



Faculty of Manufacturing Engineering

**PREPARATION AND CHARACTERIZATION OF SILICONE RUBBER
COMPOSITES FILLED WASTE MINERAL FILLERS FOR HIGH
VOLTAGE INSULATION APPLICATION**

Najwa binti Kamarudin

Master of Science in Manufacturing Engineering

2019

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COMPOSITES FILLED WASTE MINERAL FILLERS FOR HIGH VOLTAGE
INSULATION APPLICATION**

NAJWA BINTI KAMARUDIN

**A thesis submitted
in fulfilment of the requirements for the degree of Master of Science
in Manufacturing Engineering**

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2019

DECLARATION

I declare that this thesis entitled “Preparation and Characterization of Silicone Rubber Composites Filled Waste Mineral Fillers for High Voltage Insulation Application” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :



Name : NAJWA BINTI KAMARUDIN

Date :

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.



Signature

Supervisor Name : TS. DR. JEEFFERIE BIN ABD RAZAK

Date :

DEDICATION

To my beloved parents

Hj Kamarudin Bin Hj Said

Hjh Wan Rokma Binti Wan Ghani

ABSTRACT

Silicone rubber (SiR) based composites has increased demand in high voltage (HV) insulator application, due to their exceptional advantages over the existing conventional ceramic based insulator. SiR based insulator has compromised light-weight, superb breakage resistance, improved seismic performance and more flexible and manufacturable, than ceramic insulator. Hence, this research was conducted to prepare and characterize the performance of SiR based composites filled with mineral fillers, that derived from waste resources of silica (SiO_2) from waste glass, calcium carbonate (CaCO_3) from waste cockle shell and wollastonite (CaSiO_3) from combination of both. Simplified calcination heat treatment between SiO_2 and CaCO_3 (at percentage ratio of 51.70% : 48.30%), has successfully derived synthetic mineral CaSiO_3 , as confirmed by x-ray diffraction at 37.5° of 2θ peak. Later, SiR filled mineral filler was compounded by internal mixer with addition of dicumyl peroxide (DCP) as vulcanization agent, aux-heat stabilizer as colorant and mineral filler. Vulcanization via hot compression molding was performed before continuing into prolonged post-cured for complete conditioning. Two main independent variables has been tested in this research, which are the effects of mineral filler types and the effects of mineral filler loadings (at 5.00, 10.00, 20.00, 30.00 and 40.00wt.%), towards the resulted electrical, physical, mechanical and morphological performances of SiR filled composites. For electrical testing, an inclined plane test (IPT), surface resistivity and relative permittivity tests were conducted. Fracture surface observation via scanning electron microscope (SEM) was performed to relate the behavior of resulted mechanical strength of produced SiR based composites. It was interestingly found that, addition of mineral fillers caused an improvement in tensile strength about 70%, which exhibited by SiR/ CaSiO_3 at 5.00wt.% filler added. In terms of IPT test, SiR/ CaSiO_3 had maximally passed the tracking failure limit (4 out of 5 samples $< 2.50\text{cm}$) as compared than the other SiR composites. In addition, SiR/ CaSiO_3 at 40wt.% exhibits highest value of surface resistivity and relative permittivity. Not only that, it also possessed maximal hardness value which indicating complete peroxide curing, at before and after HV exposure. While stable water absorption was also obtained by SiR/ CaSiO_3 sample. In overall, SiR based composites provide absolute significant improvement as compared than unfilled especially for SiR/ CaSiO_3 composites, which has dominated the best properties almost for entire performed tests. These findings, was underlined the potential of CaSiO_3 over the other mineral fillers, to established improved filler-matrix interaction, due to their needle-like structure and protrusion condition and its extraordinary insulation effects, which benefited for electrical insulation and strength performances of SiR based composites for outstanding and reliable HV application.

