STUDIES ON SURFACE CHARACTERISTICS OF SILICONE RUBBER COATED HV INSULATION UNDER DIFFERENT NANO-FILLER CONCENTRATIONS

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by

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CHAPTER-1

INTRODUCTION

Chapter 1

1.1. Background

With the increase in power demand in every sector high voltage transmission and distribution has become inevitable, and with them the use of high voltage insulators especially in high voltage transmission line. These insulators are faced with the vagaries of nature like rain, fog, humidity, snow and pollution subject to their location, be it coastal or semi-arid regions and moreover they are also subjected to electrical and mechanical stresses. Due to these reasons insulators are considered to be the weakest link in the high voltage insulation system, failure of which results in the failure of the whole transmission system, so the point in the making is that we need to ensure reliable operation of the insulators and thereby of the whole transmission system.

Conventionally ceramic insulators (porcelain and glass) which are inert and stable materials that didn't suffered much surface degradation but these materials are possess high wettability when exposed to moist conditions as rain, fog and dew because of their high surface energy. When insulators are wet and contaminated then leakage current increases which may lead to flashover resulting in system outages. Due to these reasons polymeric insulators started to gain prominence as they are able to maintain high tensile strengths and solve the classical problems of porcelain and glass insulators. They also have the advantage of being much lighter in weight, economical at installation and maintenance level, lesser threat of vandalism are the factors at the superficial level. In view of its technical aspects it possesses very high resistance to flashover, puncture, tracking and erosion as compared to ceramic insulators. In face of contamination and pollution also polymeric insulator fares far better than ceramic insulator due to its excellent hydrophobic property that is excellent water repellency.

Due to these reasons of late silicone rubber bulk material is being used for composite insulators in new installations, the bulk material can be either room temperature vulcanised (RTV) or high temperature vulcanised (HTV) silicone rubber (SiR). But in cases of budgetary constraints or other technical reasons were replacement is not the option then the ceramic insulators are coated with RTV silicone rubber of 0.5mm thickness. This is where comes the use of room temperature vulcanised silicone rubber.

1.2. Motivation

With the recent advancement in polymeric insulator both high temperature vulcanised silicone rubber and room temperature vulcanised silicone rubber are being widely used in transmission system because of their excellent hydrophobic properties and have ample number of investigations carried on to determine variation in their hydrophobic properties of high temperature vulcanised in case of contamination and the hydrophobicity recovery. It is also essential to have a study on the hydrophobicity of room temperature vulcanised silicone rubber.

There has been a large number of study on the leakage current analysis of polymeric insulator and majorly the studies and analysis that have been carried out are through fast Fourier transform, which basically convert the leakage current signal from time domain to frequency domain, and the analysis have been carried out in respect to variation in harmonic content with respect to contamination, but a study of harmonic content with different concentration is also essential

1.3. Contribution

A comparative study on variation of leakage current (LC) magnitude with changing fumed silica nanofiller concentration in room temperature vulcanised (RTV) silicone rubber (SiR), and also corroborated the result with root mean square value of LC pattern so obtained. Thereafter fast Fourier transform (FFT) was performed on the LC pattern and the variation in harmonic content and in the ratio of 3rd harmonic content to fundamental has also been discussed in detail.

A study on hydrophobicity measurement was done on two-part RTV SiR with varying silica nanofiller concentration and the variation in contact angle was noted and justified. Further to gauge the dynamic hydrophobicity of the RTV samples were also studied by contaminating the samples and measuring the hydrophobicity transfer to the surface of contaminated insulator and recovery percentage of hydrophobicity after the removal of the contaminant by measuring the contact angle of water droplet using goniometer by sessile drop technique. Thereafter, the mechanism of hydrophobicity transfer to the surface of contaminated RTV samples with different concentration of silica filler and also the hydrophobicity recovery has been explained.

1.4. Literature Review

1.4.1. Introduction

In power systems, insulator plays a very important role by isolating live parts with live parts and ground and also providing mechanical protection. The insulators have been widely used at substations, transmissions and distribution networks also [1].

Since a long time ceramic insulators have been widely used in power system and is still quite prominent in the transmission and distribution systems. Ceramic insulators possess good mechanical and electrical properties and are quite inexpensive, but being outdoor insulators they have quite a few weaknesses especially under certain environmental circumstances such as in humid condition, during the rainy seasons and also in case of pollution, which reduces their surface resistance. The reduction in surface resistance increases the magnitude of leakage current flowing on the surface [2]. Leakage current (LC) of large magnitude flowing on the surface for long period causes degradation of the insulator surface [3]. Moreover, in case of humid condition or in rainy season the ceramic insulators being hydrophilic in nature leads to the formation of continuous water films which results in increased tracking eventually to flashover. One of the ways of overcoming this problem of ceramic insulators, be it porcelain or glass insulator, is to coat it with room temperature vulcanised silicone rubber. A study on this regard was conducted by *Suwarno and Pratomosiwi* [4], in which they compared the leakage current, hydrophobicity and surface smoothness of RTV silicone rubber coated porcelain insulator and uncoated porcelain insulator.

1.4.2. Sample Preparation

In past few years, a lot of emphasis has been given on the silicone rubber (SiR) composites for use in electrical insulation applications due to excellent results obtained for their dielectric and surface properties [5]. But to improve these properties of base polymer further it is often compounded with various fillers, depending on the property that has to be enhanced [6]. Like as shown in [7,8] that a Highly loaded SiR performed better than SiR composites having lower filler content in context of tracking and erosion resistance. And it has also been reported that higher loadings of the hydroxide filler can result in poor mechanical properties like low tensile strength [9].

Although micro and nanodielectrics open a new arena of opportunities in the electrical insulation industry, a major issue for the researchers is to accurately distinguish their dielectric properties and one of factors that has directly affected these properties is the method of processing adopted for these SiR composites preparation. But the processing method has to be commercially viable and should be a relatively easy process of manufacturing. A few of the general processing techniques adopted for dispersing the micro and nanoparticles in a polymeric base are direct mixing, mechanical stirring, ultrasonication and in-situ polymerization [10]. Moreover, for having a good dispersion of filler and for having improved characteristics for the polymer composites, a method has been proposed in [10,11]. The datasheet for the RTV SiR composite is referred to for mechanical and dielectric properties [12].

A thorough study on different organic, inorganic and metallic filler conducted by *A Khattak and M Amin* [13] has shown that silica filler is excellent for imparting mechanical strength to the polymer composite whereas alumina trihydrate was quite efficient in enhancing the electrical properties. *FANG Su et al.* did an extensive study on silica filler as an enforcing filler in the silicone rubber compound having fumed silica, precipitated silica and modified precipitated silica. In his study he found that fumed silica fared exceptionally well in terms of hardness, elongation and tear strength. It also was comparatively better in context to tensile strength. The examination of morphological features of compound with these various silica fillers using transmission electron microscopy showed had long chain aggregates that resulted in excellent mechanical properties it also revealed that fumed silica compound had less moisture content as compared to precipitated silica compound and also in its datasheet [14,18].

1.4.3. Leakage Current

High voltage insulators are extensively used in transmission and distribution lines to separate two electrical conductors and electrical conductors from towers. When insulators are located in coastal and industrial areas then they get polluted with salt deposits and industrial dusts respectively. In high humidity and rainy condition, the layers become wet and are conductive resulting in the flow of leakage current (LC) along the surface of the insulator. This development of LC results in the flash over which short circuits two lines or between line and towers. This is why leakage currents are measured and analysed under laboratory or in field conditions, to comparatively access the condition of the insulator surfaces [15]. Investigation

of leakage currents in terms of magnitude and pattern provides us with useful information regarding the condition of the insulator surfaces [16,17]. The leakage currents are initially quite small in magnitude and capacitive but over the time their magnitude increases and become resistive and later on their harmonic content also increases [16,19,20]. Increase in LC leads to the formation of dry band on the insulator surface. Dry band arcing further increases the harmonic content [19]. Thus, leakage currents are usually analysed both in time and frequency domains [15]. There are quite a number of time domain and frequency domain feature which independently or collectively can be used to investigate the LC values as shown by *D. Pylarinos et al.* [20,21].

A study conducted by *M. Amin et al.* [22] shows that the usage of composite insulators in reduces the leakage current considerably even in face of contamination, and also different LC measurement methods. An extensive survey on the the micro and nano-filler to improve the performance of outdoor silicone rubber insulator was done by *G. Momen and M. Farzaneh* [23] showed that aluminium trihydrate (ATH) and silica were the mostly used filler, of which silica being the reinforcing filler as they increase the tensile strength and ATH being the extending filler that imparts tracking resistance and acts as a flame retardant also. They have also studied the effects of other inorganic, metallic, carbonaceous and organic fillers too.

1.4.4. Hydrophobicity

Composite insulators have been in use for outdoor electric power insulations since the early 1960's, and the modern style composite insulators have been developed recently in the past 25 years [24,25]. At the starting, ethylene-propylene-diene monomer (EPDM) and epoxy resin were mostly used as insulator material, but silicone rubber (SiR) composites started gaining more attention for outdoor insulation since the 1990's mainly because of its excellent hydrophobicity [26]. Of which Room temperature vulcanized (RTV) silicone rubber is becoming popular mainly because it possesses excellent hydrophobicity and unique ability of hydrophobicity to migrate which results in a significant increase in the pollution flashover voltage for glass and porcelain insulators [27,28].

A large number of studies have shown that the degradation of the hydrophobicity of RTV SiR occurs under corona discharge and to a great extent to the span of time of exposure to harsh environmental condition [29]. To evaluate this degradation of condition of the materials, hydrophobicity indexes are affected by many parameters, like measurement temperature, surface charge, surface roughness, and volume of the water droplet etc. Hence it is very

important to note the effect of these measurement parameters for the evaluation of hydrophobicity. A few papers which evaluated these effects are [30-34].

The standard assessment methods for hydrophobicity of RTV silicone rubber are contact angle measurement, research on discharge and Fourier transform infrared spectroscopy (FTIR) analysis RTV silicone rubber [35-39]. The contact angle is basically the direct measurement of the hydrophobicity of the surface whereas FTIR shows the variation in its function groups.

The change in hydrophobicity that is the mechanism of transfer with the diffusion of low molecular weight (LMW) has been investigated in [40-42].

1.5. Thesis Organisation

The next chapter that is chapter 2 will be deals with the different outdoor insulators that have been in use, their comparative study and the different insulator coatings that are used, with emphasis on silicone rubber.

Chapter 3 presents different condition assessment techniques of high voltage insulators

Chapter 4 deals with different raw material and equipment used with a brief description about them, the sample preparation method adopted, and the experimental setup and procedure for leakage current measurement and hydrophobicity measurement.

Chapter 5 presents the experimental results of the leakage current measurement, the leakage current analysis, the static and dynamic hydrophobicity measurement of two-part room temperature vulcanised silicone rubber and discusses the findings.

Chapter 6 provides conclusion from the present study and the future scope of the work.

CHAPTER-2

BACKGROUND OF OUTDOOR

INSULATORS

Chapter 2

2.1. Introduction

Electrical insulation has been needed since the time electricity was discovered. As for the outdoor insulators mostly porcelain or glass were used, often called as ceramic insulator. Porcelain was the most used outdoor insulator and still is one of the widely used outdoor insulator but being fast replaced by the polymeric composite insulators. A detailed study of the shortcoming of ceramic insulators, the advantages of polymeric insulators, and the methods to improve the ceramic insulators for at par performance with the polymeric ones are discussed in the ensuing sections.

2.2. Ceramic Insulators

Ceramic insulators have been in use as outdoor insulator since the 1850's, the porcelain insulator which was used as telegraph insulators. But ceramic insulators for outdoor insulators remained in electrical industry because they both, porcelain and glass are very stable materials which can take substantial amount of arcing without leading to much surface degradation as they also have high withstand capacity to heat and dry-band discharges [1,2]. But the main problem with ceramic insulators are they have highly wettable surface especially when exposed to humid environmental condition like dew, rain and fog, the reason being they are very high surface energy materials.

As known that when insulators are wet and contaminated then large leakage current develops which ultimately leads to flashover and power system outages. Insulator contamination is one of the major problems in electrical power system as they are the primary cause of flashover as they increase the surface leakage current. Wet atmospheric condition often leads to water filming on the surface of the insulator and in presence of contaminants large leakage current develop on the insulator surface and this uncontrolled increase in leakage current ultimately leads to flashover [3].

Contamination is one of the serious issue ceramic insulators as they often lead to surface degradation which varies as the contaminant deposit is different for different environmental and topographical location. On the nature of contaminant deposit, contamination study can be

broadly classified into the urban or industrial area, the rural area and the coastal area. The classifications are discussed below [42]

1. In urban or industrial area: The main reason for contaminant deposit in these areas are due to the emission from industries and automobile sectors. There are different kinds of industrial pollution majorly depending on the type of industry like chemical, metallurgical, petrochemical, textile etc.

