

**CHARACTERIZATION OF VARIOUS TYPES  
OF NANOFILLERS FILLED CROSSLINKED  
POLYETHYLENE COMPOSITES FOR  
CABLE APPLICATION**

**LIM KAI SHENG**

**UNIVERSITI SAINS MALAYSIA**

**2018**

**CHARACTERIZATION OF VARIOUS TYPES OF NANOFILLERS  
FILLED CROSSLINKED POLYETHYLENE COMPOSITES FOR  
CABLE APPLICATION**

**by**

**LIM KAI SHENG**

**Thesis submitted in fulfilment of the  
requirements for the degree of  
Master of Science**

**April 2018**

## **ACKNOWLEDGEMENTS**

First, I would like to thank University Sains Malaysia (USM) for providing a good facilities, equipment and environment to conduct my research. I would like to thank to all professors, administrative and technical staffs of School of Materials and Mineral Resources Engineering, University Sains Malaysia.

Second, I would like to take this opportunity to express my deepest gratitude to my project supervisors Assoc. Prof. Dr. Azhar Bin Abu Bakar, Prof. Ir. Dr. Mariatti Binti Jaafar and Assoc. Prof. Ir. Dr. Mohamad Kamarol Mohd Jamil for the support, guidance and inspiration throughout two years of research study. The supervision, valuable comments, constructive advices and inputs given truly help the progression and smoothness of the project. Besides, I would like to thank Tenaga Nasional Berhad (TNB) for providing the grant to conduct the research.

Last but not least, I would like to thank all my colleagues, friends and family for the wise idea, invaluable supports and suggestions throughout the project.

## **TABLES OF CONTENTS**

	<b>Page</b>
<b>ACKNOWLEDGEMENTS</b>	ii
<b>TABLE OF CONTENTS</b>	iii
<b>LIST OF TABLES</b>	viii
<b>LIST OF FIGURES</b>	x
<b>LIST OF ABBREVIATIONS</b>	xvi
<b>LIST OF SYMBOLS</b>	xvii
<b>ABSTRAK</b>	xviii
<b>ABSTRACT</b>	xx
<b>CHAPTER ONE : INTRODUCTION</b>	
1.1 Background of Research	1
1.2 Problem Statement	3
1.3 Objective	5
1.4 Scope	5
1.5 Organization of The Thesis	6
<b>CHAPTER TWO : LITERATURE REVIEW</b>	
2.1 Introduction	7
2.2 Materials Used for Cable insulation	7
2.3 Polyethylene	8
2.4 Polymer Nanocomposites	11
2.5 Factor Affecting Properties of Polymeric Composites	13
2.5.1 Size, Shape and Orientation of Nanoparticles	14

2.5.2	Properties of The Matrix	16
2.5.3	Interfacial Adhesion	17
2.6	Nanofillers	19
2.6.1	Metal Oxides Nanoparticles	19
2.6.1(a)	Zinc Oxide	19
2.6.1(b)	Aluminium Oxide	21
2.6.2	Nanoclay	23
2.6.3	Surface Treatment of Nanofillers	25
2.7	Hybrid Filler Effect	29
2.8	Characterization and Properties of Nanocomposites	30
2.8.1	Chemical Structure	30
2.8.2	Microstructure and Morphology	31
2.8.3	Stress Strain Behavior	32
2.8.4	Dielectric Breakdown Strength	36
2.8.5	Volume Resistivity	37
2.8.6	Flammability	39
2.8.7	Water Resistance	40
2.8.8	Thermal Properties	43
 <b>CHAPTER THREE : MATERIALS AND METHODOLOGY</b>		
3.1	Introduction	47
3.2	Materials	47
3.2.1	Matrix	47
3.2.2	Nanofillers	48
3.2.3	Chemical	50
3.3	Fabrication of Crosslinked Polyethylene Nanocomposites	50

