Role of Micro and Nano Filler Combination in Hydrophobic Recovery of Glass Fiber Reinforced Epoxy Composites under Various Contaminants

Santhy P. Kuruvilla, N. M. Renukappa

Abstract: Use as outdoor insulators in high voltage power transmission is a potential application of epoxy composites. Vulnerability of these insulators to various stresses when exposed to external environment, limit their potential. Better pollution performance in hostile environments can be achieved in these composites by using nano and micro combination of fillers. This paper discusses the merits of using combination of micro and nano fillers consisting of silica, alumina, ATH, $CaCO_3$ and MgO in glass epoxy to demonstrate that they serve a better alternative to the conventional glass epoxy composite insulation systems. Extensive investigations have been carried out on hydrophobic recovery of these composites for understanding their pollution performance under various contaminants such as NaCl, seawater, acid and cement. The addition of filler combinations to glass epoxy enhanced the hydrophobic properties of the composites compared to the glass epoxy without fillers. The results revealed that the composite with MgO filler showed 37% increase in contact angle in as-cast form compared to contact angle of epoxy without fillers.

Keywords: Contact angle, hydrophobic recovery, glass epoxy composites, pollution performance.

I. INTRODUCTION

Extensive research investigations have been carried out on surface wetting phenomena in outdoor polymers for understanding the performance of composites under contaminated conditions [1,2,4-7]. The wettability in general describes the capability of the liquids to spread on the polymer surface. It is also recognized that a proper assessment of surface wetting characteristics of the material can be established by measurement of contact angle of liquids on the surface of solids [3]. This paper discusses the effect of different contaminants on the contact angle of silicone and glass reinforced epoxy composites. Using a combination of fillers consisting of silica, alumina, ATH, Magnesium oxide and calcium carbonate, three different epoxy composites namely GESAA, GESAC and GESAM were developed and their surface wetting characteristics/ hydrophobicity has been analyzed. The contact angle was measured by subjecting these materials to five different contaminants having different electrical conductivity and

salinity. An attempt is made to evaluate the relative recovery characteristics when subjected to ambient conditions for 2000 h after immersing them in five different contaminants.

II. MATERIALS AND METHODS

A. Materials

The details of the source of the materials, the weight percentage of epoxy, filler and glass fiber reinforcement used in the fabrication of the composites are shown in Table I and II respectively.

B. Fabrication

Epoxy composites were fabricated using epoxy resin with surface functionalized long ECR-glass fiber. The coupling agent used was 3-glycidyloxypropyl trimethoxy silane. Since nano silica and nano alumina are established to impart improvement in dielectric properties, while micrometer sized ATH, $CaCO_3$ and MgO are known to impart distinct advantages to the composites, combination of these fillers has been used and the reasons for selection of fillers is outlined in an earlier publication [8]. To reduce the viscosity and to eliminate the moister content, epoxy resin and hardener were pre-treated in a vacuum chamber for 1h 15 min at 65 °C before mixing. Similarly, before mixing, SiO_2, Al_2O_3, ATH, $CaCO_3$ and MgO filler particles were preheated in an oven for 24 h at 115 °C. The fillers were dispersed through gentle stirring in absolute ethanol. To disperse the nano/micro particles uniformly in the epoxy resin, Ultrasonication and high-speed shearing were employed. Silane coupling agent was mixed and hydrolyzation occurred after 15 min. Then epoxy resin was mixed with the composite, followed by sonication for 45 min. The accelerator, release agent and the hardener were mixed using a high shear mixer at 3500 rpm. Before pultrusion, glass fibers were modified with silane coupling agent for improving the interfacial adhesion between the resin and glass fibers. The weight was maintained at 78 wt. % in all the composites during reinforcement of ECR-glass fiber.

