

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE
ENGINEERING AND TECHNOLOGY

**EFFECTS OF ATH AND SILICA FILLERS UPON ELECTRICAL AND
THERMAL PERFORMANCES OF HTV SILICONE RUBBER COMPOSITES**



M.Sc. THESIS

Didem TÜZÜN

Department of Electrical Engineering

Electrical Engineering Programme

JULY 2020

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

**ATH VE SİLİKA KATKI MADDELERİNİN HTV SİLİKON
YALITKANLARININ ELEKTRİKSEL VE ISIL PERFORMANSLARINA
ETKİLERİ**

YÜKSEK LİSANS TEZİ

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To my family and loved ones,



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ABBREVIATIONS

HTV	: High Temperature Vulcanizing
HCR	: High Consistency Rubber
LSR	: Liquid Silicone Rubber
RTV	: Room Temperature Vulcanizing
PDMS	: Polydimethylsiloxane
SIR	: Silicone Rubber
ATH	: Alumina Trihydrate
LC	: Leakage Current
DBA	: Dry Band Arcing
TGA	: Thermo-Gravimetric Analysis
DTG(A)	: Derivative Thermo-Gravimetric Analysis
DSC	: Differential Scanning Calorimeter
FTIR	: Fourier Transform Infrared Spectroscopy
GC-MS	: Gas Chromatography–Mass Spectrometry
IPT	: Inclined Plane Test
AC	: Alternating Current
DC	: Direct Current
Sil1	: Silica with median particle size of 3.4 μm
Sil2	: Silica with median particle size of 10.5 μm
ATH1	: ATH with median particle size of 3.6 μm
ATH2	: ATH with median particle size of 9.5 μm
HPC	: Harmonic Power Component
Wt	: Weight



SYMBOLS

Ω	: Resistance
S	: Siemens
mS	: Milli Siemens
°C	: Celsius
A	: Ampere
W	: Power
mW	: Milli Power
rms	: Root mean square
V	: Voltage
μm	: Micrometer
nm	: Nanometer
mm	: Millimeter
cm	: Centimeter
min	: Minute
kV	: Kilo volt
kΩ	: Kilo ohm
Hz	: Hertz



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SUMMARY

Silicone rubber insulators are used on overhead lines due to their several advantageous, such as low weight, easy installation, superior pollution performance, etc., over conventional glass and porcelain insulators. Better pollution performance of the silicone rubber insulators is due to their hydrophobic surface features. In a polluted environment, temporary or permanent loss of hydrophobicity leads to increase the magnitude of the leakage current flowing through the insulators, which causes dry-band arcing discharges. During these discharges, temperature of the discharging regions increases, which may initiate tracking and erosion of the silicone rubber material. Therefore, loss of hydrophobicity and tracking-erosion performance of the silicone rubber insulators are important aspects that need to be considered in their design. In order to enhance thermal performance, and therefore, to improve the tracking and erosion performance, several fillers are incorporated with silicone rubbers. This study presents the impacts of filler types, particle sizes and filler loadings on the erosion, leakage current and temperature performance of High Temperature Vulcanizing (HTV) silicone rubber used as an outdoor insulator material. HTV silicone rubber have comprised of 10%, 30% and 50% weight filler concentrations that consist of alumina trihydrate (ATH) and silica (SiO_2) with the mean particle size of approximately 3.5 μm and 10 μm . Thermal Gravimetric (TG), Derivative Thermal Gravimetric (DTG), Differential Scanning Calorimeter (DSC) and Fourier Transform Infrared Spectroscopy (FTIR) analyses were performed to better understand behaviours of HTV samples. IEC 60587 standard was used to evaluate tracking and erosion performance, leakage current and temperature performance of the samples. Inclined-plane tracking and erosion tests (IPT) were conducted by using constant test voltage method, considering 3.5 kV and 4.5 kV test voltages. Eroded masses, erosion depth and time-to-failure were evaluated. Besides, the leakage current by using LabVIEW data acquisition system and the maximum temperature by using IR thermal camera were recorded during the test period.

As a result of TG analysis, the filler type, filler ratio and thermal stability of HTV samples were evaluated. There was two stage weight loss in ATH filled samples while it was seen as one stage in silica filled ones. The dehydration of the water and degradation of the polydimethylsiloxane (PDMS) are the stages of the weight loss. These stages are available if the silicone rubber has ATH filler. On the other hand, if the silicone rubber has silica filler, it has only one stage weight loss which is the decomposition of PDMS. With DSC analysis, it was observed that the enthalpy changes increased as the filler ratio for ATH filled samples increased. The increase in the enthalpy change results in a better erosion performance. FTIR analysis performed before and after the experiment, it was clearly observed that the O-H bonds were exhausted due to the dehydration of the water.

It was found that the filler type and filler concentration affect the erosion performance of HTV silicone rubber. For both filler types, test duration increased as the filler ratio or volume fraction increased. For both test voltages, the highest filler ratio used in the study was found to be sufficient for ATH filled samples to satisfy the conditions as provided in IEC 60587. However, for silica filled HTV samples, some samples failed at both voltage levels. ATH filled samples with 50% wt filler ratio showed better erosion performance than silica ones for both 3.5 and 4.5 kV test voltages. 30% wt ATH filled samples showed the worst performance in terms of erosion performance due to the formation of holes on the samples. At 3.5 kV test voltage for 50% wt loaded samples, particle size did not affect the erosion performance, while at 4.5 kV test voltage, ATH2 showed better erosion performance than ATH1. Moreover, Sil1 samples performed better than Sil2 samples in terms of erosion performance. Except for 30% wt ATH filled samples, the depth of erosion has decreased as the filler ratio increases.

For all cases at 3.5 kV test voltage, the leakage current and the maximum temperature values of ATH filled samples were higher than those of silica filled ones since silica filled samples are better in terms of thermal stability. The same leakage current values were observed in both ATH and silica filled samples for 4.5 kV test voltage. No significant effect of particle size has been observed in terms of leakage current. In terms of maximum temperature, samples with a small particle size at both the ATH and silica filled samples showed slightly better temperature performance at 3.5 kV test voltage. For 4.5 kV test voltage, ATH2 filled samples showed better temperature performance than ATH1 filled ones. For silica filled samples, this situation did not change at 4.5 kV test voltage and samples with small particle size showed better temperature performance. It was found that there was a strong correlation between the 3rd harmonic power / 3rd harmonic component of the leakage current and the maximum temperature. The increase or decrease of the temperature was directly related to the 3rd harmonic component.

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ÖZET

Yüksek ve çok yüksek gerilim enerji iletim hatlarında, gerilim altında bulunan kısımlar ile topraklı kısımlar arasındaki yalıtım izolatörler ile sağlanmaktadır. İletim hatlarındaki elektriksel yalıtım, cam ve seramik izolatörlerin yanında son yıllarda polimer (silikon kauçuk, kompozit) izolatörlerle de yapılmaktadır. Polimer izolatörlerin cam ve seramik izolatörlere göre birçok avantajı vardır. Polimer izolatörlerin en önemli avantajı hidrofobik (su tutmayan) yüzeye sahip olmaları ve geçici hidrofobiklik kaybı oluştuğunda hidrofobik özelliklerini geri kazanmalarındır. İlave hafif olmaları, kolay kurulumları, darbelere karşı dayanıklı olmaları, kaçak akımı bastırmada üstün olmaları, kimyasal ve elektriksel dayanımları, geniş sıcaklık aralığında yapılarını koruyabilmeleri ve düşük maliyete sahip olmaları polimer izolatörlerin diğer avantajlarıdır. Bu avantajlar nedeniyle, silikon izolatörlerin kullanımı gün geçtikçe artmaktadır. Diğer taraftan, cam ve porselen izolatörler hidrofilik (su tutan) yüzeye sahip olduklarından, özellikle kirli ortamlarda kötü elektriksel performans göstermektedirler.

Silikon izolatörler karbon içermelerinden dolayı organik, silisyum içermelerinden dolayı da inorganik özelliğe sahiptirler. Silikon kauçuk malzemeler silikon-oksijen ana zincirinden ve silisyuma bağlı metil gruplarından (CH_3) oluşur. Birden fazla bu zincirlerin bir araya gelmesiyle Polydimethylsiloxane (PDMS) oluşur.

Bu tez çalışmasında, yüksek sıcaklıkta kürlenmiş (High Temperature Vulcanizing / HTV) silikon kauçuk numuneleri kullanılmıştır. Çalışma kapsamında ana malzeme olarak kullanılan HTV silikon kauçuğun yanında iz-erozyon dayanımını arttırmak için ana malzemeye ilave dolgular eklenmiştir. İlave dolgu olarak Alumina tri-hydrate (ATH) ve silika kullanılmıştır. Bu malzemenin kullanılmasının amacı şu ana kadar ki yapılmış çalışmalarda silika katkılı HTV numuneleri ile ilgili az sayıda çalışmanın yapılmış olmasıdır. Mikron boyutunda kullanılan katkı maddelerinin parçacık boyutunun etkisini görebilmek için ortalama 3.5 μm ve 10 μm parçacık boyutuna sahip katkı maddeleri kullanılmıştır ve her katkı türü ve parçacık boyutu için ağırlıkça %10, %30 ve %50 oranlarına sahip numuneler hazırlanmıştır. Ana silikon ile ATH ve silika katkılar ile oluşturulmuş toplamda on üç farklı numune hazırlanmıştır. Her deney düzeneğinde, her durum için, toplam beş özdeş numune kullanılmıştır. Katkı maddelerinin türleri, parçacık boyutları ve katkı oranları dikkate alınarak elektriksel, kimyasal, fiziksel ve termal özellikleri incelenmiştir.

Deneyler öncesinde hazırlanmış olan tüm numunelerin Thermo Gravimetric (TG), Derivative Thermo Gravimetric (DTA), Fourier Transform İnfra-Red Spectroscopy (FTIR) analizleri yapılmıştır. Deney sonrası erozyon bölgelerinden tekrar örnekler alınarak FTIR analizleri yeniden yapılmıştır. Ayrıca ısı değişim entalpisini değerlendirmek amacıyla Differential Scanning Calorimeter (DSC) analizi bazı numuneler için yapıldı.

TG analizi sonucu katkı türü, katkı miktarı ve termal kararlılık hakkında bilgilere ulaşılmıştır. Çalışmada kullanılan ana silikon malzemede TG analizi sonucunda %31,5 fume-silika katkısı olduğu görülmüştür. Her iki katkı türü için de katkı konsantrasyonu arttıkça analiz sonucunda kalan kütlelerin miktarları da artmıştır. Bu durumun nedeni katkı maddelerinin inorganik yapıda olmaları ve analiz sonucunda bu maddelerin ortamda kalmalarıdır. Ayrıca katkı miktarının artmasıyla termal kararlılığın arttığı DTG eğrilerinde görülmüştür. Bu eğrilerde ATH katkılı numunelerde suyun dehidrasyonundan kaynaklı iki aşamalı bir süreç oluşurken silika katkılı numunelerde suyun dehidrasyonu olmadığından dolayı sadece tek aşamalı termal bozulma olmuştur. Suyun dehidrasyonu 240 °C civarı başlarken termal bozunma 400 °C civarı başlamıştır.

FTIR eğrilerinde ATH katkılı numunelerde O-H bağı varken silika katkılı numunelerde bu bağ yoktur. FTIR analizi ile dalga boylarına karşılık gelen kimyasal bağlar deney öncesi ve sonrası durumlar için değerlendirilmiştir. Yaşlanmaya bağlı olarak deney sonrası analiz sonucunda O-H, C-H, Si-O ve Si-C piklerinde azalma olmuştur. O-H bağlarının azalmasının sebebi suyun dehidrasyonudur. Hidrat suyunun buharlaşması numune yüzeyinde koruyucu bir tabaka oluşmasına yardımcı olur. Diğer bağların azalmasının nedeni karbonlaşmadır. Ayrıca Si elementinin deney sırasında koruma etkisi yaptığı yapılan bazı makale çalışmaları sonucu bulunmuştur.

DSC analizi yapılarak malzemenin entalpi değişimi incelenmiştir. ATH katkılı silikon yalıtkanlarda suyun dehidrasyonundan kaynaklı endotermik bir reaksiyon olarak 230 °C sıcaklık değerinde başlayan ve 350 °C civarı biten ısı alışverişi görülmüştür. Entalpi değişimi en çok ağırlıkça %50 ATH katkılı numunede görülmüş olup katkı oranı arttıkça bu değişimin arttığı gözlenmiştir. Ancak bu entalpi değişimi, suyun dehidrasyonu olmadığından dolayı hem katkısız numunede hem de silika katkılı numunelerde görülmemiştir. Ayrıca bu entalpi değişimi ne kadar fazla olursa numune yüzeyinde koruyucu bir tabaka oluşturarak erozyonu bastırmada rol aldığı söylenebilmektedir.

3,5 kV ve 4,5 kV sabit gerilim altında yapılan eğik düzlem deneyleri sonrasında malzeme analizleri yapılmıştır. Katkı türünün erozyon davranışını etkilediği ve bu farklı erozyon davranışları daha çok kritik düzeyin hemen altındaki konsantrasyona sahip numunelerde olduğu görülmüştür. Ağırlıkça %10 katkı oranı için iki katkı türü de hemen hemen aynı erozyon davranışı sergilerken ağırlıkça %30 katkı türü için ATH katkılılarda delik oluşumu ile sonuçlanmış bunun aksine silika katkılılarda erozyon yolu şeklinde numuneler başarısız olmuştur.

Erozyon performansı açısından 3,5 kV ve 4,5 kV gerilim düzeylerinde de %50 ATH katkılı numuneler %50 silika katkılı numunelere göre daha iyi performans göstermiştir. En kötü performansı delikler oluşmasından dolayı %30 ATH katkılı numuneler göstermiştir ve ATH1 (3,5 µm parçacık boyutlu) numunelerinde iki tane numune delinirken ATH2 (10 µm parçacık boyutlu) katkılı numunelerde üç tane numune delinmiştir. Bu nedenle erozyonla aşınmış kütle ortalamaları ATH2 numuneleri için daha fazla olmuştur. Erozyon performansı açısından delinen numuneleri dışarıda bırakılırsa 3,5 kV gerilim altında parçacık boyu açısından önemli bir fark görülmemiştir. Ancak, 4,5 kV gerilim altında, ATH2 grubu ATH1 grubuna göre, Sil1 grubu ise Sil2 grubuna göre daha iyi erozyon performansı göstermiştir. %30 ATH katkısı hariç, katkı oranı arttıkça erozyon performansı artmıştır. Erozyon derinlikleri açısından da benzer durum söz konusudur. Erozyon derinliği performansı her iki gerilim değeri için de yine %50 ATH katkılı numunelerde iyi olmuştur.

Deney süreleri olarak, %10 katkı oranına sahip silika ve ATH1 numuneleri ana HTV silikon kauçuk numuneleri ile benzer davranış göstermiştir. Ancak, %10 ATH2 katkı oranına sahip numuneler diğerlerine göre daha iyi performans göstermiştir. Ayrıca tüm katkı büyüklükleri ve türleri için katkı oranı arttıkça numunelerin deney süreleri artmıştır. %50 ATH katkı oranına sahip tüm numuneler hem 3,5 kV hem de 4,5 kV sabit gerilimde eğik-düzlem deneyinde başarılı olmuşlardır. İki test geriliminde de bu katkı oranının ATH katkılı numuneler için yeterli olduğu görülmüştür. Ancak, %50 silika katkılı numuneler için bu oran yeterli olmamıştır. Çünkü deneyi geçen numuneler olduğu gibi deneyden başarısız olan numuneler de olmuştur. Bu nedenle, silika katkılı numunelerin ilgili deney gerilimlerinde başarılı olmaları için ağırlıkça %50'den daha fazla katkı oranına sahip olmaları gerektiği belirlenmiştir. 4,5 kV deney geriliminde 3,5 kV deney gerilimine göre, sadece %50 katkı oranı için yapıldığından, parçacık boyutu açısından ATH katkılı numunelerde pek etki görülmezken 10,5 µm parçacık boyutuna sahip silika katkılı numunelerde deney süresi kısalmıştır ve Sil1 grubunun deney süreleri hemen hemen aynı kalmıştır. 3,5 kV deney geriliminde iki silika grubu da benzer deney süreleri göstermişken 4,5 kV deney geriliminde küçük parçacık boyutlu silika katkısına sahip numuneler daha uzun süre dayanım göstermişlerdir.

Numuneler üzerinden geçen kaçak akımlar LabVIEW programı aracılığıyla kaydedilmiştir. 3,5 kV deney gerilimde, tüm durumlar için, ATH katkılı numunelerin kaçak akım ve maksimum sıcaklık değerleri silika katkılılardan fazla çıkmıştır. Yapılan araştırmalar sonucunda bunun sebebi olarak silika katkılı numunelerin termal kararlılığının ATH katkılı numunelerden daha iyi olması ile açıklanmaktadır. Silikanın termal kararlılığının sebebi, silika katkısının ana malzeme ile güçlü ara yüz etkileşiminden kaynaklandığı vurgulanmıştır. 4,5 kV deney gerilimi için hem ATH hem de silika katkılı numunelerde aynı kaçak akım değerleri gözlenmiştir. Kaçak akım açısından parçacık boyunun etkisi görülmemiştir. Maksimum sıcaklık açısından 3,5 kV deney geriliminde hem ATH hem silika katkılı numuneler için küçük parçacık boyutuna sahip numuneler az da olsa daha iyi sıcaklık performansı göstermiştir. 4,5 kV deney gerilimi için ATH2 katkılılar ATH1 katkılılara göre daha iyi sıcaklık performansı göstermiştir. Silika katkılılar için 4,5 kV gerilimde bu durum değişmemiş ve yine küçük parçacık boyutuna sahip numunelerin daha iyi sıcaklık performansı gösterdiği görülmüştür. Fakat bu durumda, az bir farkla değil, hemen hemen Sil2 grup numunelerin sıcaklık performansına göre iki kat fark olmuştur. Sıcaklık sonuçları arasındaki ciddi farklılık erozyon miktarlarında da görülmüştür. 4,5 kV deney geriliminde Sil1 grubu daha iyi erozyon performansı göstermiştir.

Numune üzerinde sıcaklık artışıyla birlikte kirlenici sıvı buharlaşır ve kısmi kuru bölgeler oluşur. Bu kuru bölgelerde, kuru band arkları meydana gelmektedir ve kuru band arkları, kaçak akımda harmonik bileşenlerinin oluşmasına neden olmaktadır. Sıcaklık ile oluşan bu harmoniklerin ilişkisi değerlendirilmiş ve üçüncü harmonik kaçak akımının gücü ile sıcaklık arasında güçlü bir korelasyon olduğu görülmüştür. Sıcaklığın artması veya azalmasının, üçüncü harmonik akım bileşeni ile doğrudan ilişkili olduğu görülmüştür.



1. INTRODUCTION

1.1 Introduction

At present, high voltage, extra high voltage and ultra-high voltage levels are used in electric power transmission. The transmission of electricity can be done either by overhead lines or high voltage cables. Insulation between the high voltage part and the ground part is provided by using insulators. The most important problem of insulators used in outdoor environments are the pollution and discharge events caused by contamination. In addition, electrical, mechanical, extreme weather conditions, chemical and biological effects can damage insulators. In order to prevent or at least minimize the pollution-related problems in the transmission and distribution lines of electrical power systems, several solutions have been developed. One of the solutions is to use outdoor polymeric insulators. In Turkish power transmission systems, although there are several overhead lines composed of silicone rubber, porcelain and glass conventional insulators, new constructed lines consist of polymeric insulators.

At the outdoor insulation, both ceramic and non-ceramic insulators are used from past to present. Since the use of ceramic insulators dates back to the past, ceramic insulators have been used for a long time in the power grids [1]. It was reported that the usage of silicone rubber insulators has been increases in the last decades due to their several advantages over conventional insulators. In the first times, insulation was provided with porcelain and glass insulators. Due to their hydrophilic surface feature, these insulators do not perform well especially in polluted environments. In such a case, the power system performance decreases and the system cost increase. The corrosion which is on the ceramic and glass insulator may cause quickly to the cracks [2]. Due to these reasons, power utilities prefer to use silicone rubber insulators, especially in polluted environments. Silicone rubbers (SIRs) provide great advantages in terms of material and design over glass and ceramic insulators. Besides, they have longer system performance compared to glass and ceramic insulators in terms of pollution and leakage current. Silicone rubber insulators have some advantages compared to

porcelain and glass insulators: hydrophobic surface feature, being light-weight, ease of installation, low cost, and superior performance in polluted environments [3]–[5]. Silicone rubber insulators were begun to use in high voltage indoor insulation materials in the 1940s. However, they were discovered that they could be use at outdoor insulation applications in the 1950s. After that time, they have been used for outdoor applications [6].

The failure due to contamination is often encountered in porcelain and glass insulators. The reason for this situation in porcelain and glass insulators is that they do not have a hydrophobic surface. As a result of hydrophilic surface property, the glass or porcelain insulator whose surface is dirty causes the formation of a water path that cover the surface completely with the effect of moisture and rainfall, and increase of leakage current. Due to the effect of moisture and rainfall, the formation of a water path occurs and covers the surface completely in these insulators. That increases the leakage current. Then, with the increase of leakage current, dry-band arcs form and these lead to the discharge events [7]. Contrary to this, silicone rubbers have low surface energy, and they are hydrophobic surface feature [8]. The hydrophobic property ensures low leakage current of insulators in cases of environmental pollution since this property prevents the formation of a continuous water layer on the surface. On the other hand, although having several advantageous, the silicone rubbers have some disadvantages [9]. As soon as the tracking and erosion begins, they break down faster than glass and porcelain insulators and, also, they lose their hydrophobicity under corona discharges, and certain environmental conditions.