3.4	Characterization	52
3.4.1	Functional Groups Analysis	53
3.4.2	Particle Size Analysis	53
3.4.3	Fracture Surface Morphology	53
3.4.4	Mechanical Testing	53
3.4.4(a)	Tensile Test	53
3.4.5	Physical Testing	54
3.4.5(a)	Void Content Determination	54
3.4.5(b)	Degree of Crosslinking Determination	55
3.4.5(c)	Contact Angle Measurement	56
3.4.5(d)	Water Absorption Measurement	56
3.4.5(e)	Flammability Test	57
3.4.6	Dielectric Testing	58
3.4.6(a)	Dielectric Breakdown Test	58
3.4.6(b)	Volume Resistivity Test	59
3.4.7	Thermal Analysis	60
3.4.7(a)	Thermogravimetric Analysis	60
3.4.7(b)	Differential Scanning Calorimetry Analysis	60

## **CHAPTER FOUR : RESULTS AND DISCUSSION**

4.1	Introduction	62
4.2	Characterization of Nanofillers	62
4.2.1	Functional Groups	63
4.2.2	Morphology of The Nanofillers	65

4.3	Studies on ZnO, Al <sub>2</sub> O <sub>3</sub> and OMMT Nanofillers in XLPE nanocomposites	66
4.3.1	Tensile Properties and Void Content	66
4.3.2	Fracture Surface Morphology	72
4.3.3	Degree of Crosslinking	74
4.3.4	Contact Angle and Surface Free Energy	76
4.3.5	Water Absorption	78
4.3.6	Rate of Burning	83
4.3.7	Dielectric Breakdown Strength	85
4.3.8	Volume Resistivity	90
4.3.9	Thermogravimetric Analysis	91
4.3.10	Differential Scanning Calorimetry Analysis	94
4.4	Studies on Treated ZnO and Al <sub>2</sub> O <sub>3</sub> Nanofillers in XLPE nanocomposites	98
4.4.1	Tensile Properties and Void Content	99
4.4.2	Fracture Surface Morphology	104
4.4.3	Degree of Crosslinking	106
4.4.4	Contact Angle and Surface Free Energy	107
4.4.5	Water Absorption	110
4.4.6	Rate of Burning	112
4.4.7	Dielectric Breakdown Strength	113
4.4.8	Volume Resistivity	117
4.4.9	Thermogravimetric Analysis	119
4.4.10	Differential Scanning Calorimetry Analysis	120
4.5	Studies on Hybrid ZnO/Al <sub>2</sub> O <sub>3</sub> and ZnO/OMMT Nanofillers in XLPE nanocomposites	124
4.5.1	Tensile Properties and Void Content	124

4.5.2	Fracture Surface Morphology	128
4.5.3	Degree of Crosslinking	130
4.5.4	Contact Angle and Surface Free Energy	130
4.5.5	Rate of Burning	132
4.5.6	Dielectric Breakdown Strength	133
4.5.7	Volume Resistivity	136
4.5.8	Thermogravimetric Analysis	136
4.5.9	Differential Scanning Calorimetry Analysis	138

## **CHAPTER FIVE : CONCLUSION AND FUTURE RESEARCH**

5.1	Conclusion	142
5.2	Future Research	144

<b>REFERENCES</b>		145
-------------------	--	-----

## **APPENDICES**

Appendix A	: Nanocomposites: ZnO/XLPE Nanocomposites
Appendix B	: Nanocomposites: Al <sub>2</sub> O <sub>3</sub> /XLPE Nanocomposites
Appendix C	: Nanocomposites: OMMT/XLPE Nanocomposites
Appendix D	: Treated Nanocomposites: KH550-ZnO/XLPE Nanocomposites
Appendix E	: Treated Nanocomposites: TCS-ZnO/XLPE Nanocomposites
Appendix F	: Treated Nanocomposites: AE-Al <sub>2</sub> O <sub>3</sub> /XLPE Nanocomposites
Appendix G	: Hybrid Nanocomposites: ZnO/Al <sub>2</sub> O <sub>3</sub> /XLPE Nanocomposites
Appendix H	: Hybrid Nanocomposites: ZnO/OMMT/XLPE Nanocomposites



