



UNIVERSITI PUTRA MALAYSIA

**DEVELOPMENT AND CHARACTERISATION OF UNSATURATED
POLYESTER COMPOSITE INCORPORATING WASTE RUBBER GLOVE
CRUMBS AS FILLERS**

NUZAIMAH MUSTAFA

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By

NUZAIMAH MUSTAFA

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

October 2020

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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October 2020

Chairman : Professor Mohd Sapuan Salit, PhD, PEng
Faculty : Engineering

This thesis, in general, aims to explore the potential of gloves waste rubber to be incorporated into polymer composites and to help minimise waste caused by gloves disposal. This research started with first objective of characterised the rubber crumbs obtained from waste rubber gloves for its shape, size and thermal properties. The waste gloves were downsized into crumbs with size ranging from 300 μm to 2 mm. The crumbs were regular shapes in shape and has smooth surface. The TGA and DSC analyses showed that the degradation pattern of waste gloves crumbs was similar to the degradation pattern of fresh gloves crumbs, suggesting that the properties of waste gloves did not degrade even after they have been disposed of.

Second objective of the research was to evaluates the effects of different contents of rubber crumbs on the properties of composites. The rubber crumbs of 5%, 10%, 15%, 20%, 25%, 30%, 35 % and 40 % (by weight) were incorporated into unsaturated polyester. The addition of rubber crumbs has increased the impact strength of the composites by 6% and continues to increase as the content of the rubber increases. This is because the rubber gives a certain degree of elasticity to the composite and has better capacity to absorb energy from the impact load. The addition of crumbs, however, decreased the tensile and flexural strength by 80% and 75% respectively. The strength decreases as the rubber crumbs increase because the adhesion between the crumbs and the polyester becomes weaker due to the agglomeration of the crumbs prevents the polyester from binding well to the rubber surface. Many voids can be seen in the SEM micrographs of the tensile fracture as the content of the crumbs increases in the composite.

Third objective was analysed the effect of NaOH, H₂SO₄ and KMnO₄ treatment on rubber crumbs surface to the composite's properties. Composites with NaOH-treated crumbs had improved tensile strength by 2% because NaOH treatment improves the hydrophilicity of the rubber surface, which facilitates better polyester wetting and improves adhesion between rubber and polyester. The rubber crumbs with NaOH treatment have a rougher surface with numerous micropores and microcracks that allow more polyester to penetrate the rubber surface irregularities, thus improving the adhesion of the rubber-polyester. Furthermore, FTIR peak study indicates more polar functional groups and soluble elements on the NaOH-treated rubber surface. These groups initiate further linkages with the polyester polar groups and improves the interfacial adhesion of rubber-polyester. In addition, NaOH eliminated the passive layer created by rubber additives and further reduced the surface contact angle to 31%, allowing for better polyester wetting on rubber. Contrariwise, treatment with H₂SO₄ and KMnO₄ decreased the tensile strength of the composites by 2% and 11% respectively.

Finally, this research assesses the effect on the properties of composites when crumbs have been treated with different concentrations of sodium hydroxide. The 7% of NaOH treatment concentration on rubber crumbs have sufficiently modified the rubber surface and reduced the contact angle of rubber surface by 27%. It increases the hydrophilicity of the rubber and thus improves the rubber-polyester wettability and adhesion. The 7% NaOH intensively etched the rubber and made the surface rougher, more microcracks and irregularities and eliminating the passive layer on the rubber, thereby creating a greater surface area for polyester coverage and better polyester adhesion. It also induced more functional groups as FTIR revealed higher peak intensities, which generate more linkages with polar groups in polyester. These factors strengthened the interfacial rubber-polyester adhesion as the SEM micrograph tensile fracture showed that the rubber crumbs remained intact in the polyester matrix after fractured. In conclusion, waste rubber gloves can be incorporated into unsaturated polyester composites as it improves the composites toughness. Treatment of rubber crumbs with a concentration of 7% of NaOH has effectively modified the rubber surface and strengthens the adhesion between rubber and polyester resulting in improved composite properties.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PEMBANGUNAN DAN PERINCIAN BAGI KOMPOSIT POLIESTER TAK
TEPU TERISI DENGAN SISA SARUNG TANGAN GETAH**

