Study on High Voltage Polymer Insulator with Nano-Titanium Filler

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Abstract
High voltage polymer insulator material such as SiR/EPDM composite has become one of the important studies recently. The researchers discovered that SiR/EPDM with the formulation of 50:50 weight percentages ratio had yielded the optimal dielectric and mechanical strength. However, the studies on these balanced compositions with the combination of nano-filler are still few and not yet fully explored. In this paper, SiR/EPDM composite was prepared with various loading concentration TiO_2 nano-filler. The loading concentration of TiO_2 nano-filler is 1 Vol%, 2 Vol%, 3 Vol%, 4 Vol% and 5 Vol%. The nano-composite samples were prepared via heated two rolls mill, MDR and hot press machines. The dielectric strength of nano-composites was analysed with 2-Parameter Weibull distribution. The experimental result revealed that the dielectric and tensile strength of SiR/EPDM containing 1 Vol% TiO_2 nano-filler gives the highest dielectric and tensile strength of 33.01 kV/mm and 10.52 MPa as compared with other loading concentration. Therefore, the inclusion of 1 Vol% TiO2 nano-filler into SiR/EPDM was significantly has enhanced the dielectric and tensile strength of SiR/EPDM composite.

Keywords: silicone rubber (SiR), ethylene propylene diene monomer (EPDM), nano-titanium (TiO2)

1.0 Introduction

Several types of insulators have been used in high voltage overhead power lines. The older weather shed insulators were fabricated from ceramic and glass material. Since the 1960s polymeric material has been used, since if offers several advantages, e.g., higher breakdown voltage and tensile strength, reduced weight, smaller size, less frequent cleaning requirement, and lower cost (Hackam, 1999). Silicone Rubber (SiR) has been extensively used because of its excellent electrical properties, e.g., high dielectric strength and volume resistivity. However, it suffers from poor mechanical strength and low tracking resistance, and is expensive. Ethylene propylene diene monomer (EPDM) has excellent resistance to tracking and erosion, and higher mechanical strength than SiR, but suffers from low volume and surface

resistivity compared with SiR (Deepalaxmi, Balaji, & Rajini, 2013; Ehsani et al., 2004; Fairus, Mansor, Hafiz, Mariatti, & Kamarol, 2015; Rajini & Deepalaxmi, 2012; Vijayalekshmi & Abdul Majeed, 2013).

A SiR/EPDM composite has the potential to combine the outstanding electrical properties of SiR with the excellent mechanical properties of EPDM at low cost (Deepalaxmi et al., 2013; Rajini & Deepalaxmi, 2012; Vijayalekshmi & Abdul Majeed, 2013). Prabu et al. reported that a 50:50 weight percentage SiR/EPDM composite yields optimal electrical and mechanical properties (Prabu, Usa, Udayakumar, Khan, & Majeed, 2007; Rajini & Deepalaxmi, 2012); if the EPDM content is increased above 50 wt% in order to improve the mechanical properties of the composite, its electrical properties are degraded. This research group also investigated the effects of adding fillers such as Silica (SiO_2) and Alumina Trihydrate (ATH) to the composite, and found improvements in dielectric strength, volume and surface resistivity, arc resistance, tracking resistance and tensile strength (Prabu et al., 2007). More recently, Vijayalekshmi and Majeed reported that a 50:50 wt% of SiR/EPDM composite containing an organically modified montmorillonite (OMMT) nano-clay showed improved mechanical, thermal and electrical properties compared to the SiR/EPDM host (Vijayalekshmi & Abdul Majeed, 2013).

In this article, the adding of nano-titanium (TiO_2) filler to a 50:50 wt% SiR/EPDM composite more effect on electrical and mechanical properties such as dielectric and tensile strength. The TiO_2 nano-filler was chosen because of its superior material characteristics, e.g., high surface hydrophobicity, relative permittivity, and thermal conductivity (Fairus, Ha, Mariatti, & Kamarol, 2017; Huang, Jiang, & Tanaka, 2011). The new nano-composites were arranged into several nano-TiO_2 loading concentration from 0 until 5 Vol%.

2.0 Methodology

2.1 Material Specification

The nano-composite was produced with a mixture of four different types of raw materials. The materials are EPDM, SiR, DCP and TiO_2. The general specifications of the materials are as follows. The EPDM polymer with grade Kelton® 2070P with a density of 0.86 g/cm^3 was obtained from LANXESS Deutschland GmbH Company. Meanwhile, the SiR polymer with grade Elastosil® R401/60 with a density of 1.15 g/cm^3 was supplied by Wacker Chemie chemical company. The DCP with 98% active type was delivered by Bayen (M) Sdn. Bhd. Lastly, the TiO_2 nano-filler was obtained from Sigma Aldrich (M) Sdn. Bhd which have a 21 nm primary filler size, 99.5% trace metals basis, and density of 4 g/cm^3. All these materials are commercially available in the local market and used without any further material treatment

2.2 Sample Preparation Process

The composition formulations of nano-composite on SiR/EPDM with and without nano-TiO_2 fillers were tabulated in Table 1. The nano-composite was prepared with a mixture of polymer and nano-filler materials in heated two-roll mill machine. The rear and front roller two-roll machine speeds were

set at 14.6 rpm and 12.2 rpm respectively. The operating temperature is 80°C. Prior to the compounding process, the two-roll mill was warmed up for 15 minutes. The blending cycle was started by adding SiR, EPDM, and nano-filler for 30 minutes. Vulcanizing agent Dicumyl peroxide of 98% active was mixed at the final stage for 10 minutes. Face mask and glove are used during the process in order to avoid the inhaling of nano-fillers and DCP.