## LIST OF TABLES

		<b>Page</b>
Table 2.1	Changes in polyethylene after crosslinking	11
Table 2.2	Properties of alumina (purity 99.7%) used in ceramic industry	23
Table 2.3	Classification of nanoclay	24
Table 2.4	Silane coupling agent treatment for ZnO and Al <sub>2</sub> O <sub>3</sub> nanoparticles	26
Table 2.5	Glass transition and crystalline melting temperatures of polymers	45
Table 3.1	Information of raw materials used	49
Table 3.2	Formulation of different filler composites	51
Table 3.3	Formulation of treated composites	51
Table 3.4	Formulation of hybrid composites	52
Table 4.1	Vibration bands of different nanofillers	63
Table 4.2	Diffusion parameters of XLPE nanocomposites	83
Table 4.3	The Weibull parameters of XLPE nanocomposites	89
Table 4.4	Thermal parameters obtained from thermogravimetric analysis of XLPE nanocomposites	93
Table 4.5	Differential scanning calorimetry thermal properties of XLPE nanocomposites	96
Table 4.6	Summary properties of XLPE nanocomposites filled with 1.5 wt% filler loading	98
Table 4.7	Diffusion parameters of treated XLPE nanocomposites	112
Table 4.8	The Weibull parameters of treated XLPE nanocomposites	117
Table 4.9	Thermal parameters obtained from TGA analysis of treated XLPE nanocomposites	120
Table 4.10	Differential scanning calorimetry thermal properties of treated XLPE nanocomposites	122

Table 4.11	Summary properties of treated XLPE nanocomposites filled with 1.5 wt% filler loading	123
Table 4.12	The Weibull parameters of hybrid XLPE nanocomposites	135
Table 4.13	Thermal parameters obtained from TGA analysis of hybrid XLPE nanocomposites	138
Table 4.14	Differential scanning calorimetry thermal properties of hybrid XLPE nanocomposites	140
Table 4.15	Summary properties of hybrid XLPE nanocomposites filled with 1 wt% filler loading	141

## LIST OF FIGURES

		<b>Page</b>
Figure 1.1	Number of failure report for underground cable system from year 2010 to 2015 in Malaysia	2
Figure 2.1	Schematic structure of different polyethylene grades including high density polyethylene (HDPE), low density polyethylene (LDPE), linear low density polyethylene (LLDPE) and crosslinked polyethylene (XLPE)	9
Figure 2.2	Crystalline and amorphous polymer structure in polyethylene	9
Figure 2.3	The interaction zone induced by micro-sized and nano-sized particles	14
Figure 2.4	The effect of particle size on interfacial volume per total polymer volume with respect to same loading	15
Figure 2.5	Classification of nanofillers based on dimension	19
Figure 2.6	The crystal structures of ZnO where the gray spheres denote Zn atoms and black spheres denote O atoms: (a) cubic rocksalt, (b) cubic zinc blende and (c) hexagonal wurtzite	20
Figure 2.7	The crystal structure of $\alpha$ -Al <sub>2</sub> O <sub>3</sub> where the large gray spheres denote O atoms and the small black spheres denote Al atoms	21
Figure 2.8	The crystal structure of $\gamma$ -Al <sub>2</sub> O <sub>3</sub> where it shows the face centre cubic structure	22
Figure 2.9	The transformation phases of alumina from bauxite to $\alpha$ -Al <sub>2</sub> O <sub>3</sub>	22
Figure 2.10	Crystal structure of montmorillonite composed by two silica tetrahedral sheets and an alumina octahedral sheet	24
Figure 2.11	Before and after treatment of filler with silane coupling agent	27
Figure 2.12	Interaction between filler and resin via silane coupling agent	27
Figure 2.13	Schematic of nanoclay composites: (a) conventional composite, (b) intercalated composite and (c) exfoliated composite	27