2. In rural area: In these areas the major reason for contamination is the dust particle as the region are quite underdeveloped or undergoing development. To some extent the contaminant deposit can also be attributed to the extensive use of pesticides, herbicides and fertilisers too.

3. The coastal area: It is also one of the areas which suffer from severe contaminant depositing which is mainly in the form of salt deposits and salty moisture. This kind of contaminants are not only found on insulator located near to the but also upto a considerable distance away form the sea as well. Increased contaminant deposits occur on the insulator during coastal wind blowing toward the wind as they carry salty moisture and deposit them on the insulator surface aggravating the problem of contamination and increased leakage current.

In order to get rid of the problem of surface degradation and of contamination, to the great extent, in relation to contamination leading to increase wetting of the surface the main techniques employed are

1. Using polymeric insulators as they have improved resistance to weathering and erosion or

2. To apply protective coating on the insulator surface of ceramic insulator to lessen the degradation process.

These are two most viable options available economically are enlisted above of which a study on each lead us to subsequent section. The first option is dealt with in detail in section 2.3. Whereas the second option is discussed with in detail in section 2.4.

2.3 Polymeric Insulators

Polymeric insulators have been in the fray for quite a long time as outdoor high voltage electrical insulator, approximately for more than 40 years. Yet they are relatively new in comparison to ceramic insulators and are still a subject of research. The polymeric insulators have very strong cores and also possess very high tensile strength. They are seen to be modern day insulators for they have been very efficient in tackling the problem infested in classical insulators of glass and porcelain with respect to contamination performance and resistance to weathering and erosion. They have the capability to support very large mechanical loads especially for ultra-high voltage transmission and moreover they can be designed for and unconventional insulating structure also. In comparison to ceramic insulators are light in weight, have good vandalism resistance, and as said far improved contamination performance. The degradation also takes place in polymeric insulator when exposed to harsh environmental condition or electrical stress for long durations.

Polymeric insulators are majorly ethylene propylene diene monomer (EPDM) and Silicone rubber (SiR), of which EPDM possess good hydrophobicity properties but do not possess weathering resistance and hydrophobicity resistance [23]. These insulators also have excellent weathering resistance and leakage current suppression capability. Generally the composite polymeric insulator have fibre-glass rod for mechanical strength and weather shed made of EPDM or SiR, with metal endfitting at both ends for attachment. Figure 2.1 shows the composite polymeric insulator.
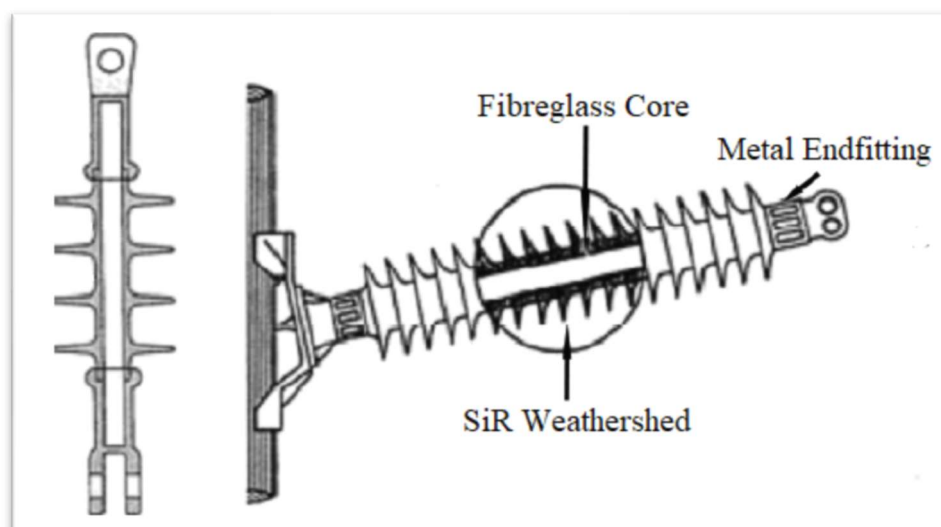

Figure 2.1: A composite polymeric insulator [23]

2.4. Coatings of Line Insulators

Weathering and degradations have been a problem for ceramic insulator from the beginning. As early as in 1920's oil bath technique was employed to prevent wetting of the insulator surface [42]. Around 1950's the hydrocarbon coating technique in the form of petroleum jelly was used extensively, but it had its drawback so an improved coating in the form of silicone grease were used. Around 1980's the silicone grease coatings were replaced by silicone rubber coatings, which are effective even today.

2.4.1. Hydrocarbon Coatings

Petroleum jellies were made from different petroleum fractions but mostly from microcrystalline and slack waxes and from hydrocarbon oils too. They are found to soften with increasing temperature and often melt at sites of electrical discharges due to heat, thereby engulfing the contaminant while on cooling they again retain their properties. Studies had revealed that a maximum thickness of about 7.6 mm and minimum thickness of 3.2 mm had shown considerable inhibition of flashover [42]. Their working life was estimated to be 3 years. So, it was found to be an economical option on that front.

Results were satisfactory with respect to number of flashover occurrence but the major issues were the inability of application at moderate or high temperature regions because of melting tendency of petroleum jelly. To remove the fouled petroleum jelly after it service life was found to be very costly and cumbersome as they had to be manually scraped which is a laborious process [42]. Moreover, with advancements silicone material were available and known for their water repellent characteristics.

2.4.2. Silicone Grease Coatings

Silicone grease like compound were used as protective coating for ceramic insulator because of the tiresome process of removal of hydrocarbon coatings. With the advancements in silicone compounds known for their excellent hydrophobicity and weathering resistance were employed for coatings of ceramic insulators as well. Silicone greases were composed of silicone oil and silicone filler that do not melt, but decompose at temperature in excess of

200°C. So, it was possible to use them practically in all climatic conditions unlike the hydrocarbon coatings [42].

The other important improvement over the hydrocarbon grease was, that even after an electrical discharge the substance remains greasy. The grease like components provided a non-wetting surface and hence maintained a high surface resistance, though salt, dust and other contaminants were deposited on the insulator surface. Silicone rubber coating suppressed leakage current and flashover voltage remained high until the coating was totally saturated with contaminant even under wet condition. It was reported that the effectiveness of the coating largely depends on the thickness and uniformity. An aluminium trihydrate filled silicone grease was found to have much better antitracking resistance but reduced water repellent characteristic.

The commonly used silicone grease coating had its limitation too, it being lesser arc resistant, though it maintained hydrophobic property for long time but the problem of water erosion persisted, the infusion and adsorption of the particle matter resulted in loss of hydrophobicity after a certain time and moreover required more frequent reapplication of the grease as compared to hydrocarbon coatings. Removing silicone grease for reapplication was also a time-consuming task and resulted in higher cost.

2.4.3. RTV Silicone Rubber Coatings

Room temperature vulcanised (RTV) silicone rubber (SiR) coatings have been used in many electrical applications. They maintain good dielectric properties and flexibility for a wide range of temperature and also provide excellent resistance to corona discharges and thermal degradations. Like all silicone rubber compounds, they possess excellent water repellency characteristic because of their low surface energy. The hydrophobic surface of RTV SiR reduces the dry band formation and leakage current developments thereby improving the contamination performance.

A lot of variation in the properties of silicone rubber can be achieved by adding filler into it, by changing the quantity of filler, size of the filler, even the type of the filler has a pronounced effect on the properties of the coatings. Especially aluminium trihydrate (ATH) and silica are known to be one of the most effective filler for imparting anti-tracking resistance and mechanical property enhancements [4,8].

The clean ceramic insulators are spray coated with a thin layer of RTV SiR of about 0.5 mm, which is cured to form a flexible elastomer having excellent dielectric properties. It is known that the coated surface of the ceramic insulators tends to remain hydrophobic even after exposure to electrical stress for long durations. Moreover, the SiR coatings have the property of hydrophobicity recovery which is attributed to low molecular weight (LMW) compounds present in the bulk of coatings [42].

Advantages of SiR coating to silicone grease coatings are that the thickness of the coating applied is greatly reduced thereby reducing the material consumption, labour saving, and longer life of the coatings also prove to be cost effective. The RTV coating application are simple and relatively economical. It is also very easy to clean and restore it water repellency. Due to all these factors, it was be concluded that RTV SiR coatings reduces the maintenance cost of insulator to a great extent.

2.4.3.1. Silicone Rubber Compounds

In humid environment and costal conditions, surface resistance of the ceramic insulators decreases drastically resulting in the increased leakage current ultimately leading to flashover rendering the insulator damaged permanently damaged. To overcome this problem the ceramic insulators are being replaced by the polymeric insulator, generally the new insulators being installed are high temperature vulcanised (HTV) silicone rubber (SiR), but in case of in-use insulators which cannot be removed from system then room temperature vulcanised (RTV) SiR coating is applied on the insulator surface [4].

Silicone rubber is a synthetic, or a man-made, compound known for its excellent weatherability and the ability to maintain its properties over a wide range of temperatures. SiR's use as an electrical insulator is due to it being resistant to oxidation, possessing low surface energy, and resisting degradation from ultraviolet (UV) radiation. These properties of silicone rubber are due to the structure of the base polymer. Resistance to oxidation, heat stability and weathering are because of the strength of silicon oxygen bonds. Silicone rubber possesses low temperature flexibility and low surface energy (excellent hydrophobicity) mainly because of its flexible polymer chains.

Silicone rubbers are 'polymers' and as polymers are made up of large number of molecules that have atoms arranged one after the another in chain like structure, while each link in the chain has the same basic structure. Polysiloxane has a basic unit of siloxane which repeats in

the chain like structure. The structures are identical, and the number of repeating units is denoted by 'n'. If this 'n' is small then it is said to be low molecular weight (LMW) compounds and they exhibit low physical properties. As 'n' increases the molecular weight also increases and the physical properties are also enhanced. The length of SiR polymer is in the range of 3000 to 10000 monomers. Organic side groups (generally containing carbon) are attached to the silicon atoms which allow for crosslinking and tailored applications, like increased tracking resistance, weathering resistance, heat resistance etc. SiR has methyl as side-group attached to the siloxane unit, which provides for water repellency characteristics, resulting in dimethyl siloxane being its basic unit and hence the name for polymer so obtained as poly dimethyl siloxane (PDMS) [6]. Hence SiR is said to be neither organic nor inorganic but rather it is classified as an organo-silicone compound it is because of the very important bond that is formed between carbon (an organic group) and silicon (an inorganic group). The basic unit of SIR that is dimethyl siloxane is shown in Figure 2.2.
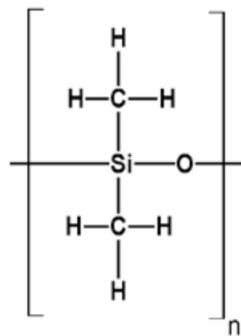
Figure 2.2: Dimethyl siloxane unit [6].

RTV silicone rubber that is commonly used in high voltage insulator coatings have poly dimethyl silicone base and a mixture of reinforcing filler, extending filler, low molecular weight silicone of improving dynamic hydrophobic properties, and structuring additive also to reduce crepe hardening.

2.4.3.2. Fillers

Fillers are always added to the silicone rubber (SiR) base to improve their electrical and mechanical properties. Fillers also help in reducing the overall cost of the composite insulators. There are variety of different types of filler for enhancing different properties to the SiR insulators, apart from that fillers of same type also differ in properties due to their different sizes like microfiller and nanofiller also have marked difference in properties. Even filler of same type and same size cater to extending different properties just due to having different

surface treatment or different surfactants [23]. The extent of property enhancement depends largely on filler particle size, structure, morphology, degree of dispersion, filler concentration, filler surface treatment and also on the degree of adhesion and orientation in the matrix.

Based on the property enhancement of the base polymer chain the filler can be broadly classified as extending filler or reinforcing filler. The reinforcing filler generally improve the mechanical strength of the polymer like tear strength, abrasion resistance tensile strength etc. Whereas the extending fillers generally enhance targeted properties of the base polymer. Fumed silica, carbon black and aerogel silica are known to be reinforcing filler whereas aluminium trihydrate, zinc oxide and titanium dioxide are known to be extending fillers. Table 2.1 represents commonly used filler and the property enhanced by it in silicone rubber [23].

Table 2.1: Fillers and the corresponding property enhanced [23]

Filler type	Property enhanced
zinc oxide	mechanical properties and electrical conductivity
Titanium dioxide	thermal strength and relative permittivity
Aluminium trihydrate	antitracking, erosion resistance
Silica	mechanical properties and antitracking
Barium titanate	thermal stability and relative permitivity
Calcium carbonate	flame retardant and hydrophobicity
Carbon black	mechanical and electrical properties

CHAPTER-3

CONDITION ASSESSMENT

TECHNIQUES

Chapter 3

3.1. Introduction

Monitoring of insulators are of prime importance for proper functioning of the power system, as the overall reliability of the system depends on the reliability of the insulators. At present there are many condition assessment techniques employed for effective monitoring of the system, to name the major ones are scanning electron microscopy (SEM), Leakage current measurements, hydrophobicity measurements and Fourier transform infrared spectroscopy (FTIR). These condition assessment techniques are discussed in this section with special emphasis on leakage current and hydrophobicity measurement technique as they have been employed in this study for the condition assessment of insulators.