Table 1: SiR/EPDM composition with and without nano-TiO_2 fillers

Formulation	Concentration	SiR	EPDM	DCP
	Vol%	Wt%	Wt%	Phr
UnF SiR/EPDM	0	50	50	2
SiR/EPDM filled with TiO_2 nano-filler	1	50	50	2
	2	50	50	2
	3	50	50	2
	4	50	50	2
	5	50	50	2

Next, a piece of uncured nano-composite sample around 4 grams of each total completed nano-composites were taken to determine the composite optimal cured time (t90) via Moving Die Rheometer machine (MDR Alpha 2000). The MDR machine was set 30 minutes testing with reference tested temperature at 150°C. The torque of the MDR machine was set at 50 dNm. Table 2 shows the optimal cure time (T90) result for each nano-composite before proceed to the compression stage.

Table 2: The optimal cure time result (T90) for each nano-composition

Formulation	Concentration	Optimal Cure Time
	Vol%	T90 (min)
UnF SiR/EPDM	0	18.75
SiR/EPDM filled with TiO_2 nano-filler	1	21.22
	2	21.45
	3	21.68
	4	21.86
	5	22.01

Later, the programmable hydraulic hot press machine was set at 1000 psi with temperature 150°C before proceed with a compression process. The uncured nano-composite from various loading concentration of TiO_2 was inserted into the flat sheet mould and compressed by referring to obtain optimal cure time (t90). The dielectric strength samples were formed into the rectangle flat sheet mould, with length, width and thickness sizes of 190 mm, 110 mm, and 1 mm. Meanwhile, the tensile strength specimens were formed

into a square flat sheet mould with the dimension of length, width and thickness are 250 mm, 250 mm and 2 mm.

The sample mould was carefully removed from the machine by wearing heat resistance glove. The sample was left at the room temperature for 10 minute to cool down before punch out from the mould. Then, each loading concentration of the cured samples were cut into 10 and 5 pieces before proceed to dielectric and tensile strength measurement.

2.3 Dielectric Strength Measurements

A short time (rapid rise) test method was selected as a mode of increased of voltage for dielectric strength evaluation. An a.c voltage with rising rate of 500 V/s was applied to each batch of sample until breakdown occurs. The a.c voltage was supplied by the 50 Hz HV transformer. The specimens were placed in a test cell between plane to plane stainless steel electrodes. The specimens were immersed in Hyrax mineral insulating oil to avoid electrical flashover during testing. The circuit diagram for dielectric strength measurement is illustrated in Figure 1.

Resistor of 10 MΩ was connected in series with secondary side of the transformer to limit the LC flowing into the system. The capacitive divider was connected in parallel with test cell to measure the applied voltage. The breakdown voltage was measured by ac digital volt meter via capacitive divider. Ten samples of same composition batches were tested for breakdown voltage, *VB* (kV). The test has been carried out at room temperature according to the IEC-60243-1 standard (IEC 60243-1:1998, 1999). The dielectric strength, *EBD* (kV/mm) was calculated from *VB/d*, where d is the thickness of the sample in millimetres. The dielectric strengths obtained from this measurement were presented in Weibull plots for further analysis. Figure 2 shows the drawing of plane to plane electrodes arrangement perpendicular aligned with the sample to the electrode surface.