Figure 2.14	Organophilization of montmorillonite with hydrocarbon chains	28
Figure 2.15	Schematic of properties of different hybrid combination	29
Figure 2.16	Deformation steps upon tensile stress	32
Figure 2.17	Stress-strain curves of brittle and ductile polymer	33
Figure 2.18	Deformation mechanisms in polymer material: (a) shear yielding and (b) crazing	33
Figure 2.19	Schematic measurement of volume and surface resistivity	38
Figure 2.20	Graphical illustration of contact angle formed on a solid surface	42
Figure 2.21	Idealized view of the effect on the deformability of polymer with increasing the temperature	45
Figure 3.1	Graphical illustration of horizontal burning test	58
Figure 3.2	High voltage equipment (model TERCO, Sweden) used for dielectric breakdown strength test	58
Figure 3.3	Position of specimen and electrodes for dielectric breakdown test	59
Figure 3.4	Resistance meter (model PRS-812 Resistance Meter, Prostat) used for surface resistivity test	60
Figure 4.1	FTIR spectra of different nanofillers (a) ZnO, (b) Al <sub>2</sub> O <sub>3</sub> and (c) OMMT	63
Figure 4.2	FTIR spectra of XLPE nanocomposites (a) unfilled XLPE, (b) ZnO(2)XLPE nanocomposites, (c) Al <sub>2</sub> O <sub>3</sub> (2)XLPE nanocomposites and (d) OMMT(2)XLPE nanocomposites	64
Figure 4.3	SEM image of the morphology of different nanofillers of (a) ZnO; (b) Al <sub>2</sub> O <sub>3</sub> and (c) OMMT	65
Figure 4.4	Tensile strength of XLPE nanocomposites as a function of filler loading	67
Figure 4.5	Void content of XLPE nanocomposites as a function of filler loading	68
Figure 4.6	Elongation at break of XLPE nanocomposites as a function of filler loading	70

Figure 4.7	Young's modulus of XLPE nanocomposites as a function of filler loading	71
Figure 4.8	Fracture surface morphology of XLPE nanocomposites of (a) XLPE; (b) ZnO(0.5)/XLPE; (c) ZnO(2)/XLPE; (d) Al <sub>2</sub> O <sub>3</sub> (0.5)/XLPE and (e) Al <sub>2</sub> O <sub>3</sub> (2)/XLPE; (f) OMMT(0.5)/XLPE and (g) OMMT(2)/XLPE observed at 1000X magnification	73
Figure 4.9	Degree of crosslinking of XLPE nanocomposites as a function of filler loading	75
Figure 4.10	Contact angle of XLPE nanocomposites as a function of filler loading	76
Figure 4.11	Surface free energy of XLPE nanocomposites as a function of filler loading	76
Figure 4.12	Water absorption of ZnO/XLPE nanocomposites as a function of square root of time in hour	79
Figure 4.13	Water absorption of Al <sub>2</sub> O <sub>3</sub> /XLPE nanocomposites as a function of square root of time in hour	79
Figure 4.14	Water absorption of OMMT/XLPE nanocomposites as a function of square root of time in hour	80
Figure 4.15	Diffusion coefficient of XLPE nanocomposites as a function of filler loading	81
Figure 4.16	Burning rate of XLPE nanocomposites as a function of filler loading	84
Figure 4.17	Dielectric breakdown strength of XLPE nanocomposites as a function of filler loading	86
Figure 4.18	Weibull statistical distribution curves of dielectric breakdown strength of ZnO/XLPE nanocomposites	86
Figure 4.19	Weibull statistical distribution curves of dielectric breakdown strength of Al <sub>2</sub> O <sub>3</sub> /XLPE nanocomposites	87
Figure 4.20	Weibull statistical distribution curves of dielectric breakdown strength of OMMT/XLPE nanocomposites	87
Figure 4.21	Volume resistivity of XLPE nanocomposites as a function of filler loading	90
Figure 4.22	Thermogravimetric analysis curves of XLPE nanocomposites	92

