Determination of Hydrophobic Contact Angle of Epoxy Resin Compound Silicon Rubber and Silica

Abdul SYAKUR¹, Hermawan², Heri SUTANTO³

¹,² Department of Electrical Engineering, Universitas Diponegoro
³ Department of Physics, Faculty of Science and Mathematics, Universitas Diponegoro, Jln. Prof. Soedarto SH Tembalang, Semarang, INDONESIA

Email : gakusei2003@yahoo.com

Abstract. Epoxy resin is a thermosetting polymeric material which is very good for application of high voltage outdoor insulator in electrical power system. This material has several advantages, i.e. high dielectric strength, light weight, high mechanical strength, easy to blend with additive, and easy maintenance if compared to that of porcelain and glass outdoor insulators which are commonly used. 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. The research towards improving the performance of epoxy resin insulation materials were carried out to obtain epoxy resin insulating material with high water repellent properties and high surface tracking to aging. In this work, insulating material was made at room temperature vulcanization, with material composition: Diglycidyl Ether Bisphenol A (DGEBA), Metaphenylene Diamine (MPDA) as hardener with stoichiometric value of unity, and nano silica mixed with Silicon Rubber (SiR) with 10% (RTV 21), 20% (RTV 22), 30% (RTV 23), 40% (RTV 24) and 50% (RTV 25) variation. The usage of nanosilica and Silicon Rubber (SiR) as filler was expected to provide hydrophobic properties and was able to increase the value of surface tracking of materials. The performance of the insulator observed were contact angle of hydrophobic surface materials. Tests carried out using Inclined Plane Tracking procedure according to IEC 60-587: 1984 with Ammonium Chloride (NH₄Cl) as contaminants flowed using peristaltic pumps. The results show that hydrophobic contact angle can be determined from each sample, and RTV 25 has maximum contact angle among others.

1. Introduction

Epoxy resin uses as an insulating material in electric power systems is still under study. The selection of epoxy resin with the base material is diglycidyl ether of bisphenol A (DGEBA), and meta phenylene diamine ripening agent as alternative isolator made by polimere insulation because it has high dielectric power (E), that is 24-25 MV/m, 10⁻¹³⁻¹⁰⁻¹⁵ Ω.m volume resistance (ρ), 50/60 Hz frequency of dielectric constant (ε), 3.5-3.9 dan low power dissipation that is (35-90) x 10⁴. To increase epoxy resin performance to be resistant to the environmental conditions, it is be added the polydimethylsiloxane which has type is silicon rubber. Properties that polysiloxan have are high power dielectric at 20-30 MV/m, dielectric constant (ε) has frequency at 50/60 Hz, 2.5-3.2 and low power dissipation (tan δ) that is (4-25) x 10⁻⁴ and could increases the performance of water denied [1].
The addition of silicone rubber in large amounts can increase the hydrophobic properties of the surface, but the mechanical strength is reduced. To improve the mechanical strength of the epoxy resin material coupled with the filler material is silica sand (SiO2) in the hope of epoxy resin insulating material has a high mechanical strength is tensile strength and compressive strength [2]. If the silica sand is added the more the mechanical strength increases, but on the other hand will reduce the surface hydrophobic properties. Therefore, it is necessary to do a study to obtain a filler material composition of silicone rubber and silica mixture right on epoxy resin material that will be used as a high voltage insulator material. To determine the performance of surface material made of epoxy resin filler mixture of silicon rubber and silica under severe pollution, then tested the electrical tracking refers to the Inclined Plane Tracking test methods according to standards IEC 587, with duration of voltage application for 6, 12 and 18 minutes. Research on epoxy resin material performance under severe conditions has also been studied by [3] using natural and artificial aging in the laboratory for 96 hours with ultraviolet light. In this report, hydrophobic contact angle will be discussed before and after surface tracking test.