Silicone rubbers show excellent hydrophobicity due to being low surface energy. This property, hydrophobicity, is explained by the low molecular weight (LMW) liquid in the silicone rubbers. The higher the LMW on the material surface, the better its hydrophobicity. The loss of hydrophobicity on the surface occurs due to the reduction of the LMW liquid silicone or reduction of the transfer of the LMW from the bulk material. The hydrophobicity recovers by diffusion of LMW to the surface of silicone rubber [7], [10]. However, recovery of hydrophobicity depends on several factors [11]. Also, in the study [11], it was stated that recovery of the hydrophobicity is effective at ambient temperature. As the temperature increased, hydrophobicity was rapidly recovery. However, when hydrophobicity disappears temporarily or dry band arcing (DBA) occurs, tracking and erosion may occur in the silicone insulator [7]. As long as

the loss of hydrophobicity continues, dry band arcing continues [12]. A lot of parameters cause the scintillation mechanism of the dry band arcing; applied voltage level, voltage type, the series pre-resistor, the conductivity of the contaminant liquid and its flow rate, and the sample surface tension [13]. Therefore, selection of these parameters is important.

In high voltage applications, three different types of silicone rubbers are used: High temperature vulcanizing (HTV)/ heat-cure rubber (HCR), room temperature vulcanizing (RTV) and liquid silicone rubber (LSR). Silicone rubber insulators are made of either HTV or LSR material. On the other hand, RTV is used as a coating material for conventional porcelain and glass insulators.

In addition to the creepage distance, the other parameters such as shed spacing, size, shape, and diameter of insulators are effective in the service time of insulators [7]. In addition to the base material of silicone rubber, several fillers are added for various purposes, such as improving the tracking-erosion performance which is caused by dry band arcing, improving thermal conductivity [14], [15].

In the inclined plane test (IPT) done for HTV silicone rubber samples with ATH filler, the tracking and erosion resistance improved as the ATH level increased [16]. Aluminum trihydrate (ATH, $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) and silica (SiO_2) are used to improve resistance to dry band arcing, partial discharge, and corona; Barium Titanite (BaTiO_3) is used to reduce surface stress by increasing the relative permittivity; Alumina (Al_2O_3), and Calcium Carbonate (CaCO_3) are used for various purposes [15]. Also, these fillers reduce the cost of material [17]. It was reported that the thermal conductivity of the silicone rubbers increases with increasing of silica and ATH filler levels in the silicone rubber [15].

The tracking-erosion resistance, thermal, and electrical properties of silicone rubbers were investigated with respect to the filler type, filler shape, size of the filler (micro or nano), filler ratio (10% wt, 40% wt etc.), being treated or untreated of filler material [10], [14], [17]–[20]. For example, the treated filler material shows better erosion resistance than the untreated filler one [18]. ATH and silica fillers are commonly used to improve the tracking and erosion resistance of the SIRs. A lot of papers have been published about the benefits of using ATH and silica fillers [5], [17], [21]–[23]. Effects of filler type, mean particle size, and filler concentrations on the tracking and erosion

resistance of the SIR material were investigated by using laboratory studies, such as inclined plane test, dynamic drop test, and needle- plane electrode system. But another paper was stated that the presence of ATH does not need to improve tracking and erosion resistance in the silicone elastomers [24]. The electrical activity that occurs during the test also depends on the base material used [24].

Silicone insulators show successful performance due to hydrophobicity feature in case of polluted environments, and they suppress the leakage currents [25]. At the salt-fog test performed for silicone rubber samples, no correlation was observed between the filler level and the cumulative leakage current in the low conductivity of fog [25]. The leakage current values increase as the test period increases. Because the variation of cumulative charge of leakage current is linear at samples that passed or failed the test, it is not a good indicator of the tracking and erosion failure [25].

The criterion of the erosion length (2.54 cm) may not be sufficient [19]. Also, the depth of erosion can be evaluated. Although the depth of erosion had lots of in the sample, it passed in the test [19]. On the other hand, the sample failed in the test even though the depth of erosion had little. Therefore, erosion length is not the only parameter in evaluating tracking and erosion performance.

Several analysis and tests are performed to evaluate the performance of silicone insulators from several perspectives: The inclined plane test (IPT) for tracking-erosion resistance, dynamic drop test (DDT) and salt-fog test for hydrophobicity, surface roughness test, scanning electron microscopy (SEM) for surface morphology, thermogravimetric analysis (TGA) for thermal analysis, leakage current analysis with data acquisition system for electrical analysis, electron spectroscopy (ESCA) for chemical analysis, fourier transform infra-red spectroscopy (FTIR) for atomic structure analysis, gas chromatography-mass spectrometry (GC-MS) to analyses change of low molecular weight (LMW) in polymer.

1.2 Review of Literature

Silicone rubber compounds have hydrophobic feature and low surface tension, and they can recover the loss of hydrophobicity. Silicone rubbers are different from other materials, such as epoxy (EP), ethylene propylene diene monomer (EPDM), and ethylene-vinyl acetate (EVA) due to their recovering property of the hydrophobicity

[9]. The silicone rubber composes of methyl groups, and Oxygen and Silicon elements [26]. The methyl groups provide hydrophobicity in silicone polymers [26]. It was reported that methyl groups [26] and diffusion of LMW maintain the hydrophobicity [9]. The regeneration of LMW liquid on the surface is directly, also, proportional to the temperature rise [27].

The partial discharge on the insulators depends on the pollution level [1]. Failure of silicone rubbers comprise of the discharge activity and loss of hydrophobicity [15]. The discharge activity starts with loss of hydrophobicity on the wet surface. Later, the magnitude of the leakage current increases, which causes the temperature rise on the material surface, and dry band arcing develops [9].

The dry band arcing and discharge mechanism were shown in Figure 1.1 [28]. As the magnitude of the leakage current increases, dry areas form due to the evaporation of the liquid. Since these dry areas create a capacitive effect, they increase the electric field, and then cause dry band arcings and hot spot temperatures.

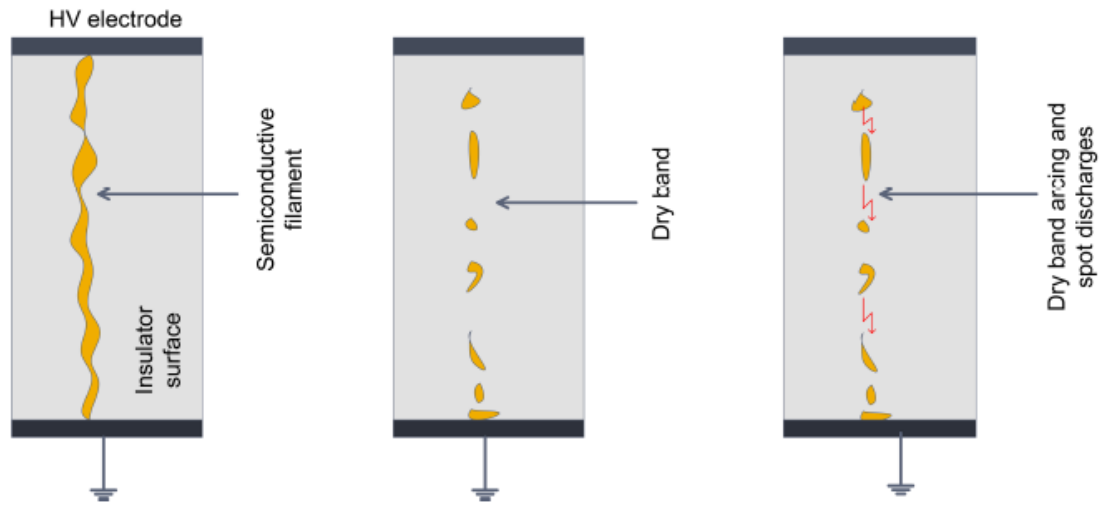


Figure 1.1 : Illustration of dry band arcing in an IPT [28].

In the study [15], it was stated that correlation is available between eroded mass and thermal conductivity. It was found that the eroded mass was less with increasing thermal conductivity. While silica filler material forms a bond with silicone polymer, ATH filler material does not form that bond [15]. The lack of ATH filler bonding causes small gaps between the filler and silicone polymer. Therefore, the gaps reduce the effective thermal conductivity of the ATH particle with respect to silica.

The exposure time of the moisture to the silicone rubber surface affects the initial value of the leakage current [8], [26]. The recovery of hydrophobicity decreases as long as water is present on the silicone rubber surface [8]. Contact angle measurements are used to determine the hydrophobicity. That is, contact angle degree is a indicator of hydrophobicity. As the advancing contact angle increases, hydrophobicity increases as well [26]. The hydrophobicity recovers in the silicone rubber that is not under voltage and allowed to dry in an air environment. However, the recovery of hydrophobicity is fast at higher temperatures [8]. The temperature increase that will not cause any physical change in the material, increases the recovery speed of hydrophobicity. Also, it has been observed that the hydrophilic OH groups appeared instead of the hydrophobic CH₃ groups under electrical causes the temporary loss of the hydrophobicity [29]. Owing of the dehydration of OH groups, the endothermic dents form in the samples with ATH filler [29].

A distorted current waveform occurs due to dry band arcing and erosion [12]. The leakage current data are smoothed in order to reduce the fluctuations of leakage current by using moving average technique. After applying this technique, the data can be analyzed more easily [12]. In the moving average technique is selected window frame according to data point determined [12].

The inclined plane test was investigated under the AC voltage and DC voltage provided that other conditions remain the same. The pollution accumulation was greater on the insulator in the DC voltage than from in AC voltage [2]. So, tracking and erosion were more severe under the DC voltage. Besides, for different voltage magnitude, the IPT under negative DC voltage showed the worst performance than positive DC voltage in terms of leakage current and failure time [12]. It was reported that the average leakage current was higher in the positive DC condition [13]. Also, for the same voltage magnitude, the DC stress having positive polarity caused more damage than that of the negative polarity [28]. Both the higher eroded mass and the deeper erosion were observed at the positive DC voltage rather than the negative DC [13], [30]. More residue was accumulated under negative polarity DC voltage than under positive polarity DC while the depth of erosion was greater under +DC than under -DC [31]. This condition was explained by DBA [31]. Also, the reason for the accumulation of more residue under the -DC voltage is due to the quick initiation of erosion.

It was obtained as 67% and 87% of the AC initial tracking voltage for + DC and –DC voltages, respectively, and they were rounded to 70% and 90% for practical purposes [13]. Positive DC is more severe than negative DC [31]. The tracking-erosion performance depends on the magnitude as well as type of the applied voltage [5]. Iron was found on the sample surface by EDAX analysis of the silicone rubber with silica filled tested under the DC stress [32]. This is a clear indication of the degradation of electrode material under DC stress [32]. DC stress is more severe as compared with AC stress [32]. It was commonly stated that DC voltage causes more corrosion of the electrodes than AC voltage, which may accelerate the test specimen's aging. Because metallic ions formed due to the electrode corrosion dissolve in the pollutant and further increase the conductivity of semiconductor tracks [28]. Therefore, DC stress is more severe than AC stress.

For HTV-SIRs, it was reported that the leakage current level on the sample surface may be forecasted by using ρV (ρ : conductivity of liquid, V : applied voltage) value [21]. At the thin samples with low ρV (<5.4) value, tracking-erosion resistance decreased as long as thickness increased, and the samples failed due to the increase in the leakage current. On the other hand, the thicker samples had higher ρV (> 5.4) value and higher volume, thus increasing erosion resistance as the thickness increased, and more leakage current was generated on the samples before the failure [21]. It is clear that, volume of the samples affects the results in IPT.

The polymer aging process due to the chemical reaction could be categorized into two groups: crosslinking reaction and decomposition reaction [33]. The crosslink reaction comprises of physical crosslink density and chemical crosslink density. In reference [33], it was reported that chemical crosslink density decreases while physical crosslink density increases during the aging process. While the chemical crosslink density decreases due to the decomposition reaction, the physical crosslink density increases due to the interaction between the filler and the PDMS molecules [33]. This showed that the decomposition and crosslinking reaction take place simultaneously, and they contribute to the aging of composite insulators together [33]. With increasing the physical crosslink density, the hardness and color fading of the samples increased and a high positive correlation occurred [33]. The color faded specimens have less thermal stability than normal specimens [33]. The tracking and erosion resistance reduces due to hardening in the silicone samples exposed the thermal aging [32], [34].

Since insulators such as the porcelain and EVA have a more wettable surface, the water behavior shows different characteristics and therefore the scintillation causes the dry band arcing on the surface, and these dry band arcing reduces the average leakage current with increased voltage [24]. The average leakage current may increase with the increased voltage on the condition that the formation of the dry band is no [24]. Therefore, it might say that electrical activity depends on the material. [24].

Two exothermic peaks were indicating that two crystallization processes may occur in HTV-SIRs [35]. Exothermic peaks, indicating that more than one crystallization may occur in this HTV-SIRs, relate to a large amount of ATH fillers [35]. It has been found that a large amount of fillers may easily cause heterogeneous dispersion, decrease HTV's crystallization temperature, and increase HTV-SIR's crystallization ratio, so that cold crystallization cannot be observed during heating [35]. For this reason, LSR is used instead of HTV-SIR in cold regions and regions with large temperature changes [35].

1.2.1 Role of fillers

Inorganic fillers increase the erosion resistance and reduce the cost of insulating material [31]. Nowadays, the various inorganic filler materials are used at about 60% weight in order to increase the tracking and erosion resistance under the AC voltage [31]. Housing of silicone insulators consists of both reinforcing and extending fillers [7], [18]. The tracking and erosion performance may depend on filler ratio, filler type, filler particle size, filler particle shape [14] and surface roughness [19].

The formulation of silicone rubber with good erosion resistance is balanced by having sufficient amount of filler, maintaining a good dispersion of the particles in the silicone matrix, as well as ensuring good bonding between the filler and the matrix [19]. However, filler material with too much filler ratio [18] or too large particle size [19] may affect the tracking and erosion resistance adversely. In a study [5], the ATH filled HTV silicone rubbers with different voltage levels (3, 3.5, 4.5 and 5 kV) and different filler ratios (10, 25, 30, 40, 50 and 60% wt) were tested. The samples showed the worst performance at 4.5 kV voltage level, and the tracking-erosion performance was found to be depend on the voltage level [5]. Moreover, ATH filled HTV-SIRs with 40% or higher filler ratios increased the tracking-erosion performance for all voltage levels,

and 40% weight filler ratio was reported as critical level in terms of tracking and erosion performance [5].

The silica filler material exhibits better mechanical properties and thermal properties than ATH filler material due to its strong interfacial interaction and good dispersion to the silicone rubber matrix [14]. Moreover, the filler shape as well as the filler type can affect the mechanical properties of the silicone rubbers. The good interfacial interaction provides a homogeneous surface morphology. Kone et al. reported the increase in the erosion resistance of the silica residue formed during the IP test for HTV SIRs with silica filled [30]. In the study [30], it was emphasized that the tracking and erosion resistance increase as both the volume fraction and the particle size increase. It was also reported that the volume fraction is the first effect, and the particle size has the second effect in the erosion performance [30].

It was found that the surface-modified precipitated 57% wt ATH filled samples performed the best in terms of tracking and erosion performance [18]. It was also emphasized that the filler type is important. Besides, if the mean particle size increases, the erosion resistance deteriorate. The tracking and erosion resistance of nanoparticle size samples increased even at the lower filler ratio according to micro particle size samples [20], [34]. The plasticizer level placed with 0-0.6% wt of the both filled and unfilled HTV silicone rubbers is not influencing the time-to-failure and tracking-erosion resistance [10]. It was reported that the addition of silicone plasticizer had positive effect in the leakage current suppression if the HTV had fillers, but no effect was observed due to the plasticizer if the HTV silicone rubber had no filler [10].

In the study done with RTV [36], the tracking and erosion performance improved significantly if the silicone rubber has 10% wt nano size fillers. The weight loss obtained for 10% wt nano size filler was almost the same for the samples having 50% wt micro size fillers. It was observed that the samples with nano size fillers showed significant resistance to erosion at low concentrations compared to micro size fillers. The thermal stability of the samples has improved significantly by using low concentration fumed-silica [36]. These results are due to the strong filler-matrix interaction in case of nano filler.

The correlation is available between eroded volume and harmonic power in the samples with RTV with ATH filled and silica filled except a few samples [17]. It

cannot be mentioned from this correlation for unfilled samples [17]. In the high filler ratio, ATH and silica show different erosion performance [17]. For instance, the erosion was formed around the bottom electrode in the ATH filled silicone rubber, whereas an erosion path from the bottom electrode to the top electrode was observed for the silica filled samples. [17]. Also, in the study [17], ATH showed better tracking and erosion performance than silica at high filler ratio. In another study for 50% filled silica, ATH, and magnesium hydroxide (MH) samples, ATH showed better tracking and erosion performance than silica. Moreover, MH filled samples showed close performance with ATH filled samples [37]. That is, it is emphasized that the dehydration of the water is a major role in suppression erosion [37].

Silicone rubber samples with hybrid particles (27% Aluminum nitride with 5-10 μm + 3% silica with 20 nm) filler were found to show better tracking and erosion resistance than samples with micron size filler [38]. The hybrid particles have excellent heat dispersion since they increase thermal stability and thermal conductivity, hence, they delay DBA [38], [39]. Also, the r.m.s. value of the leakage current was lower in the material with hybrid particles (27% Aluminum nitride with 5-10 μm + 3% silica with 20 nm), and the leakage current showed no significant increment as time got on [39]. In addition, due to their higher surface area, the enhanced scattering potential of the hybrid particles impedes the collapse of the secondary electron and limits the high energy emissions that can cause thermal depolymerization and trigger carbon tracking and erosion in hybrid composites [39].

1.2.2 Degradation mechanism

In the silicone rubber, when the temperature is less than 200 °C, there will be very little leakage current since dry band arcing will not occur [31]. However, the dry band arcing begins to occur at a value above of this temperature, and thus degradation begins, too. The degradation leads to material erosion with the formation of a conductive carbonaceous path [32]. There are many techniques to examine the degradation mechanism in materials. The chemical degradation of the substance is specified by an analysis of FTIR [32]. The ESCA technique is used to understand the status resistance to the flow of water caused by dry band arcing [25]. The flow of the water under voltage gives a lot of information related to the degradation mechanism.

An endothermic reaction indicates thermal decomposition caused weight loss, and an exothermic reaction indicates thermo-oxidation caused weight loss [5]. The endothermic reaction increases due to dehydration of ATH [30] while the exothermic reaction decreases with increasing of ATH filled level [5], [29]. Water is released with a first dehydration called endothermic reaction when sample with ATH filled is heated above 180 to 200 °C [5]. Water release plays an important role in the protection mechanism of PDMS polymers having ATH fillers [40]. Thermal decomposition starts at the temperatures above 400 °C in the silicone rubbers [5], [21].

Silica filler has good thermal stability, the weight loss due to thermal decomposition is lower than ATH filler [14]. The thermal conductivity of ATH filler is initially higher than over silica filler. After during aging process, the conductivity of silica filler is higher due to the thermal stability. The reason behind the good performance of silica filler material is due to the strong interface interaction [14]. After dry band arcing, contents of Si and O increase while the content of C decreases in the silicone rubber [14]. Furthermore, it was reported that the shape of filler material was also important in terms of electrical and mechanical properties [14].

At the samples with 10%, 30%, and 50% wt filler ratio, the third harmonic component of leakage current is proportional with filler ratio while the fundamental component of leakage current show independent behavior with respect to filler ratio [20]. As the filler ratio increases, the third harmonic component of the leakage current decreases [20]. Nevertheless, this is not the case for the fundamental component. Besides, the third harmonic component of leakage current is correlated well with surface erosion.

1.2.3 Hydrophobicity mechanism

Hydrophobicity is a feature that prevents the formation of continuous water on the polymer surface. Due to the hydrophobic feature, the leakage currents are reduced to some extent [41]. The contact angle is the best method to measure the degree of the hydrophobicity [41]. The contact angle must be the more than 90 degrees for considering the surface as hydrophobic [41]. In the study [41], all silicone compounds showed similar contact angles of around 110 degrees.

It would be a wrong consideration that the filler material affects the hydrophobicity negatively [18]. Because the use of the surface-modified filler material increases both tracking-erosion resistance and decreases loss of hydrophobicity [18]. However, the

use of the unmodified filler affects the hydrophobicity negatively. Transferring of the LMW from the bulk to the surface, or recovering the hydrophobicity would be affected adversely by considering heavy filler loading, which is a drawback especially under contaminated operating conditions [40]. It was emphasized that surface modification of the filler particles is therefore important to reduce the quantity of water absorption which is crucial for insulators [40].

It was emphasized that increasing the pollution layer and temperature can increase the hydrophobicity recovery rate [42]. Recovery of the hydrophobicity is influenced by the ambient temperature significantly, and faster recovery occurs with higher temperatures [11]. The reason for this was explained by the formation of low molecular weight (LMW) from the high molecular weight (HMW) and subsequently, higher LMW diffusion in the pollution layer, resulting in faster hydrophobicity recovery [32], [42]. Due to their higher mobility, the shorter LMW chains would be responsible for the diffusion to the surface [11]. The recovery of the hydrophobicity occurs in a short time in the silicone rubbers [32]. Nevertheless, it could be found that the hydrophobic recovery rate is significantly reduced with aging [11]. The whitish substance formed during experiment indicates that a hydrophobic surface has appeared at the ATH filled samples [28]. Another result was that the increased Si-C peak during the experiment duration played a role in the recovery of hydrophobicity [42].