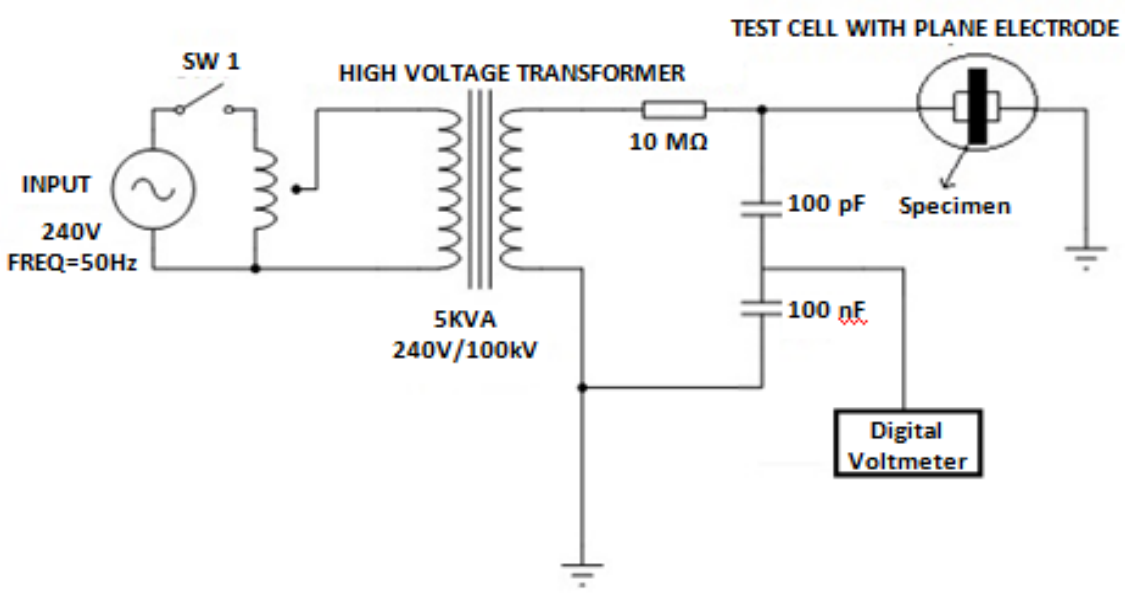


Figure 1: Dielectric strength measurement circuit diagram

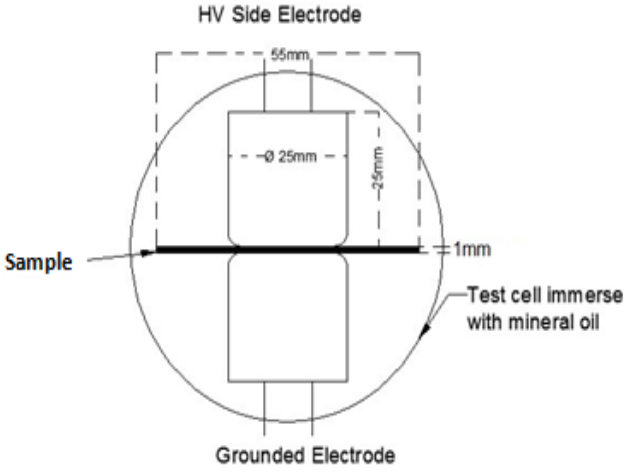
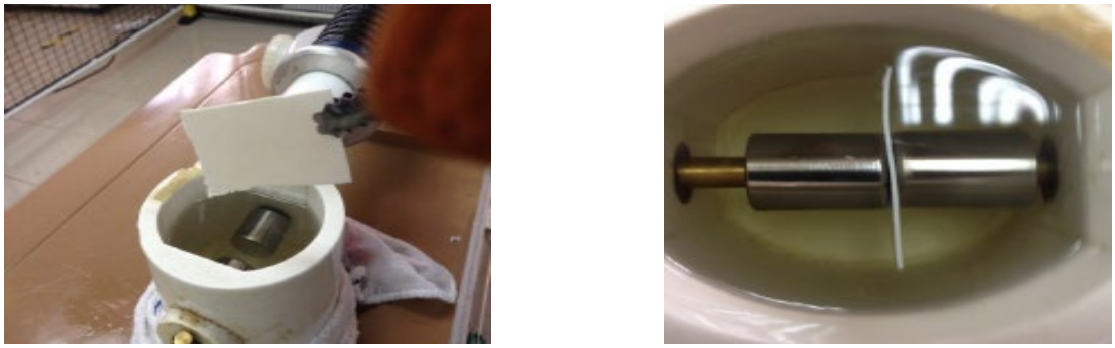


Figure 2: Plane-plane electrodes arrangement with the position of the specimen

The photographs of the specimen were inserted into the ceramic test cup and completely immersed with mineral oil are also shown in Figure 3. Figure 4 shows the photograph of the arrangement apparatus for the AC breakdown strength test.



(a) Specimen inserted into test cell (b) Specimen and electrodes completely immersed with mineral oil

Figure 3: The photographs of the ceramic test cell filled with mineral oil.