Figure 4.23	Derivative thermogravimetric analysis curves of XLPE nanocomposites	92
Figure 4.24	Differential scanning calorimetry second heating curves of XLPE nanocomposites	95
Figure 4.25	Differential scanning calorimetry first cooling curves of XLPE nanocomposites	95
Figure 4.26	Tensile strength of treated XLPE nanocomposites as a function of filler loading	99
Figure 4.27	Void content of treated XLPE nanocomposites as a function of filler loading	101
Figure 4.28	Elongation at break of treated XLPE nanocomposites as a function of filler loading	102
Figure 4.29	Young's modulus of treated XLPE nanocomposites as a function of filler loading	103
Figure 4.30	Fracture surface morphology and microstructure of treated XLPE nanocomposites of (a) ZnO(0.5)/XLPE; (b) KH550-ZnO(0.5)/XLPE; (c) TCS-ZnO(0.5)/XLPE; (d) Al <sub>2</sub> O <sub>3</sub> (0.5)/XLPE and (e) AE-Al <sub>2</sub> O <sub>3</sub> (0.5)/XLPE observed at 2000X magnification	105
Figure 4.31	Degree of crosslinking of treated XLPE nanocomposites as a function of filler loading	107
Figure 4.32	Contact angle of treated XLPE nanocomposites as a function of filler loading	108
Figure 4.33	Surface free energy of treated XLPE nanocomposites as a function of filler loading	108
Figure 4.34	Water absorption of treated XLPE nanocomposites as a function of square root of time in hour	110
Figure 4.35	Diffusion coefficient of treated XLPE nanocomposites filled with 1 wt% filler loading	111
Figure 4.36	Rate of burning of treated XLPE nanocomposites as a function of filler loading	113
Figure 4.37	Dielectric breakdown strength of treated XLPE nanocomposites as a function of filler loading	114

Figure 4.38	Weibull statistical distribution curves of dielectric breakdown strength of KH550-ZnO/XLPE nanocomposites	114
Figure 4.39	Weibull statistical distribution curves of dielectric breakdown strength of TCS-ZnO/XLPE nanocomposites	115
Figure 4.40	Weibull statistical distribution curves of dielectric breakdown strength of AE-Al <sub>2</sub> O <sub>3</sub> /XLPE nanocomposites	115
Figure 4.41	Volume resistivity of treated XLPE nanocomposites as a function of filler loading	118
Figure 4.42	Thermogravimetric analysis curves of treated XLPE nanocomposites	119
Figure 4.43	Derivative thermogravimetric analysis curves of treated XLPE nanocomposites	119
Figure 4.44	Differential scanning calorimetry second heating of treated XLPE nanocomposites	121
Figure 4.45	Differential scanning calorimetry first cooling curve of treated XLPE nanocomposites	121
Figure 4.46	Tensile strength of hybrid XLPE nanocomposites as a function of filler loading	125
Figure 4.47	Void content of hybrid XLPE nanocomposites as a function of filler loading	126
Figure 4.48	Elongation at break of hybrid XLPE nanocomposites as a function of filler loading	127
Figure 4.49	Young's modulus of hybrid XLPE nanocomposites as a function of filler loading	128
Figure 4.50	Fracture surface morphology of hybrid XLPE nanocomposites of (a) XLPE; (b) ZnO/Al <sub>2</sub> O <sub>3</sub> (75/25)/XLPE; (c) ZnO/Al <sub>2</sub> O <sub>3</sub> (50/50)/XLPE; (d) ZnO/Al <sub>2</sub> O <sub>3</sub> (25/75)/XLPE (e) ZnO/OMMT(75/25)/XLPE; (f) ZnO/OMMT(50/50)/XLPE and (g) ZnO/OMMT(25/75)/XLPE observed at 1000X magnification	129
Figure 4.51	Degree of crosslinking of hybrid XLPE nanocomposites as a function of filler loading	130