1.3 Aim of the Thesis

The purpose of the artificial aging test is to simulate the long-term operating condition of composite insulators in a relatively short time. There are a lot of artificial aging tests in the literature. In the present study, inclined plane test according to IEC 60587 was used to evaluate the silicone rubber samples consisting of several fillers in terms of tracking/erosion, leakage current, and thermal performances.

Aim of this study is to evaluate the best performance with the inclined plane test of silicone insulators used at high voltage applications. Many studies have been conducted on this purpose in the literature. In this thesis, the performance of the samples was evaluated by using inclined plane tests. Moreover, several analyses, such as TGA, DTG, DSC and FTIR were conducted to characterize the silicone rubber samples, and to explain the test results. In this study, fillers were added to polydimethylsiloxane high temperature vulcanizing (PDMS HTV) silicone rubber

material. The silicone rubber samples with two different particle sizes of silica and ATH fillers were tested at 3.5 kV and 4.5 kV constant test voltages. The best erosion performance, temperature distributions, leakage current variations, correlation between temperature and third harmonic power component of leakage current, the erosion depth and test duration were evaluated for the HTV silicone rubbers. Both material and surface analysis were performed to understand the fundamental mechanisms which have substantially improved the performance of micro composites. It was determined to the selecting high-performance material to understand the electrical, mechanical, and chemical degradations with these tests and analyses.

1.4 Hypothesis

The aim is to find the best insulation material with appropriate filler type and filler ratio of HTV-SIR material used in the high voltage insulation system, and to find the optimal combination showing the best tracking and erosion performance, leakage currents and temperature distributions. Two common fillers, ATH and silica, with two different particle sizes and three different concentrations in the HTV silicone rubbers are considered in this work.



2. TEST SETUP

2.1 Silicone Rubber Samples

Silicone rubber is formed from a mixture of several ingredients. Silicone rubber is resistant against oxidation, has low surface energy, and resistant against ultraviolet (UV) radiation. The resistance ability of silicone rubber originates from the silicon-oxygen bond. The polymer is formed from large molecule including atoms which consist of same base units. The same units repeated are identical and the number of the repeated units shows with number n . When the number n is small, the polymer shows low physical properties and has a low molecular weight (LMW). While number n is increasing, molecular weight increases and develops physical properties of the polymer. The main material for the silicone rubber is sand converted to silicone element by heating of carbon. The silicone rubber consists of the silicon-oxygen main chain and methyl groups attached to silicon-carbon bonds [43]. The number n polydimethylsiloxane (PDMS) structure is shown in Figure 2.1. The base silicone rubber can be consist of fillers such as extending and reinforcing fillers, one or more silicone gum, curing agents, vulcanizing agents, plasticizers, and softeners [10], [41], [43]. Plasticizers are used both to aid in mixing and to provide elasticity at low temperatures [43]. The filler material is chosen according to the property desired to reinforce the base material.

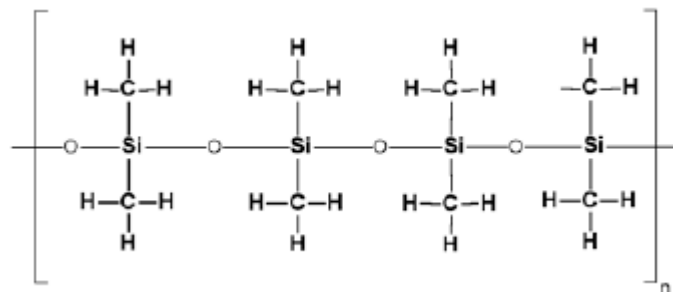


Figure 2.1 : The structure of PDMS [43].

Silicone rubber is organic due to consisting of carbon, and it is inorganic due to consisting of silicone [43]. Also, the filler materials such as ATH or silica are inorganic

[44]. The silicone gum is the pure material in silicone. MQ (dimethylsiloxane) as standard silicone rubber and VMQ (methyl vinyl siloxane) as modified silicone rubber are commonly used in high-voltage insulators. The silicone base consists of silicone gum and some filler materials. There have been two kinds of fillers namely reinforcing and extending fillers. Extending fillers consist of alumina trihydrate (ATH), titanium dioxide, ground quartz, clay, whiting, and zinc oxide while the reinforcing fillers consist of fumed silica, carbon black and aerogel silica [43].

In this study, heat cured HTV silicone rubber was used as the base material. The size of the test specimens was 120x50x6 mm with respect to IEC 60587 standard. HTV silicone rubber was designed from two mean particle sizes of ATH and silica fillers, with three different filler concentrations (10%, 30% and 50% wt). A total of thirteen cases were tested in the IP test. In order to prepare the HTV silicone rubber samples for IP tracking and erosion tests, a curing temperature of 160 ± 10 °C was maintained at around 8 minutes. The mentioned properties of the samples used in the tests are given in Table 2.1.

Table 2.1 : Features and designation of the filler materials.

	Designation	Median Particle Size (μm)	Specific Gravity (gr/cm^3)	Average Filler Ratio (wt%)
ATH-SB632	ATH1	3.6	2.42	10,30 and 50
ATH-HN434	ATH2	9.5	2.42	10,30 and 50
Min-U-Sil10	Silica1	3.4	2.65	10,30 and 50
Min-U-Sil40	Silica2	10.5	2.65	10,30 and 50

2.1.1 Base material

Momentive brand Silplus 50 MP was used as the base material. The specific density of the uncured base material is $1.15 \text{ gr}/\text{cm}^3$ at 23 °C, as provided in the technical document [45]. The base material was a polydimethylsiloxane (PDMS) mixture reinforced with fumed silica particles. The base silicone rubber used in the study consists of approximately 30% wt fumed silica. This was explained in more detail in Section 3.

2.1.2 Filler materials

In this study, alumina trihydrate (ATH) and silica (SiO_2) were utilized as the filler material. ATH filler is the Hubber brand, and silica filler is the U.S. Silica brand. The

ATH-SB632 and ATH-HN434 have the median particle size of 3.6 μm and 9.5 μm , respectively, and have the specific density of 2.42 g/cm^3 as provided in the technical document [46], [47]. Min-U-Sil10 and Min-U-Sil40 have the median particle size of 3.4 μm and 10.5 μm , respectively, and have the specific density of 2.65 g/cm^3 . The features of ATH ve silica were shown in Table 2.1. ATH-SB632, ATH-HN434, Min-U-Sil10 and Min-U-Sil40 were named as ATH1, ATH2, Sil1 and Sil2, respectively. Also, the % filler volumes and % wt filler ratios were calculated for the specimens consisting of ATH and silica fillers.

The volume fraction calculation for ATH filled or silica filled HTV SIRs

For a 1 cm^3 volume; ATH or silica have $x \text{ cm}^3$ volume and PDMS has $1-x \text{ cm}^3$ volume.

$$\frac{m_{ATH}}{m_{ATH} + m_{PDMS}} = 0.1 \rightarrow 10\% \text{ wt ATH filled sample}$$

$$\frac{x * 2.42}{x * 2.42 + (1 - x) * 1.15} = 0.1$$

$$x = 0.05015 \text{ cm}^3 \text{ volume for ATH1 - 10}$$

$$0.05015 * 100 = 5.015\% \text{ volume fraction ATH filler}$$

The specific gravity of silica has 2.65 and the volume fractions were calculated from the same formula. X-10, X-30, and X-50 are represented by filled samples corresponding to silicone rubbers for 10%, 30%, and 50% in weight filler ratios, respectively. The volume fraction calculated for ATH1-10, ATH1-30, and ATH1-50 filled HTV silicone rubber samples have 5.02%, 16.92%, and 32.21% vol and, for Sil1 filled samples, Sil1-10, Sil1-30, and Sil1-50 filled HTV samples have 4.6%, 15.68% and 30.26% vol, respectively. Table 2.2 shows the % wt and % volume filler fractions. Also, as can be seen in Table 2.2, the material hardness increased as the amount of filler increased. In the present study, explanations are done by giving designation according to the % wt filler ratio.

Table 2.2 : Features of the HTV samples.

	Designation	Filler Ratio (% weight)	Calculated Volume Fraction (%)	Hardness (Shore A)
ATH1	ATH1-10	10	05.02	61
	ATH1-30	30	16.92	66
	ATH1-50	50	32.21	77
ATH2	ATH2-10	10	05.02	60
	ATH2-30	30	16.92	65
	ATH2-50	50	32.21	70
Silica1	Sil1-10	10	04.60	56
	Sil1-30	30	15.68	64
	Sil1-50	50	30.26	75
Silica2	Sil2-10	10	04.60	56
	Sil2-30	30	15.68	63
	Sil2-50	50	30.26	74

2.2 Inclined Plane Test Setup

There are many test methods for evaluating the performance of insulators. Inclined plane test, dynamic drop test, salt-fog test, round robin test, and corona test are some of these methods. In this study, the inclined plane test was used for evaluating the tracking and erosion performance of the above mentioned HTV silicone rubber samples.

Tracking and erosion resistance tests of ATH and silica filled HTV silicone rubber samples were conducted according to IEC 60587 standard [48]. Assembly of the test setup was conducted according to this standard. The IP test setup is provided in Figure 2.2. To supply the required test voltage to the samples, a medium voltage power transformer with 30 kVA power rating, 50 Hz frequency and 220/22 kV voltage level were used. An oscilloscope was used to confirm the test voltage by using a high voltage probe. A variac was used to supply the voltage to the primary side of the transformer. Contaminant liquid was flowed from the upper electrode to the bottom electrode by adjusting the flow rate corresponding to the voltage level as stated in the standard on the sample surface via a peristaltic pump. Contaminant liquid was prepared using 0.1% by weight of ammonium chloride (NH₄Cl) and 0.02% by weight of non-ionic wetting agent (TRITON X100). In order to measure and monitor the leakage current flowing through each sample, the LABVIEW data acquisition system (National Instrument brand) was used. The leakage currents were recorded through 50 Ω shunt resistors to

the computer by using 10 kHz sampling frequency. The voltage values across the shunt resistors were inputted to the data acquisition system. Test voltages were selected as 3.5 kV and 4.5 kV. During the experiment, 22 k Ω and 33 k Ω pre-resistors were used corresponding to 3.5 kV and 4.5 kV test voltages, respectively. For 3.5 kV test voltage, the contaminant liquid flow rate was set to 0.3 ml/min, and 22 k Ω pre-resistors were selected. For 4.5 kV test voltage, the contaminant liquid flow rate was set to 0.6 ml/min, and 33 k Ω pre-resistors were selected. The inclined plane test setup with basic components was given in Figure 2.2.

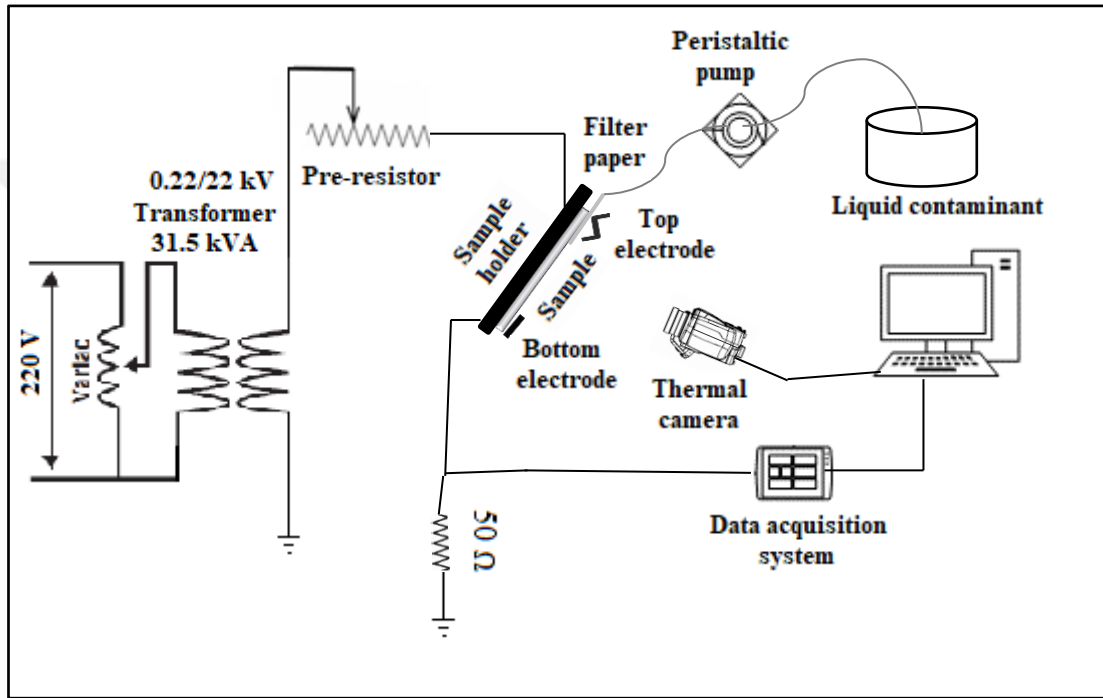


Figure 2.2 : Inclined plane test setup.

All samples were cleaned with isopropyl alcohol and distilled water, and remained at room temperature at least 24 hours prior to the test. Five identical test samples were used for each test condition. The electrode and filter paper were used according to the IEC standard while assembling samples. Filter papers were wetted completely by a peristaltic pump before applying test voltage. The test voltage was applied to the sample after a continuous water flow was provided from the top electrode to the bottom electrode. The distance between the two electrodes is 5 cm and the angle of the sample with horizontal is $45^\circ \pm 2^\circ$. A photo from the experiment is given in Figure 2.3. Criteria to terminate the experiment according to IEC-60587 standard:

- The test is terminated when the leakage current exceeds 60 mA for 4 seconds and above. (The relays were adjusted to open after exceeding 4 seconds).

- The test is terminated when a hole is developed on the sample, or the erosion path exceeds 2.5 cm.

If the criteria for terminating the test do not occur, the test is continued for 6-hours.



Figure 2.3 : A photo of the inclined plane test setup.

3. RESULTS

3.1 Introduction

In this section, physical and chemical analyses of the HTV silicone rubber test samples were explained. To evaluate physical and chemical properties, thermo gravimetric, TGA, derivative thermo gravimetric, DTG, differential scanning calorimeter, DSC, and Fourier transform infra-red spectroscopy analyzes, FTIR, analysis were conducted. In order to define the electrical and thermal performances, the specimens were evaluated with respect to test duration, the leakage current behavior, the eroded mass, the depth of erosion, the temperature behavior.

3.2 Material Analysis Results

The thermal decomposition occurs in polymeric material with increasing temperature. This decomposition is caused by dry band arcing, which is closely related to the tracking and erosion performance of materials. The Thermo-Gravimetric analysis (TGA) and Derivative Thermo-Gravimetric analysis (DTGA) are performed to understand the effect of the fillers and the reason for the residual weight [30]. Also, TGA and DTGA curves explain the thermal decomposition mechanism [5]. That is, these curves show the chemical and physical states as a function of temperature [49]. On the other hand, Differential Scanning Calorimeter (DSC) analysis gives information about the endothermic and the exothermic reactions [35] while Fourier Transform Infrared Spectroscopy (FTIR) analysis gives information about the chemical structure [32]. Therefore, the TGA analysis assesses the thermal stability of the material while the FTIR analysis shows with functional groups [28]. Analysis of FTIR and TGA are carried out to confirm both the filler amounts and filler types.

3.2.1 Thermal gravimetric and derivative thermal analysis

To test the thermal stability of the material, the Thermo Gravimetry-Derivative Gravimetric (TG-DTG) analysis is performed [34]. The DTG curve displays the first order derivative weights on the y-axis, and temperature on the x-axis while the TG curve gives the sample weight loss change regarding to temperature [34]. The TG curve gives the change of the mass in a stable atmosphere with temperature. Also, it explains the degradation mechanism. The eroded mass due to increasing of the temperature indicates that the sample is deteriorated or evaporated at these points. The areas where there is no weight loss are those where the material is stable. The weight loss occurs in two stages for ATH filled HTV-SIR. While it occurs decomposition of ATH in the first phase, it occurs decomposition of PDMS in the second phase [50]. The decomposition of ATH filled silicone rubber material starts at 220-250 °C and ends at 340-360 °C [33]. The decomposition of PDMS starts at 340-360 °C and ends at about 600 °C [33]. For silica filled samples [34], the weight loss begins at 400 °C due to the thermal decomposition and ends at around 600 °C. After 600 °C, the TGA curves are stable [42]. The analyzed sample can be taken about 10-20 mg [30], [37], and can be heated up to 1200 °C in a stable heating rate [5].

As showed in the results of the TGA analysis, the base silicone rubber has less residual weight than the samples with ATH filled. As seen in the TGA curves, the weight loss occurs in a stage for silica filled silicone rubbers while it occurs in two stages for ATH filled silicone rubbers. ATH filled silicone rubbers showed two stages of weight loss due to ATH filler as mentioned above. The effect of the dehydration of ATH was observed at the first stage. On the other hand, single stage weight loss occurred in the silica filled silicone rubbers since there was no any dehydration.

In present study, the thermal stability was evaluated in nitrogen atmosphere by thermogravimetric analysis (TGA). TGA curves of the test samples are shown in the Figure 3.1. Black, blue, red and green lines in the TGA curves correspond to base, 10, 30 and 50% wt ATH and silica filled silicone rubbers, respectively. In this study, TGA analysis was carried out in the temperature ranging from 30 °C to 900 °C at a controlled heating rate of 20 °C / min. Similar TGA curves were obtained for the other samples. In another study [36], The samples with nanoparticle and microparticle sizes were showed similar TGA curves for the same filler type and filler ratio. In another study

[28], FTIR and TGA curves after the samples tested in different voltage types were found to be similar [28].

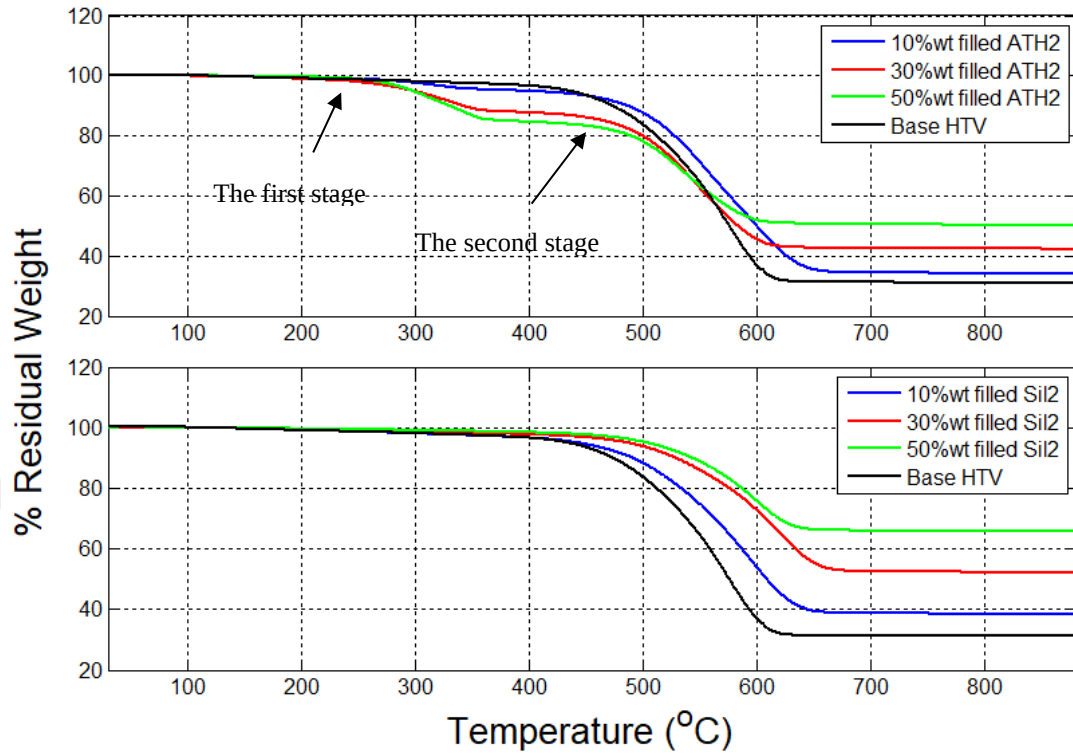


Figure 3.1 : TGA analyses of ATH2 and Sil2 filled HTV-SIR samples.

In the study [28], it was stated that the first peak in the DTG curve occurs around 340 °C, and it has been observed that the aged samples are about twice as much weight loss due to decreased ATH with respect to unaged samples. The second peak caused decomposition of SIR occurs above 400 °C, and it is the same in both aged and unaged samples [28]. For samples with ATH and silica filler, Table 3.1 is given peak temperature values of the decomposition points, and residual weights at the TGA analysis result. For the other particle size samples with the same filler loading and filler type, it was obtained similar results. It can be seen in the Figure 3.1 and in Table 3.1, the residual weight ratio increased when the filler ratio increased. That is, weight loss reduces as concentrations of fillers increase [20]. TGA curves and Table 3.1 show that in the base silicone rubber, around 31.15% residual weight is available. As showed in DTG curve in study [36], methyl groups were released from the main silicone chain (Si-O-Si) at around 500 °C. It was observed that the decomposition of water caused by the release of hydrate water in ATH filled materials occurs at temperatures at around 220 °C. But, the decomposition of PDMS for both ATH and Silica filled samples started at around 400 °C and finished at around 650 °C. That is, the DTG curves given

in Figure 3.2 were used to see the temperature range where the silicone rubber decomposes. The DTG curves are intended to validate the temperature points where maximum weight losses occur. As can be seen, the temperature values at which peak point of degradation of the samples with silica filler were higher. This is due to the strong interface interaction with the silicon matrix of the silica material. Therefore, its thermal stability is better than ATH one [14]. It was said that the decomposition peak decreases with the decrease in particle size in samples with silica filled [30].