Oleh

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Tesis ini, secara amnya, bertujuan untuk meneroka potensi sisa getah dari sarung tangan untuk dimasukkan ke dalam komposit polimer dan membantu meminimumkan pembuangan sisa getah yang disebabkan oleh sarung tangan terpakai. Penyelidikan ini dimulakan dengan objektif pertama untuk pencirian getah cebis yang diperolehi dari sisa sarung tangan getah bagi bentuk, saiz dan sifat termalnya. Sisa sarung tangan dikecilkan menjadi getah cebis dengan ukuran antara 300 μm hingga 2 mm. Getah cebis mempunyai bentuk yang sekata serta permukaannya licin. Analisis TGA dan DSC menunjukkan bahawa pola degradasi sisa sarung tangan serupa dengan pola degradasi sarung tangan baru, menunjukkan bahawa sifat-sifat sisa sarung tangan tidak merosot walaupun setelah dibuang.

Objektif kedua penyelidikan adalah untuk menilai kesan kandungan getah cebis yang berbeza terhadap sifat komposit. Getah cebis sebanyak 5%, 10%, 15%, 20%, 25%, 30%, 35% dan 40% (dalam ukuran berat) dimasukkan ke dalam poliester tak tepu. Penambahan getah cebis telah meningkatkan kekuatan hentaman komposit sebanyak 6% dan ia terus meningkat apabila kandungan getah meningkat. Ini kerana getah memberikan tahap keanjalan tertentu pada komposit dan mempunyai keupayaan yang lebih baik untuk menyerap tenaga dari beban hentaman. Penambahan getah cebis, bagaimanapun, menurunkan kekuatan tegangan dan lenturan masing-masing sebanyak 80% dan 75%. Kekuatannya semakin berkurang apabila getah cebis meningkat kerana lekatan antara cebisan dan poliester menjadi lemah kerana penggumpalan getah cebis menghalang poliester daripada melekat dengan baik ke permukaan getah. Banyak kekosongan yang dapat dilihat dalam mikrograf SEM bagi fraktur tegangan komposit yang mempunyai kandungan getah cebis yang lebih banyak.

Objektif ketiga adalah menganalisa kesan rawatan NaOH, H₂SO₄ dan KMnO₄ terhadap getah cebis kepada sifat komposit. Komposit dengan getah cebis dirawat NaOH mempunyai kekuatan tegangan yang lebih baik sebanyak 2% kerana rawatan NaOH meningkatkan sifat hidrofilik permukaan getah, ia memudahkan kadar pelekatan poliester dan memperbaiki lekatan antara getah dan poliester. Getah cebis dengan rawatan NaOH mempunyai permukaan yang lebih kasar dan mempunyai lebih banyak pori dan retak mikro yang membantu lebih banyak poliester menembusi liang-liang permukaan getah, sehingga meningkatkan lekatan antara getah dan poliester. Selanjutnya, analisa FTIR menunjukkan terdapat lebih banyak kumpulan berfungsi berpolar dan unsur-unsur boleh larut pada permukaan getah yang dirawat dengan NaOH. Kumpulan-kumpulan ini menghasilkan ikatan polar lebih banyak dengan kumpulan polar di dalam poliester dan memperkuat lagi lekatan antara getah dan poliester. Di samping itu, rawatan NaOH menghilangkan lapisan pasif yang terhasil daripada serapan bahan didalam getah dan seterusnya mengurangkan sudut sentuhan permukaan menjadi 31%, yang mana menghasilkan lekatan poliester lebih baik terhadap getah. Sebaliknya, rawatan dengan H₂SO₄ dan KMnO₄ menurunkan kekuatan tegangan komposit masing-masing sebanyak 2% dan 11%.