ABSTRAK

Komposit berasaskan getah silikon (SiR) telah mendapat permintaan yang tinggi dalam aplikasi penebat voltan tinggi (HV), kerana kelebihannya yang luar biasa berbanding penebat berasaskan seramik konvensional yang sedia ada. Penebat berdasarkan SiR telah menjanjikan berat badan ringan, rintangan pecah yang hebat, prestasi seismik yang lebih baik dan lebih fleksibel dan boleh dihasilkan daripada penebat seramik. Oleh itu, kajian ini dijalankan untuk menyediakan dan mencirikan prestasi komposit SiR yang diisi dengan pengisi mineral, yang diperolehi daripada sumber silika (SiO_2) dari sisa kaca, kalsium karbonat (CaCO_3) dari sisa kulit kerang dan wollastonite (CaSiO_3) dari gabungan kedua-duanya. Rawatan haba kalsinasi yang sederhana antara SiO_3 dan CaCO_3 (pada nisbah peratusan 51.70%: 48.30%), telah berjaya memperoleh mineral sintetik CaSiO_3 , seperti yang disahkan oleh difraksi sinar-x pada 37.5° puncak 2θ . Kemudian, pengisi mineral yang diisi SiR dikompaun oleh pengadun dalaman dengan penambahan dicumyl peroksida (DCP) sebagai ejen pemvulkanan, penstabil aux-haba sebagai pewarna dan pengisi mineral. Pemvulkanan melalui pengacuan mampatan panas telah dilakukan sebelum diteruskan dengan pra-pemvulkanan yang berpanjangan untuk kitaran penyaman yang lengkap. Dua pembolehubah bebas utama telah diuji dalam kajian ini, iaitu kesan terhadap pengisi mineral dan kesan terhadap kuantitipengisian mineral (pada 5.00, 10.00, 20.00, 30.00 dan 40.00wt.%), ke arah prestasielektrik, mekanikal, fizikal dan morfologi SiR berasaskan komposit. Bagi ujian elektrik, ujian satah cenderung (IPT), ujian permukaan dan ketelusan relatif dijalankan. Pemerhatian permukaan patah melalui mikroskop elektron scanning (SEM) dilakukan untuk mengaitkan tingkah laku kekuatan mekanikal terhadap komposit SiR yang dihasilkan. Ia menarik perhatian bahawa, penambahan pengisi mineral menyebabkan peningkatan kekuatan tegangan sekitar 70%, yang dipamerkan oleh SiR/ CaSiO_3 pada 5.00wt.%. Dari segi ujian IPT, SiR/ CaSiO_3 telah mencapaikegagalan penjejakan maksimum (4 daripada 5 sampel $<2.50\text{sm}$) berbanding komposit SiR yang lain. Di samping itu, SiR/ CaSiO_3 pada 40wt.% mempamerkan nilai tertinggi permukaan rintangan dan ketelusan relatif. Selain itu, ia juga mempunyai nilai kekerasan maksima yang menunjukkan pengawetan peroksida yang lengkap, sebelum dan selepas pendedahan HV. Tambahan pula, penyerapan air yang stabil juga diperolehi oleh sampel SiR/ CaSiO_3 . Secara keseluruhannya, komposit SiR memberikan pembaikan mutlak yang ketara berbanding yang tidak terisi terutamanya untuk komposit SiR/ CaSiO_3 , yang telah menguasai sifat-sifat terbaik hampir untuk seluruh ujian yang dilakukan. Penemuan ini menggariskan potensi CaSiO_3 berbanding pengisi mineral lain, untuk membentuk interaksi matriks pengisi yang lebih baik, disebabkan oleh struktur dan keadaan tujahan seperti jarum dan kesan penebat luar biasa yang memberi manfaat kepada penebat elektrik dan prestasi kekuatan SiR berdasarkan komposit untuk aplikasi HV yang cemerlang dan boleh dipercayai.