Table 3.1 : For a group of samples with ATH and silica filled, peak points of the decomposition temperatures and their residual weights.

Samples	Peak Point of Dehydration of ATH (°C)	Peak Point of Decomposition of PDMS (°C)	Residual Weight (%)
ATH1-10	322.8	586.39	33.70
ATH1-30	352.2	544.07	40.68
ATH1-50	351.6	523.60	50.43
Sil1-10	-	597.35	39.42
Sil1-30	-	592.85	52.55
Sil1-50	-	580.53	65.94
ATH2-10	319.3	561.19	34.12
ATH2-30	328.3	544.67	42.29
ATH2-50	325.6	541.17	50.21
Sil2-10	-	595.01	38.44
Sil2-30	-	620.88	52.21
Sil2-50	-	601.06	65.88
Base HTV	-	573.40	31.15

Residual weights were about 33.7%, 40.675% and 50.43% wt respectively for ATH1 samples with 10%, 30% and 50% wt filler loading. In the first phase, weight losses with water release decreased 4.6%, 9.52%, and 15.52% wt, respectively. For ATH2 samples with 10%, 30% and 50% wt filler loading, the weight losses with the release of water at the first stage, which started about 220 °C, were 4.6%, 11.76% and 14.73%, respectively. With Sil1 samples, weight losses occurred in a single stage due to the decomposition of the silicone content and residual weights were present at 39.42%, 52.55%, and 65.94% for 10%, 30% and 50% silica filled samples, respectively. The second stage of weight losses started around 400 °C by degradation of silicone material. DTG curves of ATH2 and Sil2 samples are illustrated in Figure 3.2. All data in Table 3.1 was obtained according to the results of TG-DTG analysis.

As it is seen in Figure 3.2, the amount of water released increased as the ATH filler ratio increased. On the other hand, the weight loss of PDMS decreased with increasing ATH or silica filler ratio.

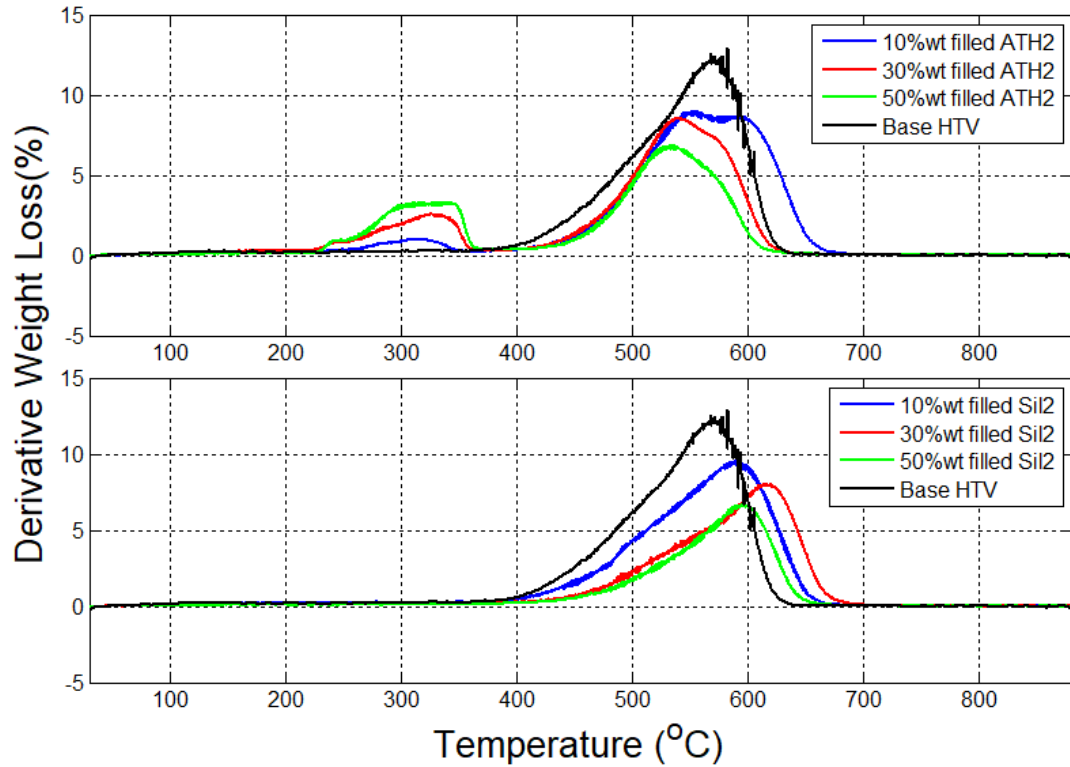


Figure 3.2 : DTG curves of ATH2 and Sil2 filled HTV-SIR samples.

3.2.2 Differential scanning calorimeter

Differential scanning calorimeter (DSC) analysis is explained by the heat exchange of the system in a stable atmosphere. The DSC analysis is the amount of energy given by unit volume and unit time, (W/g). It is determined the amount of energy liberated with this heat exchange. Also, it determines endothermic and exothermic reactions corresponding to the heat of the material. These reactions determine the thermal changes such as oxidation, polymerization, evaporation, dehydration, and melting occurring in the structure by temperature changes. It was reported that the first weight loss begins around 210 °C in the ATH filled sample at the DTA curve and associated with endothermic [37] dent due to the dehydration of ATH [29], [37]. It was reported that the enthalpy change caused by dehydration of the water increased the heat capacity and thus the temperature was suppressed [37]. Besides, the dehydration of the water was said to form a protective layer in the sample [37]. In the DSC curve, it is found

that the peak temperature value of the endothermic dent increases as the temperature rising per minute increases [50].

The thermal degradation for the silica filler, the first weight loss begins around 400 °C in the TGA curve [30]. Si-O bonds are the depolymerization and then volatile cyclic oligomers [30]. After that, in this case of the temperature exceeding 600 °C in the DTGA curve, it was said that cross-linking may have the effect of scission of Si-C bonds. In the study, that is carried out with silica filled samples, it was emphasized that the mobility decreased with the decrease in the particle size, and subsequent evaporation decreased and therefore the exothermic peak decreased [30]. In addition, exothermic peak decreased with increasing of the filler ratio.

The higher the filler ratio in samples filled with ATH, the greater the energy released by the enthalpy change [5], [51]. It was also found that the temperature due to water dehydration has been suppressed by the increase of enthalpy in ATH filled samples [5], [51]. In the present study, the enthalpy change is 433.85 J / g in the sample filled with 50% ATH while it is 279.58 J / g in the sample with 30% ATH filler. The higher endothermic peaks were seen in the samples containing 50% wt ATH filler as shown in Figure 3.3. The reason for this situation is due to the amount of ATH filler in the silicone rubber since endothermic areas relate to the ratio of ATH filler directly. Due to the absence of oxidation of water in the base and silica filled samples, these endothermic peaks were not observed.

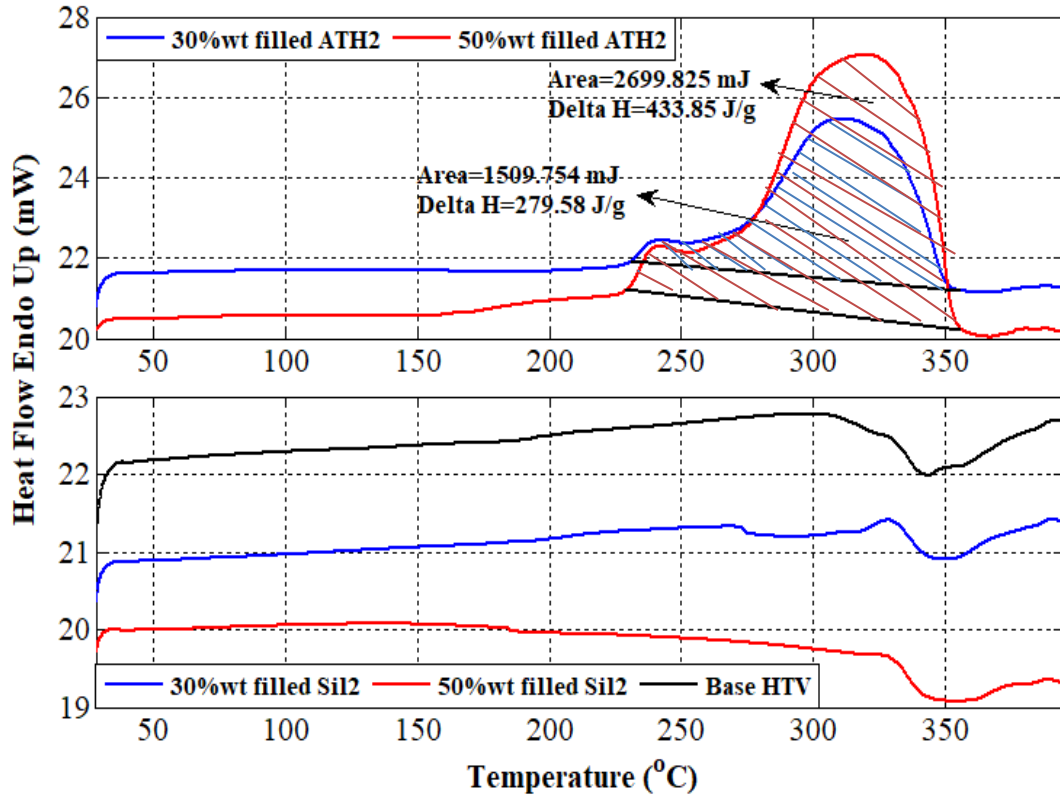


Figure 3.3 : DSC curves of ATH2 filled, Sil2 filled, and base HTV-SIR samples.

3.2.3 Fourier transform infrared spectroscopy

The FTIR analysis demonstrates the chemical bond properties found in composites. [29]. It is used in material analysis before and after aging through absorption bands [28]. This is often used to describe the chemical changes on the surface of the SIR material due to the aging [34]. The chemical bonds in silicone rubber are: O-H (hydroxyl) groups at $3700\text{--}3200\text{ cm}^{-1}$, symmetric C-H bonds at 2960 cm^{-1} , Si-CH₃ bonds at $1270\text{--}1255\text{ cm}^{-1}$, Si-O-Si bonds at $1100\text{--}1000\text{ cm}^{-1}$ and C-Si bonds at 786 cm^{-1} wavelengths [29], [32]. The vibration of H₂O molecules is 1629 cm^{-1} [34]. This band is also observed at 868 cm^{-1} due to Si(CH₃)₃ [34]. At these wavelengths, the downward changes in the FTIR curves show the bonds that are broken in the main chain [32]. Figure 3.3 shows the FTIR analysis of the HTV silicone rubbers. The FTIR spectrum used in the present work is in the range of $4000\text{--}500\text{ cm}^{-1}$ wavelengths.

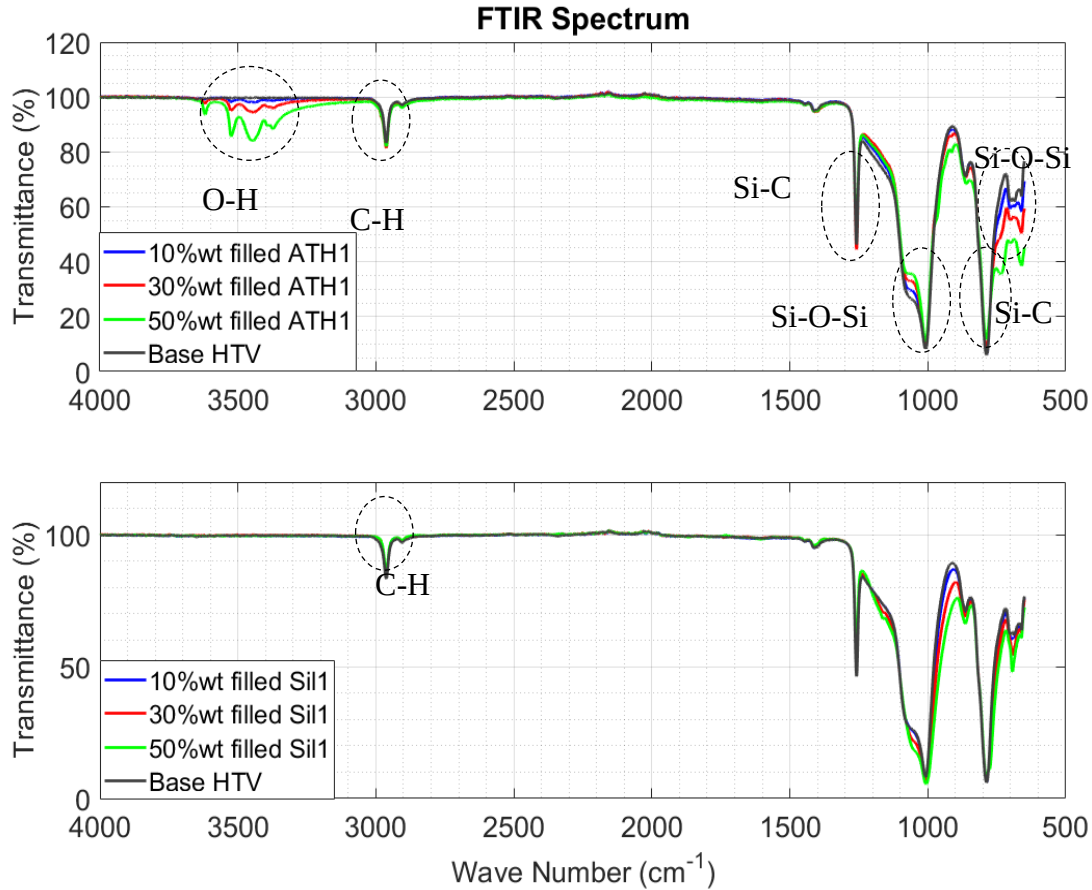


Figure 3.4 : FTIR analyses of ATH1 and Sil1 filled HTV-SIR samples before the test.

As can be seen in the FTIR curves, the ATH filled silicone rubber contains O-H bonds due to the water molecules. Water release plays an important part in the PDMS polymer protection mechanism [40]. It was recorded that it could be inferred from the IR spectrum that Si-C bond decomposition contributes to the major weight loss with a significant reduction in IR spectrum absorption peaks [33].

In the study done ATH filled samples [28], the O-H group situated in the ATH decreased as a result of FTIR analysis performed after the test. Tracking formation due to the dry band arcing of the aged samples changes the structure of the SIR polymer by reducing the polymer chain, resulting in a harder point on the SIR surface, resulting in less vibration of the CH bond, thereby reducing the CH tensile density [28]. Si-C bond does not change because it forms of the structure of the polymer matrix. The decrease of the Si-O bond is due to arc and heat. Whereas in another study [36], it was shown that the increase of Si-O-Si bond in the samples with silica filler can form a protective silicate layer on the sample.

For 50% in weight ATH and silica filled silicone rubbers, the FTIR analyzes of before and after the test were compared. As shown in the Figure 3.5., in the ATH filled sample, the spectrum changed due to the O-H bond while in the silica filled HTV silicone rubber, no change was observed. The O-H bond decreased due to the dehydration of water in the ATH filled sample and it can be seen in Figure 3.5. For ATH filled samples, it decreased the absorption peaks of the C-H, Si-C, and Si-O bonds due to aging. But this decrease was very less in the silica filled samples, and the reduction did not ever become at the Si-O bonds for the absence of the water molecule.

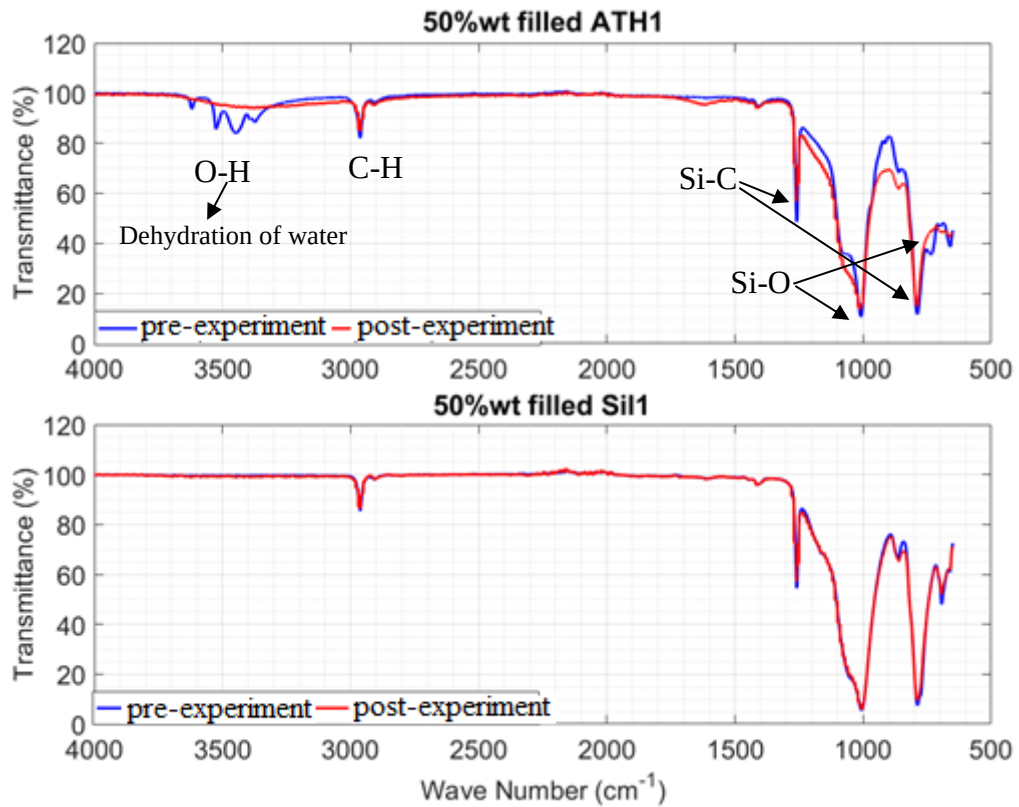


Figure 3.5 : FTIR analyzes of samples before and after the experiment.

3.3 Experimental Results

3.3.1 Inclined plane test

Figure 3.6 and 3.7 show the photos of the test samples after IP tests. For each type of filler, 10% wt of filled silicone rubbers had the same erosion behaviour with base silicone rubber. The erosion path from the bottom electrode to the upper electrode was available for 30% wt silica filled samples while 30% wt ATH filled samples, holes occurred near the bottom electrode. Due to intense discharges, the holes formed at 30%

wt ATH filled samples, but surface erosion was observed for silica-filled samples, this situation may be explained by the mechanism of thermal decomposition [17]. While all the samples filled with 50% ATH passed the tests, two of the samples filled with 50% silica failed in the tests. This is due to the fact that hydration of water in high-filled ATH materials increases erosion performance for RTV samples [37]. It is emphasized that the released water forms a protective layer, and this layer suppresses discharges from the dry band arcing. For both 50% ATH1 and 50% ATH2 filled samples, the dehydration of water formed a protective layer in this study, and they passed the tracking and erosion test. Nevertheless, samples from both the Sil1 and Sil2 groups showed similar performance for 50% wt filler loading. For samples with silica filled, it can be said that more than 50% wt filler ratio may be required for desired inclined plane tracking an erosion test.

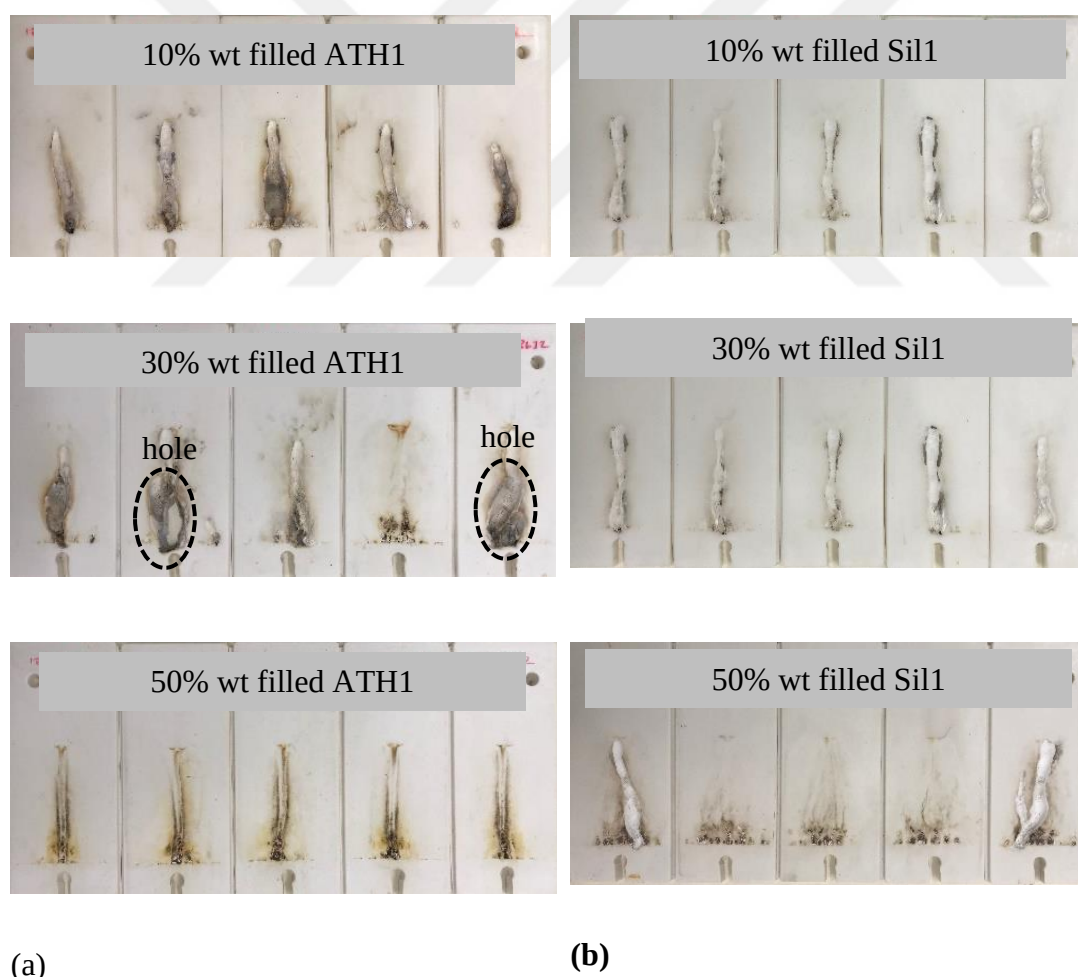


Figure 3.6 : HTV samples after the inclined plane tests at 3.5 kV (a) 10, 30 and 50% wt ATH1 filled HTV samples (b) 10, 30 and 50% wt Sil1 filled HTV samples.