Figure 4.52	Contact angle of hybrid XLPE nanocomposites as a function of filler loading	131
Figure 4.53	Surface free energy of hybrid XLPE nanocomposites as a function of filler loading	131
Figure 4.54	Rate of burning of hybrid XLPE nanocomposites as a function of filler loading	132
Figure 4.55	Dielectric breakdown strength of hybrid XLPE nanocomposites as a function of filler loading	134
Figure 4.56	Weibull statistical distribution curves of dielectric breakdown strength of ZnO/Al <sub>2</sub> O <sub>3</sub> /XLPE nanocomposites	134
Figure 4.57	Weibull statistical distribution curves of dielectric breakdown strength of ZnO/OMMT/XLPE nanocomposites	135
Figure 4.58	Volume resistivity of hybrid XLPE nanocomposites as a function of filler loading	136
Figure 4.59	Thermogravimetric analysis curves of hybrid XLPE nanocomposites	137
Figure 4.60	Derivative thermogravimetric analysis curves of hybrid XLPE nanocomposites	137
Figure 4.61	Differential scanning calorimetry second heating of hybrid XLPE nanocomposites	139
Figure 4.62	Differential scanning calorimetry first cooling curve of hybrid XLPE nanocomposites	139



## LIST OF ABBREVIATIONS

XLPE	Crosslinked polyethylene
LDPE	Low density polyethylene
HDPE	High density polyethylene
LLDPE	Linear low density polyethylene
ZnO	Zinc oxide
Al <sub>2</sub> O <sub>3</sub>	Aluminium oxide
OMMT	Organo-montmorillonite clay
KH550-ZnO	3-aminopropyltriethoxysilane treated zinc oxide
TCS-ZnO	Triethoxycaprylylsilane treated zinc oxide
AE-Al <sub>2</sub> O <sub>3</sub>	Aluminic ester treated aluminium oxide
FE-SEM	Field emission-scanning electron microscope
TGA	Thermogravimetric analysis
DSC	Differential scanning calorimetry
FTIR	Fourier transfer infrared spectroscopy
ASTM	American Society for Testing and Materials
DBS	Dielectric breakdown strength

## LIST OF SYMBOLS

<b>Symbol</b>	<b>Description</b>
wt%	Weight percent
MPa	Mega pascal
$\sqrt{t}$	Square root of time in hour
$M_m$	Maximum moisture
K	Initial slope
T	Thickness
mm <sup>2</sup> /h	Millimetres square per hour
mm/min	Millimetres per minute
kV/mm	Kilovolts per millimetres
$\alpha$	Scale for dielectric breakdown distribution
B	Shape for dielectric breakdown distribution
$\Omega$ cm	Ohm centimetres
$T_m$	Melting temperature
$T_c$	Crystallization temperature