Using a table-top lab-scale pultrusion machine, the composite rods were fabricated. The machine consisted of a resin bath, pultrusion die and a puller. Skeins with the required amount of fibers for the given wt. % of the primary reinforcement were made by winding the glass fiber roving on a former. After soaking in resin, the skeins were pulled into flared glass tubes by allowing the resin to settle prior to post-curing at a temperature of 80 ºC for 4 h.

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Table- I: Details of materials used in fabrication of Composites

No	Materials	Source	Density (g/cm³)
1	Epoxy	Huntsman, USA	1.16
2	ECR glass fiber	Owens Corning, India	2.62
3	Alumina (Nano)	Sigma Aldrich, USA	4.0
4	Silica (Nano)		2.6
5	$CaCO_3$ (Micro)		2.9
6	MgO (Micro)		3.6
7	ATH (Micro)	Akrochem Corp.	2.4
8	Silicone	Navitas Insulators, India	2.1

Table- II: Details of epoxy, glass fiber and filler percentages used (weight percentages used are indicated by the numbers in parenthesis)

Sample	Details
GE	ECR glass fiber reinforced (78) epoxy (22)
GESAA	ECR glass fiber (75) reinforced epoxy (15) with nano-silica (2) + nano alumina (3) +micro ATH (5)
GESAC	ECR glass fiber (75) +epoxy (15) + nano silica (2) + nano alumina (3) + micro $CaCO_3$ (5)
GESAM	ECR glass fiber (75) + epoxy (15) + nano silica (2) + nano alumina (3) + micro MgO (5)

C.   Selection of Contaminants

The contaminants used in this investigation are listed in Table III along with their electrical conductivity, salinity and pH values. In this study, NaCl solution, cement solution, sea water, tap water and H_2SO_4 were also used as contaminants [9-15], keeping in mind wide range of applications of the fiber reinforced composites. Sea water is different in its constituents from NaCl since it has ions of Na, Mg. Ca, K. Br, Fl, sulphates, HCO_3 and Bromine. Sea water is alkaline and Ca ++ and HCO_3^- forms insoluble $CaCO_3$. Al and Iron Oxide coagulate to form colloidal solutions [16]. The constituents of tap water [17] are different from that of sea water in that it has Cl, Fl, trihalomethanes, in addition to As, Pb, Hg, Al, Ba, Cd, some sulphates, pesticides, organic matter, microbiological contents like algae and bacteria. It has a pH of 8 and compounds like SO_2 and NOx dissolve easily in water and are carried by wind to form acid fog with pH of 4.2 to 4.4. Hence the effect of acid was also studied. The composites were immersed in the contaminants for 1000 h and measurement of the contact angle was carried out at regular intervals.

The details of the contaminants selected with their electrical conductivity, salinity and pH values are shown in Table III.

Table- III: Details of the contaminants used

No.	Contaminant	Conductivity (mS) (at 25 ºC and 55% RH)	pH	Salinity kg/m³
1	NaCl	15	8.44	8.875
2	Cement	15	11	8.875
3	Sea Water	46	7.56	30.03
4	Tap Water	1.2	7.94	0.57
5	Acid	0.95	4.0	0.44

For the measurement of the contact angle, Hollmarc instrument was used. The samples were initially conditioned at a temperature of 25±2 ºC and 55±5% RH before commencement of the measurements. Stability of the applied drop on the specimen was ensured by orienting the samples were horizontally oriented so that the applied drop was stable on the specimen. Using a hypodermic syringe, a 2μL drop of the liquid was placed on the horizontal surface of the sample. A high-resolution digital camera was used to take photographs of drops. The recorded data of the contour of the drop was then analyzed using "ImageJ" software [18]. For measurements, samples measuring 5 cm x 10 cm were used. During the long duration immersion experiments, the specimens were removed periodically and were wiped with a clean dry cloth and immediately subjected to measurements. The measurements were completed within 5 min after removal from the container with the contaminants. In this investigation the surface wetting angle of polymer has been evaluated as a function of duration of exposure to the contaminants. To assess the capability of composites to recover the hydrophobicity which in turn is related to the wetting angle, the polymers were conditioned at ambient temperatures of 25±5 °C and RH of 55±5 % for a further period of 2000 h and contact angle measurements were carried out.