3.2. ESDD Measurement

For simulation of the polluted environmental condition and pollutant on the insulator surface, researchers have suggested to have equivalent salt deposit density (ESDD) and non-soluble salt deposit density (NSDD) as measure of the contamination severity. Generally, kaolin $(Al_2Si_2O_5(OH)_4)$ and sodium chloride (NaCl) are mixed in distilled water to get the desired ESDD values

ESDD calculations are based on the temperature and the conductivity of the contaminant. Based on IEC 60507 [38] the formula for conductivity at 20°C is given in equation (3.1)

$$\sigma_{20} = \sigma_\theta[1 - b(\theta - 20)] \tag{3.1}$$

Where

$\sigma_{20} =$ is the volume conductivity at 20°C (S/m)

$\sigma_\theta =$ is the volume conductivity at θ°C (S/m)

$\theta =$ is the solution temperature in °C

$b =$ is a factor depending on θ.

The relationship between salinity (Sa) and volume conductivity at 20°C is given in equation (3.2) as

$$Sa = (5.7\sigma_{20})^{1.03} \tag{3.2}$$

And ESDD is calculated from the value of salinity using the equation (3.3)

$$ESDD = Sa \times V/A. \tag{3.3}$$

3.3. Scanning Electron Microscopy

SEM technique has been in use for more than 40 years which gives us information of the surface topography of insulating materials. It gives us a magnified image of the surface, much like the electron microscope. In SEM method and electron beam is accelerated and directed towards the surface whose topography is to be analysed. On striking the surface the electrons divide into four different groups namely, the stopped electrons, the absorbed electrons, the deflected or secondary electrons and the reflected or we can say the back scattered electrons. The stopped electrons are those electrons which are stopped on striking the surface resulting in transfer of energy to the sample which in turn results in exciting the material electrons and producing a luminance. The second group of electrons that is absorbed electrons are absorbed by the sample resulting in ejection of other electron from the surface leading to X-rays being produced. The deflected or secondary electrons move in their direction and reflected electron move towards the source. All of these electron groups are detected and collected to give a comprehensive study of the surface roughness, surface deterioration and the shape of the surface upto micrometer range [26].

3.4. Fourier Transform Infrared Spectroscopy

It is also condition assessment technique for insulators, but unlike SEM it does not focus on the topographical changes rather on the structural changes in the insulator [26]. It is quantitative measurement technique and used prominently for organic compound, nut can be used for inorganic compound as well. It is based on the principle that when any material is subjected to infrared radiation then they absorb radiation of particular frequency which match with the frequency of vibration of atoms present in the material.

So, in this method the samples are subjected to infrared radiation, the samples absorb the radiation of frequencies that match with the vibrating frequency of the constituent atoms. Hence a dip is there for that frequency range in the infrared spectrum obtained. The infrared

spectrum is then compared with a set of standard spectrum curves and the constituent elements identified. For sample degradation studies also it is helpful, as we can measure the FTIR response for virgin samples and then compare it with FTIR response of aging or degraded samples and the changes in it can be analysed.

3.5. Leakage Current Measurements

The performance of an electrical power system depends mainly on its insulation. Today, polymeric insulators are being widely used in both the transmission and distribution of high voltage and are steadily capturing a wide share of the market. The phenomenal growth in the application of polymeric insulator is because of their advantages over the glass and porcelain insulators. These advantages are specifically low surface energy, lighter weight for same capacity, higher mechanical strength, vandalism resistance and much better performance in wet and contaminated condition. The long-term ability of polymeric composites as a high voltage insulator is judged by design test, which is accelerated aging test and normal aging test. The polymeric insulator performance is tested by using different measurement techniques for the diagnostics of the insulators. The equivalent salt deposit density (ESDD), surface conductance, the leakage current measurement and non-soluble deposit density (NSDD) are the most commonly used methods for monitoring and investigating the polymeric insulator performance. The hydrophobicity and aging are the other important factors that affects the performance of insulators under environmental and electrical stress [22]. Due to the increasing leakage current (LC) and discharges, the degradation process develops significantly on the polymeric insulator's surface. The measurement of LC is performed frequently to assess the performance and aging of polymeric insulators in laboratory and field studies. The leakage current analysis plays a very important role in helping us to understand the surface condition and deterioration of the polymeric insulators. The leakage current development mechanism and thereafter, its time domain and frequency domain analysis are discussed in detail.

3.5.1. Leakage Current Analysis

The LC is driven by a source voltage and is collected at the ground end of the insulator, which provides quite a lot of useful information on describing the current state of a contaminated polymeric insulator. When the applied voltage is low or the contamination is less the LC is usually capacitive in nature and the waveform is sinusoidal. Once the applied voltage increases beyond a certain value or the contamination increases above a certain level then LC becomes

more resistive. Dry-band discharging causes the appearance of spikes on the LC crest. This results in the deformation of LC waveform and results in the increase in the harmonic content. It has been reported that the changes in the LC waveforms, until flashover occurs is classified into five different stages. The possibility of flashover occurring increases when the magnitudes and the harmonic contents exceed a certain level [17]. Surface discharge activity ultimately leads to deterioration in the electrical and mechanical properties of the materials due to the chemical reactions occurring on their surfaces. The modes of degradation can be seen by the formation of tracking as well as loss in material weight. The changes on the surface condition of polymeric insulators affect the magnitude and the shape of the LC. Mathematical analysis results in a nonlinear relationship between the leakage current time variations, surface discharge and length of the arc. If the extension of surface discharge is more than electrical breakdown stress of air then the arc will take place. This leads to change in the magnitude and shape of the LC. So, RMS and peak value of current are not sufficient parameters to judge the surface condition. This leads to the concept of frequency domain analysis of the LC waveform [15]. But before the application of frequency domain analysis tool moving average method is applied to the LC waveform to smoothen the time variation and get a clear view of the LC pattern.

3.5.2. Moving Average Technique

The method of moving average technique is used to understand then characteristic variation of LC with time. By having an average magnitude of the LC that moves with the addition of new data points, the characteristic variations are smoothened out resulting in reduced fluctuations with time and hence there is a stronger indication of the trend in the variation of LC magnitude over the period. The basic meaning of a moving average is that it is the average magnitude of the current value at a particular point of time. There are many different types of moving average techniques like the simple moving average technique, the exponential moving average, the time series moving average, etc. In this work the simple moving average technique has been used. A window size consisting of k points is chosen to calculate the moving average using the expression as shown in equation (3.4)

$$X_i = \frac{\sum_{N=i}^{N=i+k} Y_n}{k} \tag{3.4}$$

where X_i are the calculated moving average points corresponding to each Y_N the raw data points. In this work window frame is of 25 data points.

3.5.3. Fast Fourier Transform

Frequency domain analysis is the analysis done to calculate the exact harmonic content of the leakage current signals. The harmonic content in the signals are extracted by using Fast Fourier Transformation (FFT). FFT approach to analysis is an efficient one and produces realistic results for large scale of signal processes.

To transform the signal from time domain to frequency domain Fourier series or Fourier transform can be used. Discrete Fourier Transform (DFT) is one of the powerful computation tools and it also allows evaluating the Fourier Transform of a discrete signal. DFT requires about N^2 complex multiplications, which can be reduced by using symmetric properties of the trigonometrical functions. In 1965, Tukey and Cooley proposed a method for computing DFTs reducing the number of operations proportional to $N \log_2 N$, which is referred to as the FFT. Therefore, the FFT is claimed to be an $N \log(N)$ algorithm. Hence the speed increase is in the order of $(\log_2 N)/N$ where N is the time series length of the sample.

Let x(t) be the time domain representation of the given signal x then the frequency domain representation of signal x is given by X(f) in the equation (3.5)

$$X(f) = \int_{-\infty}^{\infty} \chi(t)e^{-j2\pi} \; dt$$

(3.5)

the amplitude spectrum of the leakage current in amperes rms is computed by the equation (3.6)

$$Amplitude \; LC \; (I_{rms}) = \sqrt{2}\frac{Magnitude[FFT(A)]}{N} \qquad for \; i = 1 \; to \; \frac{N}{2} - 1$$

(3.6)

where A is the leakage current in the frequency domain.

3.6. Hydrophobicity Measurements

Hydrophobicity of a material is its resistance to flow of water on its surface. A material is said to be hydrophobic if it resists to flowing of water dropped on it and is said to be least hydrophobic if the dropped water flows leaving a track on its surface. A hydrophobic surface is water repellent, in contrast to a hydrophilic surface that can be easily wetted. Hydrophobicity of any material is described by the contact angle on its material surface (θ_C) that a liquid drop makes on coming into contact with a solid surface; this angle is often used as a measure of the surface wettability. The material that is easily wettable means it allows water to come in contact with a large surface area of the material and therefore has a contact angle of much less than 90°, whereas, hydrophobic material allows lesser water contact with the surface resulting in a contact angle of greater than 90°.

From the contact angle we can get information about surface roughness, surface energies, and surface heterogeneity. Generally solid surfaces are characterised as high energy or low energy surfaces. A high energy surface is wetted easily and the water spreads over the entire surface forming a continuous film and contact angle is zero whereas, a low energy surface repels water resulting in separate droplets being formed on the surface and the contact angle is greater than 90°. Contact angle can also be used as a measure of the extent of surface contamination. The hydrophobicity of an insulation material is quantitatively evaluated by the contact angle formed between a water droplet and the material surface, which is a direct representation of the tension between the material atoms and water interface. The shape of the liquid droplet and the contact angle largely depends on the type of the solid material, of which hydrophobicity is to be measured and the chemical and physical state of its surface [32].

Surfaces are said to be hydrophilic when the contact angle between the water droplet and the surface is less than 35° and for angles in excess of 90°, the surface is said to be hydrophobic whereas, the surfaces having contact angles in between 35° to 90° are said to be partially wettable. It should be noted that the term 'hydrophobicity' means resistance to water, but it is generally used to represent resistance to any of the liquid.

Hydrophobicity performance of a substance is determined by two types of measurement: static hydrophobicity measurements and dynamic hydrophobicity measurements. The static measurement is the hydrophobicity of the raw material or of the material under normal stresses

[40]. The dynamic measurement means the measurements of hydrophobicity loss in event of long exposure electrical stress, hydrophobicity transfer in case of contamination and finally the hydrophobicity recovery when the electrical stress is removed or when the contamination is washed off, as the case may be.

3.6.1. Hydrophobicity Phenomenon

Before going onto the understand the hydrophobicity phenomenon the understanding of surface energy and surface tension is necessary. Surface tension is defined as the tendency of fluid surface to shrink into the minimum surface area it can. For liquid-air it is explained as: surface tension results from the greater force of attraction between liquid molecules (due to cohesion) than to the molecules in the air (due to adhesion) the result is that the surface comes under tension from the imbalanced forces and hence the term 'surface tension'. Surface tension has the dimension of either force per unit length, or energy per unit area. The two dimensions are equivalent, but when using energy per unit of area, then it is better to use surface energy, which is a more general as it applies to the solids also. The surface energy may be defined as the excess energy at the surface of a material compared to the bulk. Or another way of saying is that the surface energy is the work required to cut a bulk sample, creating two surfaces.

 The contact angle of water droplet with the surface is dependent on the free energy of the surface and the wettability of the surface is related to its contact angle [40-42]. The free energies are inter-related by the *Young*'s equation given by equation (3.7). The surface energy and surface tension can be used interchangeably. Figure 3.1 shows a water droplet, the contact angle formed and the surface tension at interface.

$$\cos\theta_y = \sigma_s - \sigma_{sl} \,/\, \sigma_l \qquad\qquad (3.7)$$

where θ_y = *Young*'s contact angle

σ_s = surface tension of the solid

σ_{sl} = surface tension of the solid-liquid interface

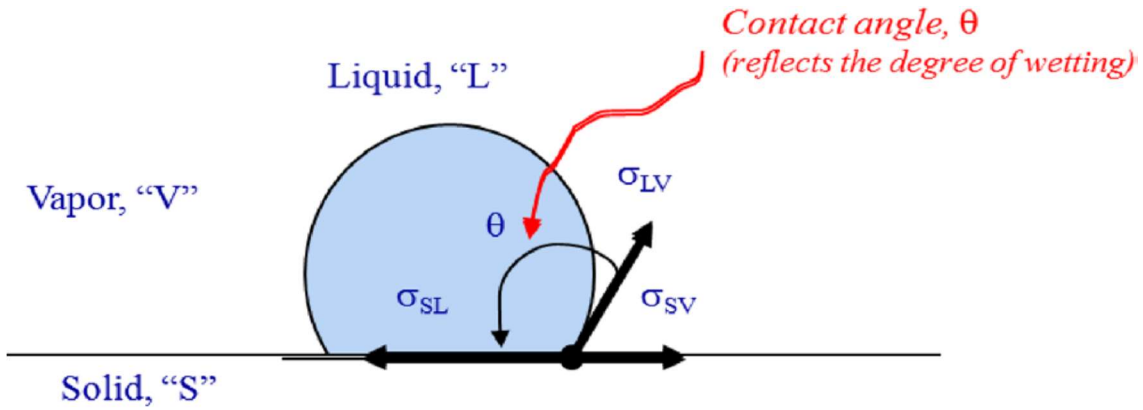σ_l = surface tension of the liquid.