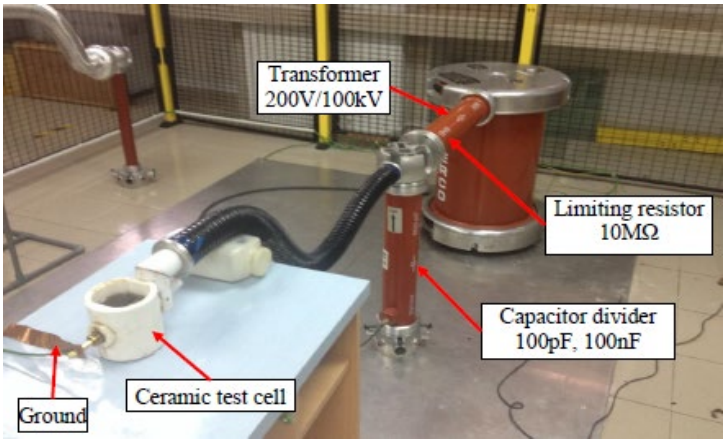


Figure 4: The arrangement of the apparatus for AC breakdown strength test

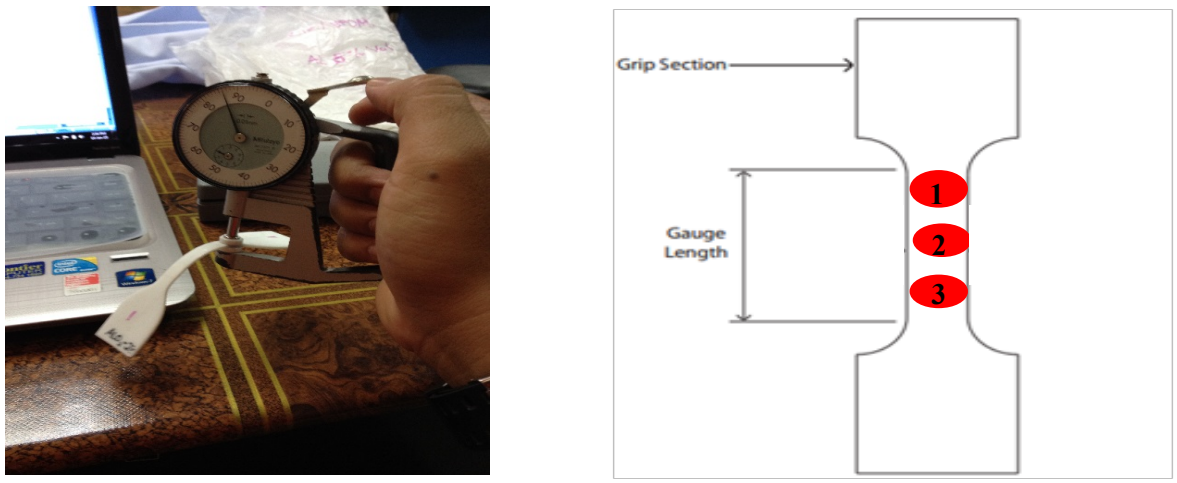
2.4 Tensile Strength Measurement

The dumbbell specimens shape and dimension were formed by the standard dies according to ASTM D412-06 standard. The photograph of the dumbbell specimen preparation is shown in Figure 5. The thickness of dumbbell specimens was measured at three different points along the gauge length of the specimen as shown in Figure 6. The obtained points from the specimen thickness were averaged and recorded for input parameter of the testing machine. Tensile strength measurement was carried out by using Instron 3360 Universal Testing Machine (UTM) at room temperature as shown in Figure 7. The machine crosshead speed was set at 500 mm/min. Dumbbell specimens must be properly clamped between two grippers before testing begins as shown in Figure 8. Five specimens of each nano-composite were measured and the averaged result is obtained accordance to ASTM D412-06 standard.

(a) Cutting process

(b) Dumbbell specimen after cutting

Figure 5: The dumbbell specimen preparation



(a) Thickness gauge measurement

(b) Measure 3 different points along gauge length

Figure 6: Thickness measurement on the tensile dumbbell specimen

Figure 7: Instron 3360 universal testing machine (UTM)

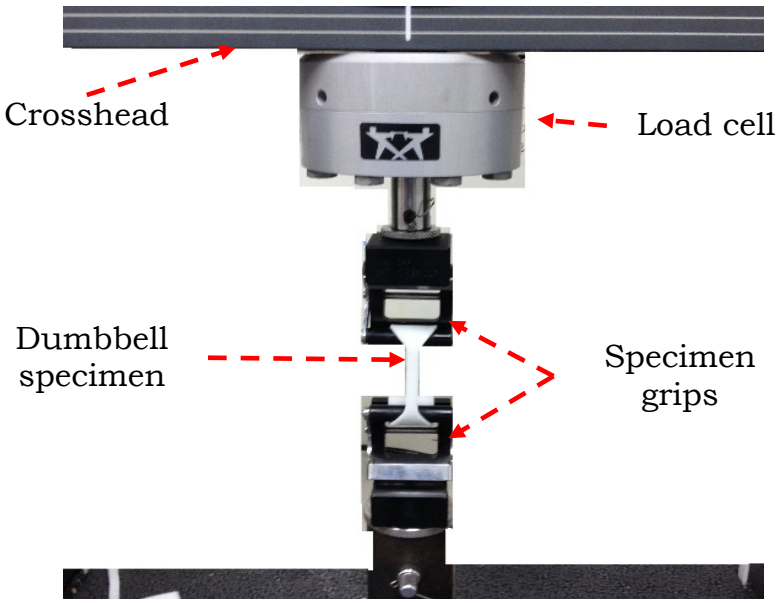
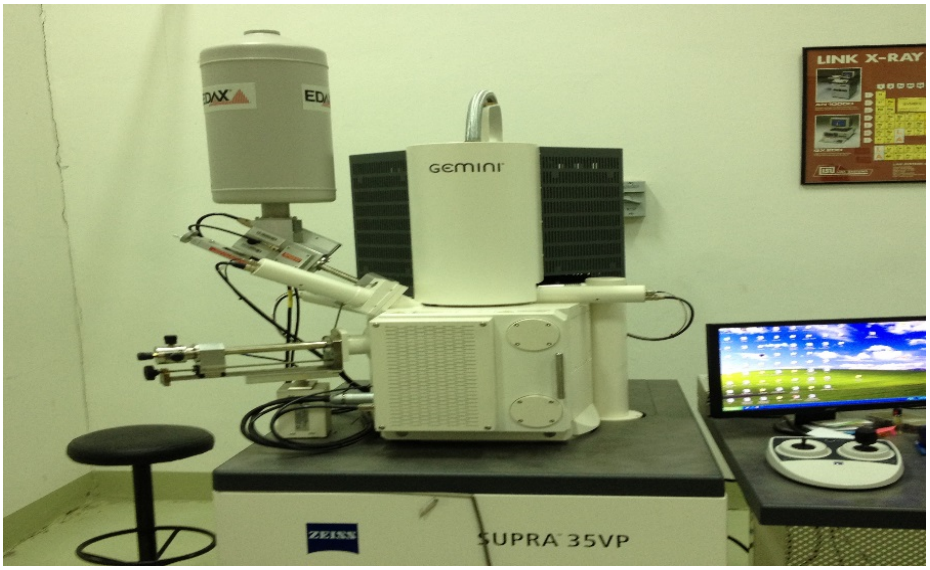