III.   RESULTS AND DISCUSSION

A. Assessment of Influence of Contaminants on CA Of Composites

The initial surface contact angle (CA) of the composites (virgin) is furnished in Table IV. The results revealed that the composite with MgO filler showed 37% increase in CA as compared to CA of glass-epoxy without fillers. The method of fabrication of composites and uniform distribution of the two nano fillers and the micro filler is critical for improving the CA and also for its recovery. Use of saline coupling agent improves the interfacial bonding between nano alumina, silica particles and the incorporation of micro sized fillers namely ATH, $CaCO_3$ and MgO further enhances the interfacial strength. Depending upon the relative sizes, shape and placement of the filler in the epoxy matrix, the pathway for water diffusion will be different. The distribution of the nano and micro fillers within the epoxy matrix will ultimately decide the pathway for transport of contaminants.

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It is also essential to understand that the free volume in these epoxies will be different because of the differences in the filler particle

size of ATH (2.6 µm), $CaCO_3$ (5 µm) and MgO (8 µm) and their distribution in the matrix.

Table- IV: Initial CA with Standard Deviation in percentage

Composites	CA (as-cast) (°)	Standard Deviation (%)
GE	86	1.95
GESAA	100	1.86
GESAC	110	1.83
GESAM	118	1.76
Silicone	114	1.65

The free space length [18] in the material microstructure is computed to be maximum in GE (1800 µm) and it is very much reduced (60-80 µm) in filled GE. Analysis of free volume GE composites using Positron Annihiliation spectrocopy have revealed that the GE has higher free volume (of about 60 Å3) as compared to GESAA, GESAC an GESAM (of 50 Å3) leading to higher water absorption in GE. Thus nano-Al_2O_3, nano-SiO_2 and micro-ATH, $CaCO_3$ and MgO fillers improve the cross linking and thus help in reducing the chain mobility. Further, from the data of Positron spectroscopy, the fillers due to their relative

dimensions appear to be located in between the chain segments and not in free volume cavities. Thus, the water molecules occupy the free volume in the epoxy materials and the results agree with the reported studies [1-2].

The data of CA variations with NaCl, cement solution, sea water, tap water and acid are shown in Table V. Except silicone all the GE materials studied lose their hydrophobicity due to long-duration exposure to contamination. However, with increase in duration of exposure GESAA and GESAM tend to recover partially their hydrophobicity.

In fact, GESAM regains its hydrophobicity to a better extent than the other two GE materials with fillers. Therefore, the performance of the five materials on a comparative scale show that silicone is least affected by NaCl. On the other hand, GESAM exhibits a better performance followed by GESAA, GESAC and GE when exposed to NaCl. The data of CA variations with NaCl, cement solution, sea water, tap water and acid is shown in Table V. It is observed that throughout the 1000 h period of immersion in cement, silicone is able to retain its contact angle. The results also show that GESAC and GESAM retain their hydrophobicity during the 1000 h period. Thus, the CA of silicone, GESAC and GESAM has shown that their surfaces are not influenced by cement.

From results of 1000 h immersion in seawater, silicone and GESAC appear to perform relatively better as compared to GE and GESAA and GESAM. However, all the GE composites lose their hydrophobicity when exposed to tap water for 1000 h. Further, with acid, Silicone and GESAM retains hydrophobicity at the end of 1000 h. Thus, the variation in contact angle depends upon the contaminant type, particle size and free volume in the bulk of the GE composite.