Akhirnya sekali, kajian ini menilai kesan terhadap sifat komposit apabila getah cebis dirawat dengan kepekatan natrium hidroksida yang berbeza. Rawatan berkepekatan 7% NaOH terhadap getah cebis berjaya mengubah permukaan getah dengan baik dan mengurangkan sudut sentuhan permukaan getah sebanyak 27%. Ini meningkatkan sifat hidrofilik getah dan dengan itu meningkatkan kemampuan lekatan poliester terhadap getah. 7% NaOH mengubah permukaan getah secara intensif dan menjadikan permukaannya lebih kasar dengan lebih banyak retak-retak mikro, ia turut menghilangkan lapisan pasif pada getah, lalu menyediakan permukaan yang lebih luas untuk liputan poliester dan meningkatkan kadar lekatan poliester. Ia juga menghasilkan lebih banyak kumpulan berfungsi berpolar pada permukaan getah sepertimana ditunjukkan oleh kadar puncak-puncak FTIR yang lebih tinggi, kumpulan fungsi ini menghasilkan lebih banyak hubungan dengan kumpulan fungsi berpolar pada poliester. Faktor-faktor ini menguatkan lekatan poliester terhadap getah seperti yang ditunjukkan dalam mikrograf SEM pada permukaan patah tegangan yang mana getah cebis tetap utuh terikat di dalam matriks poliester. Kesimpulannya, sisa sarung tangan boleh dimasukkan kedalam poliester tidak tepu kerana ia membantu meningkatkan kekuatan impak bagi komposit. Rawatan getah cebis dengan menggunakan kekuatan larutan NaOH sebanyak 7% dapat membantu memperbaiki sifat-sifat bagi komposit.

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TABLE OF CONTENTS

	Page
ABSTRACT	i
ABSTRAK	iii
ACKNOWLEDGEMENTS	v
APPROVAL	vii
DECLARATION	ix
LIST OF TABLES	xiv
LIST OF FIGURES	xv
LIST OF ABBREVIATIONS	xx
CHAPTER	
1 INTRODUCTION	1
1.1 Background	1
1.2 Problem Statements	2
1.3 Research Objectives	3
1.4 Significance of the Study	3
1.5 Scope and Limitation of the Study	4
1.6 Structure of Thesis	4
2 LITERATURE REVIEW	7
2.1 Introduction	7
2.2 Rubber	7
2.2.1 Rubber Vulcanisation	8
2.2.2 Rubber Properties and Applications	9
2.2.3 Types of Rubber	10
2.2.4 Rubber Production and Consumption	11
2.2.5 Rubber Export	12
2.3 Waste Rubber Output	13
2.3.1 Waste Rubber Impact on Environment	14
2.3.2 Recycling of Waste Rubber	15
2.4 Rubber Glove	16
2.4.1 Rubber Glove Manufacturing	17
2.4.2 Rubber Glove Properties	19
2.4.3 Rubber Gloves Toxicity	19
2.4.4 Rubber Gloves Demand	20
2.5 Development of Composites Using Waste Rubber	21
2.5.1 Waste Tyres in Composites	23
2.5.2 Waste Rubber Gloves in Composites	25
2.5.3 Other Types of Waste Rubber in Composites	29
2.6 Development of Waste Rubber in Various Types of Matrix	31
2.6.1 Waste Rubber in Cement and Asphalt Matrix	31
2.6.2 Waste Rubber in Rubber Matrix	33
2.6.3 Waste Rubber in Polymer Matrix	36
2.6.4 Waste Rubber in Thermoplastic Matrix	36