2. Fundamental Theory

2.1 Characteristics of Solid Polymer Insulation

The electron will have the electric pressure when the solid polymer insulation applied in E electric field. This electrical stress is a force that works on the electron, and raises a work. Due to the force, the electron will moves as far as dx and need an effort by W. If a different potential between two point, for example point A and point B on the solid polymer insulation material, is VAB, amount of effort required to move an electron from point A to point B has formulated by [4],

$$\int_{B}^{A} Edx = -V_{AB} \quad (2.1)$$

Then, the value of electric field strength or electric pressure is

$$E = -\frac{\partial V_{AB}}{\partial x} \quad (2.2)$$

2.2 Epoxy Resin Insulating Materials

Epoxy resin is a plastic which including thermoset category, but it is hard as phenolic and polyester. Epoxy resin offers the incredible function, low shrinkage rate, good chemical resistant and good sticking power.

The strength of epoxy resin is located on the hardness, resistance to friction and chemical resistance. Solid resin at medium molecular weight can be combined with similar polymerization or non-similar polymerization through the epoxy group. The property of epoxy resin adhesive which is perfect, malleable, good mechanical strength and strong chemical resistance are the most important benefits from the solid resin [5]. Surface degradation caused by a reaction which causes broken chain of the main molecular bond and the change of polymer base material happens. Surface degradation of polymer insulation material as insulator material on electricity power system, caused by high pollutant which came from the water sea pollution around the beach which contains salt, industrial pollution is chemical pollution because of the soot, industrial fog; desert contains sand and dust, volcanic dust pollution, and cement factory’s dust pollution. Pollutant which sticks on polymer insulation material as high voltage insulator material, influenced by the electric field and tropical climate, those are temperature, humidity, air pressure, wind velocity, rain and ultraviolet ray, causes aging of insulation, that is aging of physically aging, chemical, electrical, and combination of mechanical and electrical [6]

2.3 Metaphenylene Diamine (MPDA)

Ripening of the epoxy resin can be divide into three main groups, there are: Hydroxyl group (R-OH), Amine group and Acid Anhydride group. Each group has so many types. Every type has different
level on the mixture process. Ripening reaction occurs through the epoxy groups from base material and reactive groups from reinforcement material. Reaction between base-material of epoxy resin with ripening agent of acid anhydride a group is a more complex reaction than the ripening agent of amine group. The test uses ripening agent from amine group that is Metaphenylene-diamine (MPDA) because MPDA included the most chemical that is used as ripening agent of epoxy resin [5].

2.4 Silicon Rubber (SiR)
Silicon rubber is a material resistant to high temperature; usually it is used to cable insulation and high voltage insulator material. Physical property of this thing can be improved by mix the filler material, is like silica sand. Silicon rubber is safe to use at -55°C – 200°C temperature. It has good barrier to the ozone, corona, and water. This has well resistant against the alcohol, salt and oil. Silicon rubber ([(CH₃)₂SiO]n) is formed by cyclic siloxane monomers that created polydimethylsiloxane. The hydrophobic property of it gives a high surface obstacle in the humid condition and contaminated. It decreases the leakage current. Uncontrolled leakage current can increase the intensity of spark in dry condition, when the crack occurred, and erosion (flashover) in the polymer material. Silicon rubber has very good insulation property, there are: has tan δ = 3-30.10⁻³, dielectric constant ε = 2-4, resistivity ρ = 1015 Ωm (without filler material), resist to light (metil group absorbs light in λ > 300 nm area) and it can be stabled until the temperature is 250°C by resists the elasticity in the low temperature because PDMS has glass transition temperature around -120°C (long thermal stability).

2.5 SiliconDioxide (SiO₂)
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 sand or quartz having a combined composition of SiO₂, Fe₂O₃, Al₂O₃, TiO₂, CaO, MgO and K₂O translucent white or other colours depending on the compound impurities. Combination of silicon with oxygen to form crystal formation depends on the temperature of the called silica. Silica is an insulating material that is often used in ceramics. 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.