Table- V: The data of CA variations with NaCl, cement solution, sea water, tap water and acid

Contaminant	Composite	CA of virgin surface	Average CA During 50-1000h of exposure
NaCl	Silicone	114	109
	GE	86	71
	GESAA	100	89
	GESAC	110	85
	GESAM	118	92
Cement	Silicone	114	114
	GE	86	65
	GESAA	100	85
	GESAC	110	101
	GESAM	118	92
Seawater	Silicone	114	105
	GE	86	63
	GESAA	100	80
	GESAC	110	90
	GESAM	118	83
Tap water	Silicone	114	108
	GE	86	56
	GESAA	100	64
	GESAC	110	54
	GESAM	118	57
Acid	Silicone	114	106
	GE	86	60
	GESAA	100	82
	GESAC	110	82
	GESAM	118	92

B. Assessment of Recovery of Contact Angle After The Exposure to Contaminants

The ability of materials to recover hydrophobicity after their exposure to contaminants for 1000 h was investigated and the results are discussed in this section. All the materials were exposed to ambient conditions at 25±5° C and a RH of 55±5 % for 2000 h and variations in the CA were recorded.

a. Recovery of contact angle of silicone

The data of CA during 2000 h of exposure to ambient conditions after 1000 h conditioning with NaCl, sea water, cement, tap water and acid in case of silicone is shown in Fig.1. It is observed that silicone recovers its hydrophobicity completely since contact angle reaches a value of 114 with NaCl. Thus, silicone has a remarkable ability to retain and maintain a relatively higher contact angle. It is observed that in case of other four contaminants, namely cement, sea water, tap water and acid there is a recovery from 112±2° to 113±1° during exposure to ambient conditions for 250 to 1000 h and the CA settles down to 113±1° irrespective of the contaminants used.

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Thus, silicone exhibits unique characteristics in that the reduction in contact angle is from 114º (virgin) to 107º (lowest in case of seawater). However, the data of contact angle as a function of time duration under ambient conditions has demonstrated that silicone has the capability to recover during the off periods of contamination. The recovery of CA also demonstrates that the pollutants do not penetrate deeper in to the bulk and are easily removed because of their presence at the top surface.
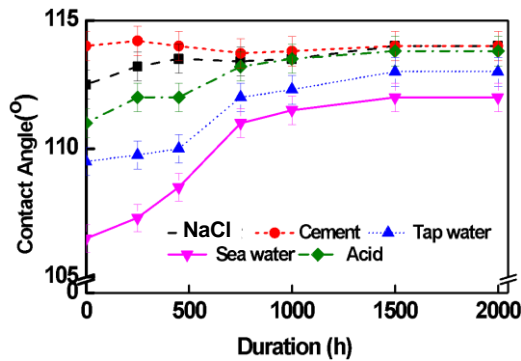

Fig. 1. Variation of contact angle of Silicone with duration of exposure to ambient conditions after 1000 h conditioning

b. Recovery of Contact Angle of GE

The variations of CA during 2000 h of exposure to ambient conditions after 1000 h conditioning with NaCl, sea water, cement, tap water and acid in case of GE is shown in Fig.2.
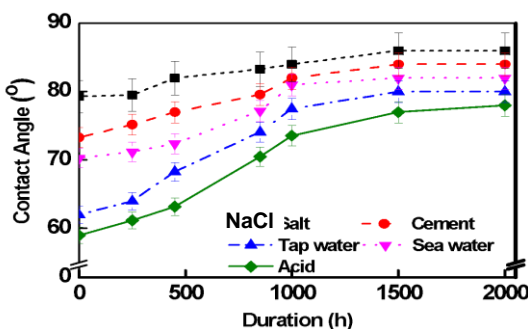

Fig. 2. Variation in CA for GE with duration of exposure to ambient conditions after 1000 h exposure to NaCl, cement, tap water, sea water and acid