Figure 3.1: Contact angle and surface tension for water droplet [30]

The energy to form two new surface is given by σ, and the energy of cohesion is given by W_C, then for a pure phase is $W_C = 2\sigma$, when different phases of of solid and liquid will be brought together there will be some adhesion and the energy associated is given as W_{sl}. The relationship between the energy required to separate the liquid from the solid and their surface energy per unit area of contact is given by Dupre equation (3.8).

$$\sigma_{sl} = \sigma_s + \sigma_l - W_{sl} \tag{3.8}$$

For two identical phases which adhere perfectly, $\sigma_s = \sigma_l$ and $W_{sl} = W_C$ therefore $\sigma_{sl} = 0$.

On combining equation (3.7) and (3.8) the resulting equation (3.9),

$$W_{sl} = \sigma_l(1 + cos\,\theta_y) \tag{3.9}$$

All solid surfaces that have higher values of σ_s than the surface free energy of water will be wetted by it, and those that have lower values will not be wetted. The lower the value of σ_s the greater the contact and angle.

3.6.2. Hydrophobicity Measurement Methods

From the above discussion it can be well ascertained that hydrophobicity measurements are essential in analysing the condition of outdoor insulators especially those in the coastal and humid environments. Hydrophobicity is estimated by majorly two methods namely, sessile drop technique that is contact angle measurement and the other is from the STRI classification guide.

3.6.3. STRI Classification Method

Swedish transmission research institute (STRI) proposed a novel method of hydrophobicity measurements by classifying different stages hydrophobicity. STRI is a very simple method of spraying a cleaned surface, of which hydrophobicity is to be measured, of about (50-100 cm^2) with distilled water. The drop pattern formed on the surface is attributed to one of the seven classes of hydrophobicity form HC1 to HC7 [27]. Of which HC1 being for most hydrophobic surface and HC7 being the least hydrophobic surface. Refernce guide for STRI classification is shown in Figure 3.2 for HC1 to HC6.

3.6.4. Contact Angle Measurement

STRI classification method suffers from the drawback of being subject to human judgement, as a result it may vary from person to person. To overcome this, the sessile drop technique or conatct angle measurement is employed to measure the hydrophobicty [35]. In this method a water drop is placed on the surface whose hydrophobicity is to be measured, by a syringe. The contact angle is measured using a goniometer. The contact angle measurement may be classified as the static contact angle measurements when the volume of the drop is fixed. For more detailed analysis of advancing and receding contact angle of water droplet is measured, while increasing the volume of water droplet until it just begins to flow for advancing contact angle whereas, reducing the water content until the droplet starts to recede for receding contact angle measurement. Figure 3.3 shows the advancing and receding contact angles.
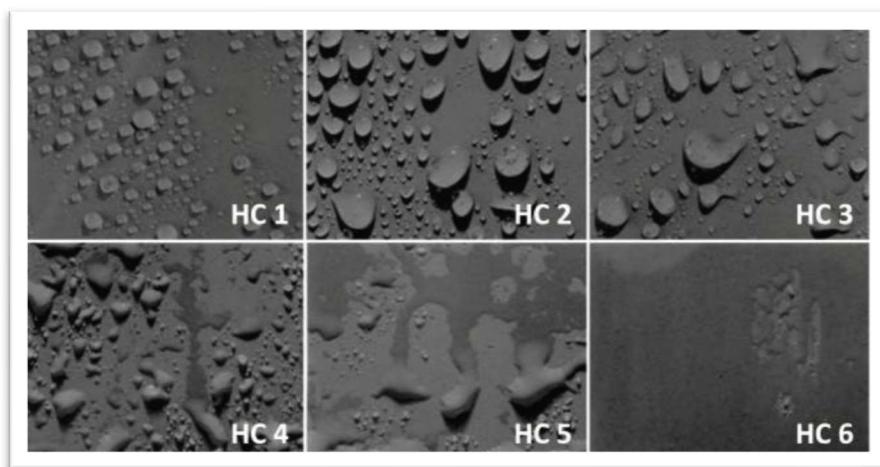

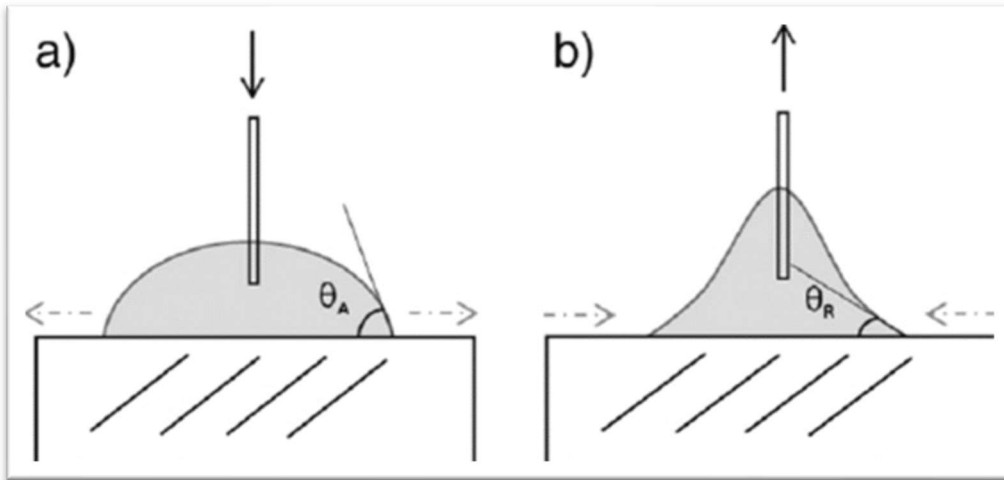*Figure 3.2: Hydrophobicity classification from HC1 to HC7 [39]*

Figure 3.3: (a) Advancing contact angle; (b) Receding contact angle. [43]

CHAPTER-4

SAMPLE PREPARATION AND EXPERIMENTAL PROCEDURE

Chapter 4

4.1. Introduction

The room temperature vulcanised silicone rubber samples prepared with varying concentration of fumed silica nanofiller were subjected to majorly two tests, the leakage current measurements and the hydrophobicity measurements. The details of the raw materials used, the laboratory equipment employed, the experimental setup, their schematic diagram and the experimental procedures are discussed in this section.

4.2. Material Used

In the previous chapter the detailed structure of the base polymer of Silicone Rubber (SiR) has been shown and the usage of different extending and reinforcing fillers has been discussed. In this study a two-part room temperature vulcanised (RTV) SiR has been used without any preloaded filler which was procured from a local distributer of Wacker Chemie AG, as Metroark Silicone Potting Gel, which comes with catalyst for curing Metroark 27 Catalyst. Figure 4.1 and Figure 4.2 shows the potting gel and the catalyst used. This potting gel is used as a coating for ceramic insulators as well as for embedding and potting electrical and electronic equipment. It is a thick viscous liquid and clear transparent in uncured state with a specific gravity of 0.97 at 25° C and has shelf life of six months. For a detailed understanding of its usage and working time the technical data sheet has been referred to [12].

Next to evaluate the performance of SiR with the addition of filler, of the different filler that are commercially used like aluminium trihydrate, silica, zinc oxide, titanium oxide etc. we zeroed on to silica filler which has been conventionally used as reinforcing filler for providing mechanical strength. Silica filler used as reinforcing filler can be categorised into three different types as fumed silica, precipitate silica and a combination of fumed and precipitate silica. A study of the effect of these three different fillers on the mechanical strength of RTV SiR has been evaluated by *FANG Su et al* [14]. Table 4.1 shows their finding on mechanical strength imparted by different types of silica fillers. The focus of this study is to see the effect

of fumed silica filler on the dielectric and surface properties of SiR, by varying its concentration. In this study fumed silica gel has been used as a filler provided by distributer of Aerosil. Figure 4.3 shows the image of fumed silica gel and Table 4.2 shows it technical specification as found from Aerosil technical overview sheet [18].

A CRC silicone releaser is used as a releasing agent for the sample from the cast. Paraffin oil, wax polishes or grease can also be used as releaser for potting gel. Figure 4.4 is the image of releasing agent. Stainless steel casts of diameter of 90mm have been used. Figure 4.5 represents the stainless-steel cast.
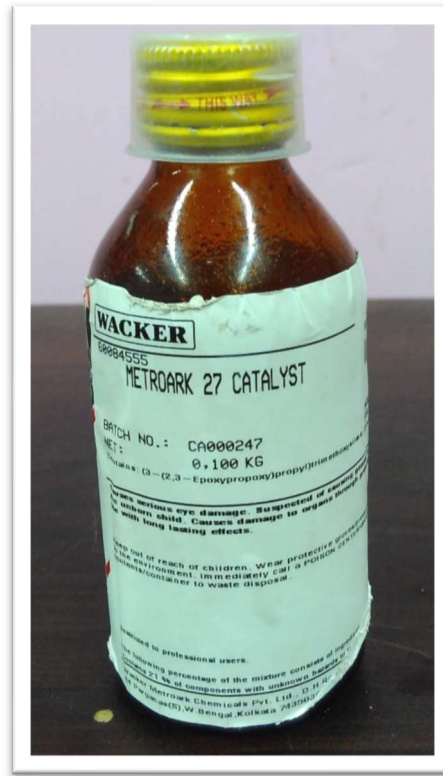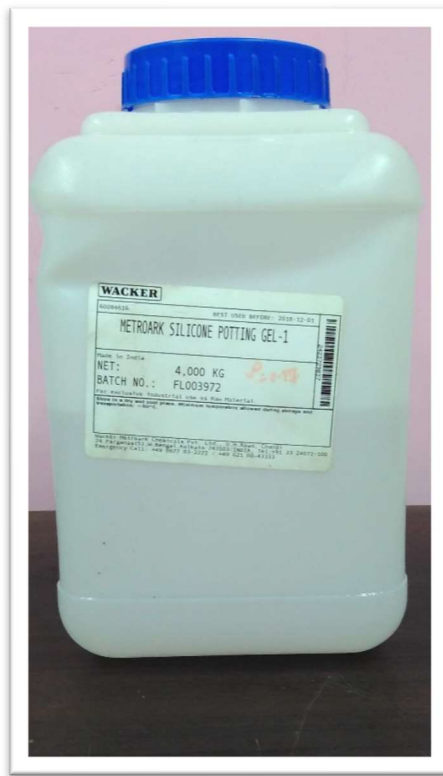

Figure 4.1: RTV SiR potting gel   *Figure 4.2: Platinum based catalyst*

Figure 4.3: Fumed silica as filler   Figure 4.4: CRC Silicone Releaser

Figure 4.5: 90mm diameter stainless steel cast

Table 4.1: Comparison of mechanical properties of SiR with different silica fillers [14]

Mechanical properties	Fumed silica	PPT silica	Fumed + PPT silica
Hardness	61	55	60
Tensile strength	6.5	5.5	6.2
Elongation (%)	230	210	200
Tear strength (kN/m)	19.5	17.5	18.5

Table 4.2: Silica filler properties [18]

Key properties	Values
specific surface area (m^2/g)	175-225
mean particle size (nm)	5-50
tapped density (g/l)	50
pH value	3.7-4.5
SiO_2 content (%)	99.8

4.3. Equipment

In this experimental study a lot laboratory equipment was needed right from the sample mixing, degassing to leakage current and hydrophobicity measurement, all of which are available at Jadavpur University, high Tension Laboratory. The list of the equipment used, their intended purpose and pictorial representation has been provided in this segment.

4.3.1. Heating Chamber

Heating chamber is a device which maintains a constant temperature inside by means of a thermostat (Thermostat is a device which senses the temperature of the system and then takes necessary action to maintain the temperature at a particular level) was used during the initial stage of sample preparation. Fumed silica nanofiller which has been used in the experimental purpose has to be demoisturized for 30 mins. Presence of particle leads to agglomeration of the filler and increase in surface area resulting in inadequate dispersion of filler in the RTV silicone rubber. It has a digital temperature indicating setup. Figure 4.6 shows the heating chamber setup.

