the time set was 100 seconds. For each specimen, the contact angle was measured at five different locations and the average value was considered for assessment and analysis.

4. Results and Discussions

4.1 Electrical Parameters

The parameters tested are the comparative tracking index, time of track initiation, depth of erosion, the electric strength (flatwise and edgewise), volume resistivity and surface resistivity.

The CTI of the composites as per IEC 112 (2009-10) method did not show much difference and all the composites withstood 50 drops of the electrolyte at 600 V and the results are shown in Table 4.

Table 4. CTI values of the composites by method a of IEC 112

Sl. No.	Composite	CTI	Depth of erosion (mm)
1	GE	600	0.12
2	GESAA	600	0.10
3	GESAC	600	0.08
4	GESAM	600	0.08

Since it was difficult to distinguish the differences in the ability of the composites to resist surface tracking by method A of IEC 112, method B of the specification was used, and the results are shown in Table 5. The CTI of the base composite was reduced to 550 when method B of IEC 112 procedure was used. The depth of erosion was measured after the test. The CTI of the specimens were measured on 3.1 mm thick specimens.

Table 5. CTI values of the composites by method b of IEC 112

Sl. No.	Composite	CTI	Depth of erosion (mm)
1	GE	550	0.25
2	GESAA	600	0.15
3	GESAC	600	0.14
4	GESAM	600	0.13

The results of inclined plane tracking and erosion by the method ASTM D2303 (IEC 60587-2007) are shown in Table 6. By the step by step voltage method, the voltage across the electrodes was increased by 250 V, starting from 2.5 kV and the voltage at which tracks were initiated were determined. For tracking initiation, a minimum length of 12.5 mm from the high voltage electrode was the yardstick. Since the composites withstood 4 kV for 6 h without tracking, the experiments were repeated by the time to track method at a proof tracking voltage of 6 kV (RMS) and the time required for initiation of tracks on the surface of the composite was determined. In addition, the erosion depth of the composites was also determined.

Table 6. Time to track of the composites at a voltage of 6 kv

Sl. No	Composite	Time for track initiation (h)	Average Depth of erosion (mm)
1	GE	Tracking not initiated	< 0.20
2	GESAA	Tracking not initiated	< 0.12
3	GESAC	Tracking not initiated	< 0.10
4	GESAM	Tracking not initiated	< 0.11

Though tracking was not initiated in any of the composites, the base epoxy composite suffered relatively higher depth of erosion as compared to the other three hybrid composites. The average of five readings was considered for measurement of the depth of erosion of the composites. For this experiment, the thickness of samples used was 6 mm. The depth of erosion was measured in the vicinity of the HV electrode. Thus, from CTI and Inclined Plane tracking and erosion test data, it was ensured that the composites had good tracking and erosion resistance.

The results of the electric strength of the composites across the thickness (flat wise) and across the surface (edgewise) are shown in Table 7. The electrodes used were in accordance with the ASTM specification D149-9 (2013). The measurements were carried out in oil to avoid flashovers and the temperature of measurement was 90 C. For flat wise electric strength measurements, 3.1 mm thick specimens were used and for edge wise electric strength, 6 mm thick specimens were used. Due to limitations of the

Table 7. Electric strength data of the composites

Sl. No.	Composite	Flat wise (kV/mm)	Edgewise (kV/mm)
1	GE	23	Withstood 80 kV for only 30 seconds
2	GESAA	28	Withstood 80 kV for 1 min
3	GESAC	30	Withstood 80 kV for 1 min
4	GESAM	32	Withstood 80 kV for 1 min

experimental set up, the composites were evaluated for a proof voltage of 80 kV and breakdown levels could not be reached within 100 kV.

The volume resistivity and surface resistivity measurements of the composites were undertaken in accordance with ASTM D 257-14 specification. The composites with the fillers incorporated, showed higher volume and surface resistivities as compared to the base composite. The composites were immersed in tap water for 500 h and the volume resistivity was measured to estimate the effect of diffused water. The results of volume resistivity are shown in Table 8.