The base material remains hydrophilic even after 2000 h of exposure to ambient conditions though it shows marginal recovery from 58 to 78 in case of acid contamination and to a maximum of 80 to 86°in case of sample with cement contamination. Thus, influence of cement on CA of base material is marginal and is closer to the lower end of hydrophobicity. After contamination of 1000 h it is observed that the recovery is better in the first 800 h and there after only a marginal improvement beyond 800 h is observed. Ultimately, it is observed that the contact angle values converge towards an average value of 73 to 83°.

c. Recovery of CA of GESAA

The results of variation of CA of GESAA during 2000 h of exposure to ambient conditions after 1000 h conditioning with NaCl, sea water, cement, tap water and acid are shown in Fig.3. Interestingly after 2000 h of conditions under ambient conditions GESAA regains its hydrophobic nature

though the values are marginally lower (by 10%) than the virgin material. At the end of 2000 h of exposure to ambient condition GESAA is able to reach a CA level of 93 with cement, tap water and salt contaminants which is closer to the lower end of hydrophobicity. However, in the case of sea water and acid, the CA falls slightly below 90°. In this case, beyond 1500 h of exposure, the contact angle converges to an average value of 85°. Thus, exposure of GESAA to ambient conditions helps in recovery of contact angle to a level lower than the range of hydrophobicity range.
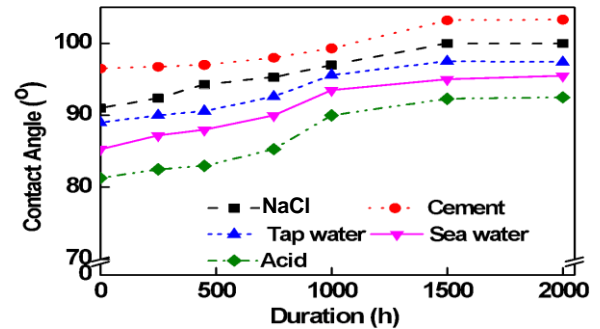

Fig. 3. Variation in CA for GESAA with duration of exposure to ambient conditions after 1000 h exposure to NaCl, cement, tap p water, sea water and acid.

d. Recovery of CA of GESAC

The data of recovery of CA of GESAC is shown in Fig. 4. In GESAC, significant recovery of CA is observed during the first 500 h of exposure and the composite regains the hydrophobic nature. During 250 h of exposure to ambient conditions significant recovery of CA is seen in case of NaCl, tap water, and acid. However, in the case of cement, the average value of CA is around 107 with a variation of ±3 during the 1000 h period. After 2000 h of conditions under ambient conditions, the recorded values of CA average to 105°.
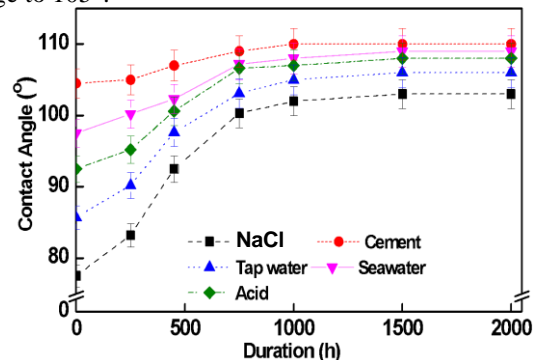

Fig. 4. Variation in CA for GESAC with duration of exposure to ambient conditions after 1000 h exposure to NaCl, cement, tap water, sea water and acid.

