International Journal of Scientific Research & Engineering Trends
Volume 6, Issue 4, July-Aug-2020, ISSN (Online): 2395-566X

Investigation the Impact of Inorganic Fillers on the Electrical and Physical Properties of Resin Epoxy

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Abstract – Epoxy micro composites with high loading of micro (silicon dioxide, magnesium hydroxide and mica) were fabricated and their electrical and physical properties were characterized .The main aim of this research is to enhance the performance of resin epoxy as an insulation materials. The micro and nano silicon dioxide are used as inorganic fillers to support the resin epoxy. The flashover voltage test was carried out to investigate the difference between the two fillers. The contact angle measurement was carried out to investigate the hydrophobicity among three types of micro fillers (silicon dioxide, magnesium hydroxide and mica).

Keywords–epoxy, nano filler, silicon dioxide, flashover voltage, contact angle.

I. INTRODUCTION

Epoxy resin is a thermosetting polymeric material. This material has several advantages, i.e. high dielectric strength, light weight, easy to blend with additive, and easy maintenance if compared to that of porcelain and glass outdoor insulators which are traditionally used. All these merits qualify epoxy to be very good as high voltage insulators in electrical power system. However, this material also has several disadvantages, i.e. hydrophilic property, very sensitive to aging and easily degraded when there is a flow of contaminants on its surface. Despite many merits of epoxy resin, the modications of epoxy resin are very vital to prolong the service life and enhancing the operating reliability of high voltage insulators[1]. The priority for improving the properties of epoxy also is due to their exposure to different climatic conditions and industrial pollutants. Our studies show that nano composites with silicon dioxide have superior improvement over micro silicon dioxide in enhancing the electrical performances under various conditions. Also the addition of inorganic micro particles such as silicon dioxide, magnesium hydroxide and mica into epoxy matrix improves the electrical and physical properties.

II. EXPERIMENTAL DETAILS

1. Material Specimen

Epoxy is a thermosetting chemical compound. It consists of oxygen and carbon atomic bond which is generated by chemical reaction between epichlorohydrin and bisphenol A .Specimens were fabricated as cylindrical rods having 1cm diameter and different lengths varied 5mm to 25mm.Then finally the filler with the required amount ha been added to the blend.

The filler is added to the resin /hardener mixture which is again stirred slowly till the particles have been uniformly mixed with the resin under magnetic field and the air bubbles have vanished. The mixture is then poured carefully into a mold and left at room temperature (25oC±1 oC) until curing after 8 days for material stability. The mixture of epoxy composite takes the mold shape. There are different percentages of each filler 30%,50% and 50% for hydroxide magnesium Mg (OH)2 ,silicon dioxide SiO2 and mica respectively in micro size and 7% by weight for SiO2 in nano size as shown in table1 and table 2.

Table 1. Abbreviation for epoxy micro composite samples with various lengths.

Length (mm)	Resin epoxy	$Mg(OH)_2$ 30%	SiO_2 50%	mica 50%
5	$E_{R/5}$	$E_{Mg/5}$	$E_{Si/5}$	$E_{M/5}$
10	$E_{R/10}$	$E_{Mg/10}$	$E_{Si/10}$	$E_{M/10}$
15	$E_{R/15}$	$E_{Mg/15}$	$E_{Si/15}$	$E_{M/15}$
20	$E_{R/20}$	$E_{Mg/20}$	$E_{Si/20}$	$E_{M/20}$
25	$E_{R/25}$	$E_{Mg/25}$	$E_{Si/25}$	$E_{M/25}$

Table 2. Abbreviation for epoxy nano composite samples with various lengths.