In this section, the situations described so far were for the 3.5 kV voltage level. However, the similar results were obtained for the 4.5 kV voltage level. In the study [30], the silicone rubber with 50% wt silica filled failed under the critical AC voltage (4.5 kV), and it formed the erosion path. In this study, since 50% wt silica filler ratio was insufficient, the samples were failed for both voltage levels. On the other hand, all samples with 50% ATH filler passed the tests in the IPT under both 3.5 and 4.5 kV voltage level.

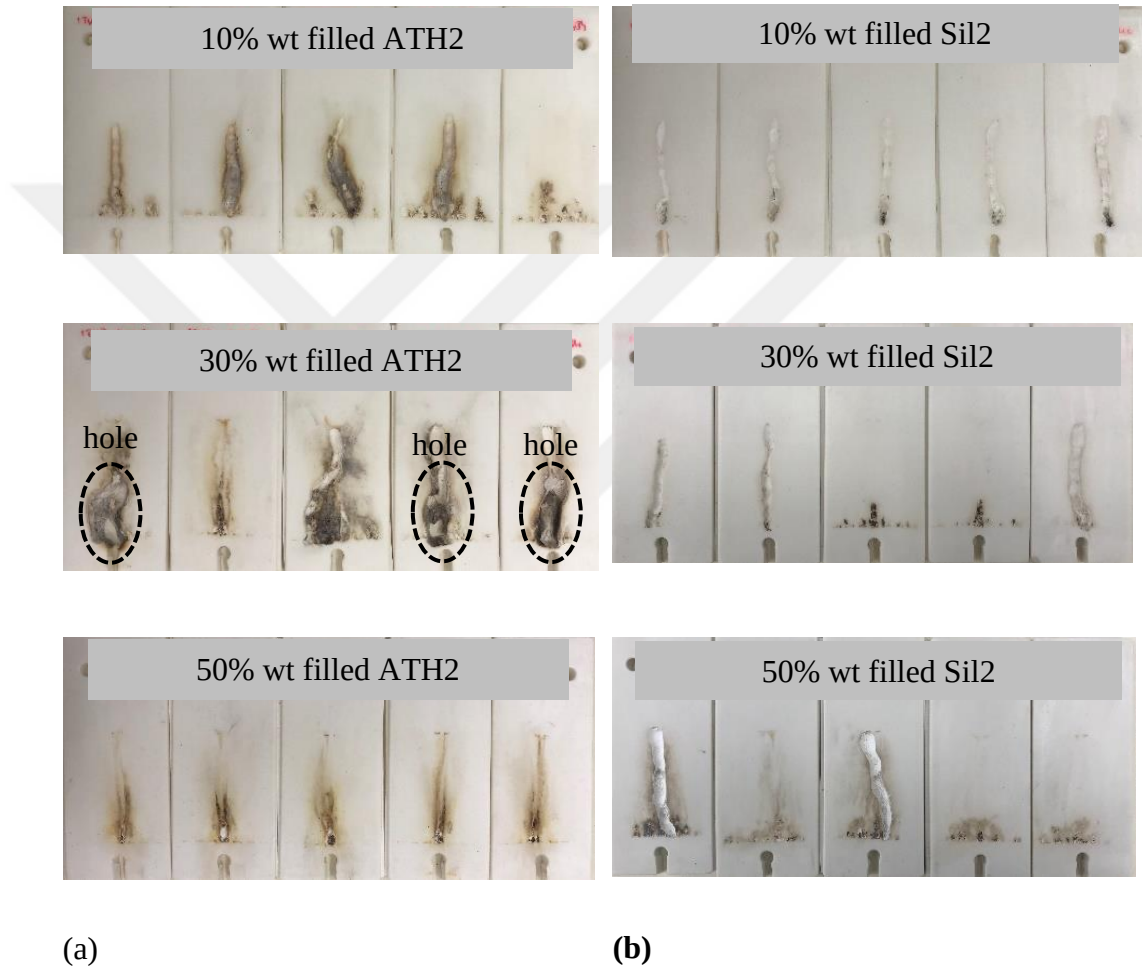


Figure 3.7 : HTV samples after the inclined plane tests at 3.5 kV (a) 10, 30 and 50% wt ATH2 filled HTV samples (b) 10, 30 and 50% wt Sil2 filled HTV samples.

3.3.2 Test duration

The test duration is an important parameter to evaluate the material performance. A lot of studies were made comparing test durations of samples [5], [18]. The test duration was carried out for six hours for the samples according to standard [48]. The test was terminated for failed samples considering the criterion, and then the test was continued

for the remaining samples. Figure 3.8 and Figure 3.9 indicate the specimens' test durations, corresponding to test voltages of 3.5 kV and 4.5 kV levels. The test durations increased slightly with increasing the particle size for the ATH samples having 10% and 30% wt filler amounts.

Test duration increased as the filler ratio increased for all samples in the voltage level of 3.5 kV. Base silicone rubber and ATH1, Sil1 and Sil2 samples having 10% wt filler ratios showed almost similar time durations behaviors. However, the 10% wt loading ATH2 samples performed better than ATH1. The test duration of silicone rubbers with 30% filler ratio improved compared to 10% filler loading for both samples filled with ATH and silica. In the 30% wt filler ratio, ATH2 and Sil2 samples performed slightly better than ATH1 and Sil1 ones. For the 50% wt filler ratios, there was a significant increase in failure times of both ATH and silica filled samples. While all of the samples filled with ATH1 and ATH2 of 50% wt filler ratio passed the IP tests, there were two failed samples under 3.5 kV test voltage in the Sil1 and Sil2 filled samples.

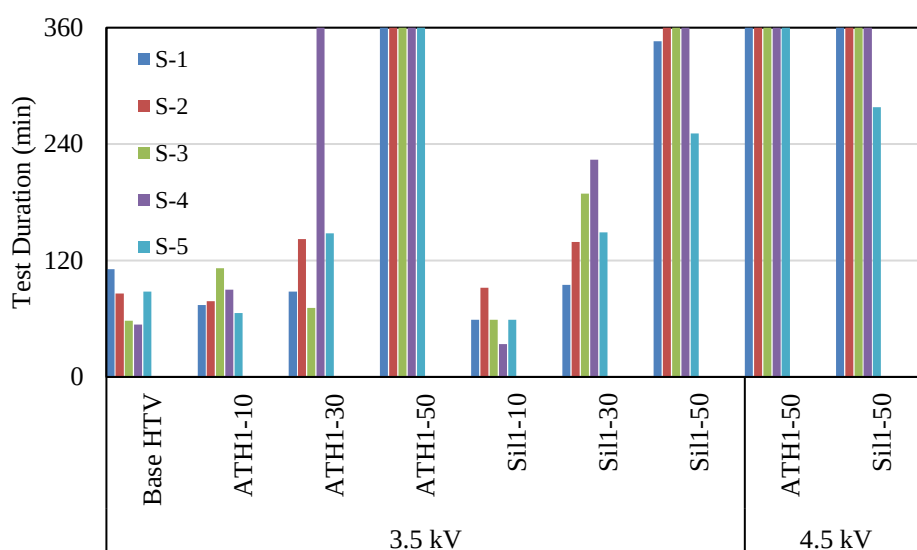


Figure 3.8 : Test durations for the samples with small particle size in IPT.

Under both filler conditions, 10% and 30% wt filled samples failed at 3.5 kV, the samples with these filler ratios were not tested again at 4.5 kV voltage level. For 4.5 kV voltage level, only the samples with 50% wt filler were tested. The samples filled with ATH1 and ATH2 showed similar test duration performances at 4.5 kV, and all samples withstood this voltage level. For 50% wt Sil1 and Sil2 filled samples, one sample failed in Sil1 group, and three samples failed in Sil2 group test samples. Test

durations of Sil1 samples were better at 4.5 kV test voltage compared to Sil2 samples. While 50% wt filler ratio was sufficient to pass the test for ATH filled samples, it was concluded that higher filler ratio is required for samples with silica filler. The differences in the test duration are due to the duration and magnitude of the leakage current as well [34].

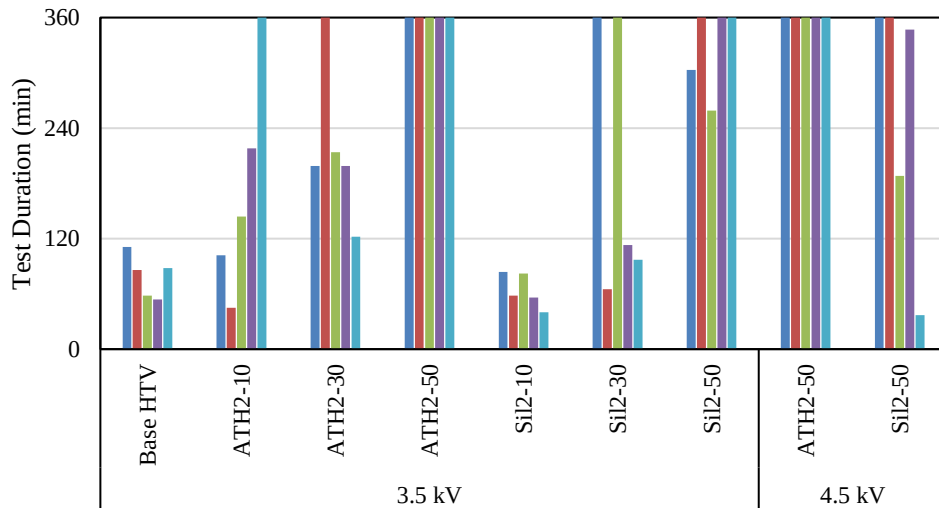


Figure 3.9 : Test durations for the samples with large particle size in IPT.

Average test duration of five samples tested for each group is given in Figure 3.10. As can be seen, for two filler types, the test duration increased gradually by increasing the filler concentration.

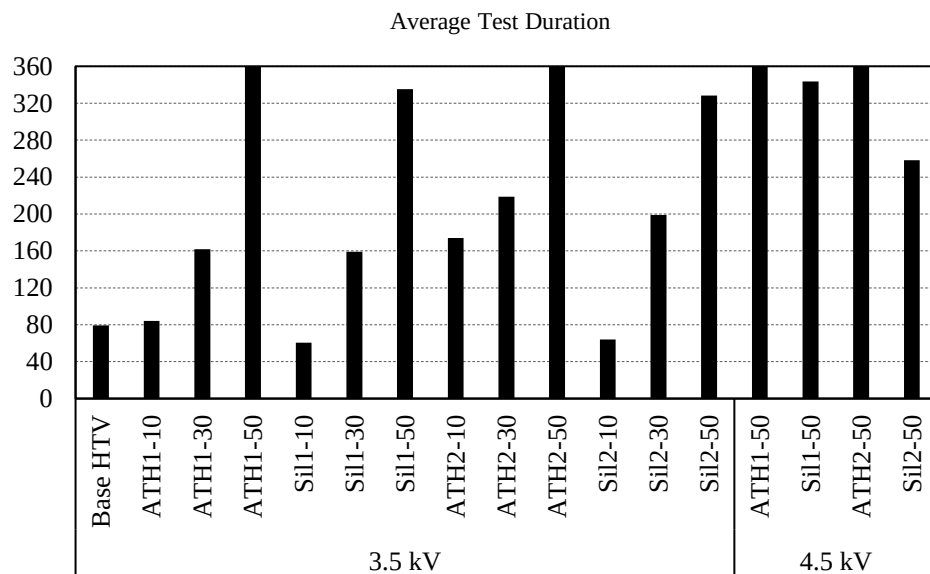


Figure 3.10 : Average test duration of five samples tested for each group.

3.3.3 Eroded mass and erosion depth

All of the samples' weight was recorded before and after tests. Figure 3.11 demonstrates the eroded mass for SIRs filled with ATH and silica after IP test. All of the 10% wt ATH and silica filled samples except one of the ATH2 samples, a full erosion path bridging from the bottom to the top electrode occurred as soon as the erosion initiated. As shown in Figure 3.11 for 10% wt filler ratio, the eroded masses were less because intensive discharges did not occur around the bottom electrode, and it took less time from the initiation up to the full erosion bridging the electrodes. The erosion performance of ATH2 samples was better than ATH1 ones, and erosion performance of Sil2 samples was slightly better than those of Sil1 ones at 3.5 kV voltage level. ATH filled and silica filled samples showed different behaviors in terms of test duration and eroded masses for 30% wt filler loadings. For Sil1 and Sil2 samples having 30% wt silica filler, the erosion performance was similar, and the erosion formed in the form of erosion path from the bottom electrode to the top electrode. ATH filled SIRs showed the worst performance at 30% wt filler ratio in terms of eroded masses. In the 30% wt ATH filled samples, the holes occurred on some samples due to intensive local discharges around the bottom electrode. Large amounts of eroded masses were observed for 30% wt ATH filled samples due to the formation of holes. Two samples failed due to the holes in the ATH1 samples having 30% wt loading while three samples in the ATH2 ones. Therefore, the eroded mass was higher for ATH2 samples. It was observed that the ATH filled samples showed good erosion performance at 50% wt filler ratio as shown in Figure 3.11. For 50% wt ATH filled samples at 3.5 kV test voltage, no noticeable effect was observed with respect to the particle size. For 50% wt silica filled samples, the right side of the bars appeared high since eroded masses of the failing samples increased. It was observed that the eroded masses were less in the samples that passed the test as the filler ratio of silica filled samples increased. On the other hand, the eroded mass was generally high at high filler ratios of silica-filled samples since failing samples showed large amounts of eroded masses. For 50% wt silica filled samples at 3.5 kV test voltage, Sil2 samples showed slightly better erosion performance from Sil1 ones.

ATH and silica filled silicone rubbers were also tested for 4.5 kV voltage level with a filler ratio of 50% wt. For ATH-filled samples, the samples of ATH2 group showed

similar eroded masses at both voltage levels, but eroded masses for ATH1 samples were slightly higher at 4.5 kV than the 3.5 kV. That is, the erosion performance of ATH2 samples with 9.5 μm mean particle size was better than ATH1 samples with 3.6 μm mean particle size at 4.5 kV test voltage. For silica filled samples, it was observed that the eroded masses of Sil1 samples decreased as compared to 3.5 kV at a voltage level of 4.5 kV, and eroded masses of Sil2 samples were almost the same at both voltage levels. At silica filled samples, the erosion performance of Sil1 samples with small mean particle size was better than Sil2 samples with large mean particle size. According to the test result with at 4.5 kV voltage level, the erosion performance was better in samples with large particle size with ATH filler. However, silica filled samples with small particle size performed better than that of the large particle size.

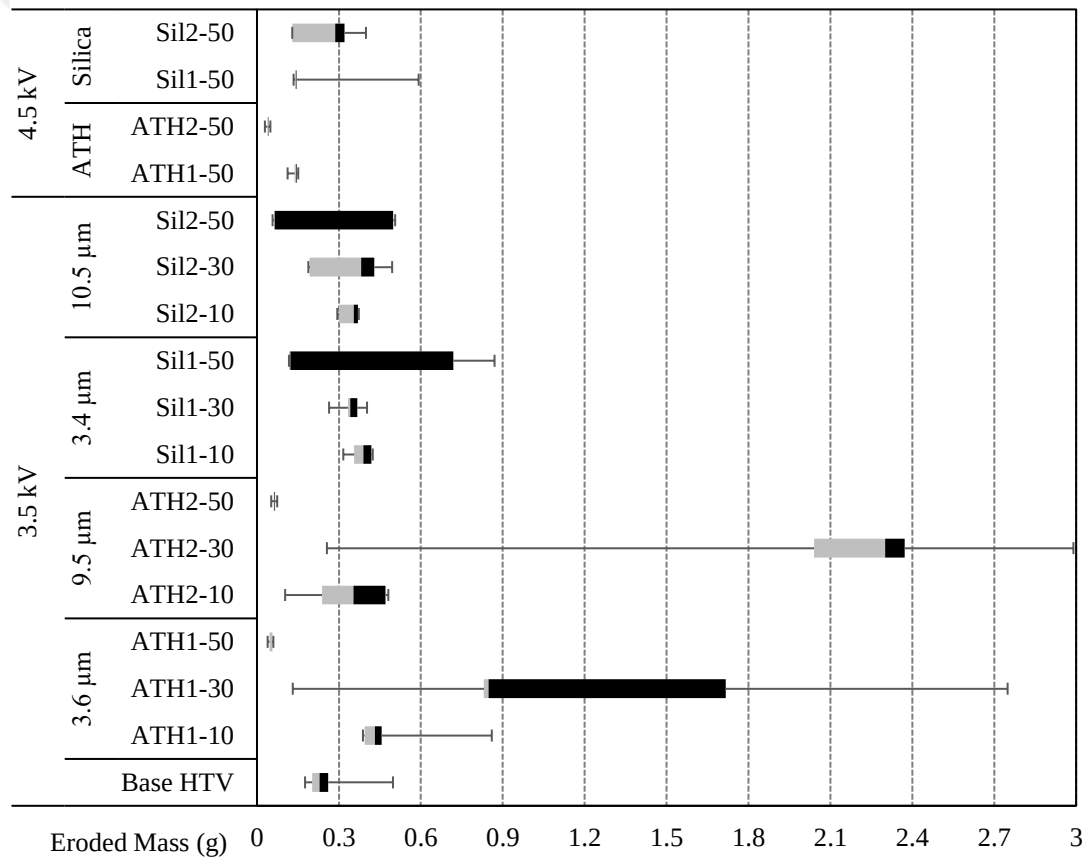


Figure 3.11 : Eroded masses of HTV-SIRs (The end portions of the bars indicate the minimum and maximum amounts of erosion, the end portions of the hatched bars indicate the amount of erosion at 25 percent and 75 percent, and the center line indicates the median eroded mass of the data).

The eroded masses obtained for all filler type, concentration ratio, particle size, and voltage level are shown in Table 3.2. Also, the average, median, and standard deviation values of the eroded masses are given in table. As can be seen in the Table 3.2, the

samples with the lowest average weight loss and standard deviation were samples with 50% ATH filler while the samples with the highest weight loss and standard deviation were samples with 30% ATH filler. The average weight loss and the standard deviation for silica filled samples did not provide a reliable information.

Table 3.2 : Average, median and standard deviation of the eroded mass and eroded masses of HTV silicone rubbers after IP tests.

Filler		Eroded Masses (g)							standard deviation
Type	S-1	S-2	S-3	S-4	S-5	average	median		
3.5 kV	ATH1-10	0.394	0.432	0.860	0.388	0.457	0.506	0.432	0.200
	ATH1-30	0.848	2.749	0.831	0.131	1.717	1.255	0.848	1.007
	ATH1-50	0.057	0.039	0.046	0.060	0.057	0.052	0.057	0.009
	ATH2-10	0.238	0.353	0.471	0.481	0.103	0.329	0.353	0.161
	ATH2-30	2.990	0.256	2.040	2.372	2.301	1.992	2.301	1.031
	ATH2-50	0.052	0.064	0.074	0.066	0.062	0.064	0.064	0.008
	Sil1-10	0.390	0.356	0.316	0.424	0.419	0.381	0.390	0.045
	Sil1-30	0.264	0.342	0.333	0.367	0.403	0.342	0.342	0.051
	Sil1-50	0.719	0.118	0.122	0.117	0.870	0.389	0.122	0.374
	Sil2-10	0.294	0.373	0.300	0.354	0.370	0.338	0.354	0.038
	Sil2-30	0.188	0.381	0.193	0.430	0.495	0.337	0.381	0.140
	Sil2-50	0.506	0.065	0.499	0.063	0.057	0.238	0.065	0.241
Base HTV	0.176	0.498	0.261	0.202	0.229	0.273	0.229	0.130	
4.5 kV	ATH1-50	0.138	0.151	0.144	0.146	0.112	0.138	0.144	0.015
	ATH2-50	0.029	0.038	0.042	0.041	0.049	0.040	0.041	0.007
	Sil1-50	0.142	0.145	0.140	0.134	0.592	0.231	0.142	0.202
	Sil2-50	0.129	0.130	0.321	0.399	0.286	0.253	0.286	0.120

Figure 3.12 shows the erosion depths which were formed on the samples after the test for both 3.5 kV and 4.5 kV test voltages. All of the samples with 10% wt filler ratio and base silicone rubber showed almost similar performance in terms of erosion depths. For the ATH filled samples at the 3.5 kV voltage level, the maximum erosion depth was observed for samples with 30% wt filler due to the existence of holes, and the minimum erosion depth was observed for samples with 50% wt filler. For at 3.5 kV test voltage, in the case of silica filled samples, the average erosion depth decreased as the filler ratio increased. The performance of the erosion depth in silica filled samples with 30% wt filler ratio was better than samples filled with ATH. For 50% wt filler ratio, this performance was better in ATH filled SIRs than silica filled SIRs.