Figure 8: Photograph of tensile dumbbell specimen position

2.5 Scanning Electron Microscope (SEM)

The dispersion of the TiO_2 nano-filler in SiR/EPDM composites was examined using a field emission scanning electron microscope (SEM) (ZEISS SUPRA 35 VP). Five samples with the dimension of length, width and thickness are 1 mm, 1 mm, and 1 mm from dielectric strength sample (one sample of each composition) were immersed in liquid nitrogen and fractured to reveal the internal structures. The samples were mounted on stubs using double-sided conductive tape and inserted into a sputter coater machine model Polaron SC 515. The fracture surfaces were coated with gold/ palladium layers to avoid electrostatic charging during SEM. The photographs of the equipment are shown in Figure 9 (a) and (b).

(a) Sputter coater model Polaron SC 515



(b) FE-SEM model ZIESS SUPRA 35 VP

Figure 9: Photograph of sputter coater and SEM machines.

3.0 Results & Discussion

The results of breakdown strength of SiR/EPDM filled with various loading concentration TiO_2 nano-fillers were analysed using a 2-parameter Weibull model. The relevant equation is expressed as follow Eq. 1 (Hamzah, Jaafar, & Mohd Jamil, 2014):

$$P(E_{BD}) = 1 - exp\left[-\left(\frac{E_{BD}}{\alpha}\right)^{\beta}\right] \qquad \text{(Eq.1)}$$

Where, E_{BD} is the measured breakdown strength, $P(E_{BD})$ is the cumulative probability of breakdown strength, α is a scale parameter equal to

the breakdown strength at 63.2% failure probability of breakdown. The shape parameter β is related to the width of the breakdown distribution. The values of α and β for the UnF SiR/EPDM composite and various loading concentrations of TiO_2 nano-filler in SiR/EPDM is tabulated in Table 3. Figures 10 illustrate the Weibull distribution plot for UnF SiR/EPDM and various loading concentration of TiO_2 nano-filler in SiR/EPDM with confidence interval of 95%.

Table 3: Value of α and β parameters in the 2-parameter Weibull distributions of breakdown strength for the nano-composites

Samples	2- parameter Weibull distribution		
	Concentration Vol%	Shape, β	Scale, α (kV/mm)
UnF SiR/EPDM	0	41.51	32.09
SiR/EPDM filled with TiO_2 nano-filler	1	41.38	33.01
	2	32.47	31.40
	3	40.91	29.50
	4	17.64	28.12
	5	17.15	26.10

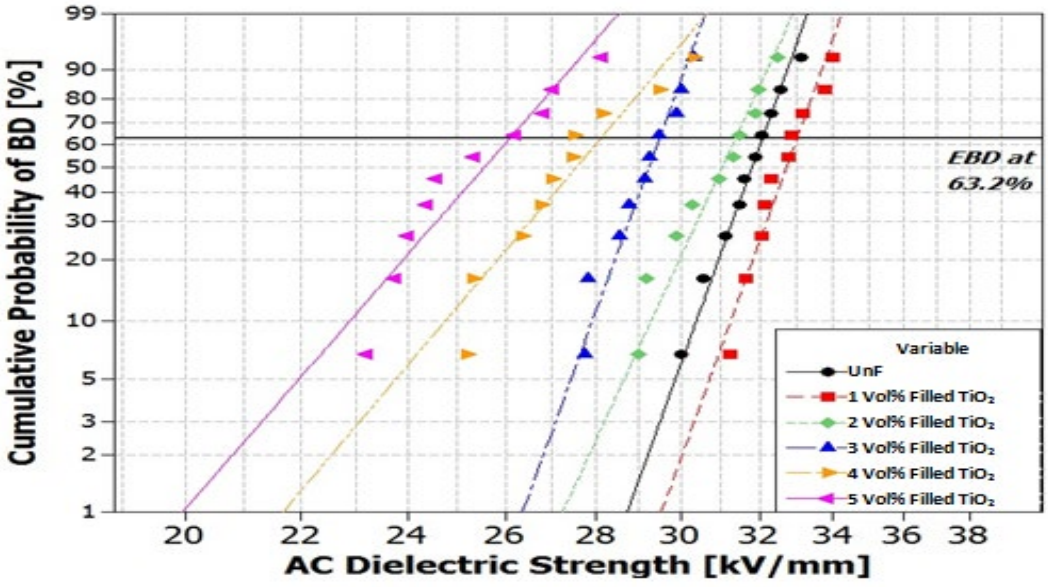


Figure 10: Weibull distribution plots for UnF SiR/EPDM and various loading concentrations of TiO_2 nano-filler in SiR/EPDM (Confidence Interval – 95%)

The result from Table 3 and Figure 10 show that the breakdown strength of 1 Vol% TiO_2 nano-filler in SiR/EPDM is relatively higher compared with the breakdown strength of UnF SiR/EPDM composite. When the loading concentration of the nano-filler increases from 2 Vol% to 5 Vol%, the breakdown strength of TiO_2 nano-filler in SiR/EPDM gradually decreased

from 31.40 kV/mm to 26.10 kV/mm. The result indicates that the breakdown strength of SiR/EPDM containing 1 Vol% TiO_2 nano-filler is relatively higher than UnF SiR/EPDM composite. The result also indicates that breakdown strength superior at 1 Vol% TiO_2 nano-filler compared to other loading concentration.