Length (mm)	SiO_2(7gm)
5	$E_{SiN/5}$
10	$E_{SiN/10}$
15	$E_{SiN/15}$
20	$E_{SiN/20}$
25	$E_{SiN/25}$

The mixture between epoxy and micro filler is prepared in lab using mixer at high speed to get micro composite while nano composite is manufactured by mixing at high speed and facing to magnetic or ultrasonic at the same time of mixing. Nature and size of filler affect physical, the mechanical and electrical properties of epoxy composites. Recently, various types of multi-functional composites with nano particles have been introduced in advanced materials science. When the size of materials decreases to the nanometer scale, the ratio of its surface area to volume critically increases[2,3].

2. Testing Samples

Samples are manufactured with dimension according to the requirements of the electrical and physical tests .The samples were cut and prepared with dimensions that best suited each testing method according to ASTM. The dimensions of the samples were limited due to the limitations of the capabilities of AC supplies available in the high voltage laboratory. Samples were prepared by using hand tools. The resulting samples for pure resin, micro and nano fillers are presented in figures 1-3.

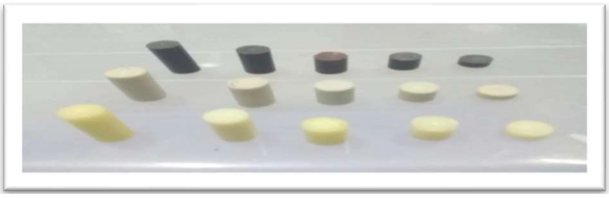

Fig. 1. Samples of epoxy with micro inorganic fillers.

The fillers are 30 % hydroxide magnesium Mg(OH)2 (middle samples), 50 % silicon dioxide SiO2(front samples) and 50% mica (back samples).The samples are of cylindrical shape with diameter 1cm and length 25mm (left) to 5 mm (right) as shown in figure(1).

Fig. 2. Samples of resin epoxy .

The resin samples are of cylindrical shape with diameter1cm and length 5mm(left) to25mm(right) as shown in figure(2).

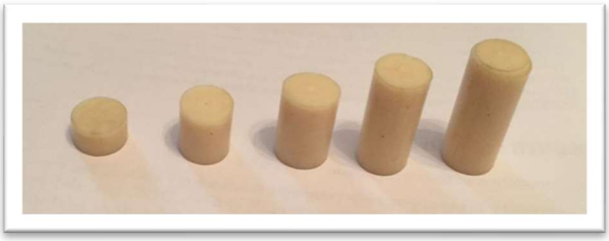

Fig. 3. Samples of resin epoxy+7%nano SiO2.

The resin epoxy+7%nano SiO2 samples are of cylindrical shape with diameter 1cm and length 5mm (left) to 25 mm (right) as shown in figure(3).

The sample will be a practical polymer insulator in the flashover test. Each sample has been tested several times to ensure the results. The study of industrial pollution for different types of insulators reflects that the electrical and physical performance of polluted insulators decreases with increasing the degree of pollution that depends on the forms and materials of insulators. The various testing conditions are classified as follows:

- The first set of the samples have been tested using AC voltage under dry condition.
- The second set of samples has been tested using AC voltage under wet condition.
- The third set of samples was immersed in sodium chloride condition to simulate the coastal areas.
- The fourth set of samples was immersed in Naphtha condition to simulate the hydrocarbon solvents from exhaust fumes of vehicles near railway lines.
- The fifth set of samples was immersed in Hydrochloric acid condition as Hydrochloric acid (hCl) is listed as a Title III Hazardous Air Pollutant.

Hydrochloric acid is released from various sources. Hydrochloric acid is one of the emissions of incineration plants, CFC(Chlorofluorocarbon) atmosphere as well as in the cross-emissions.

3. Types of Tests

3.1 Flashover test

Flashover can be defined as an unintended disruptive electric discharge over or around the insulator, connecting those parts, which normally have the operating voltage between them according to ASTM D-2303-64T. Flashover voltage is an external disturbance on insulator surface or fire arcing process on insulator surface. It may occur in solid surface or gas. Flashover voltage value is smaller than puncture voltage on an insulator. Several factor affect flashover voltage, such as: surface resistance of a material, surface condition and electric field shape between electrodes and insulator[4]. The previous flashover tests in our study [5] were concluded that the three types of micro inorganic fillers is that the silicon dioxide is the best filler among them. Therefore, the following nano test will be taken from that filler and mixed with raw epoxy, and then the flashover voltage will be measured.