Figure 4.6: Heating chamber setup

4.3.2. Goniometer

For the measurement of hydrophobicity there are two methods one is the STRI classification and the other is the contact angle measurement. Goniometer is the device which helps us in contact angle measurement. Goniometer setup in lab is shown in Figure 4.7. It captures the image of the water droplet on the sample and then with the pixel calibration technique it distinguishes triple point of contact of air, water droplet and sample. There after it calculates the angle of contact between the water droplet and the sample surface through the selected methods available like the half angle, tangent, ellipse. The setup has a manually controllable water dispensing machine with a minimum volume controllable of 0.20µl. The setup employs a regulated illumination source for contrast and accurate contact angle measurement for different colour variant of samples. It also has tilting facility for uneven and deformed sample's contact angle measurement. It has an image resolution of 744× 480 pixels. The magnification power is 5 times and frame rate of 75fps. Goniometer provided by Apex instrument boasts of accuracy upto $\pm 0.5°$.
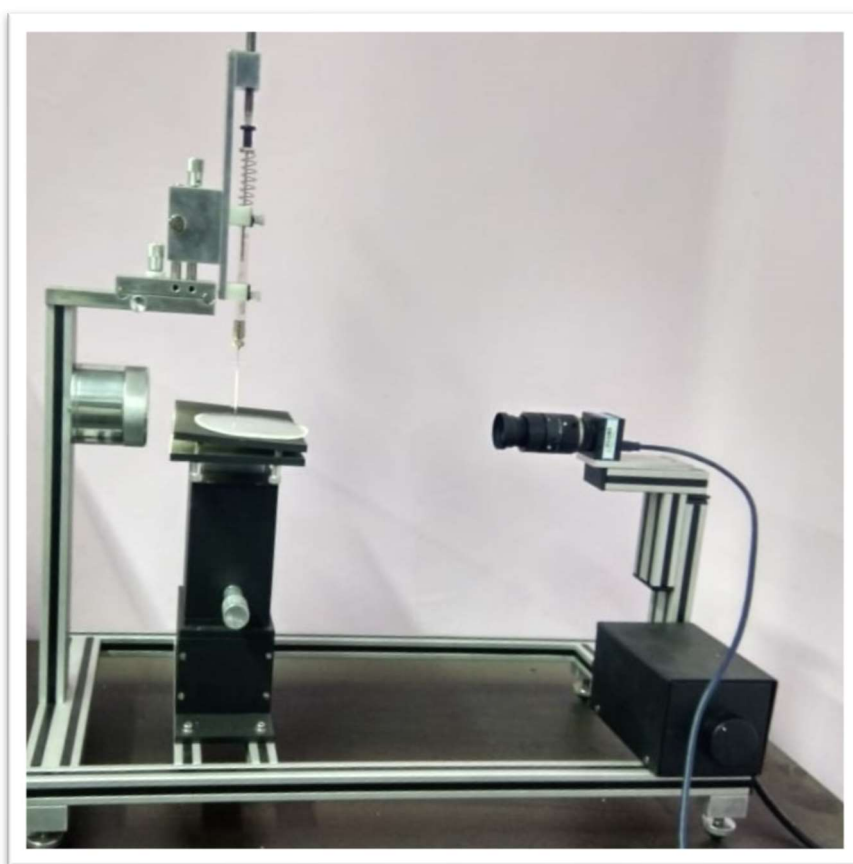
Figure 4.7: Goniometer

4.3.3. Degassing Unit

Degassing or vacuuming is a very important process in sample preparation as voids or the presence of trapped gases bubbles reduce the insulating property to a large extent. In degassing, moulds with elastomers are placed inside the degassing unit. Applying vacuum reduces the pressure inside the chamber resulting in the evolution of gases and air bubbles because by Henry's Law fall in pressure reduces the solubility of air bubbles and gases in the elastomers, resulting in their evolution. Vacuuming is achieved by attaching a pump to outlet of the chamber and sucking out air from it resulting in drop in pressure. Vacuuming is done twice during the sample preparation, once just after pouring the uncured rubber and then again after adding the filler and the catalyst. The degassing unit can attain a pressure of upto -1atm. Figure 4.8 shows the degassing unit available in the laboratory.

Figure 4.8: The Degassing unit

4.3.4. Electrode Setup

The electrode setup used for measurement of leakage current is shown in figure 4.9. It is a two-electrode setup of circular base having a diameter of 50mm. The top electrode is used as a high voltage electrode and the bottom electrode as the ground electrode. The electrodes are bevelled at the edges in order not to induce partial discharge. The electrode setup is enclosed in an acrylic glass chamber.

4.3.5. Ultrasonic Bath Sonicator

Ultrasonic bath sonicator works on the principle of applying sound energy as high frequency pressure waves to agitate particles in a sample resulting in uniform dispersion and bond formation between the nanofiller and the base polymeric matrix. It also has the advantage of having the desired temperature during the sonication. During sample preparation it was kept at room temperature. Ideally it is found that 30 minutes of sonification is enough for uniform dispersion in liquid bases. But in gel like substance or where the viscosity of the base substance is high then 1 hour of sonification is better. Figure 4.10 depicts the ultrasonic bath sonicator available in laboratory.

Figure 4.9: Electrode setup   *Figure 4.10: Ultrasonic bath sonicator*

4.3.6. Digital Storage Oscilloscope

For measurement for leakage current TEKTRONIX TBS1152 digital storage oscilloscope was used. It is used for quick and easy storage of leakage current data into the flash drive through the USB 2.0 host port on the front panel. It has a bandwidth of 150 MHz and sampling rate of upto 1GS/s and a data record length of 2500 points on both channels. Figure 4.11 shows the image of digital storage oscilloscope in use.

Figure 4.11: Digital storage oscilloscope

4.4. Sample Preparation

In case of laboratory prepared samples, the steps followed always has an impact on the dielectric and surface properties of the insulating material. So as discussed earlier in section the vital steps followed are discussed as below

1. The mixing can and the casts are thoroughly cleaned with acetone, so as not have any kind of impurity. Thereafter the circular casts are sprayed with a uniform layer of CRC silicone releasing agent and allowed to dry for 10 minutes. CRC silicone releasing agent is used to allow easy release of cured samples from the cast, without sticking to the edges.

2. The RTV silicone rubber is poured slowly into the mixing can so as not to entrap much air.

3. The measured amount of weight percentage of filler is kept in the heating chamber for 30 minutes at a constant temperature to dehumidify the nanofiller and then mixed with the RTV silicone rubber in the mixing by mechanical stirring for 15 minutes, after that the mixing can is placed in the degassing unit again for 15 minutes at a constant pressure of -1 atm.

4. As degassing results in removal of trapped air pockets the effect of uniform dispersion by the ultrasonic bath sonicator is enhanced and it placed in it at room temperature for 1 hour. There after the measured amount of catalyst (1 ml of catalyst for 30 ml of RTV silicone rubber) is added and mechanically mixed for 10 minutes and then kept in degassing unit for 15 minutes at -1 atm pressure.

5. After degassing 18.5 gm of samples was poured into each cast to maintain a uniform thickness of 3 mm of the samples. Each of the cast were again put in the degassing unit for 5 minutes to remove any trapped air while pouring of the samples.

6. The casts are kept in a protected environment lest any dust particles etc doesn't settle on the surface. The skin formation starts to take place after around 6 hrs after the samples are left undisturbed. The samples are fully cured after 24 hours and are removed from the cast.

7. It is advisable to perform test on samples after 48 hours of samples being fully cured as sample settling and internal bond formations might still be going on.

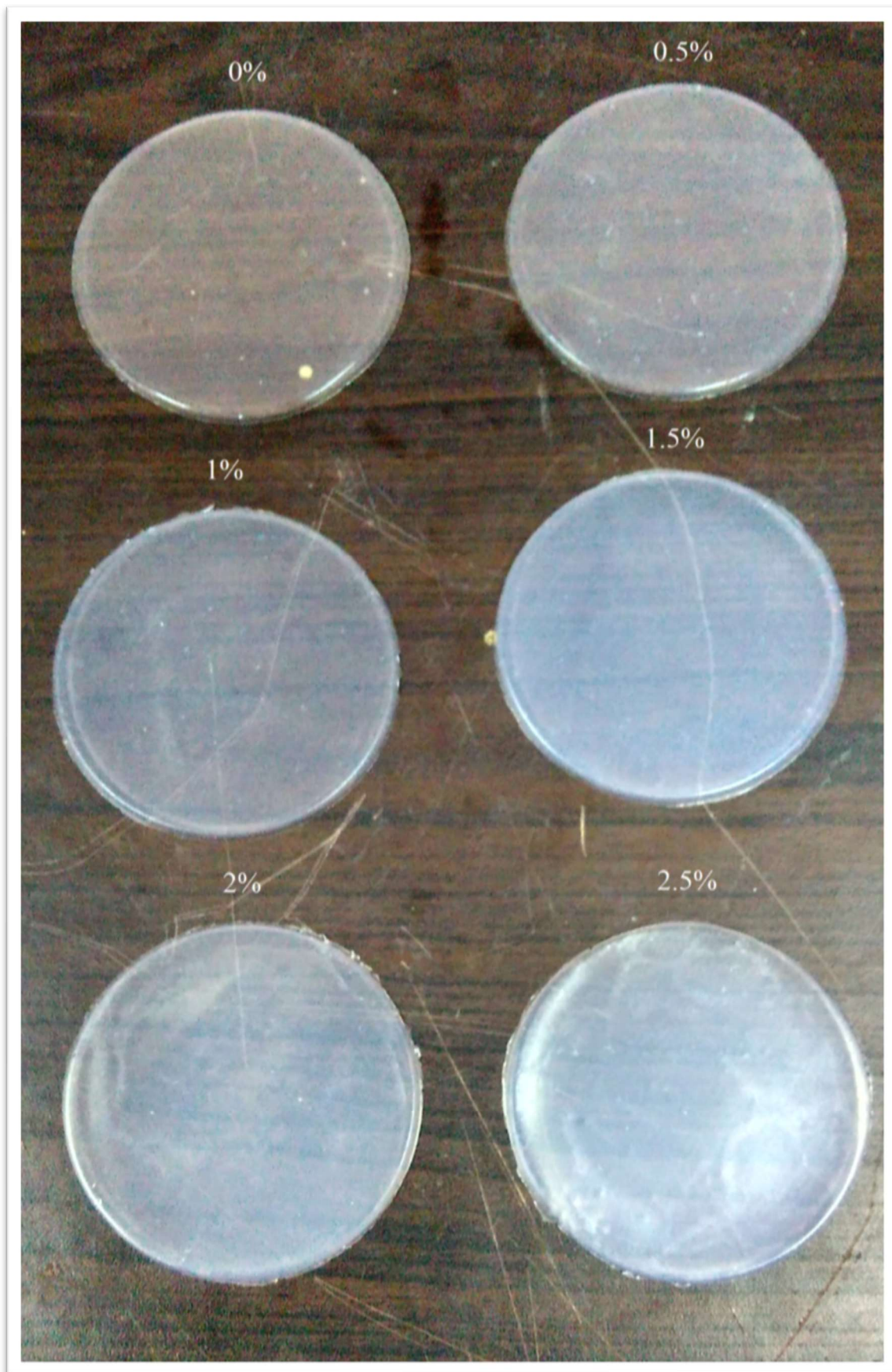Figure 4.12 shows the six samples of different filler concentration of fumed silica prepared in the lab.

Figure 4.12: RTV SiR samples with different filler concentration

4.5. Leakage Current Measurement

A schematic diagram of the experimental setup is shown in figure 4.13 and the experimental setup is as shown in Figure 4.14. The high voltage source of 0-10 kV range is connected to the top electrode of the electrode setup. The sample is placed in between the electrode with utmost precision so that the centres of the electrode and the samples are perfectly aligned lest it may result in the different length of leakage current path on different sides. The leakage current flows from the earth electrode to the current divider circuit, from which the voltage magnitude across the series resistor is fed to the Digital storage oscilloscope (DSO) measuring the LC across the samples between the electrode. The data stored in the flash drive of the DSO is transferred to the personal computer for further analysis of the LC waveform obtained. An overvoltage protection circuit is also connected across the measuring device to prevent it from any overvoltage due to accidental failure in the experimental setup.
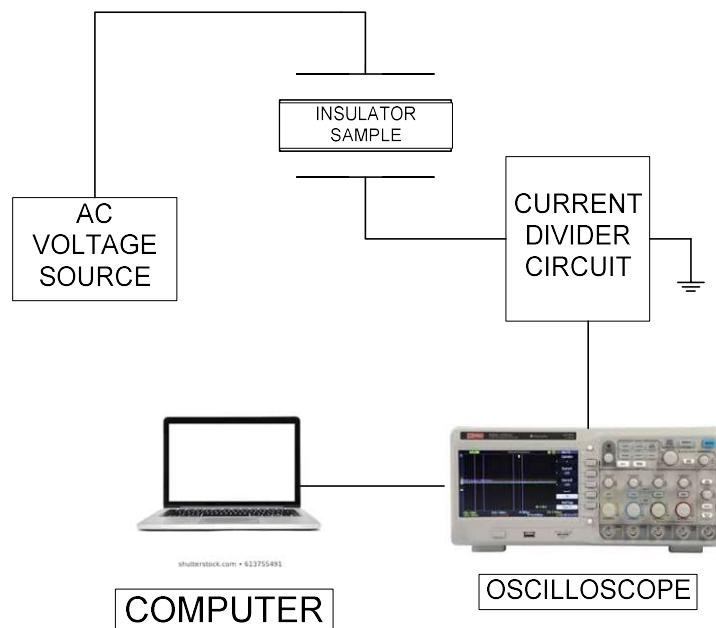

Figure 4.13: Schematic diagram of the LC measurement setup

Figure 4.14: Experimental setup for the LC measurement

4.6. Hydrophobicity Measurement

Hydrophobicity measurements were performed in the laboratory with the help of the goniometer. Figure 4.15 shows the experimental setup. Prior to measurements the surface on which contact angle measurements will be done, were thoroughly cleaned for any dust particles are not there and then the surface was discharged by placing a grounded electrode for 5 minutes so that no charge remains on the surface as contact angle measurements are severely affected by presence of charges on the surface [39].