Table 8. Volume resistivity of the composites at 25 ± 2 °C

Composite	Volume Resistivity (Ω-cm)	
	As cast	Water aged
GE	4.2×10^{14}	3.2×10^{12}
GESAA	5.3×10^{15}	5.48×10^{14}
GESAC	6.1×10^{15}	1.07×10^{14}
GESAM	6.7×10^{15}	1.57×10^{14}

The results of surface resistivity of the composites in the as received condition and after water immersion for 500 h are shown in Table 9. From the results of volume and surface resistivity, it is evident that the composites with fillers perform in a better manner than the base epoxy composite. Further, the reduction in either volume resistivity or surface resistivity after immersion in water, was less as compared to the base composites mainly due to the presence of fillers which do not permit diffusion of water into the composites.

Table 9. Surface resistivity of the composites at 25 ± 2 °C

Designation of Composites	Surface Resistivity (Ω/Square)	
	As cast	Water aged
GE	7.68×10^{13}	1.513×10^{11}
GESAA	9.86×10^{13}	2.80×10^{12}
GESAC	12.3×10^{13}	4.10×10^{12}
GESAM	13.5×10^{13}	4.50×10^{12}

The properties of the composites in the form of laminates, is thus observed to meet the requirements of the relevant specifications as basic material stage. However, it is more important that the materials also show superior performance as a composite insulator.

4.2 Contact Angle Measurements

Contact angle measurements of the composites were determined to categorize the type of the surface as hydrophobic or hydrophilic in nature and to compute the surface energy. Increased hydrophobicity of the polymer composites is characterized by higher contact angle which in turn leads to the reduction in the surface energy. This is very well correlated as seen from the data in Table 10. The decrease in surface energy is due to the decrease in free energy resulting from the fillers. Further, the chain mobility is restricted with the addition of fillers. Relatively, GESAM composite is superior to the other composites considered in this study.

Table 10. Data on contact angle of the composites

Composite	Contact Angle(°)		Surface energy (mN/m)
	Tap Water	Formamide	
GE	90	55	46.25
GESAA	95	64	42
GESAC	100	68	39
GESAM	120	85	38

The surface energy of G-E composites is generally high as compared to other composites due to the formation of carboxyl group by the reaction between oxygen in water and the epoxy molecular chains. The decrease in the surface energy of the hybrid composites is due to the decrease in free energy resulting from the addition of fillers. Further, the chain mobility is restricted with the addition of fillers. The fillers occupy the space between the epoxy molecular chains and thus restrict the electronic affinity of oxygen with epoxy[16,17].

4.3 Evaluation of Composite Insulator Rods

In order to translate the good characteristics of the composites achieved on the laboratory scale, composite rod insulators were fabricated. The composites evaluated in the laboratory were used as the central rod and using silicone rubber weather sheds, insulators were fabricated for voltage rating of 11 kV, 33 kV and 66 kV respectively. Due to ease of handling and availability of resources, 11 kV composite insulators were subjected to some of the critical tests as per IEC 62217 (2012-09) and IEC 61109 (2008-05) specifications and the results are briefly discussed in this section.

4.3.1 Water Diffusion Electrical Testing of Central Rod

The water diffusion tests were carried out on the central core of the composite insulators. The tests were carried out in accordance with IEC 62217 (2012-09) specification. Six samples were used for each test and were prepared as per clause 9.4.2.2 of the specification. The maximum leakage current at an applied voltage of 12 kV across the rods did not exceed 1 mA as required by the specification. The composite rods with filler showed significant decrease in current as compared to the unfilled composite mainly because of the filler and the hydrophobic surface of the composites due to the fillers. The magnitude of the maximum leakage current recorded for different composite rod insulation is shown in Figure 1.