3.2 Contact angle measurements

The hydrophobicity on surface of insulator material in outdoor is very important. In humid and the rainy conditions, the insulator surface must not easily wet, so that the surface conductivity of insulators remain low, thus the surface leakage current is very small. Therefore the polymeric insulator must be hydrophobic and withstand against environmental condition[6].

Four samples of cylindrical shape are measured by placing water of volume 10µL on the surfaces with an Eppendorf reference 10µL syringe.The contact angle can be calculated by dripping water on the surface of insulator material and observing its ability to form water droplets.(Swedish Transmission Research Institute, STRI Guide 1,92/1, Hydrophobicity Classification Guide).

Fig. 4. Overview of the four cylindrical samples.

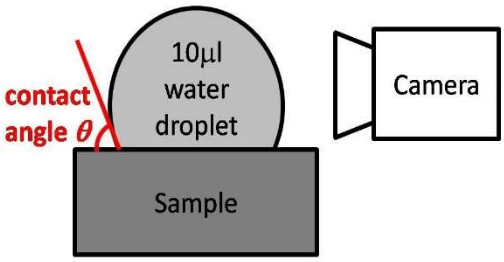

Fig. 5. Schematic draw of the contact angle method.

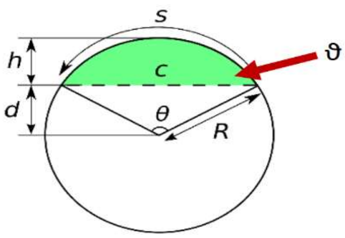

Fig. 6. The dimensions of the water droplet.

$\sin \vartheta = \sin(\ /2) = 4Ch/(C^2+4h^2)$

III. FIGURES AND TABLES

1. Flashover Voltage Results :

1.1. Studying the flashover voltage for epoxy composite Samples loaded with 7% of nano SiO2 fillers at dry condition:

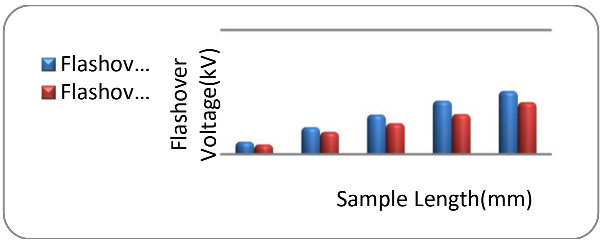

Fig. 7. Flashover voltage of the nano composites 7% SiO2 and the resin epoxy versus the sample length under dry condition.

The results of the above test for dry condition and the effect on flashover voltage are shown in Fig.2, the following can be observed:

- The dry condition is the perfect state which is the criterion for the other polluted conditions.
- In all the specimens the flashover voltage of the composites insulators increases with sample length.
- The maximum flashover voltage for the nano ESiN/25 is 51kV, although the maximum flashover voltage for the ER/25 is 41.97kV.
- At all lengths, the nano scale achieves the best results in flashover voltage values compared to the resin.
- The rate of increase at the nano scale is about 21.5% and this rate of improving increases by increasing the sample length.
- The nano SiO2 improves the flashover voltage value of epoxy nanano composite.

1.2. Studying the flashover voltage for epoxy composite Samples loaded with 7% of nano SiO2 fillers at wet condition:

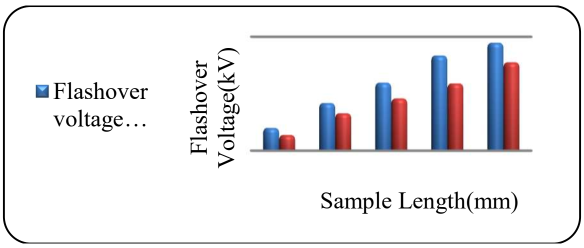

Fig. 8. Flashover voltage of the nano composites 7% SiO2 and the resin epoxy versus the sample length under wet condition.