The sample is placed on the surface and the water droplet of volume 4.97 to 5.17 µl is placed on the sample. The image is captured on by the camera and the image is analysed by a software ACAM provided by the apex instruments. Figure 4.16 depicts the image transmitted by the goniometer. The base line is fixed by the user and then the fitting method is chosen, in this case

the method was tangent method as it is most suitable for contact angle greater than 90°. The measurement shows the contact angle of the droplet as measured from the left side as well as from the right side and the final contact angle is the average of the two and the reading was exported to excel sheet. It was always ensured that there are no other particles in the vicinity of the droplet as it hampers the measurement and results in erroneous contact angle measurement. Secondly the value of contact angle very much dependent on the volume of the water droplet, so for this comparative study it was kept in the said interval. Further to remove any discrepancy or erroneous readings, average of five readings for each sample were taken.
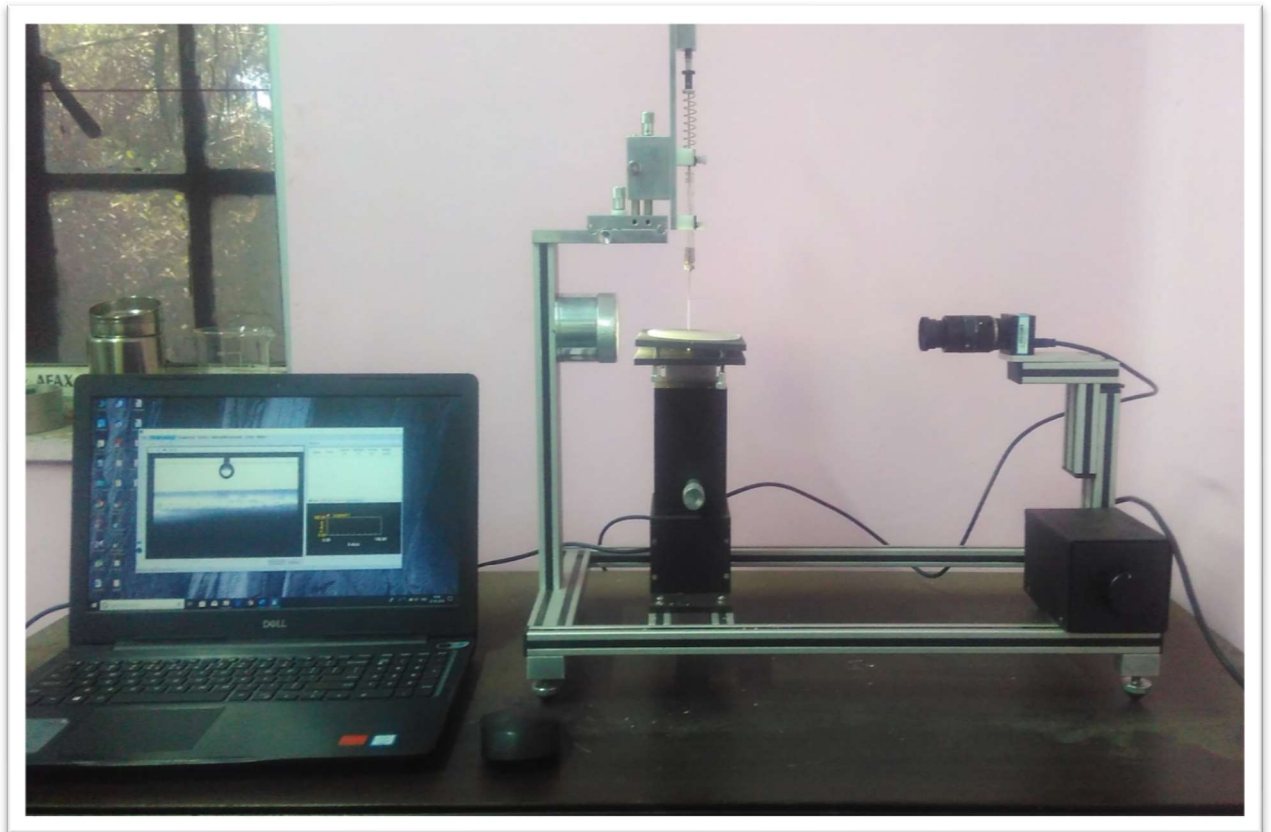

Figure 4.15: Contact angle measurement setup
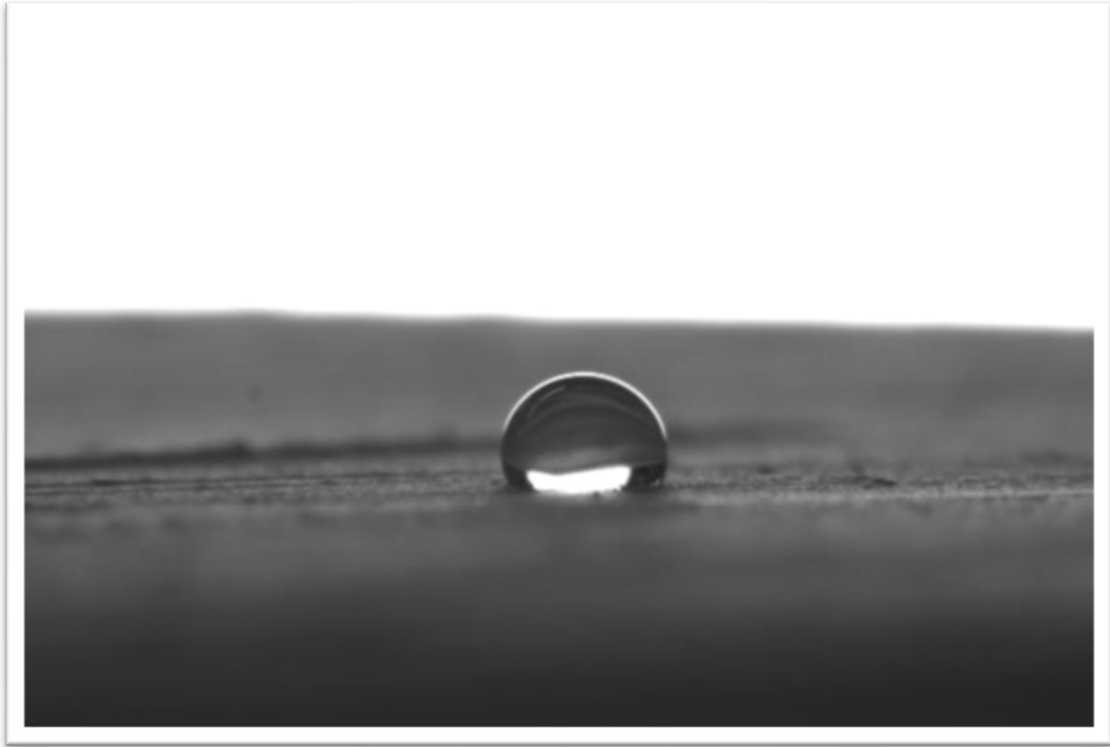
Figure 4.16: Image transmitted to ACAM software

For dynamic hydrophobicity measurements the samples were contaminated artificially as per IEC 60507 standards for contamination level of equivalent salt deposit density (ESDD) of about 0.1 mg/cm^2, and non-soluble salt deposit density (NSDD) of about 1 mg/cm^2 signifying pollution severity of medium level. The contaminated sample were kept to dry, and after 96 hours the hydrophobicity transfer was measured. For the measurement of hydrophobicity recovery, the contaminant was washed off and the samples were cleaned with acetone. The contact angle was measured again after 48 hours of removing the contaminant.

CHAPTER-5

RESULTS AND DISCUSSIONS

Chapter 5

5.1. Introduction

Silicone Rubber (SIR) insulators have been used in electrical power systems for long time mainly because of there ability to suppress leakage current (LC) even in case of heavily polluted and humid conditions. Apart from this SiR insulators are known to exhibit excellent hydrophobic properties be it static hydrophobicity or dynamic hydrophobicity. In this chapter the variation in these properties of SiR has been studied with changing silica filler concentration and the results obtained are discussed in detail. The experimental results thus obtained are broadly classified into

  I.   Analysis of leakage current waveform.

 II.   Hydrophobicity estimates.

5.2. Analysis of Leakage Current Waveform

Leakage current waveform signatures are used to judge the dielectric suitability of an insulation system. Detailed analysis of the leakage current data reveals the condition of electrical insulation system, it also depicts the behaviour of insulator with respect to the variation of voltage level and changes in filler concentration. Leakage current analysis is done with respect to

   I.   Leakage Current waveforms for different voltage level.

  II.   Variation of RMS value of Leakage current.

 III.   Harmonic content analysis of Leakage current.

5.2.1. Leakage Current Waveforms for Different Voltage Level

The leakage current plot for a voltage amplitude of 2kV across the samples of different concentration of fumed silica micro filler has been shown in Figure 5.1.
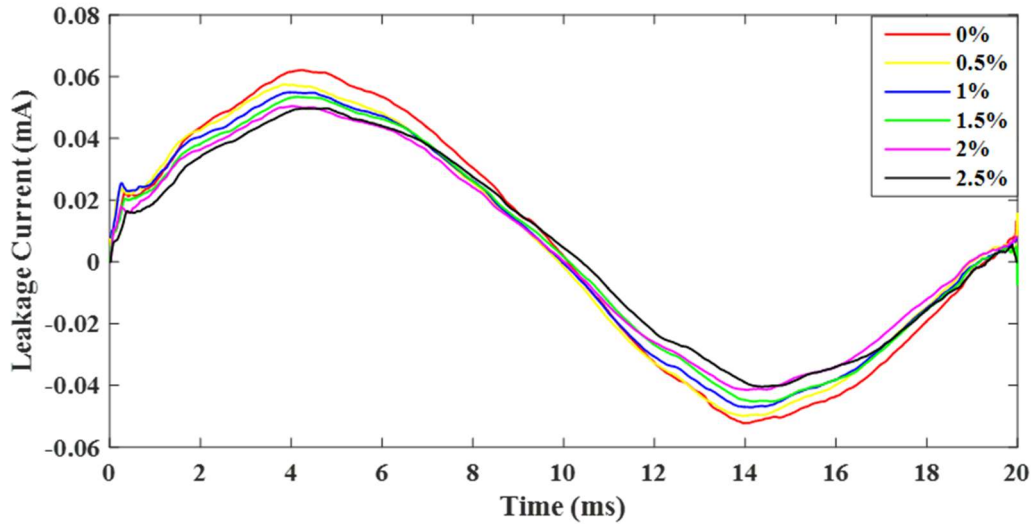
Figure 5.1: Leakage Current magnitude of samples of different filler concentration at 2kV.