ACKNOWLEDGEMENTS

First and foremost, I would first like to thank my research supervisor Ts. Dr. Jeefferie bin Abd Razak of the Faculty of Manufacturing Engineering at Universiti Teknikal Malaysia Melaka. The door to Dr. Jeefferie office was always open whenever I ran into a trouble spot or had a question about my research or writing. He consistently allowed this research to be my own work, but steered me in the right direction whenever he thought I needed it.

I would also like to thank my co-supervisor Puan Nurbahirah Binti Norddin who was involved in this research as an expert in electrical testing. Without her passionate participation and input, the electrical testing could not have been successfully conducted.

Finally, I must express my very profound gratitude to my parents and to my friends for providing me with unfailing support and continuous encouragement throughout my years of study and through the process of researching and writing this thesis. The accomplishment would not have been possible without them.

Thank you.

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LIST OF ABBREVIATIONS

AC	-	Alternate current
Al ₂ O ₃	-	Alumina
ASTM	-	American standard test method
ATH	-	Aluminum tetahedra
B	-	Boron
BaTiO ₃	-	Barium Titanate
C	-	Carbon
Ca	-	Calcium
CaCO ₃	-	Calcium carbonate
CaSiO ₃	-	Wollastonite
Cu	-	Copper
Cm	-	Centimetre
CNT	-	Carbon nanotechnology
DBA	-	Dry band arcing
DC	-	Direct current
DCP	-	Dicumyl Peroxide
E _{AB}	-	Elongation at break
EMI	-	Electromagnetic interference
Fe	-	Iron
GHz	-	Gigahertz
GNR	-	Graphene nanoribbon

HRS	-	Hours
HTVSR	-	High temperature vulcanized silicone rubber
HV	-	High voltage
IRHD	-	International Rubber Hardness Degree
IPT	-	Inclined Plane Tracking
K	-	Potassium
kN	-	KiloNewton
kV	-	Kilovolt
Mg	-	Magnesium
mins	-	Minutes
ml	-	Mililitre
mm	-	Milimetre
MPa	-	Mega Pascal
Na	-	Sodium
NH ₄ Cl	-	Ammonium chloride
Ni	-	Nickel
O	-	Oxygen
P	-	Phosphorus
PDMS	-	Polydimethylsiloxane
PSA	-	Particle size analysis
RPM	-	Rotation per minute
RT	-	Room temperature
RTV	-	Room temperature vulcanized
S	-	Second
SE	-	Shielding effectiveness
SEM	-	Scanning electron microscope

Si	-	Silicone
SiO ₂	-	Silica
SiR	-	Silicone rubber
TiO ₂	-	Titanium Oxide
T _s	-	Tensile strength
UTM	-	Universal tensile machine
UV	-	Ultraviolet
XRD	-	X-Ray diffraction
XRF	-	X-Ray fluorescence
Zn	-	Zinc

LIST OF SYMBOLS

Ω	-	Ohms
wt	-	Weight
%	-	Percentage
Σ	-	Sum up
$^{\circ}\text{C}$	-	Degree celcius
Ω/sq	-	Ohm per square
\emptyset	-	Theta
μm	-	Micrometer

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CHAPTER 1

INTRODUCTION

1.1 Background of study

An electrical insulation has been recognized as an important aspect to be considered for high voltage outdoor application (Karthik et al., 2013). Previously, there are several types of high voltage insulators on transmission lines for outdoor applications, have been manufactured. The history of high voltage insulator has began in 20th century, with porcelain as the only material being utilized for insulator applications. In 1940s, the development of insulator was then continued with utilization of polymer insulator that replacing conventional materials like porcelain, glass and ceramic. This transformation had started with manufacturing of high voltage insulator using epoxy resins as based raw materials (Hall, 1993).