The samples with 50% wt filler ratio was also tested for at 4.5 kV test voltage. It was found that the average erosion depth for ATH1 and ATH2 filled samples increased with increasing the voltage level. However, it was found that the average erosion depth

of silica filled samples decreased for Sil1 samples, and there was no significant change for Sil2 sample. No noticeable effect both 3.5 kV and 4.5 kV test voltages were observed with respect to the particle size of the fillers.

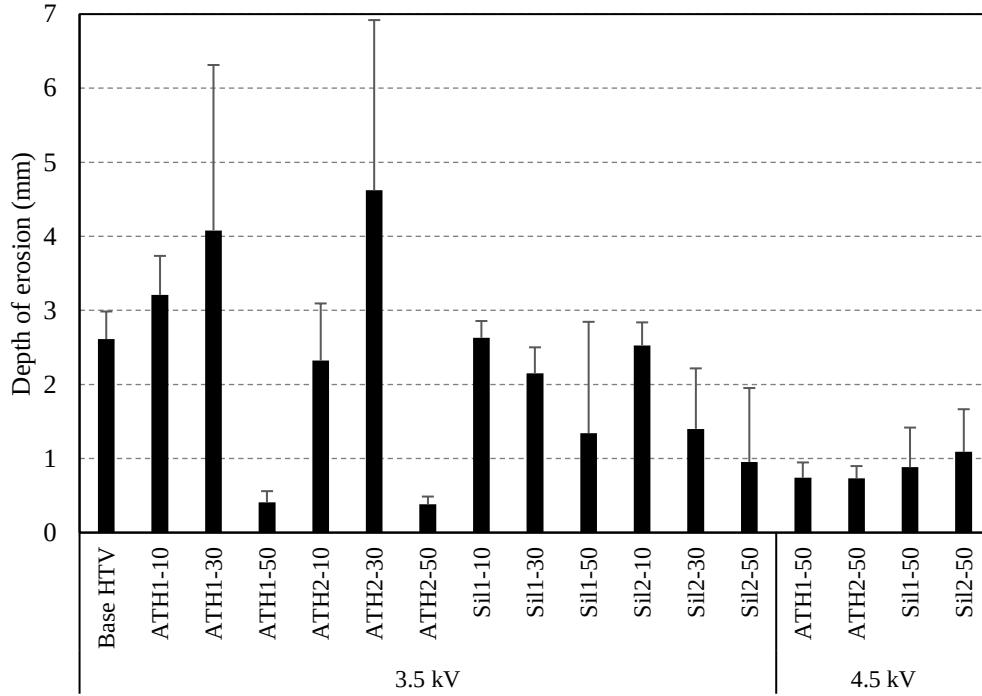


Figure 3.12 : The average erosion depths of all tested samples and standard deviation (The end portions of the bars indicate the standard deviation of erosion depth; the end portions of the hatched bars indicate the average of erosion depth).

Comparing the erosion depths, test durations, and the eroded masses of HTV samples after the tests, it was found that the samples filled with ATH2 showed better performance than.

3.3.4 Leakage current behavior

At the data acquisition system, sampling group size was determined as 2000 and, sampling frequency value was selected as 10 kHz. This recording was performed with the block diagram created in LabVIEW software, and one section of the block diagram is given in Figure 3.14. Leakage current data were recorded by taking 5 samples per second through 50 Ω shunt resistors at regular intervals for 6 hours test duration. After 6-hour test duration, 108000 data were obtained for a sample. The maximum, r.m.s. and harmonic components of leakage currents were recorded. USB-6341 data card was used for this purpose [52]. Figure 3.13 shows an instantaneous leakage current of the samples received through the 50 Ω shunt resistors.

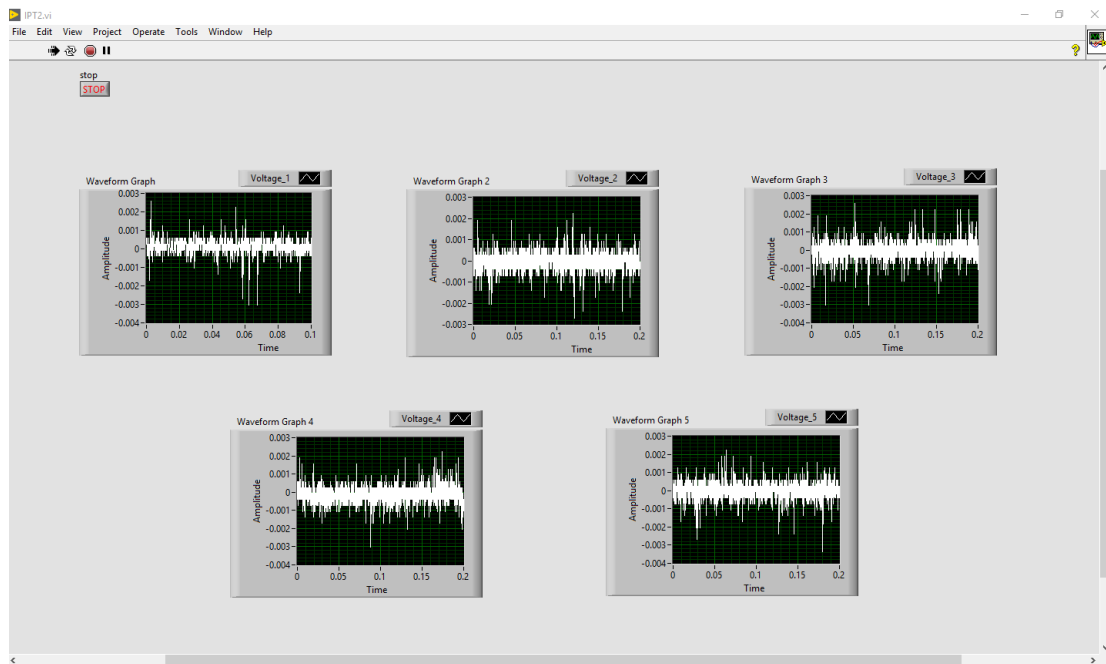


Figure 3.13 : Instantaneous leakage current in the program interface.

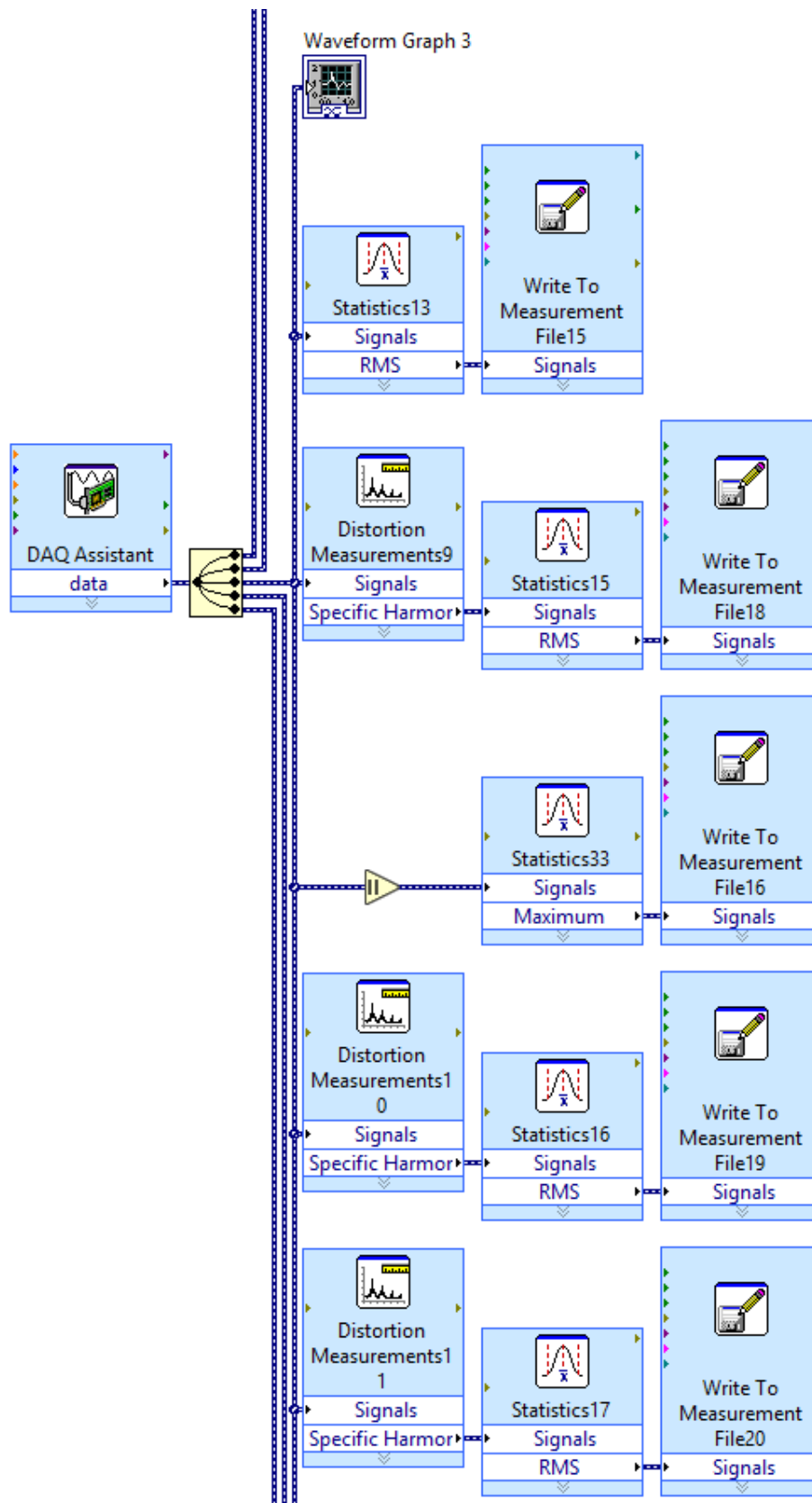


Figure 3.14 : Block diagram created in LabVIEW software.

The average values of the leakage current of all samples are given in Table 3.3. For all filler ratios at the 3.5 kV test voltage, it was observed that silica filled samples were effective than ATH filled samples at the suppression ability of the leakage current. But, both ATH and silica filled samples showed similar leakage current performance at the 4.5 kV voltage level. It was observed that the leakage current increased with increasing of the test voltage. It was reported that, regardless of the ATH level, leakage current values increase with increasing voltage levels [5]. Effects of filler particle size were not observed in the leakage current behaviours for the samples.

Table 3.3 : The average leakage current values of all samples.

	Type	Filler		Average r.m.s. Leakage Current (mA)					Average R.M.S. LC (mA)
		Wt (%)	Size (µm)	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	
3.5 kV	ATH	10	3,6	5,66	5,86	6,43	5,36	5,54	5,8
			9,5	5,82	5,55	5,85	5,37	5,90	5,7
		30	3,6	6,28	6,16	6,29	6,03	6,52	6,3
			9,5	7,16	6,55	6,83	5,81	6,22	6,5
		50	3,6	6,32	6,59	6,58	6,03	5,62	6,2
			9,5	6,34	6,15	6,84	6,38	5,85	6,3
	Silica	10	3,4	5,57	5,39	5,34	5,48	4,92	5,3
			10,5	5,47	5,28	5,49	4,98	5,23	5,3
		30	3,4	5,72	5,98	6,05	5,79	5,52	5,8
			10,5	6,15	5,68	6,29	5,46	5,48	5,8
		50	3,4	6,03	5,95	6,04	5,56	5,68	5,9
			10,5	5,91	6,00	6,04	5,63	5,47	5,8
	Base HTV	--	--	5,33	5,89	5,86	5,35	5,29	5,5
4.5 kV	ATH	50	3,6	9,66	9,89	9,95	9,52	8,96	9,6
			9,5	9,11	9,40	9,88	9,40	9,28	9,4
	Silica	50	3,4	9,58	9,85	9,98	9,10	9,39	9,6
			10,5	9,61	9,80	9,26	9,20	8,27	9,2

The leakage currents were variable due to discharges and dry band arcing during the experiment, and harmonic components were available in the current due to the dry band arcing. For 10% wt silica filled and base HTV silicone rubber samples, r.m.s value of the leakage current was around 15-20 mA while the maximum value of it is around 30 mA. But r.m.s leakage current value of 10% wt ATH filled samples was around 20-25 mA, and it was around 25-30 mA for 30% wt ATH filled samples while it was around 20-25 mA for 30% wt silica filled samples. For 50% wt ATH and silica filled samples, it was around 15 mA. For all samples with 50% wt filler ratio in the 4.5 kV test voltage, r.m.s of leakage current was found around 25-30 mA. The raw and smoothed data of the leakage current are given in Figure 3.15 and 3.16.

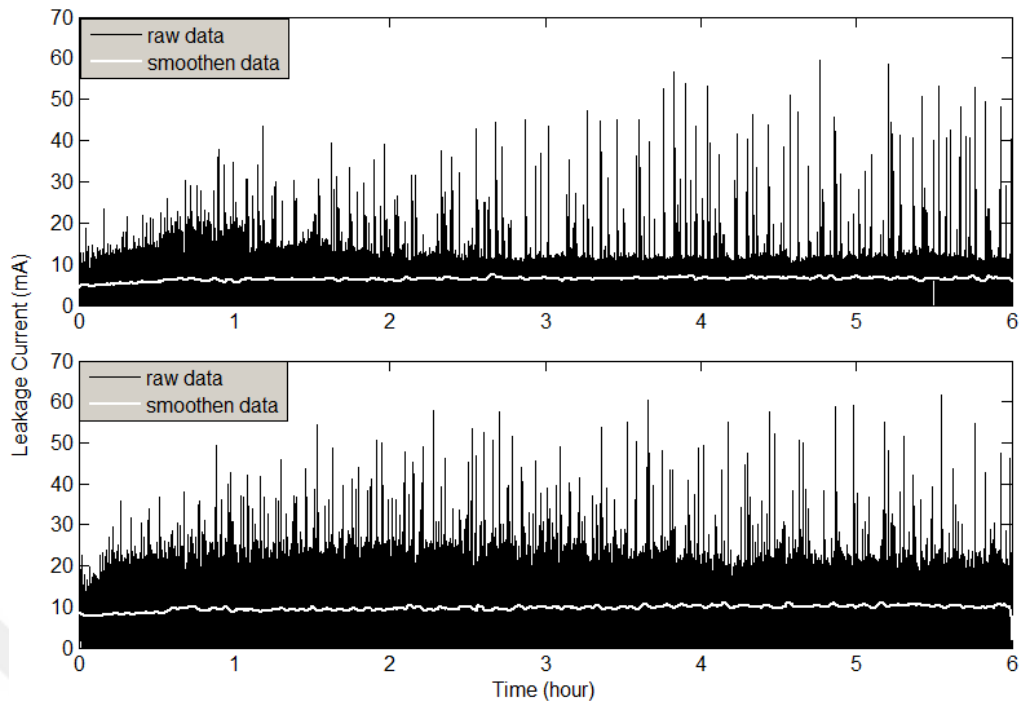


Figure 3.15 : R.m.s. leakage current for ATH1 with 50% wt filled at 3.5 kV and 4.5 kV (Sample-1). The smoothed data with 750 data points.

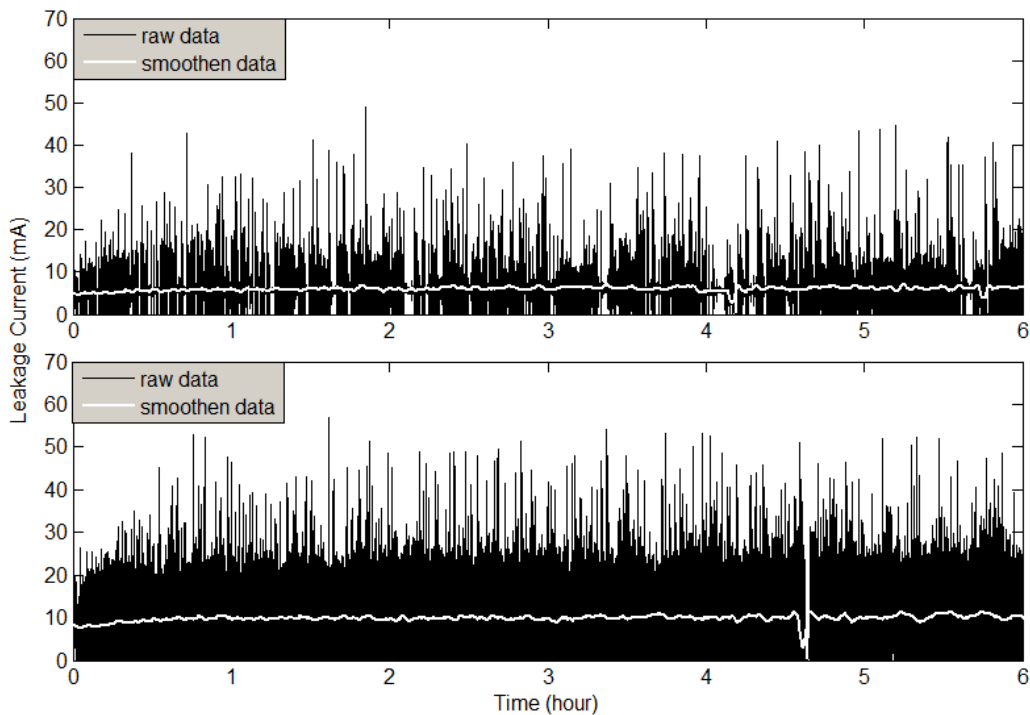


Figure 3.16 : R.m.s. leakage current for Sil1 with 50% wt filled at 3.5 kV and 4.5 kV (Sample-2). The smoothed data with 750 data points.

The leakage current values in the sample with 50% wt ATH1 filler for the 3.5 kV test voltage were mostly 15 mA and also, the LC values in the sample with 50% wt Sil1 filler were mostly 15 mA as seen in the Figure 3.15 and Figure 3.16. Higher current

values were also available for both filler ratios. However, due to the thermal stability of the silica, these values were less than the ATH filled sample. For at 4.5 kV test voltage, the LC values of the samples having both ATH1 and Sil1 filled were about 30 mA. The current values increased with increase in the test voltage. In the study [24], the current values increased with increasing of the test voltage for the polymer material.

3.3.5 Temperature behavior

The FLIR T420 brand thermal camera was used to monitor the temperature rise caused by leakage current, dry band arcing and partial discharge on the sample surface [53]. It was recorded the maximum temperatures on the samples surface by using thermal camera. The thermal camera sensitivity used is between -20 °C and 670 °C. The maximum temperatures on the sample surfaces were recorded for each second. 21600 temperature data were obtained for each sample during 6-hourr test period. The image of the thermal camera in the computer interface is as given in Figure 3.17.

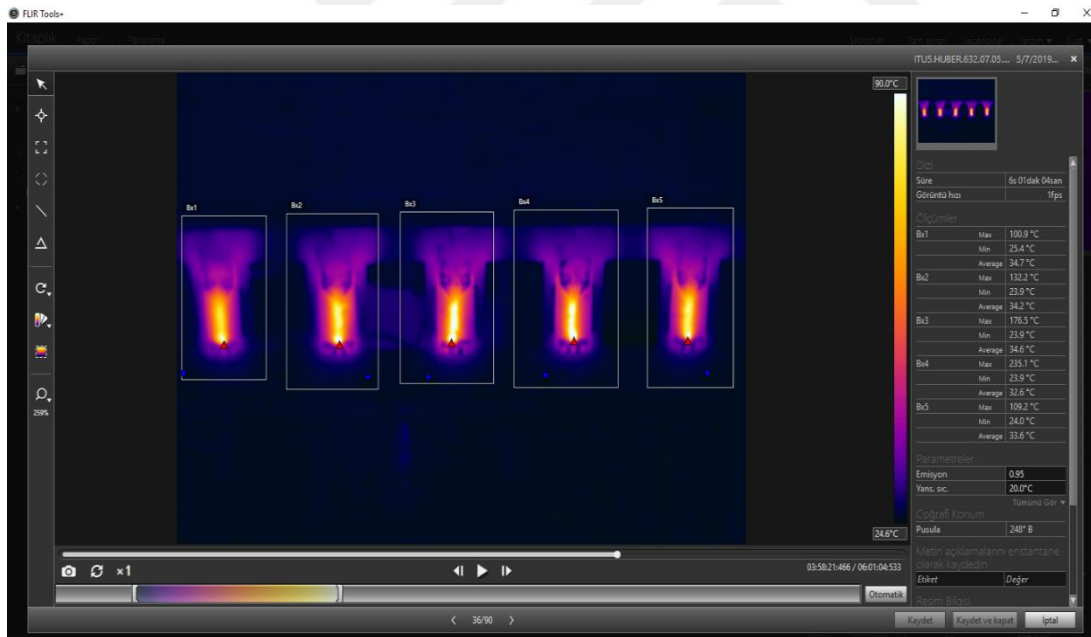


Figure 3.17 : Thermal camera image on computer interface.