Fumed silica has been used in the polymeric insulators for a long time as reinforcing filler but from the Figure 5.1 it can be seen that, as the filler concentration is increasing, the magnitude of leakage current is decreasing. So, it is clear that fumed silica also inhibits the flow of leakage current apart from imparting mechanical strength to silicone rubber samples. This point is further acknowledged from the LC plot of different samples at 3kV shown in Figure 5.2 and at 4kV shown in Figure 5.3.
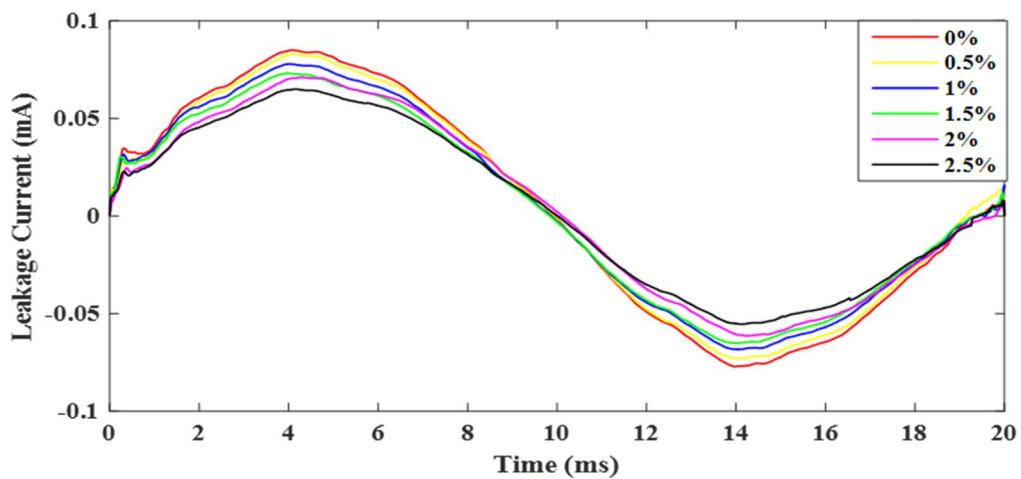
Figure 5.2: Leakage Current magnitude of samples of different filler concentration at 3kV.
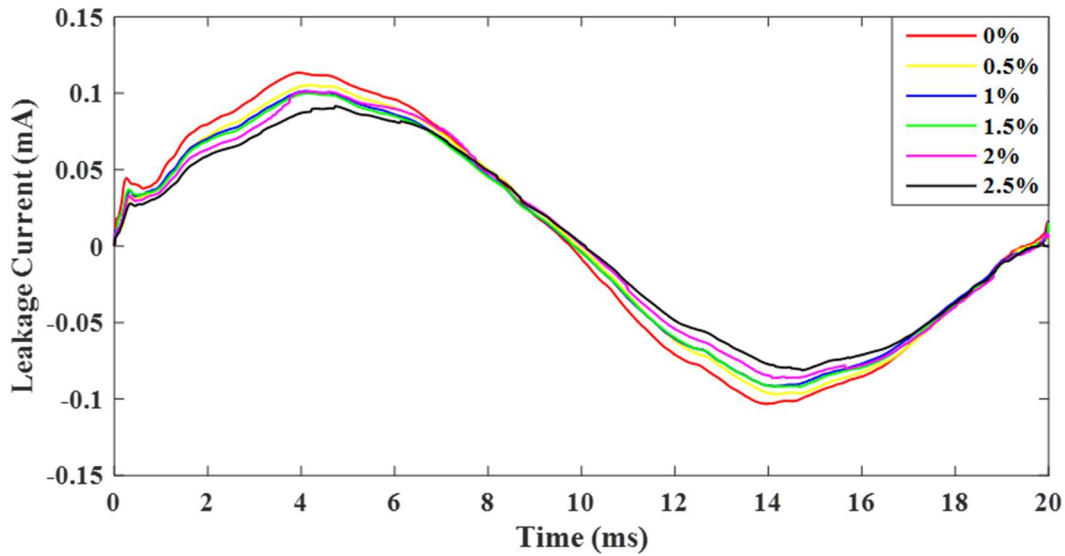
Figure 5.3: Leakage Current magnitude of samples of different filler concentration at 4kV.

5.2.2. Variation of RMS Value of Leakage Current

From Figure 5.1, 5.2 and 5.3 it can be said that, as the filler concentration is increasing the magnitude of leakage current (LC) is proportionately decreasing. Moreover, observing the root mean square (RMS) values of the LC for different filler concentration, there was a linear decreasing trend for RMS values with increasing filler concentration. The RMS value of the LC at 3kV for samples of different filler concentration is shown in Figure 5.4. Similar pattern was observed for RMS values of LC at both 3kV and 4kV.
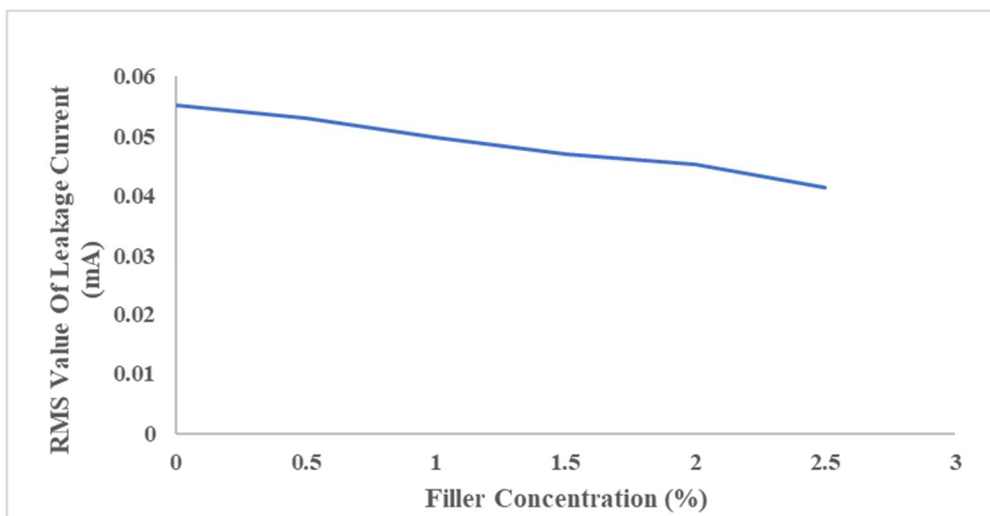
Figure 5.4: Nature of the RMS value of Leakage Current for different filler concentration.

With a clear pattern of decreasing amplitude of leakage current with increasing silica filler concentration can be attributed to the tracking resistance imparted by silica filler, since a lower magnitude of voltage is applied tracking was not observed but the increase in filler has led to a gradual decrease in the LC unlike the exponential decrease in tracking.

5.2.3. Harmonic Content Analysis of Leakage Current

Fourier Transform is performed on the leakage current data obtained for the samples at three different voltage level. The results obtained from the Fast Fourier Transform (FFT) of LC at 2kV is shown in Figure 5.5, it can be observed that the increase in the fumed silica filler concentration leads to decrease in LC harmonic components and it is also justified by Table 5.1 which shows the values of odd harmonics up to 9th harmonics.
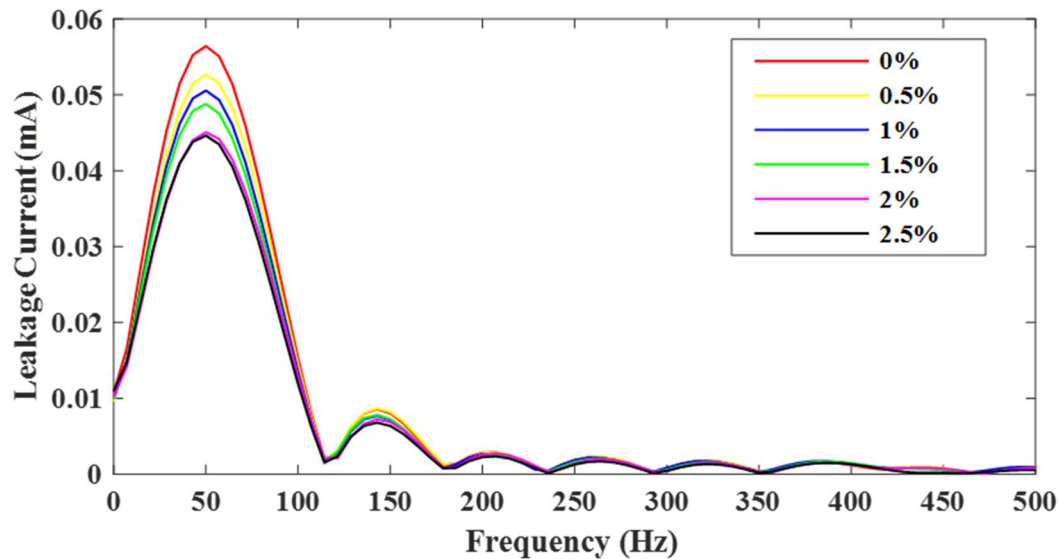

Figure 5.5:FFT of the Leakage Current data at 2kV for different filler concentrations.

Table 5.1: Variation in harmonic content of Leakage Current with increase in filler percentage at 2kV.

Harmonic order	0%	0.5%	1%	1.5%	2%	2.5%
fundamental	0.056429	0.052689	0.050569	0.048803	0.045074	0.0446306
3rd harmonic	0.008198	0.008025	0.007268	0.007129	0.006864	0.0063562
5th harmonic	0.00187	0.001849	0.0018	0.00172	0.001395	0.0012753
7th harmonic	0.000832	0.000741	0.00066	0.000573	0.000561	0.0001813
9th harmonic	0.000313	0.000332	0.00033	0.000289	0.000234	0.0001578

Table 5.2: Variation in harmonic content of Leakage Current with increase in filler percentage at 3kV.

Harmonic order	0%	0.5%	1%	1.5%	2%	2.5%
fundamental	0.080369	0.076761	0.072463	0.068441	0.065793	0.0603031
3rd harmonic	0.011003	0.010971	0.009926	0.009472	0.008451	0.0076407
5th harmonic	0.002669	0.002503	0.002269	0.002155	0.00208	0.0019523
7th harmonic	0.000955	0.000876	0.000779	0.000694	0.000687	0.0004674
9th harmonic	0.000382	0.000299	0.000291	0.000285	0.000262	0.0000845

Table 5.3: Variation in harmonic content of Leakage Current with increase in filler percentage at 4kV.

Harmonic order	0	0.5	1	1.5	2	2.5
fundamental	0.107429	0.100171	0.095665	0.095625	0.093522	0.0859014
3rd harmonic	0.013742	0.013665	0.012334	0.01223	0.011187	0.0099755
5th harmonic	0.003121	0.003057	0.002683	0.002677	0.002572	0.0024271
7th harmonic	0.00116	0.001019	0.00079	0.000753	0.000696	0.0003116
9th harmonic	0.000666	0.000393	0.00035	0.000341	0.000187	0.0001487

From Table 5.1, 5.2 and 5.3 it can be seen that with increasing filler concentration there is a significant decrease in fundamental harmonic content. The other odd harmonics components are also decreasing. These observations are in perfect tandem with observation from the leakage current plot. The values of 3rd harmonics are less than 20% of the fundamental harmonics, so it can be safely assumed that dry band formation has not yet started taking place [17]. From the decreasing nature of the 3rd, 5th and 7th harmonic content, it can be asserted that the electrical stress is decreasing. 5th harmonic too is decreasing, increase of which is seen as a sign of the development of surface discharge. Analysis of 3rd harmonic values of the leakage current data are of utmost importance in the investigation of the insulator properties because they give a greater insight into the condition of the insulator. Studies on the condition monitoring of insulator has revealed that the leakage current across the insulators are mostly resistive barring the initial low magnitude leakage current which is of capacitive in nature. It is further found that, the increase in the resistive component of the leakage current is proportionate to the 3rd harmonic. Due to these reasons, 3rd harmonic variations have special significance. The ratio of 3rd harmonic to fundamental was calculated for all the readings of

different samples and the average value obtained for the ratio at different voltage level is shown in Table 5.4. A graphical representation of the average value of 3rd to fundamental for different voltage level is shown in Figure 5.6.

Table 5.4: Variations in the ratio of 3rd harmonic to the fundamental component of leakage current.

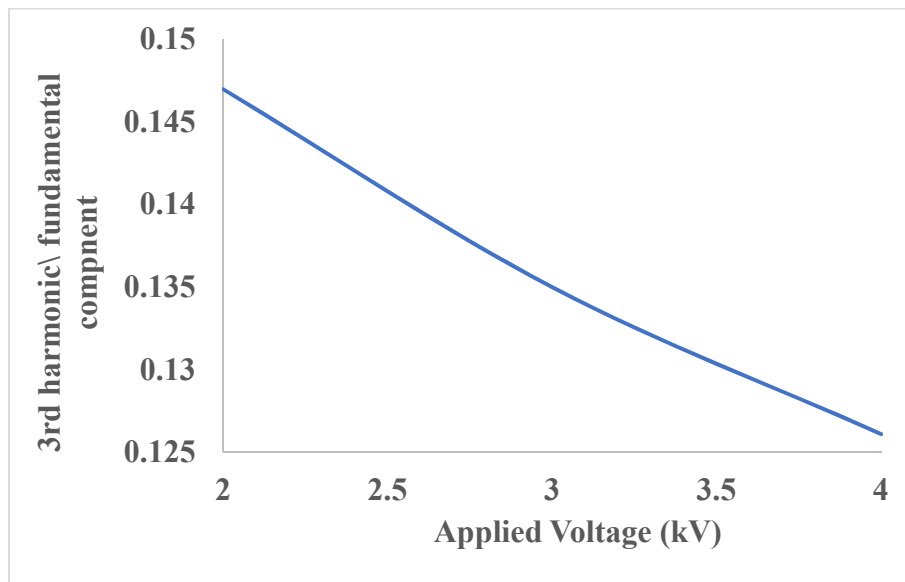| Voltage	0%	0.5%	1%	1.5%	2%	2.5%	Average
2kV	0.145279	0.152306	0.143722	0.146076	0.152274	0.1424173	0.147
3kV	0.13691	0.142918	0.136983	0.138393	0.128451	0.1267047	0.135
4kV	0.127917	0.136418	0.128924	0.127894	0.119618	0.1161269	0.1261

Figure 5.6: Variation of average value of 3rd harmonic to fundamental component with different voltage level.