The need for this major replacement was to eliminate the usage of expensive porcelain material, reduce the cost of manufacturing and most important to increase the tracking and erosion resistance of insulator which has very important for high voltage insulator application (Khan et al., 2017). Polymer material has chosen to replace previous type of insulator, due to their hydrophilic properties, which aids the process of dry band arcing that led into flashover phenomena (Prasenjit et al., 2015). Furthermore, polymeric insulators addressed most of the problems faced by the porcelain insulators. Their advantages includes light weight and flexible characteristics, which allows easier erection and commissioning of insulators, even at distant areas including places with resistance to vandalisms (Xuguang et al., 2000). These advantages over ceramic insulators have made them very attractive enough to be used for high voltage insulator industry.

Nowadays, in most countries, polymeric based insulator has growing demand due to technological growth and attractive potential benefits to their end consumers (Ramirez and Hernandez, 2016). Since polymeric insulations are well accepted in high voltage application, large number of important studies and research activities for improvement on their performances had been performed by various researchers at the global worldwide. One of key indicators for polymeric insulation surface performance was determined by its tracking and erosion effects since it has been well-known reason for common insulation failure (Vasudev, 2012). Tracking could be defined as formation of surface carbonaceous path, while erosion was a weight loss experienced by the tested insulator material (Ghunem, 2015). Tracking and erosion are considered as important aspects to be looked into, since it leads to a better strength of insulator (Kannan et al., 2015). Furthermore, polymeric insulators exhibit a hydrophobicity property which has suitable to be used in highly polluted areas. These kinds of properties make polymeric insulators good for their usage. However, polymeric insulators also possessed certain drawbacks. While they are having commercial success, an obstacle occurred due to fluctuate hydrocarbon cost, limited manufacturing versatility and the utmost important part was an inadequate performance for outdoor field high voltage application (Sundhar et al., 1992). Since the usage of polymeric insulators has not been developed until the late 1960s and 1970s, their expected life was still unknown (Hall, 1993). Furthermore, polymeric insulators are vulnerable to tracking and erosion under electrical stress, and also to degradation under corona and weathering (Vas et al., 2012). If the polymeric insulator agonize from tracking and erosion for a long period of time, it may eventually lead into failure of the insulators (Yaacob et al., 2013). Thus, in order to overcome these problems, an initiative should be created. Since silicone rubber (SiR) possessed lower stiffness, an alternative has been made by adding some functional fillers into it, for polymeric based composites production,

for the sake to enhance their resulted end properties of electrical insulation and mechanical strength (Amin and Salman, 2006). As reported by many previous researchers, by adding functional fillers into the polymer matrix, it could improvised certain properties and also could lowering the cost of manufacturing process (Venkatesulu and Thomas, 2010; Aman et al., 2013; Ghunem et al., 2015; Ali et al., 2017).

Over the past few years, polymeric based composite insulators have growing into higher demand and has already known worldwide (Momen, G. and Farzaneh, 2011). According to Rowland et al. (2010), a failure would occurred earlier for polymeric based composite insulators due to poor design and improper manufacturing processes. However, an improvement has been established for over 20 years, by enhancing the design and manufacturing techniques to overcome the problem in early application. Surprisingly, their usage has increased rapidly and known worldwide for over three decades. Among the benefits, polymeric composite designs for high voltage insulators has compromised lighter weight, less breakage, improved seismic performance and more flexibility at real application than ceramic insulators. These features provide advantages of lower installation cost, better durability and more aesthetically pleasing design. To counter these advantages, a comprehensive understanding of synthetic and mineral fillers roles and involvement are yet to be realized. It should be noted that an involvement of filler in polymeric composite for HV application was important since it could enhance the mechanical strength and other electrical insulation attributes of resulted polymer composites (Bian et al., 2013).

Basically, reinforcements material or fillers are regularly used to enhance the polymeric composite properties and also to reduce the end cost of final products (Ansoorge et al., 2012; Aman et al., 2013). The most common fillers that often been used for electrical insulation application are alumina, silica, calcium carbonate and wollastonite mineral fillers. In this research work, the later three mineral filler types were chosen based