In addition, tensile strength is a measure of the material ability to resist breaking under tensile stress. It is one of the most important measured properties of materials used in insulation applications. Figure 11 depicts the relationship between tensile strength measurement result of UnF SiR/EPDM and various loading concentrations of TiO_2 nano-filler in SiR/EPDM composite.

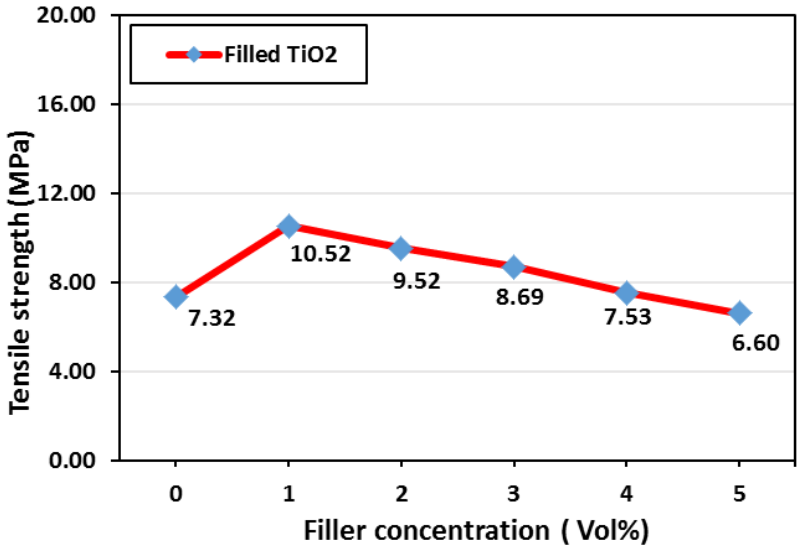


Figure 11: Tensile strength measurement results of UnF SiR/EPDM and various loading concentrations TiO_2 nano-filler in SiR/EPDM

The result shows that the 1 Vol% of TiO_2 nano-filler in SiR/EPDM composite produce higher tensile strength compared with the UnF SiR/EPDM and other loading concentrations. The tensile strength results for both nano-composites were gradually reduced when the nano-filler concentrations were increased from 2 Vol% to 5 Vol%. The maximum tensile strength of 1 Vol% nano-filler in both nano-composites may be attributed to a strong interaction between SiR/EPDM and the nano-fillers, and also homogenous dispersion of nano-fillers in the composite (Acharya & Srivastava, 2014). On the other hand, the decreases of tensile strength in the nano-composites may due to non-homogeneous dispersion of nano-fillers, which lead to a weak region of interacting between polymer and nano-fillers in the system. The regions where the loops in several chains are in close proximity, but do not entangle each other filler (Ali, El-Nemr, Youssef, & Mohamad Bekhit, 2008) . These aggregate of chain ends cause also micro cracks at the interface and lower the available interaction surface between the SiR/EPDM and the nano-filler. Also at high nano-filler amounts, the agglomerates are present to low interaction between SiR/EPDM and nano-filler. Furthermore, all of these possibilities may lead to lower strength values as the nano-filler amount increases.

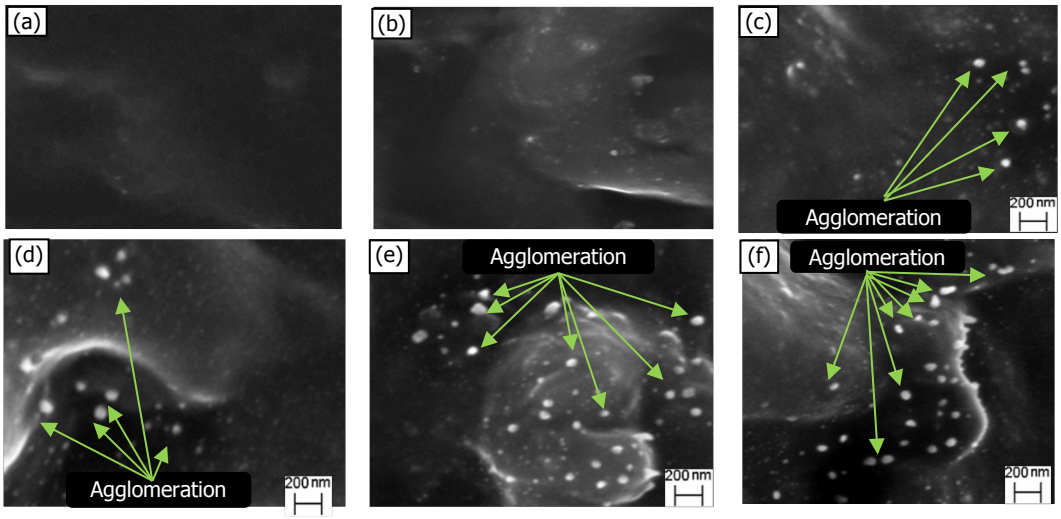


Figure 12: SEM images of TiO_2 nano-filler dispersion in SiR/EPDM at 20K magnification (a) 0 Vol% (b) 1 Vol%, (c) 2 Vol%, (d) 3 Vol% (e) 4 Vol% (f) 5 Vol%