The following can be concluded:

- This test is extremely necessary to simulate the damp environmental conditions that insulators are often exposed to. It represents the nature of rain, dew, fog, and humidity in general.

- In all the specimens the flashover voltage of the composites insulators increases with sample length.
- The maximum flashover voltage for the nano ESiN/25 is 47.39 kV, although the maximum flashover voltage for the ER/25 is 38.77kV.
- At all lengths, the nano scale achieves the best results in flashover voltage values compared to the resin.
- The rate of increase at the nano scale is about 22%.
- The nano SiO2 still resists the moisture effect and improves the performance of the epoxy.

1.3. Studying the flashover voltage for epoxy composite samples loaded with 7% of nano SiO2 fillers at salt wet condition:

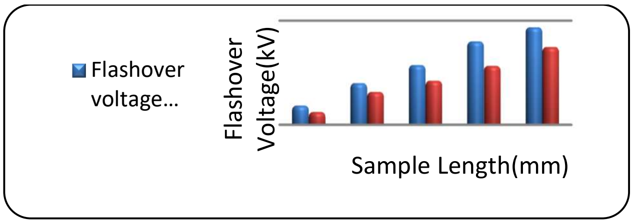

Fig. 9. Flashover voltage of the nano composites 7% SiO2 and the resin epoxy versus the sample length under salt wet condition.

From Fig.4. the following could be noticed:

- The goal of this test is to simulate the coastal environment.
- In all the specimens the flashover voltage of the composites insulators increases with sample length.
- The maximum flashover voltage for the nano ESiN/25 is 46.85 kV, although the maximum flashover voltage for the ER/25 is 37.38kV.
- At all lengths, the nano scale achieves the best results in flashover voltage values compared to the resin.
- The rate of increase due to the nano silicon is about 25%.
- The nano SiO2 still resists the salinity effect and improves the performance of the epoxy.

1.4. Studying the flashover voltage for epoxy composite samples loaded with 7% of nano SiO2 fillers at Naphtha condition:

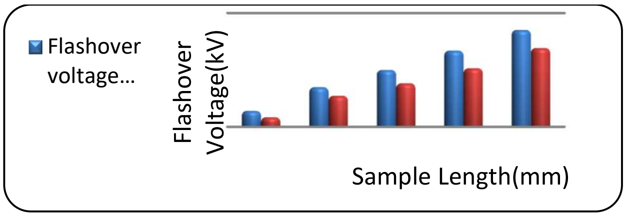

Fig. 10. Flashover voltage of the nano composites 7% SiO2 and the resin epoxy versus the sample length at Naphtha condition.

The following could be remarked from Fig.5:-

- There is a noticeable reduction in flashover voltage for resin epoxy due to the high level of Naphtha pollution subjected to the samples.
- This kind of pollution appears through vehicle exhaust fumes and near railroad tracks.
- silicon dioxide improves markedly the electrical properties of resin epoxy and resists the negative impact of this pollution.
- The maximum flashover voltage for the nano ESiN/25 is 42.60 although the maximum flashover voltage for the ER/25 is 34.65kV.
- The rate of increase due to the nano silicon is about 23%.