From the Figure 5.6 it can be seen that with increasing magnitude of voltage level the ratio of 3rd harmonic to fundamental component is decreasing linearly which is an excellent positive sign. As explained earlier that monitoring of 3rd harmonic content of Leakage Current is of prime importance and a decreasing nature of it (ratio of 3rd harmonic to fundamental) means its resistance to flow leakage current is increasing with increase in voltage magnitude. As can be seen from Table 5.4 that the decrease in percentage component of 3rd harmonic to fundamental is uniformly present across the table irrespective of the filler concentration, so it can be attributed to the SiR's inherent property which is intact even with increasing silica filler concentration, while on a closer study it can be said that this property is being further enhanced on addition of filler.

5.3. Hydrophobicity Estimates

Hydrophobicity being one of the important properties of polymeric insulator more so in case of Silicone Rubber insulator has been measured using a goniometer and its variation with filler concentration has been explained. For each percentage of filler concentration 3 samples were taken and on each sample 5 readings of contact angle were taken in a controlled environment, the average of these readings was taken for analysis. The static and dynamic hydrophobicity measurements were taken:

    I.   Contact angle for different filler concentration.
   II.   Hydrophobicity measurements of contaminated sample.
 III.   Mechanism of hydrophobicity transfer and recovery.

5.3.1. Contact Angle for Different Filler Concentration

Figure 5.7 shows the variation of raw (uncontaminated) state contact angle, for SiR samples with different concentration of silica fillers.
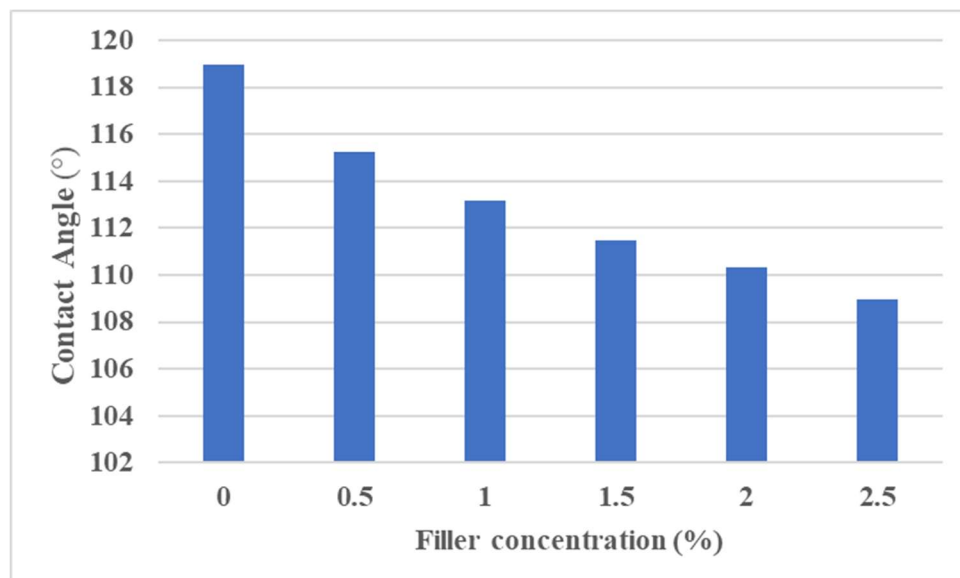

Figure 5.1: Variation in contact angle with increase in filler concentration.

It can be inferred from Figure 5.7 that an increase in the filler concentration decreases the contact angle thereby decreasing the hydrophobicity. It can also be seen from the bar-graph that with the addition of 0.5% of filler to a pure sample results in a steep decrease in the contact angle and thereafter with increment in filler concentration there is a gradual decrease in the contact angle. The reason behind this is attributed to the fact that silicone rubber has base polymer of poly dimethyl siloxane (PDMS) which is a low surface energy material and as explained earlier that a low surface energy substance exhibits excellent hydrophobicity that is it has very high contact angle with water droplets [42]. Fumed silica is an inorganic filler and has high surface energy resulting in the decrease in hydrophobicity of silicone rubber samples with increasing filler concentration.

5.3.2. Hydrophobicity Measurements of Contaminated Sample

Table 5.4 shows the different contact angles measurements of dynamic hydrophobicity and their variation with increasing filler. For further analysis a graphical representation is provided on the effect of fumed silica filler in silicone rubber insulator's hydrophobicity, by measuring the hydrophobicity transfer to the surface of contaminated silicone rubber insulator and hydrophobicity recovery in the event of removal of the contaminant is shown in the Figure 5.8. It can be seen that with increase in filler percentage there is an increase in the percentage of hydrophobicity transfer and hydrophobicity recovery.

Table 5.4: Contact Angle at different stages of hydrophobicity measurements

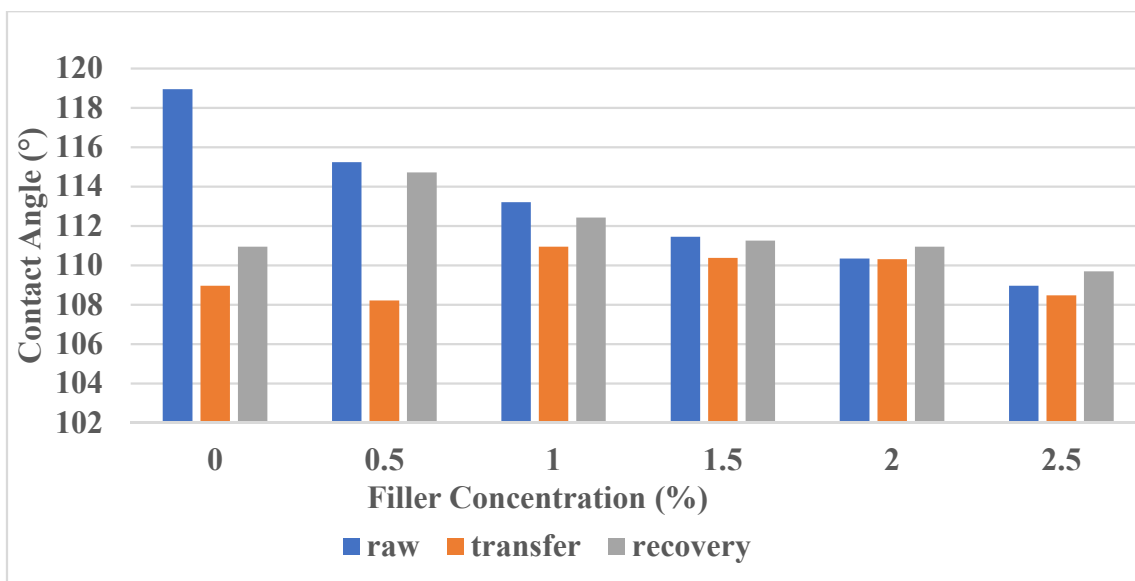| Filler (%)	Raw hydrophobicity (degree)	Hydrophobicity Transfer (degree)	Hydrophobicity Recovery (degree)
0	118.9522	108.9552	110.94
0.5	115.2376	108.2163	114.7167
1	113.1967	110.9435	112.4304
1.5	111.4552	110.3684	111.2593
2	110.3464	110.3108	110.9435
2.5	108.9552	108.4769	109.6984

Figure 5.2: Variation in hydrophobicity transfer and hydrophobicity recovery with increase in filler concentration.

Table 5.5: Percentage of hydrophobicity Transfer and Recovery.

Filler (%)	Hydrophobicity Transfer (%)	Hydrophobicity Recovery (%)
0	91.59578	93.26435
0.5	93.90707	99.64798
1	98.00948	99.32304
1.5	99.0249	99.82419
2	99.96774	100.5411
2.5	99.56101	100.6821

5.3.3. Mechanism of Hydrophobicity Transfer and Recovery

Before going on to analyse the data presented on dynamic hydrophobicity measurements by the Figure 5.8 or more specifically Table 5.5, the understanding of mechanism of hydrophobicity transfer and recovery is very important. Basically, hydrophobicity of any substance is because of it having a low surface energy than the surface energy of water droplet, so that the water does not settle on the surface or form continuous film rather is in discrete drops. This property is exhibited by silicone rubber. The presence of methyl group in the poly

dimethyl siloxane is main reason for silicone rubber possessing the hydrophobic property as methyl groups are low energy molecules, hence restricts water filming on the surface. Hydrophobicity or water repellency is also imparted because of the presence of low molecular weight (LMW) compounds in silicone rubber. The more is the LMW on the surface of a material, the larger is its hydrophobicity and vice versa [42].

Loss of hydrophobicity in case of silicone rubber on being exposed to contaminated environment is mainly due to the reorientation of the methyl group away from the surface, and also due to the oxidation of PDMS resulting in formation of hydrophilic silanol groups rendering the surface hydrophilic. Hydrophobicity loss occurs in case of dry band arcing, corona discharging and environmental pollution also.

Hydrophobicity transfer is a phenomenon exhibited in the face of persistent environmental condition in which the hydrophobicity is transferred to surface of the pollutant layer. In this process LMWs play a much important role than the transfer of movement of methyl groups. The LMWs diffuse from the bulk of the insulator to surface of the insulator all the while following the gradient as the concentration is more in the bulk of the insulator than that on the surface. In case of long exposure to polluted environmental condition the LMWs are lost from the surface or the chains are broken, in this case the silica filler plays a two pronged role of forming new chains of LMWs and also attracting the oxidised groups away from the surface towards the bulk of the material resulting in increased hydrophobicity transfer with increasing filler.

Hydrophobicity recovery an important characteristic of silicone rubber which occurs after the removal of the polluted environmental condition or the electrical stress to which it was subjected, is because of the reorientation of the hydrophobic methyl group towards the surface and the hydrophilic oxidised groups away from the surface, and to some extent recovery also takes place because of the LMWs diffusion towards the surface. Presence of silica filler (SiO_2) results in the development of inter-molecular forces that attract the oxidised groups of silanol away from the surface, which justifies the increased percentage of hydrophobicity recovery with increase in filler concentration.

CHAPTER-6

CONCLUSIONS AND FUTURE

WORK

Chapter 6

6.1. Conclusion

Room temperature vulcanised (RTV) silicone rubber (SiR) samples with varying filler concentration of fumed silica nanofillers were successfully prepared in the laboratory. The samples prepared were used to investigate the variation in the leakage current (LC) waveform and the changes in hydrophobicity with different concentration of the silica nanofiller.

While investigating the leakage current waveform for samples of SiR with different silica filler concentration, the important findings are, that the increase in filler concentration leads to a consequent decrease in leakage current magnitude which is further substantiated by the measure of RMS value of the leakage current, that showed linear decreasing nature with increasing filler concentration. For further analysis, Fourier Transform was performed on the obtained LC data which revealed that the odd harmonics (namely the fundamental, 3rd, 5th, 7th and 9th) all showed decreasing trends which means that the increase in filler concentration suppresses the formation of dry band also, apart from decreasing LC magnitude. Another interesting point that was noted is that the average value of 3rd to fundamental component ratio was decreasing with the increasing magnitude of voltage level, which is an excellent positive sign that the leakage current suppressing capability increase with increasing voltage magnitude. So overall it can be concluded from the LC analysis that silica filler not only acts as a reinforcing filler but also suppress the LC current and increase in filler concentration yields better results, in terms of LC suppression.

The hydrophobicity estimates of SiR samples also gave positive results on addition of silica nanofiller, though the initial hydrophobicity measurements or the raw hydrophobicity measured after 96 hrs of sample preparation showed that the increasing filler concentration resulted in decrease in the contact angle measured reason being the silica, an inorganic filler which has high surface energy, hence permits surface wetting. The dynamic hydrophobic properties were also investigated for the samples by contaminating it with medium level of pollution as per IEC 60507 standards and after 96 hrs the hydrophobicity transfer was measured. It was noted that the increase in filler led to an increase in the percentage of hydrophobicity transfer to the surface of contaminant, which can be attributed to increase in silica filler for forming new chains of low molecular weight (LMW) compounds and for

attracting oxides away from the surface towards the bulk. After 48hrs of removal of the contaminant, the hydrophobicity recovery was measured which also showed an increase percentage of recovery with increasing filler concentration. Increased hydrophobicity recovery is mainly due to the reorientation of methyl groups towards the surface of the insulator and the oxidised groups away from the surface which is propelled due to the presence of silica fillers as they develop intermolecular forces which attract the oxidised group towards the bulk. So overall it can be said that addition of fumed silica nanofillers suppresses the LC development and enhances the dynamic hydrophobicity of room temperature vulcanised silicone rubber.

6.2. Future Work

In this study the focus was primarily on the effect of fumed silica nanofiller on RTV SiR, and it has shown good enhancement of leakage current suppression and hydrophobic properties but further research needs to be carried out to ascertain its suitability under different environmental conditions. Further research work on this can be considered on these topics enlisted below

1. Tracking and erosion test, being an important measure of the performance of outdoor insulators, can be performed and effect of silica filler on tracking and erosion resistance can be studied in detail.
2. Effect of temperature variation on sample's dynamic hydrophobicity properties with different filler concentration could be a subject of further study.
3. A study on the combined effect of aluminium trihydrate (a very commonly used filler for anti-tracking properties) and different concentrations of silica filler on the LC suppression and hydrophobic property enhancements.

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