Figures 12 indicate the morphology analysis on the dispersion of TiO_2 nano-fillers in SiR/EPDM that were carried out by SEM with 20K magnification. Figures 12 depicts the morphology images of UnF SiR/EPDM composite. The figures obviously show the non-existence of nano-filler in UnF SiR/EPDM composite. Meanwhile, the presence of the TiO_2 nano-fillers in the SiR/EPDM composite has been identified as white dots in the spherical shape. The TiO_2 nano-filler in white dots spherical shape can be observed in Figures 12 (b), (c), (d), (e) and (f).

Table 4 shows the calculated and measured inter-filler distance of TiO_2 nano-filler in SiR/EPDM composites. The inter-filler distance of nano-filler was calculated using the Eq. 2, which was introduced by Tanaka et al. (Tanaka, Kozako, Fuse, & Ohki, 2005):

$$D = \left[\left\{\frac{\pi}{6}\left(\frac{\rho_n}{\rho_m}\right)\frac{100}{wt\%}\left[1-\frac{wt\%}{100}\left(1-\frac{\rho_m}{\rho_n}\right)\right]\right\}^{\frac{1}{3}}-1\right]d \qquad \text{(Eq.2)}$$

where D is the inter particle distance, d is the diameter of nano-fillers, ρ_m and ρ_n are the volume density for SiR/EPDM composite and nano-fillers, respectively, and $wt\%$ is a weight percentage of nano-filler content in the composite.

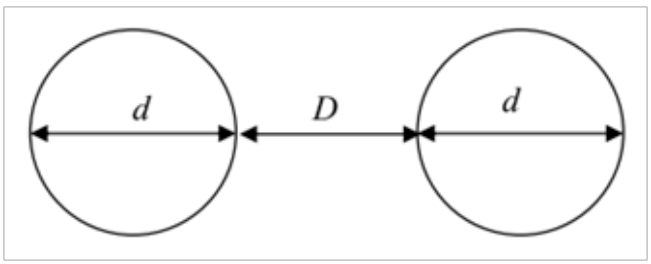


Figure 13: Nano-filler dispersion schematic for inter-filler calculation

The calculation result revealed that the SiR/EPDM containing 1 Vol% of TiO_2 nano-filler produce relatively higher inter-filler distance compared with other loading concentration of TiO_2 nano-filler. When the loading concentration of the nano-filler increases from 2 Vol% to 5 Vol%, the inter-filler distance of TiO_2 nano-filler in SiR/EPDM gradually decreased from 35.54 nm to 20.19 nm. The measurement results also confirmed that 1 Vol% TiO_2 has a higher inter-filler distance than other loading concentration. The average inter-filler distance of 1 Vol% TiO_2 nano-filler is 82.3 nm. However, when the loading concentration of nano-filler is increased further to 5 Vol% in nano-composites, the inter-filler distance of nano-filler could not be measured since most of the nano-fillers has been overlapped each other and usually it produces the agglomeration of nano-filler in the composite

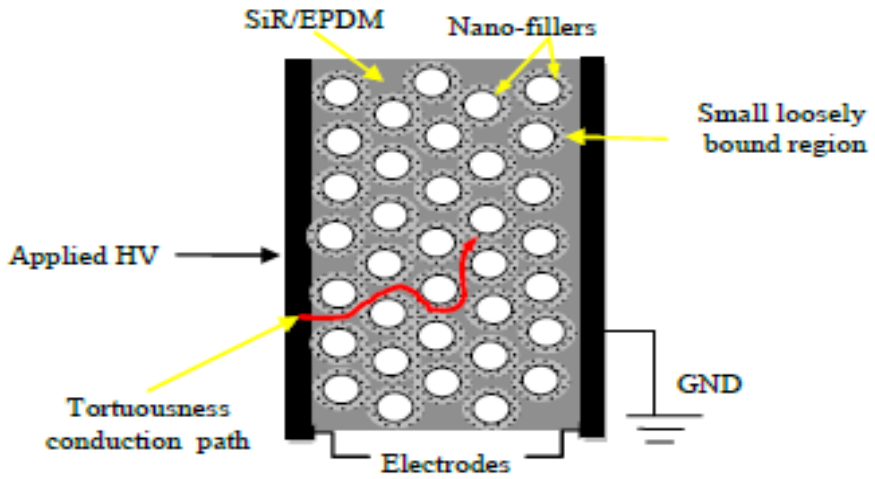
Table 4: The inter-filler distance of TiO_2 nano-filler in SiR/EPDM

Samples	Concentration		Inter-filler distance	
	Vol%	Wt%	Calculated (nm)	Average measured (nm)
UnF SiR/EPDM	0	4	50.53	82.3
SiR/EPDM filled with TiO_2 nano-filler	1	7.78	35.54	Agglomeration
	2	11.35	28.20	Agglomeration
	3	14.74	23.20	Agglomeration
	4	17.91	20.19	Agglomeration
	5	4	50.53	82.3