The temperature changes on the sample surface were recorded by the thermal camera during the test, and two examples are given in Figure 3.18. The image on the left is the thermal camera image of the failed sample due to the hole. The maximum temperature formed on the sample at any time instantly before no failed was 486 °C. As can be seen, the maximum temperatures occurred around the bottom electrode. The image on

the right is the thermal camera image of the sample that passed the test. The temperature evenly distributed throughout the area under the voltage. In this case, the instantaneous measured maximum temperature value was 193 °C.

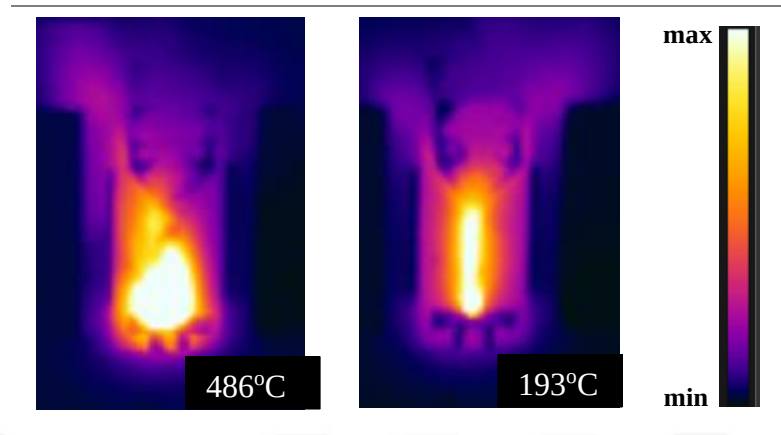


Figure 3.18 : FLIR IR image of ATH silicone rubbers. ATH2 sample with 30% wt filler ratio on the left, ATH1 sample with 50% wt filler ratio on the right.

Table 3.4 shows the average of the maximum temperature of each sample and all samples in the same group. At test voltage of 3.5 kV, the samples filled with 10% wt silica displayed a slightly better thermal performance than samples filled with 10% wt ATH. For a loading condition of 30% wt, silica filled samples obviously had better thermal performance than ATH filled samples, and the average of the maximum temperatures of the ATH filled samples were about twice than those of silica-filled samples. The maximum temperature values on the sample surfaces increased due to the intensive dry band discharges around the bottom electrode in ATH filled HTV samples. For the samples with 50% wt filler ratio in the 3.5 kV voltage level, silica had lower temperatures than the ATH filled samples again, but this difference was not as high as the 30% wt filler condition.

Table 3.4 : The average temperature values of all samples.

	Filler		Average Temperature (°C)					Average Temperature (°C)
	Type	Wt (%)	Size (μm)	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
3.5 kV	ATH	10	3,6	149,0	156,8	160,8	167,9	181,1
			9,5	148,4	164,3	163,5	137,7	181,3
		30	3,6	175,0	276,3	191,0	139,3	209,6
			9,5	301,3	197,2	206,3	182,3	205,2
		50	3,6	107,1	145,6	143,8	159,5	123,1
			9,5	161,8	168,4	228,1	166,2	133,9
	Silica	10	3,4	115,6	128,5	137,2	183,4	141,6
			10,5	110,6	150,5	138,9	150,4	145,0
		30	3,4	82,1	87,0	90,4	90,1	90,3
			10,5	120,9	103,6	90,8	88,4	87,9
		50	3,4	104,3	111,7	127,5	129,5	124,0
			10,5	128,9	146,1	160,8	158,4	136,2
	Base HTV	--	--	79,8	108,6	110,6	101,7	99,4
4.5 kV	ATH	50	3,6	132,7	172,5	172,4	283,6	132,3
			9,5	125,7	130,1	193,8	154,8	142,1
	Silica	50	3,4	78,0	83,3	88,1	84,8	85,8
			10,5	139,2	192,0	153,9	197,6	133,4

No noticeable effect was observed in temperature with respect to particle size for both ATH and silica filled samples having 10% wt filler loading under the 3.5 kV test voltage. However, in samples containing small particle size of fillers, the temperature performance was better for both 30% and 50% wt filler conditions.

The Sil1 group performed better than Sil2 group at a voltage level of 4.5 kV while the ATH2 group performed better than the ATH1 group. The results showed that all the silica-filled samples showed better thermal performance for all filler ratios, except for the Sil2 group at 4.5 kV test voltage.

The specific thermal activity of silicone rubbers with ATH and silica filler is due to the different interface interaction between the filler material and the rubber matrix. In the case of silica filled samples, this interaction is strong while it is weak in the case of ATH filled ones. In silica filled samples, the temperature does not rise excessively due to the strong interface interaction. The samples filled with silica are, therefore, said to perform better in terms of thermal stability, as explained in [14].

For the ATH1 (sample-2) and Sil1 samples (sample-5) maximum temperature and r.m.s. leakage currents are given for both instantaneous and smoothed values in Figure 3.19 and in Figure 3.20. For the leakage current, there were 108000 data points, and

window width was selected as 1500 points for the smoothed the data. For the temperature values, there were 21600 data points, and window width was selected 300 points for the smoothed the data. If the sample temperature reaches a certain level, then the erosion begins, the leakage current rises and the sample fails. As shown in Figure 13.19, the surface temperature of the sample increased after four hours, and erosion occurred within a few minutes, and the temperature exceeded 600 °C. The third harmonic value of the leakage current is directly related to the temperature changes that occur on the sample surface. In this situation, on the sample surface, the erosion occurred quickly from the bottom to the upper electrode, and the sample failed in the IPT. For the samples filled with silica, the initiation of the erosion and the sample failure occurred in a shorter time than the samples filled with ATH. The reason for the excessive mass losses in the samples that do not have sufficient ATH filler is the prolonged erosion activity as seen 30% wt ATH filler samples.

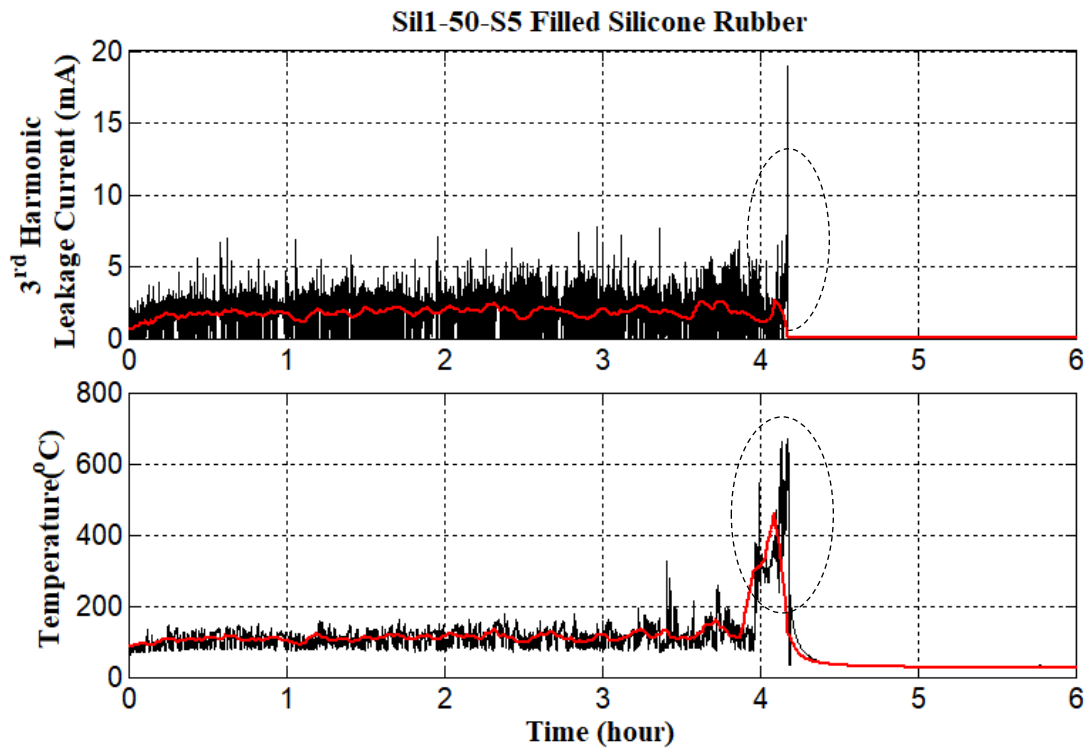


Figure 3.19 : 3rd harmonic leakage current and maximum temperature for 50% wt Sil1 filled sample. Raw (black) and smoothed (red) data.

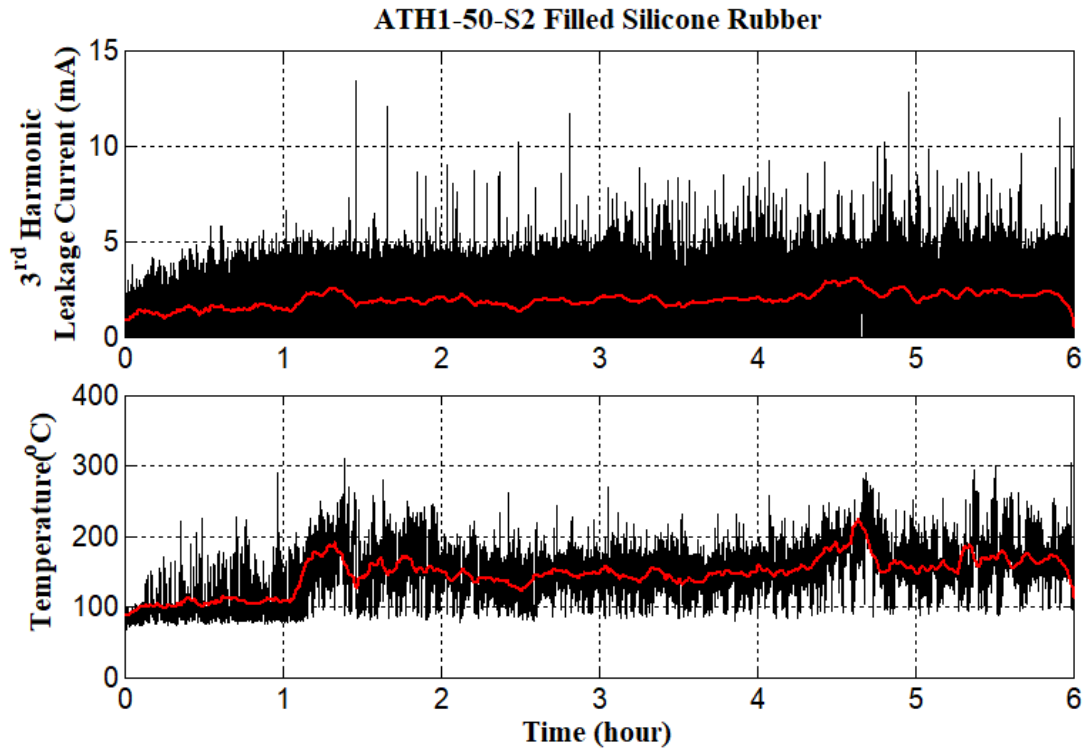


Figure 3.20 : 3rd harmonic leakage current and maximum temperature for 50% wt ATH1 filled sample. Raw (black) and smoothed (red) data.

3.3.6 Correlation between temperature and 3rd harmonic component of leakage current

The dry areas that occurred by increasing the temperature cause to occur dry band arcing. The dry band arcings occur harmonic components. For observing the correlation, the test result was evaluated with the data obtained. As seen in Figure 3.21, 3rd harmonic power component and temperature data were found to be correlated. In Figure 3.21, it is given an example for both ATH and silica filled specimens.

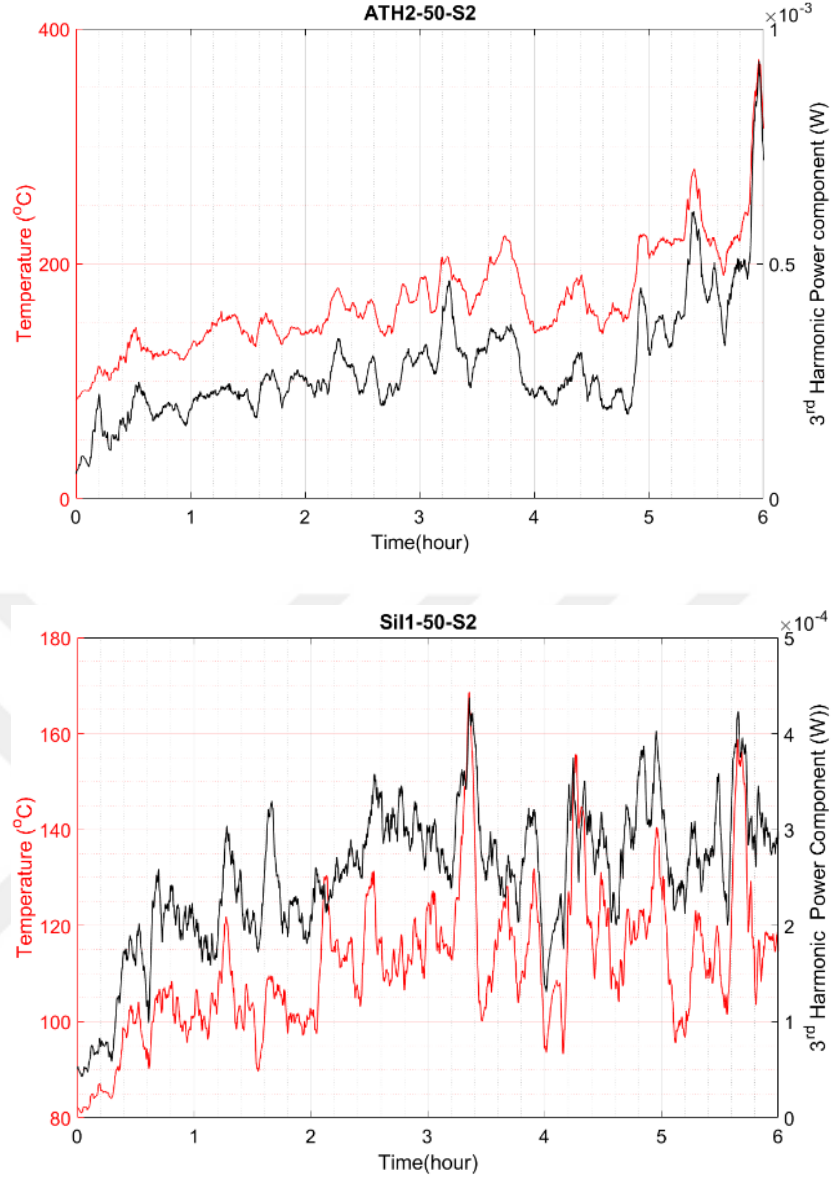


Figure 3.21 : Correlation between temperature and third harmonic power component for 50% wt filled ATH2 (sample 2) and 50% wt filled Sil1 (sample2) under 3.5 kV test voltage. Temperature (red) and 3rd harmonic power component (black).

As seen in the Figure 3.21, the correlation is the best between the third harmonic power component (HPC) and the maximum temperature (T_m). The next correlated harmonic is the by 7th HPC. But correlation is the worst in the fundamental (1st) and 5th HPC. These results are supported the study of Meyer et al [3]. Also, the power component values decreased as the number of the harmonic component increased.

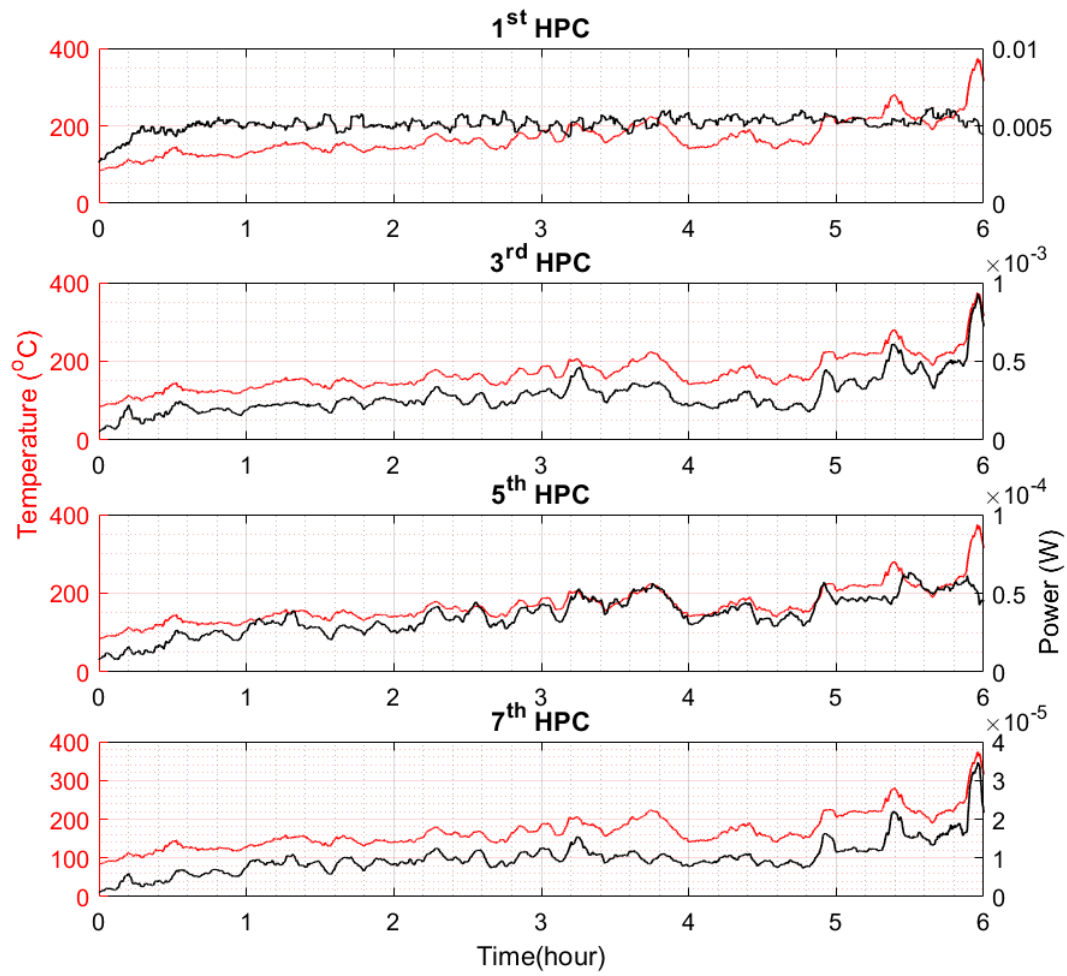


Figure 3.22 : Correlation between the maximum temperatures and the harmonic components of the sample having 50% wt ATH2 filled (Sample-2). Temperature (red) and power components (black).

4. DISCUSSION

In this study, HTV silicone rubber samples with different concentration and type of filler were exposed to inclined plane test. In addition, all physical, chemical and electrical behaviors of the materials were evaluated.

Different physical and electrical features of ATH and silica have. Both fillers dilute the polymer amount and they decrease the cost. ATH filler material has high heat capacity and this high heat capacity increase the amount of thermal energy that is used to vaporize the crystal water. The water vapour makes the formed gas less flammable, also, char produced as a result of discharges creates an insulation effect [19]. As for silica, it has good interface interaction and forms a good bonding with the silicone polymer matrix. Also, it performs well in terms of thermal stability [14], and it is an important filler to obtain good mechanical properties in the silicone rubbers [54]. Silica material has strongly bonded to the silicone rubber matrix due to molecular proximity, but this is not the case for ATH particles [9]. This study was conducted to evaluate the effects of two filler types originating from these properties. Thermal conductivity of ATH is better than silica, but the bonding of silica with the silicone matrix is better than ATH [14], [22].

In the study [17], ATH showed better tracking and erosion performance than silica at high filler ratio. In another study for 50% wt filled silica, ATH, and magnesium hydroxide (MH) samples, ATH showed better tracking and erosion performance than silica. Moreover, MH filled samples showed close performance with ATH filled samples [37]. That is, it is emphasized that the dehydration of the water is a major role in reducing erosion [37].

In order to evaluate the material performances, not only the criterion of passing or failing of the test, but also characteristics such as mass loss, erosion depth, erosion length, eroded volume is considered to be good indicators for determining the material performance [18], [19], [54].

At the high filler ratios, the erosion formed around the bottom electrode in the silicone rubber filled with ATH while it formed as an erosion path from the bottom electrode to the top electrode in silicone rubber filled with silica [17]. In the present study, it was observed a similar condition for 30% wt ATH and 30% wt silica filled samples. In terms of eroded masses and erosion depth for 30% wt filler ratio, the performance of ATH filled samples was poorer than that of silica filled samples because of the formation of holes in ATH filled samples (see Figures 3.11 and 3.12). The biggest eroded mass due to hole occurred the samples with 30% wt ATH filler. ATH samples having 20% wt filler ratio showed poor performance compared to unfilled samples [40]. Therefore, a sufficient quantity of ATH filler must be added to ensure that ATH has a positive effect [40]. In the present study, the eroded mass of the samples having 30% wt ATH filler was higher than base HTV and 10% wt ATH filled HTV samples. It was emphasized that in samples with the same filler ratio and type, the difference in mass losses due to erosion is also high due to irregular arc movements [40]. In the present study, there were differences in the eroded masses of the samples. As can be seen in Figure 4.1, the standard deviations are high. Also, although dehydration of the water with ATH filled samples serves as a protective function, this is not the case for low concentrations [22].