1.5. Studying the flashover voltage for epoxy composite samples loaded with 7% of nano SiO2 fillers at HCL condition:

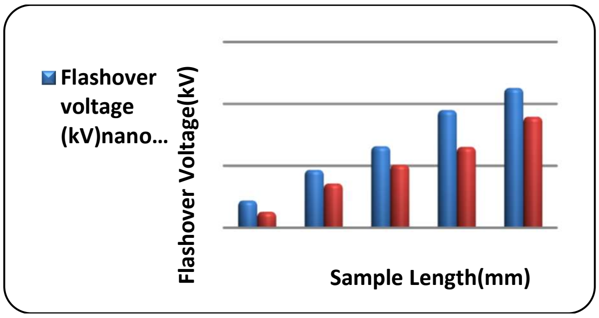

Fig. 11. Flashover voltage of the nano composites 7% SiO2 and the resin epoxy versus the sample length at HCL condition.

The following can be drawn from Fig.6:

- The diffusion of acidic electrolyte into thermo set (epoxy) matrix leads to non equilibrium swelling behavior .This behavior leads to physical erosion and the a corresponding degradation in material properties[7].
- If moisture finds its way to acid crystals or residual hardener on or near the surface of the insulator, it will act as a carrier for the acid allowing it to seep into cracks and reach the surface of the fibers, Thereby starting the brittle fracture process[8].
- Clearly silicon dioxide upgrades the electrical properties of resin epoxy and fights the negative impact of this pollution.
- The maximum flashover voltage for the nano ESiN/25 is 45.12 kV although the maximum flashover voltage for the ER/25 is 35.84kV.
- The rate of increase due to the nano silicon is about 26%.

2. Contact angle Results:

These experimental measurements are done for the resin epoxy sample and the composite samples after flashover exposure, so the samples had affected by the electrical sparks and the passed leakage current through it. Due to

International Journal of Scientific Research & Engineering Trends
Volume 6, Issue 4, July-Aug-2020, ISSN (Online): 2395-566X

the flashover test, there are cracks and simple holes on the samples surface. The samples are cylindrical rods, so they have three types of surfaces, top flat surface, bottom flat surface and the curved surface, varying in dimensions and in texture. Mostly, the average value of the measuring angle for the top and bottom surfaces can be calculated, as in many times the water droplet slides down the curved surface.

1.1. Contact angle of resin samples:

By substituting in the above equation:

First flat surface:
$\sin(\vartheta) = \sin(\theta/2) = 4\times858\times150/(858^2 + 4\times150^2) = 0.62$
Second flat surface:
$\sin(\vartheta) = \sin(\theta/2) = 4\times898\times114/(898^2+4\times114^2) = 0.48$

Fig. 12. Side view of water droplet on the resin surface after flashover test.

The contact angle on the flat surfaces is between: :
$38o \pm 5o$ and $28o \pm 5o$.

1.2. Contact angle of epoxy-hydroxide magnesium composites samples:

The flat surface:
$\sin\vartheta = \sin(\theta/2) = 4\times425\times145 /(425^2+4\times145^2)= 0.93$
The first rough surface had a contact angle of 68o ± 5o.
Due to crack and roughness, the drop spread over a large part of the second flat surface

Fig. 13. Side view of water droplet on the hydroxide magnesium composits surface after flashover test.

1.3. Contact Angle Of Epoxy-Silicon Dioxide Composites Samples:

$\sin\vartheta = \sin(\theta/2) = 4\times533\times168 /(533^2 + 4\times168^2)= 0.9$
The contact angle on the rough surface is $64o \pm 5o$.

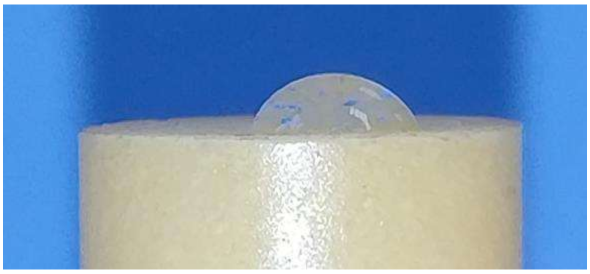

Fig. 14. Side view of water droplet on the mica composites surface after flashover test.