**PENCIRIAN PELBAGAI JENIS PENGISI NANO TERISI  
KOMPOSIT POLIETILENA TERSAMBUNG SILANG UNTUK  
APLIKASI KABEL**

**ABSTRAK**

Kajian ini adalah untuk mengkaji kesan pengisi nano terhadap sifat mekanikal, fizikal, dielektrik dan terma matriks polietilena tersambung silang (XLPE). Pengisi nano yang tidak dirawat adalah zink oksida (ZnO) dan aluminium oksida ( $\text{Al}_2\text{O}_3$ ). Pengisi nano yang telah dirawat adalah tanah liat (OMMT), ZnO dirawat dengan *3-aminopropyltriethoxysilane* (KH550-ZnO), ZnO dirawat dengan *triethoxycaprylylsilane* (TCS-ZnO) dan  $\text{Al}_2\text{O}_3$  dirawat dengan *aluminic ester* (AE- $\text{Al}_2\text{O}_3$ ). Komposit nano XLPE telah disediakan dengan pencampuran leburan dengan ekstruder skru tunggal diikuti dengan penekan panas. Peratusan berat yang berlainan (0.5, 1, 1.5 dan 2 wt%) telah dikompaun bagi komposit nano yang tidak dirawat dan telah dirawat. Gabungan ZnO/ $\text{Al}_2\text{O}_3$  dan ZnO/OMMT dengan nisbah yang berbeza (75/25, 50/50 dan 25/75) dalam jumlah 1 wt% berat pengisi telah dikompaun bagi komposit nano hibrid. Komposit nano telah diuji dengan mengikut kaedah piawaian ASTM dan dicirikan dengan pemeriksaan mikroskop elektron (SEM), analisis termogravimetrik (TGA) dan kalorimetri pengimbasan berbeza (DSC). Keputusan menunjukkan penambahan pengisi nano yang tidak dirawat dan dirawat meningkatkan sifat tegangan, kadar pembakaran, sifat dielektrik dan suhu penguraian. Walau bagaimanapun, ia menunjukkan kesan terhad terhadap sifat rintangan air, suhu leburan dan suhu penghabluran. Berat pengisi optimum adalah 1.5 wt% dan kesan  $\text{Al}_2\text{O}_3$  lebih baik daripada pengisi nano ZnO dan OMMT berdasarkan kebanyakan sifat. Rawatan permukaan dengan ejen gandingan meningkatkan

kawasan antara muka pengisi dan matriks dengan ikatan kimia. Interaksi pengisi-matriks yang kuat meningkatkan lagi sifat komposit. Berat pengisi optimum adalah 1.5 wt% dan kesan AE-Al<sub>2</sub>O<sub>3</sub> adalah lebih baik daripada pengisi nano KH550-ZnO dan TCS-ZnO berdasarkan kebanyakan sifat. Dalam komposit nano hibrid, nisbah pengisi yang terbaik adalah 50/50 bagi gabungan ZnO/Al<sub>2</sub>O<sub>3</sub> dan 75/25 bagi gabungan ZnO/OMMT disebabkan oleh sifat sinergi. Secara keseluruhan, komposit nano AE-Al<sub>2</sub>O<sub>3</sub>(1.5)/XLPE mempunyai keputusan yang terbaik. Berbanding XLPE yang tidak terisi, ia telah menunjukkan peningkatan ketara dalam kekuatan tegangan (59%), pemanjangan pada takat putus (51%), modulus Young's (60%), sudut sentuh (4%), kekuatan pecahan dielektrik (35%), rintangan isipadu (55%), suhu penguraian dan pengurangan kadar pembakaran (14%). Ia sesuai untuk aplikasi penebat kabel kerana ciri-ciri mekanik, fizikal, dielektrik dan haba yang luar biasa.

**CHARACTERIZATION OF VARIOUS TYPES OF NANOFILLERS  
FILLED CROSSLINKED POLYETHYLENE COMPOSITES FOR  
CABLE APPLICATION**

**ABSTRACT**

This research is to study the effect of nanofillers on mechanical, physical, dielectric and thermal properties of the crosslinked polyethylene (XLPE) matrix. Untreated nanofillers used are zinc oxide (ZnO) and aluminium oxide ( $\text{Al}_2\text{O}_3$ ). Treated nanofillers used are organoclay (OMMT), 3-aminopropyltriethoxysilane treated ZnO (KH550-ZnO), triethoxycaprylylsilane treated ZnO (TCS-ZnO) and aluminic ester treated  $\text{Al}_2\text{O}_3$  (AE- $\text{Al}_2\text{O}_3$ ). XLPE nanocomposites were prepared by melt mixing with a single screw extruder followed by hot press moulding. Different weight percentages (0.5, 1, 1.5 and 2 wt%) were compounded in untreated and treated nanocomposites. Combinations of ZnO/ $\text{Al}_2\text{O}_3$  and ZnO/OMMT with different ratios (75/25, 50/50 and 25/75) in total of 1 wt% filler loading were compounded in hybrid nanocomposites. Nanocomposites were tested as per ASTM standard methods and characterized with scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The results showed that the addition of untreated and treated nanofillers improved tensile property, burning rate, dielectric property and decomposition temperature. However, it has limited effect on the water resistance property, melting and crystallization temperatures. The optimal filler loading was 1.5 wt% and the effect of  $\text{Al}_2\text{O}_3$  is better than ZnO and OMMT nanofillers based on most properties. Surface treatment with coupling agent enhanced the interface between the filler and the matrix with chemical bonding. Strong filler-matrix interaction further improved the properties of composite. The