For the two filler types having 50% wt filler ratio, the tracking and erosion performance increased. For the 50% wt filler ratio, the some of the silica filled samples passed the test while all of ATH filled samples passed the test. For ATH filled samples, approximately 45-55% wt ATH filler is used to improve HTV silicone rubber tracking and erosion performance [54]. With RTV silicone rubber with 50% wt filler ratio, ATH filled samples performed better tracking and erosion resistance than the silica filled ones [37]. The first reason behind this behavior was explained by the hydration of water in ATH, and the second reason was char formation that might suppress the erosion [37]. HTV silicone rubbers [5] requires about 40% ATH filler by weight to improve tracking and erosion performance. RTV silicone rubbers requires about 50% silica loading by weight to improve erosion performance [44]. In the present study, since 50% filler concentration was sufficient in ATH filled samples, all samples passed the test at both 3.5 and 4.5 kV test voltage, and also eroded mass and depth of erosion were considerably reduced. However, for samples with 50% silica filler, this concentration was not sufficient and there were samples that failed the test. In addition,

the silica filled samples compared to ATH filled samples, there was no significant decrease in eroded mass and erosion depths. Thus, we can say that different types of fillers cause different structural changes unless the filler ratio is chosen above the critical value. Average test duration of five samples tested for each group increased gradually by increasing filler concentration as seen in Figure 3.10. In the IPT done with 57% unmodified (particle size 1.1 μm) and 57% modified (particle size 1.1 μm) ATH filled samples under 6 kV test voltage, while all of the modified samples passed the test, some of the unmodified samples failed the test [18]. In another study [31], the time-to-failure increased by increasing filler ratio of HTV-SIRs having ATH filler.

In the study with RTV [44], it has been found that surface erosion decreases with the increasing particle size of silica filled samples under 4.5 kV test voltage. It was specially obtained a significant reduction in the samples having 5 μm mean particle size [44]. In silica filled samples [44], as a result of the reduced interaction between the filler and the base polymer, the amount of char has been reported to increase as the filler particle size increases. The char has been found to play an important role in increasing erosion resistance by acting as a heat shield [44]. However, the increase in char amount in ATH filled samples was found to have had a negative effect on erosion resistance [31].

In the study [30], the silicone rubber with 50% silica filler indicated better erosion depth performance with increasing filler particle size. In the present study, it found better erosion performance of the Sil2 samples having 10.5 μm particle size according to Sil1 having 3.4 μm particle size under the 3.5 kV test voltage. However, Sil1 samples was better in terms of erosion performance at 4.5 kV test voltage. Another study found that the voltage level affected the erosion volume in ATH filled samples [18]. In this study, the effect of the particle size was not seen at ATH filled samples under 3.5 kV voltage, but it was effective under 4.5 kV voltage.

There is a tendency that larger particles perform better than smaller ones provided that the specific surface area is less than 5 m^2/g even though the effect of the particle size on the eroded volume is quite weak [40]. Boehmite formation, would be the reason which enables a release of water up to higher temperatures. [40].

If the same sample size are used, the size of the confidence interval may be considered as consistency indicator between different materials [18]. In samples filled with ATH,

the confidence limit decreases as the sample size increases [18]. As shown in Figure 4.1, there was the lowest the confidence limit in the samples having 50% wt ATH filled, and they showed the best tracking and erosion performance. There was the highest the confidence limit in the samples having 30% wt ATH filled, and they showed the worst tracking and erosion performance. But, since the silica filled samples did not have an effective filler ratio, the confidence limit did not provide any important information.

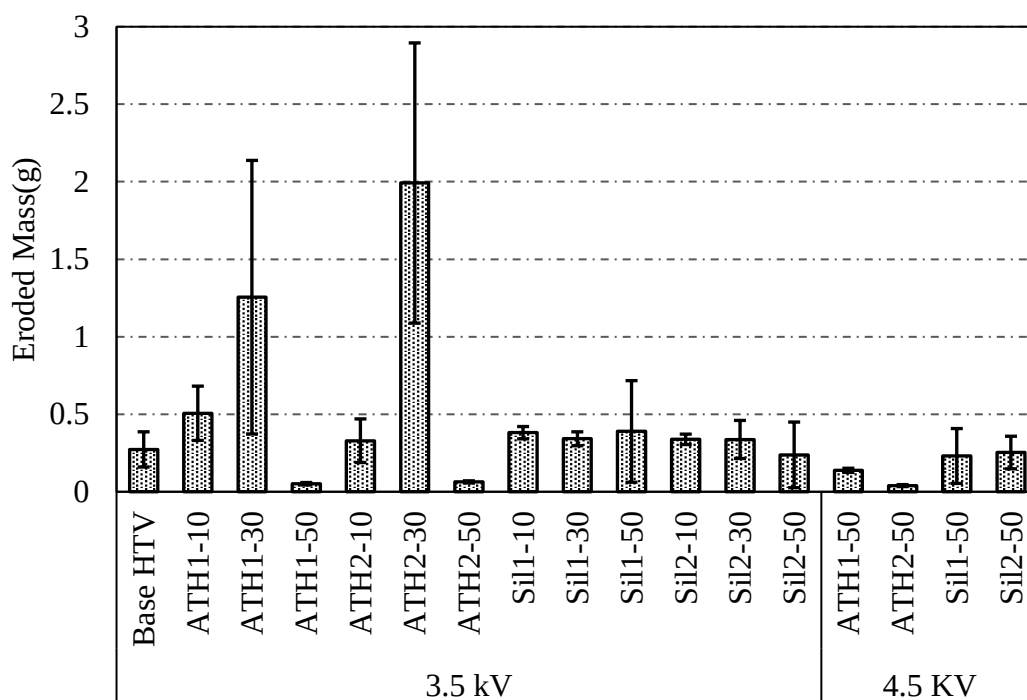


Figure 4.1 : Eroded mass of samples having different fillers in the IPT. The sample size was 5. Error bars indicate 95% confidence interval and hatched bars indicate the average value.

The thermal conductivity of fillers was found to be less important than filler-matrix bonding [55]. In the study made with RTV [55], the eroded mass decreased with decreasing mean particle size in the both ATH and silica samples having 50% wt filler. In the study done with RTV hot-pressed samples having 30% and 50% ATH and silica fillers, the silica samples showed better performance than in terms of the erosion resistance and thermal conductivity [55].

In low filled HTV silicone rubbers (<40% wt), the formation of thermal spots (>400 °C), and increase of leakage current values were attributed tracking-erosion failure [5]. Conversely, in high filled HTV silicone rubber (>40% wt), the tracking and erosion failure does not allow though composed of thermal spots and increase of leakage

current values [5]. HTV-SIRs produce low unit cyclic silicone oligomers and a lot of CO_2 seeing that the dry band arcing forms at the high temperature ($>400^\circ\text{C}$). Carbonized areas increase electrical conductivity, and leading to tracking and erosion of these areas [5]. Because ATH material reacts with methyl groups, it provides suppression of the tracking and erosion by undergoing a chemical modification [5].

It is reported that the leakage current values increase with increasing magnitude of the applied voltage [28]. In the present study, the leakage current values formed at 4.5kV test voltage were higher than those at 3.5 kV. This increase occurred for two filler type (ATH and silica) as well.

Both the leakage current and the temperature data were smoothed to clearly see the variations. For this purpose, the moving average technique was used with 1500 and 300 data points respectively for current and temperature values.

The TGA curve gives important information about thermal stability [38]. In the present study, silicone rubbers with silica fillers showed better behaviour in terms of thermal stability for all of the particle size and filler concentrations.

In the study [34], it was said that O-H groups in fillers help to reduce chemical changes by stretching the surface and prevent chemical changes in the SIR samples when samples are exposed to aging. In another study, in the suppression of tracking formation, the first effect is to replace with the filler of the burnt polymer and the second effect is that the formation of hydration water [37].

Water vapor emitted from ATH reacts with methyl ($-\text{CH}_3$) groups during dry band arcing, rather than with Si-O bonds in PDMS [5]. PDMS is converted to CH_4 and CO_2 gases and SiO_2 , meaning fewer silicone oligomer precursors and less residual carbon and thus HTV-SIR with ATH filler is an excellent suppressor of tracking and erosion [5].

It is emphasized that there is a good correlation between the third harmonic component of the LC and the eroded mass [36]. The both eroded mass and the third harmonic component of the LC decreased while filler ratio increased [36]. There is a strong correlation between the temperature caused by the dry band arc and the third harmonic power component [22], [31].

Almost all peaks in harmonic power increase temperature on the surface of the test specimens, but harmonic power peaks corresponding to all peaks in temperature may

not be present. Therefore, it was emphasized that not only harmonics but also exothermic reaction can increase the degradation [3].

Fundamental components of the leakage current increase by developing as sinusoidal immediately after starting the test. But, in the harmonic components of the LC increase with less a rate of change than according to fundamental components. [56]. However, in beginning, harmonic components develop around μA due to not occur dry band arcing [56]. However, in beginning, harmonic components developed around μA due to not occur dry band arcing [56]. The harmonic components increased immediately during the test after dry band arcing increased by the rise of the temperature [56]. For this reason, it can be emphasized that the harmonic components are a better indication of arcing than the fundamental component of the leakage current [56].

In Figure 4.2, the harmonic components of power generated by the dry band arcing during IP test are given. The raw data is black color and the smoothed data is white color. The third, fifth, and seventh harmonic power component values had much lower values than fundamental. The third harmonic power component had much lower absolute values than the fundamental, or the fifth harmonic power component had much lower absolute values than the third one. The study supports this decrease in harmonic component values [3]. Also, it is emphasized that the harmonic currents caused by the dry band arcing exhibit an unstable behavior [3].

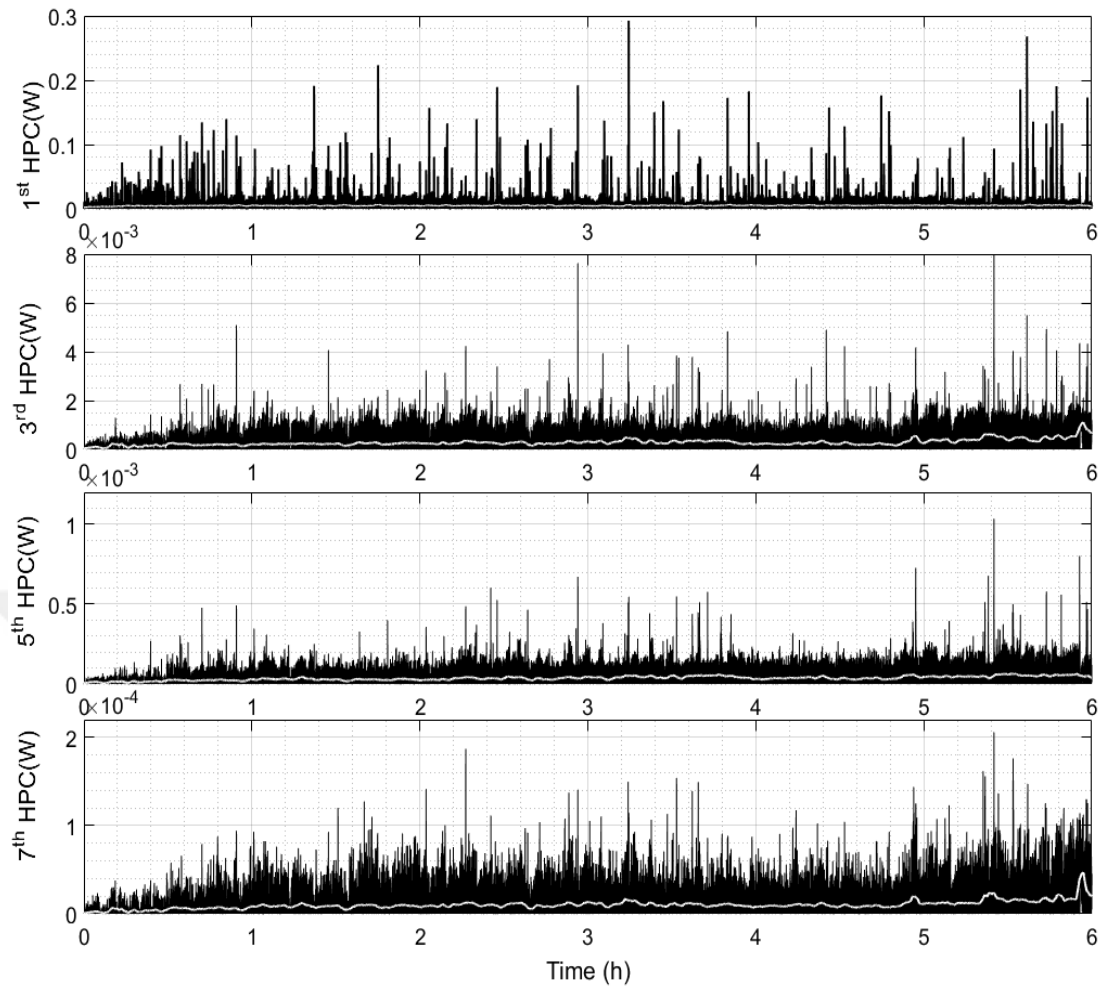


Figure 4.2 : The harmonic power components for ATH2-50 sample (Sample-2).
Raw data (black) and smoothed data (white).



5. CONCLUSION

The most important advantage of polymer insulators is that they have a hydrophobic surface and recover again after they occur temporary hydrophobic loss. However, since glass and ceramic insulators have a hydrophilic surface, this is not such a case. Other advantages of polymer insulators are that they have light, easy to install, resistant to impacts, superior in suppressing leakage current, the chemical-electrical resistances, conserving their structure at a wide temperature range, and low cost. Due to these advantages, silicone insulators' usage is increasing day by day and new polymer insulator types are emerging as their usage increases.

Silicone insulators have organic and inorganic properties due to their carbon content and their silicon content respectively. Silicone rubber materials consist of the silicone-oxygen main chain and the silicon bound methyl groups (CH_3). Polydimethylsiloxane (PDMS) occurs, when more than one of these chains come together.

In the study, the High Temperature Vulcanizing (HTV) silicone rubber samples were used. Under the study, the additional fillers were used for increasing the tracking-erosion resistance in addition to HTV silicone rubber used as the main material. Aluminum-tri-hydrate (ATH) and silica were used as additional fillers. Besides, the fillers with mean particle size of $3.5\ \mu\text{m}$ and $10\ \mu\text{m}$ were used to see effects of the particle size of additional fillers used in the micro size. The samples with 10%, 30% and 50% concentration by weight were prepared for each filler types and mean particle size. A total of thirteen different sample situations were prepared, which were made by adding in the fillers to the base silicone and the base silicone. In each test setup, a total of five identical samples were tested for each condition. Electrical, chemical, physical and thermal properties of filler types were examined.

Thermo-Gravimetric (TG), Derivative Thermo-Gravimetric (DTA), Fourier Transform Infra-Red Spectroscopy (FTIR) analyzes were performed for all samples prepared before the tests. After the test, FTIR analyzes were repeated by taking samples from the erosion areas. In addition, differential scanning calorimeter (DSC) analysis was performed for some samples to evaluate the heat exchange enthalpy.

As a result of TG analysis, information about the type of filler, the ratio of filler and thermal stability were reached. The main material used in the study was found to have 31.5% in weight filled fume-silica to result of TG analysis. For both filler types, as the concentration of fillers increased, the amounts of residual weight increased after the analyzes. The reason for this is that fillers are inorganic and these substances remain in the environment after the analysis. In addition, it was seen in DTG curves that the thermal stability increased with the increase of the filler amount. In these curves, there was a two-stage process due to the dehydration of water in ATH filled samples when only one-stage thermal degradation occurred due to the absence of water dehydration in the samples with silica filler. The dehydration of ATH starts around 240 °C, and the thermal decomposition starts around 400 °C.

ATH filled samples have O-H bonds in FTIR curves. But these bonds are not seen in silica filled samples. With FTIR analysis, chemical bonds corresponding to wavelengths were evaluated samples for before and after the test. As a result of post-test analysis due to aging, there was a decrease in O-H, C-H, Si-O and Si-C peaks. The reason for the decrease of O-H bonds is the dehydration of water, but this helps to form a protective layer on the sample surface. The reason for the decrease of other bonds is carbonization. In addition, it was found as a result of other article studies that the silicon element had a protective effect during the tests.

Enthalpy change of the material was examined by DSC analysis. Heat flow that started at 230°C and ended at 350°C was observed as an endothermic reaction resulting from the dehydration of water in silicone rubbers with ATH fillers. The enthalpy change was observed mostly in the sample with 50% wt ATH filler and the enthalpy change increased as the filler ratio increased. However, this enthalpy change has not been observed in both the base sample and the silica filled samples due to being no the dehydration of water. Also, the more enthalpy changes mean that the better suppressing erosion. It can be said that it acts as a protective layer by suppressing erosion because a protective layer on the sample is created by that.

The samples were evaluated after inclined plane tests performed under 3.5 kV and 4.5 kV constant voltage. The filler type was found to affect erosion behavior, and these different erosion behaviors were observed to be mostly in samples with concentrations just below the critical level. Both filler types exhibit the almost same erosion performance for the 10% wt filler ratio. The hole occurred in samples with 30% wt

ATH filler ratio. On the contrary, the erosion path occurred in samples with 30% wt silica filler ratio. In addition, a yellowish layer has formed on the sample surfaces caused by the dehydration of water in ATH filled samples. There are not any differences in the image was observed when compared in terms of average particle size.

In terms of erosion performance, samples with 50% wt ATH filler showed better performance than samples with 50% silica filler at both voltage levels. The samples with 30% wt ATH filler showed the worst performance due to the formation of holes. While two samples were punctured in ATH1 samples, three samples were punctured in ATH2 filled samples. Therefore, averages of the eroded masses were higher for the ATH2 samples. If it was excluded the punctured samples in terms of erosion performance, there was no significant difference in particle size under 3.5 kV voltage. However, under 4.5 kV test voltage, the ATH2 group showed better erosion performance than the ATH1 group, and the Sil1 group showed better performance than the Sil2 group. The erosion performance increased as the filler ratio increased, except for 30% wt ATH filler. This is the case in terms of erosion depths as well. The erosion depth performance was the better again 50% wt ATH filled samples for both voltage values.

Sil1, Sil2 and ATH1 samples with 10% wt filler ratio as the test duration shown almost similar property with the base HTV silicone rubber. But, ATH2 filled samples with 10% wt filler ratio shown better test duration performance than according to other. In addition, as the filler ratio increased for all filler particle sizes and types, the test durations of the samples increased. All samples with 50% wt ATH filler ratio passed the test at both 3.5 kV and 4.5 kV constant voltage. Under two test voltages, this ratio was found to be sufficient for samples with ATH filler. However, this ratio was not sufficient for samples with 50% wt silica filler. Because there were samples that failed the test as well as the samples that passed the test. Therefore, it was concluded that the filler ratio for samples with silica filler may be slightly higher from 50% wt. According to the 3.5 kV test voltage at 4.5 kV test voltage, for 50% wt filler ratio, the particle size did not have much effect considering test duration on ATH filled samples. The duration of failure was shortened for silica filled samples with 10.5 μm particle size and, test durations of Sil1 samples were almost the same. The two silica groups at 3.5 kV test voltage had similar test durations. The samples with silica filler with small

particle size have a higher test duration than the samples with large particle size one at 4.5 kV test voltage.

The leakage currents passing through the samples were recorded via the LabVIEW data acquisition program. For all cases at 3.5 kV test voltage, leakage current and maximum temperature values of ATH filled samples exceeded the higher over silica filled samples. As a result of the research done, it may be said by the fact that the thermal stability of silica filler is better than the ATH filler. It was emphasized that the reason for the good of the thermal stability of silica is that silica filler is caused by strong interface interaction with the base material. The same leakage current values were observed in both ATH and silica filled samples for 4.5 kV test voltage. There is no significant effect of particle size has been observed in terms of leakage current. In terms of the maximum temperature, samples with small particle size at both the ATH filled and silica filled samples showed slightly better temperature performance at a test voltage of 3.5 kV. For 4.5 kV test voltage, ATH2 filled samples showed better temperature performance than ATH1 filled ones. For silica filled samples, the situation did not change at 4.5 kV test voltage, and samples with small particle size showed better temperature performance again. Also, there was not a small difference between the temperature performances. Sil1 group samples were obtained almost twice the temperature performance according to the Sil2 group ones. This is clearly seen in erosion amounts. Because Sil1 group samples that passed the test showed good erosion performance at 4.5 kV test voltage.

The contaminant liquid evaporates with the temperature increase on the sample, and dry areas occur. Dry band arcing occurs in these dry regions, and the dry band arcs cause the formation of the harmonic component in the leakage current. The relationship between harmonics and temperature was evaluated. As a result of the evaluations, a strong correlation was found between the third harmonic power and the third harmonic leakage current components and the temperature. The increase or decrease of the temperature was related to the third harmonic component directly. It was obtained that the value of the third harmonic component increased with increasing temperature and decreased with decreased temperature.

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- **Ilhan S., Tuzun D., Özdemir A.** 2019. Electrical Performance of HTV Silicone Rubber under Different Fillers and Filler. International Congress – EIC: Electrical Insulation Conference, June 16-20, 2019, Calgary, Alberta, Canada.

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