1.4. Contact Angle Of Epoxy-Mica Composites Samples

$\sin\vartheta = \sin(\theta/2) = 4\times467\times162 /(467^2 + 4\times162^2)= 0.94$
$\sin\vartheta = \sin(\theta/2) = 4\times425\times153/(425^2 + 4\times153^2)= 0.95$
The contact angle on the rough surfaces is between:
70 o ± 5 o and 72 o ± 5o.

Fig. 15. Side view of water droplet on the mica composites surface after flashover test.

IV. DISCUSSION AND RESULTS

1. Discussion the results of the flashover Voltage with 7% nano SiO2 fillers, under different environmental conditions.

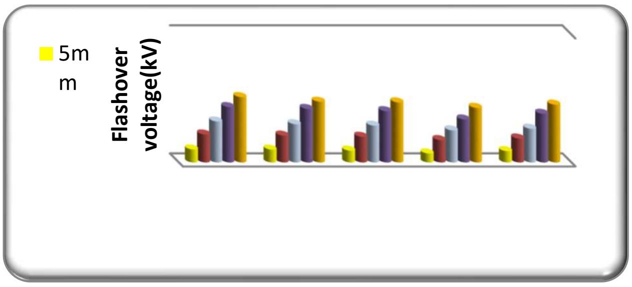

Fig. 16. Flashover voltage versus the nano composites 7% SiO2 under various environmental conditions.

The following can be emphasized:-

- In all the specimens the flashover voltage of the composites insulators increases with sample length, as the resistivity proportional to the length.

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Volume 6, Issue 4, July-Aug-2020, ISSN (Online): 2395-566X

- Type of reagents and environmental conditions surrounding the insulators influence on the flashover voltage as shown in Fig.4.16.
- The dry condition is the perfect state which is the criterion for the other polluted conditions.
- There is a drop in the values of the flashover voltage around 7% due to the wet condition compared to the dry condition.
- There is deterioration in the values of the flashover voltage around 8% due to the salt wet condition compared to the dry condition.
- There is a marked decrease in the values of the flashover voltage around 17% due to the Naphtha condition compared to the dry condition
- The flashover voltage values also decay when the samples are subjected to the hydrochloric acid pollution. This reduction is around 12% compared to the dry condition.

2. An Analytical Discussion of the difference between Micro silicon Dioxide and Nano silicon Dioxide on the Flashover Voltage for epoxy

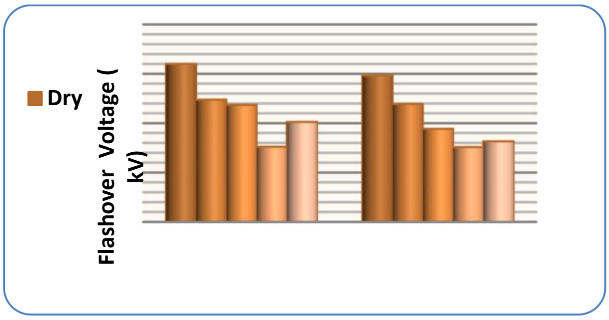

Fig. 17. the overall effect of the nano size and the micro size of silicon dioxide particles as inorganic filler on the resin epoxy.