3. Experimental Set Up

3.1 Materials
Materials composition used in this research are Diglycidyl Ether Bisphenol A (DGEBA), Metaphenylene Diamine (MPDA) as hardener with stoichiometric value of unity, and nano-silica mixed with Silicon Rubber (SiR) with 10% (RTV21), 20% (RTV22), 30% (RTV23), 40% (RTV24) and 50% (RTV25) variation.

| Test sample code | DGEBA (% pbw) | MPDA (% pbw) | Filler (% pbw) |
|------------------|---------------|---------------|----------------|
|                  |               |               | Silicone Rubber | Silica sand | Filler Total |
| RTV₂₁            | 45            | 45            | 5              | 5           | 10           |
| RTV₂₂            | 40            | 40            | 10             | 10          | 20           |
| RTV₂₃            | 35            | 35            | 15             | 15          | 30           |
| RTV₂₄            | 30            | 30            | 20             | 20          | 40           |
| RTV₂₅            | 25            | 25            | 25             | 25          | 50           |
3.2 Contact Angle Measurement

Equipment for research performance hydrophobic contact angle include: conductivity meter, pipette, lights, cameras, and computers as shown at Figure 1.

![Figure 1 Equipment for surface contact angle measurements](image)

3.3 Leakage Current Measurement

Equipment for surface leakage current performance study, include: transformer test with 220 Volt input voltage and output 50 kV with a capacity of 5 kVA, table controls to adjust the applied voltage, high voltage probes, voltmeter, voltage divider, arrester, oscilloscope, peristaltic pumps, hoses, measuring cups, digital camera, flash and computer as shown at Figure 2.

![Figure 2 Equipment for measuring of leakage current](image)

4. Results and Discussion

Contact angle measurements aims to determine the surface properties of materials, water-repellent (hydrophobic) or absorb water (hydrophilic). Hydrophobicity of the surface of the test material is influenced by the type of material and the composition of the filler constituent [7,8]. The hydrophobic properties can be determined by measuring the contact angle between the tests materials with distilled water dripped on the material surface. Value of surface contact angle of the droplets of liquid material obtained by direct observation through the digital camera scene which is then stored on the computer. The photographs processed using software to obtain the contact angle on the right side and the left side of the test sample is measured. Epoxy resin material contact angle increased after the addition of filler material silicon rubber and silica. Surface hydrophobicity properties of the epoxy resin material
increases with silicone rubber. This happens because the more a methyl group (CH3) on the hydrophobic silicon rubber. Methyl group is derived from the polysiloxane [3]

**Figure 3** Correlation graph for contact angle of epoxy resin

Surface hydrophobicity of epoxy resin composition is influenced by the chemical structure of polysiloxane more dominated by the polydimethyl functional group (n [CH3]2) are hydrophobic. The presence of methyl groups CH3 rotating freely and Si-O bond polarizability causing siloxane chains are able to straighten them and produce on their surface hydrophobicity.

**Figure 4** Hydrophobic properties of freely rotating methyl group

Figure 4 shows that Polydimethylsiloxane molecules randomly shaped zigzag. Chain configuration can curl because the valence angle is relatively large silicon (Si-O-Si ~ 143o and 109o ~ O-Si) and the radius of the elements Si = 1.46 Angstroms larger than the carbon element radius 0.91 Angstroms so that the distance between the axis of methyl compound Si-O-Si greater. If the configuration chain structure curl then reoriented methyl group (CH3) and siloxane bond in opposite
directions. When a methyl group oriented to the surface, then the surface will be hydrophobic [9]. The increasing the contact angle of an epoxy resin material after aging for 6 minutes because the surface of the resin material and RTV23 and RTV22 able to withstand sparks during an electrical process tracking, the surface does not have a fire. However, as a result of sparks on the surface, causing the surface to become rough and formed pores filled with air.

When the surface is rougher, then the cohesive forces between water molecules also greater than the adhesion force between water molecules at the surface of the insulating material, thus making the surfaces more hydrophobic insulation material. The more rough the surface of the insulating material contact angle greater hydrophobic [10].

5. Conclusion
The results of the contact angle measurements before aging, show that the test sample RTV\textsubscript{21} to RTV\textsubscript{24} have contact angle below 90° means that the test sample is normally hydrophilic, only RTV\textsubscript{25} test sample that has reached the contact angle 94°, meaning that the test sample is normally hydrophobic. The contact angle of 76° epoxy resin in the composition of the mixture of silicone rubber and silica RTV\textsubscript{21} to be the contact angle 94° after the addition of filler material silicone rubber and silica mixture of RTV\textsubscript{25} showed an increase in the hydrophobicity properties of epoxy resin material.

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