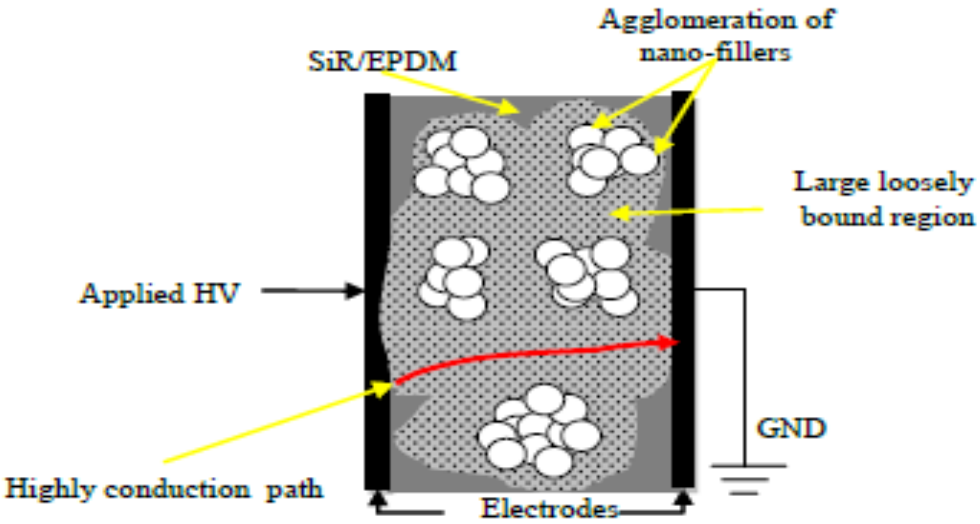
On the other hand, the effect of the various nano-fillers loading concentration into the SiR/EPDM composite has played an important role in determining the breakdown strength of the nano-composite. With 1 Vol% of TiO_2 nano-filler loading concentration, the amount of nano-filler in SiR/EPDM is higher, which has caused the strong interaction between the nano-filler and the polymer matrix. Where at this point, the inter-filler distance of nano-filler become closer to each other and the volume fraction of the loosely bound region in the nano-composite is smaller. In this situation, when the HV is applied, the nano-fillers in the sample will act as a barrier to the flow of the applied charge carrier to penetrate through bulk of nano-composite between the electrodes. Thus, the presence of 1 Vol% TiO_2 nano-filler can result in hindrance to the flow of the applied charge carrier in the SiR/EPDM composite and increase the breakdown strength (Singha & Thomas, 2008). In addition, Singha and Thomas also mentioned that the presence of a suitable nano-filler loading concentration in the nano-composite that acts as a barrier for the applied charge carriers between electrodes will cause the electrical conduction path becomes highly tortuous in nature (Singha & Thomas, 2010).

Meanwhile, the increasing of nano-filler loading concentration from 2 Vol% to 5 Vol% had also caused the decreases in the SiR/EPDM breakdown strength. In which, it had resulted in the weak interaction between the nano-filler and the polymer matrix. Where at this stage, the inter-filler distance of nano-filler will further decrease and the reduction of inter-filler distance may

contribute to the overlapping of loosely bound region. The combination of above effects had caused the nano-filler in the nano-composite tend to agglomerate with one another in the polymer region, as shown in Figures 14 (a) and (b). Eventually, agglomeration of nano-filler might produce the large volume fraction of loosely bound region around the nano-filler. In this situation, when the HV is applied, the overlapping loosely bound region of nano-filler will form a highly conductive path that permits the applied charge carriers to move easily through the nano-composite (Wang & Chen, 2014). Therefore, the presence of 2 Vol% until 5 Vol% TiO_2 nano-filler can yield the lower breakdown strength in the SiR/EPDM composite. The illustration models of the electrical conduction path in the SiR/EPDM nano-composites during the applied HV are shown in Figure 14.



(a) SiR/EPDM + 1 Vol% nano-filler



(b) SiR/EPDM + 2,3,4,5 Vol% nano-filler

Figure 14: Electrical conduction path in SiR/EPDM nano-composite.

4.0 Conclusion

In this study, the dielectric strength of the SiR/EPDM with TiO_2 nano-fillers was compared with unfilled composite. It was found that SiR/EPDM filled with 1 Vol% of TiO_2 nano-fillers showed the highest dielectric strength at 35.28 kV as compared to UnF SiR/EPDM only at 32.09 kV. The dielectric strength between the SiR/EPDM filled with 1 Vol% TiO_2 nano-filler and UnF SiR/EPDM has shown minor differences around 2.86%. However, the dielectric strength of nano-composites begins to decrease when the nano-filler loading concentration increased more than 1 Vol%. The addition of TiO_2 nano-fillers led to a remarkable increased in tensile strength on SiR/EPDM composite. Nano-composites with 1 Vol% loading concentration exhibits the highest tensile strength at 10.52 MPa (TiO_2) compared to UnF SiR/EPDM at 7.32 MPa and with differences around 43.72% (TiO_2). Nevertheless, the tensile strength for nano-composites decreased when the loading concentration of filler was increased from 2 Vol% to 5 Vol%. Therefore, the inclusion of 1 Vol% TiO_2 nano-filler into SiR/EPDM was significantly has enhanced the dielectric strength of SiR/EPDM composite

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