It's clear from this figure the following:-
- The added filler changes the physical properties of insulating material.
- Micro and Nano composites on epoxy basis with SiO2 filler were successfully fabricated.
- Fillers are used to improve such the electrical and physical properties of epoxy and to reduce their cost.
- Epoxy nano composite systems with inorganic silicon dioxide fillers display some advantageous in electrical behaviors at low nano filler loadings. The permittivity and tan delta values in the nano composites are found to be lower than that of micro composites as well as unfilled systems (for few filler loadings)[9].
- Nano composite Insulator has smooth surface while micro composite insulator has roughness surface. If the surface insulator is roughness or depredated, the flashover voltage decreases in flashover test for micro composite insulator comparing with nano composite insulator at the same concentration [10].
- The surface state is very important in surface resistance and essential factor in surface leakage current. The leakage current depends on the field, ambient relative humidity and degree of contamination[11].
- SiO2 composite insulator which contains only 7% from silicon dioxide in nano size has a high electrical performance against different contamination comparing with micro composite insulators which contains 50% filler .
- Nano scale particle manufacturing has become standard. The reason for this is a measure of their length that can be compared to a scale of polymer molecules and the high specific area of particle surfaces within a compound. Nano particles have proven new properties as fillers. Recently, the scientific research incorporating various nano particles in dielectric systems present in a cost-effective way the confirmation of the improved benefits of these systems over conventional padding systems[12].
- Silica sand or quartz sand is excavated materials composed of crystals – crystalline silica (SiO2) and contains a compound impurities carried during the deposition process. Silica is an insulating material that is often used in ceramics. Combination of silicon with oxygen to form crystal formation depends on the temperature of the called silica. Packaging atoms enclosed in the bonding of silicon and oxygen, making the structure tends to be stable in general does not decline to environmental changes. This means that the insulator is not easily damaged by ultraviolet, electrical treatment such as providing a voltage gradient on the insulator and environmental conditions such as temperature, humidity and so forth. Insulator will be more resistant to aging[13].

3. The analysis of the Contact Angle Results:

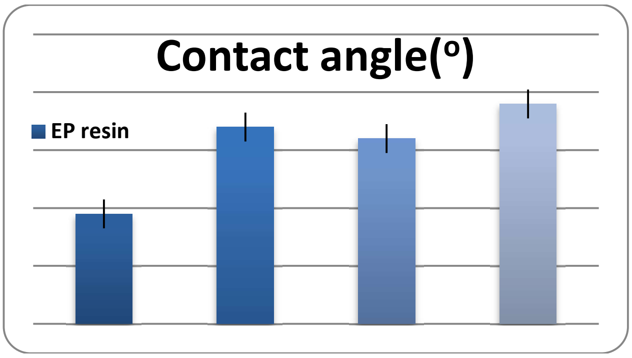

Fig. 18. The effect of filler type on the contact angle after flashover test.

- Comparing the contact angle of pure epoxy resin with epoxy micro composite, it is observed that epoxy micro composites has high contact angle which indicates to increase in hydrophobicity of the material.
- The molecular structure of epoxy contains many atoms of oxygen.

- SiO_2 contains also many atoms of oxygen.
- The existence of these oxygen atoms in the resin epoxy and silicon dioxide composite tend to result in the hydrophilicity on the surface of insulator materials. If there is H_2O molecule on the surface, O atoms released from the bond will react to form hydroxyl group (OH). Oxygen atoms tend to be oriented to the surface of material which causes the balance of molecular forces change, thus increase of the surface energy, resulting in the decrease of contact angle [14].
- This improvement , due to the addition of inorganic fillers , in the contact angle ranges between 41% to 50% over the resin sample.

V. CONCLUSION

- The type and percentage of filler have pronounced effects on the electrical and physical performance of epoxy composite insulators.
- In all the specimens the flashover voltage of the composites insulators increases with sample length.
- Fillers are used to improve such the electrical and physical properties of epoxy, also for economic issues.
- Incorporation of SiO_2 in micro and nano size modifies the electrical properties of epoxy composites.
- Flashover voltage is strongly dependent on the types of contamination and environmental conditions: the Naphtha is considered as the most polluted condition.
- The hydrophobicity was influenced by the molecular structure of the compound.
- Comparing the contact angle of pure epoxy resin with epoxy micro composite, it is observed that epoxy micro composites has high contact angle which indicates to increase in hydrophobicity of the material.

Acknowledgment
Sincere thanks are extended to the staff of High Voltage Laboratory in at Aswan Faculty of Engineering and Physical Laboratory at German University in Cairo (GUC)where the experimental work were carried out. Also thanks for Polymers & Pigments Department where the samples were prepared.

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