optimal filler loading was 1.5 wt% and the effect of AE- $\text{Al}_2\text{O}_3$  is better than KH550-ZnO and TCS-ZnO nanofillers based on most properties. In hybrid nanocomposites, the best filler ratio was 50/50 in ZnO/ $\text{Al}_2\text{O}_3$  and 75/25 in ZnO/OMMT combinations which induced synergistic properties. Overall, AE- $\text{Al}_2\text{O}_3$ (1.5)/XLPE nanocomposite has the best results. As compared with the unfilled XLPE, it has led to the significant improvement in tensile strength (59%), elongation at break (51%), Young's modulus (60%), contact angle (4%), dielectric breakdown strength (35%), volume resistivity (55%), decomposition temperature and reduced burning rate (14%). It is suitable for cable insulation application due to its extraordinary mechanical, physical, dielectric and thermal properties.

# CHAPTER ONE

## INTRODUCTION

### 1.1 Background of Research

Electricity is the key component to modern technology used for domestic appliances in our daily life. By the rapid of urban and city growth, demand of electrical energy is ever increasing and the power system is becoming more complex. The reliability of power supply to the consumers must be ensured with a trustworthy insulation system in order to has greater efficiency and cost effectiveness.

The growing of power cable technology had beginnings in the 1870 where the cables were insulated with natural rubber or porcelain (Ponniran, 2005). After that it was insulated with gutta-percha, oil and wax, jute, hemp and cotton. New evolution had beginnings in 1942 where polyethylene has been used as insulator in cable. It has been launch throughout the years until crosslinked polyethylene (XLPE) was first invented in Canada and United State in the late 1960s. Nowadays, XLPE has been extensively used throughout the world as electrical insulating material in underground distribution and transmission class. In Malaysia, underground power cables are the primary pathway of distributing electrical energy to the houses or industry area. Tenaga Nasional Berhad (TNB) is the largest electricity utility in Malaysia which responsible in keeping the lights on for all residents (Zainal, 2016). TNB has been switched to XLPE cable for the 11 kV underground systems since 1990s (Osman et al., 2005). It was reported that huge number of failures for underground cable system from year 2010 to 2015 as shown in Figure 1.1.



**Figure 1.1:** Number of failure report for underground cable system from year 2010 to 2015 in Malaysia (Zainal, 2016)

Based on Figure 1.1, it shows the reduction failure number for the past years for medium voltage underground cable. Recently, much effort has been carried out to find the solutions to overcome the problems in particularly insulation part. The existing cable mainly failed due to the breakdown of insulation and cable joint. At high water ground area, function failed due to the water ingress of internal part of cable.

A good insulation system is often considered by its electric strength as a dominant characteristic. Nevertheless, thermal and mechanical properties are crucial factors which could critically affect the performance or even failures of electrical insulation (Nelson, 2010). The approach towards seeking the long-term solution to the choice of cable insulation technology is to develop a better insulation system. It has been raised the risk and get attention from the Institute of Electrical and Electronics Engineers (IEEE) and also Dielectrics and Electrical Insulation Society (DEIS) where they are currently focus on the research of improving insulation by adding additives including nanofillers to existing materials. Even little gains in performance can bring commercial significance on electrical insulation usage.