e. Recovery of CA of GESAM

The variation in CA for GESAM with duration of exposure to ambient conditions for 2000 h after immersion in NaCl, cement, tap water, sea water and acid is shown in Fig 5. There is consistent recovery of CA during first 1000 h of exposure and beyond this point; the rate of recovery of CA is reduced. After 1000 h of exposure to ambient condition, the CA is observed to settle around 110 ° in case of NaCl, cement and acid. This is in contrary to previous cases, in which recovery was observed only during the first 250 h of exposure. GESAC is observed to regain its hydrophobicity in a better manner up to 800 h and the average value of CA of GESAM material is 108°.
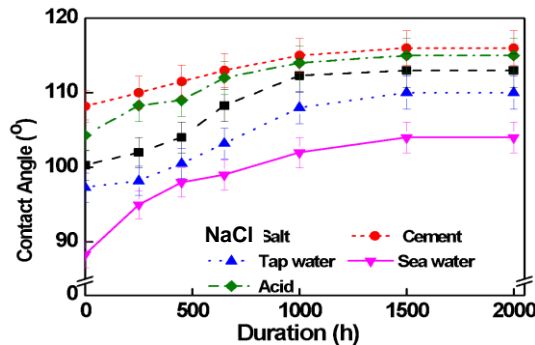

Fig. 5. Variation in CA for GESAM with duration of exposure to ambient conditions after 1000 h exposure to NaCl, cement, tap water, sea water and acid

Silicone has remarkable ability to recover hydrophobicity under ambient conditions. It recovers hydrophobicity completely in case of NaCl at higher salinities. GESAA exhibits good ability to recover hydrophobicity with cement, NaCl, tap water, sea water and acid. GESAC shows improvement over GESAA in its ability to recover hydrophobicity irrespective of the contaminants. GESAM also shows comparatively better ability to recover hydrophobicity as compared to GESAC. However, after 1000 h of exposure to ambient conditions, there is total recovery of hydrophobicity. There is a considerable reduction in hydrophobicity with NaCl, in all GE based material.

C. Water Diffusion Dynamics

Water diffusion dynamics in reinforced epoxy is determined by three different mechanisms [16], namely (i) diffusion through the micro gaps of the polymer chains, (ii) capillary transport through the gaps present at the interfaces of the fillers and the matrix and (iii) the transport through the micro-cracks, if present. Diffusion is broadly classified into Fickian, non-Fickian or anomalous diffusion, and a case in between the Fickian and non- Fickian. Some authors have used the Langmuir diffusion model to explain diffusion of water into silicone [19] and which is classified under anomalous diffusion which accounts in a better way for the presence of bound water molecules.

Fickian diffusion is characterized by a slow rate of diffusion of the liquid. This is due to mechanical, structural and similar modes of penetrant–polymer system interactions. Non-Fickian type of diffusion occurs faster than the segmental relaxation processes. The long-duration drift in absorption kinetics of polymers is attributed to the mass uptake of water, which is controlled by the visco-elastic relaxation of the polymer chains which help to accommodate the penetrant [20].

It is observed from water absorption measurements that GE, GESAA, GESAC and GESAM follow the Fickian diffusion to the extent of 70, 72, 77 and 90% and it goes further higher in silicone. Epoxy absorbs water more than the glass fibers at a slightly higher rate, and the absorbed water molecules bond either with the amine or hydroxyl groups. The unbonded or the free water molecules cluster around the free volume inside the epoxy [2]. The reduction in CA during the initial period of immersion (< 200 h) in contaminants demonstrates the time dependence and the role of micro structure in the diffusion mechanisms. The recovery of CA is also observed to depend on the nature of the contaminant and the microstructure which in turn dependence on the size, shape and placement of the fillers in the epoxy matrix. Incorporation of two nano and one micro filler leads to micro structural changes and the pathway for transport of water inside the bulk is made difficult as a consequence of the tortuous pathway.

IV. CONCLUSION

Some of the important conclusions drawn from the investigations are:

1. Silicone retains its hydrophobicity with different contaminants.
2. The time dependence of CA variations on immersion in contaminants is attributed to the involvement of different diffusion mechanisms.
3. In case of the epoxy composites, the fillers help to enhance the contact angle and impart different surface wetting properties under contaminations.
4. The use of nano and micro filler combination in the GE matrix leads to considerable improvement in the initial CA and retention of hydrophobicity.
5. Calcium carbonate and Magnesium oxide filers hold promise when combined with alumina and silica.

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