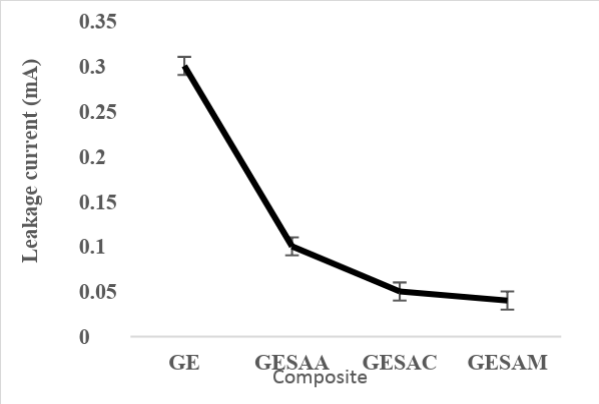

Figure 1. Maximum leakage current during water diffusion tests of the composites.

4.3.2 Dye Penetration Test

The tests were carried out on the core material as per clause 9.4 of the specifications (IEC 61109 (2008-05)). In all, ten samples were used for evaluation and the results of the tests are summarized in Table 11.

Table 11. Summary of the results of dye penetration test

Sl. No	Composite	Number of failures out of 10 samples
1	GE	One
2	GESAA	None
3	GESAC	None
4	GESAM	None

It can be observed that even in case of the unfilled insulator, only one sample out of ten failed but the other composites satisfactorily met the requirements. The filler systems thus help the composites to meet the acceptance criteria of the specification. Thus, the usefulness of the fillers in the manufacture of the central rod is very much emphasized by this result. The tests were repeated on five different batches of the filled epoxy composites and a high degree of consistency was achieved. The results are attributed to the optimum method of preparation of the composites using the base resin with fillers and the curing agent and the pultrusion method of fabrication.

4.3.3 Brittle Fracture

This is accepted as a screening test which measures the resistance of the composite rod to acids. For this evaluation, a free length of the insulator was selected to avoid interference from the superimposed mechanical stresses in the area of crimping.

Since the tensile strength in unidirectional fiber composites is typically a fiber-dominated phenomenon, transverse failures can occur across fiber bundles normally at the ultimate tensile strength of the composite. This failure is termed the brittle fracture and it takes place at unusually low loads of the string, when the composite is exposed to adverse chemical environments, and it is therefore generally referred to as the acid attack brittle fracture.

In the testing procedure, the composite rod with 16- and 20-mm diameter is subjected to a constant tensile load corresponding to the load at 75 % of the measured tensile strength of the composite rods. The tensile strength of the base epoxy was 720 MPa and that of the hybrid composites was 760 MPa on an average. A portion of the gauge section was immersed in Nitric Acid (HNO3) solution for a period of 96 hours. This test was performed at ambient temperature to determine the mechanical resistance against corrosion stress of the FRP core and the method followed the procedure outlined in IEC Project 36-6-2 of WG 36-07. The rods are expected to withstand the test conditions and the insulation resistance should not fall below 1 GΩ. The results of the test are shown in Tables 12 and 13.

Table 12. Results of brittle fracture test

Composite	Observations after 96 hours of testing
GE	No visible damage was observed
GESAA	No visible damage was observed
GESAC	No visible damage was observed
GESAM	No visible damage was observed

Table 13. Insulation resistance of composites

Composite	Insulation Resistance (GΩ)
GE	12.0
GESAA	12.2
GESAC	10.5
GESAM	11.0

4.3.4 Lighting Impulse Voltage Tests

The composite insulators were subject to lightning impulse test as per IEC 60383-2 (1993). In all, five samples were subjected to lightning impulse test at 82.5 kV for an insulator rated 11 kV. All the composites withstood the applied impulse voltage.

5. Conclusions

The investigation has established that insulators fabricated using epoxy composites with combination of nano and micro sized fillers perform very satisfactorily in all the tests carried out in the laboratory as per international standards. The advantages of the individual nano and micro filler particles help to strengthen the overall characteristics of the composite insulators when used in combination and hence are a viable alternative to the nanocomposites which serves the purpose of improving a single targeted property, which is invariably the mechanical or thermal strength. The use of hybrid combination of micro and nano fillers thus, would help in a more holistic way to achieve better performance of the composite rod insulators.

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