	2.6.5	Waste Rubber in Thermoset Matrix	37
2.7		Thermoset Unsaturated Polyester (UP) as Matrix in Composites	38
	2.7.1	Fillers and Reinforcements for Unsaturated Polyester Composites	42
	2.7.2	Waste Tyres as Fillers in Unsaturated Polyester	42
2.8		Downsizing the Waste Rubber	43
	2.8.1	Surface Treatment of Waste Rubber Crumbs	49
	2.8.2	Effects of Waste Rubber Crumbs on Polymer Composite Mechanical Properties.	55
2.9		Aspects Involved in The Development of Polymer Composites Incorporating Waste Rubber	57
2.10		Summary	68
3		MATERIALS AND METHODS	69
	3.1	Introduction	69
	3.2	Materials	70
	3.2.1	Waste Rubber	70
	3.2.2	Cryogenic Grinding of Waste Rubber Crumbs	70
	3.2.3	Unsaturated Polyester	71
	3.2.4	Sodium Hydroxide (NaOH), Sulphuric Acid (H ₂ SO ₄) and Potassium Permanganate (KMnO ₄).	72
	3.3	Fabrication of Composite	72
	3.3.1	Fabrication of Composite with Different Rubber Crumbs Content in The Unsaturated Polyester	73
	3.3.2	Rubber Crumbs Surface Treatment with Different Chemicals	73
	3.3.3	Fabrication of Composite with Rubber Crumbs of Different Surface Treatments	74
	3.3.4	Rubber Crumbs Surface Treatments with Different Concentrations of NaOH	75
	3.3.5	Fabrication of Composites with Rubber Crumbs of Various NaOH Concentrations	75
	3.4	Characterisation of Rubber Crumbs	75
	3.4.1	Size of Rubber Crumbs	76
	3.4.2	Shape and Surface Characteristic Analysis of Rubber Crumbs	76
	3.4.3	Thermal Properties	76
	3.4.4	Fourier Transform Infrared Spectrometry (FTIR) Analysis of Rubber Surface	77
	3.4.5	Contact Angle Measurement	77
	3.5	Characterisation of Composites	78
	3.5.1	Mechanical Testing	78
	3.5.2	Tensile Testing	79
	3.5.3	Flexural Testing	79
	3.5.4	Impact Testing	79
	3.5.5	Scanning Electron Microscope (SEM)	80
	3.6	Summary	80

4	PHYSICAL AND THERMAL PROPERTIES OF RUBBER GLOVE WASTE AS POTENTIAL FILLER FOR POLYMER COMPOSITES	81
	Article 1	81
	Acceptance Letter	92
5	MICROSTRUCTURE AND MECHANICAL PROPERTIES OF UNSATURATED POLYESTER COMPOSITES FILLED WITH WASTE RUBBER GLOVE CRUMBS	93
	Article 2	93
	Acceptance Letter	111
	Copyright Permission	112
6	EFFECT OF SURFACE TREATMENT ON THE PERFORMANCE OF POLYESTER COMPOSITE FILLED WITH WASTE GLOVE RUBBER CRUMBS	113
	Article 3	113
	Acceptance Letter	131
	Copyright Permission	132
7	SODIUM HYDROXIDE TREATMENT OF WASTE RUBBER CRUMB AND ITS EFFECTS ON PROPERTIES OF UNSATURATED POLYESTER COMPOSITES	133
	Article 4	133
	Acceptance Letter	151
	Copyright Permission	152
8	DISCUSSION CHAPTER	153
	8.1 Introduction	153
	8.2 Effect of 30% rubber crumbs on tensile and flexural strength of composite	153
	8.3 The effectiveness of NaOH, H ₂ SO ₄ and KMnO ₄ treatment on rubber crumbs	156
	8.4 Effect of 10% NaOH concentration on the composite strength	158
	8.5 Conclusions	159
9	CONCLUSIONS AND RECOMMENDATIONS	160
	9.1 Conclusions	160
	9.2 Recommendations for Future Research	163
	REFERENCES	164
	APPENDICES	182
	BIODATA OF STUDENT	186
	LIST OF PUBLICATIONS	187

LIST OF TABLES

Table		Page
2.1	Malaysia rubber products exports in year 2014 – 2019	13
2.2	Estimated quantity of waste tyres generated in a year	13
2.3	Mechanisms of managing waste rubber	15
2.4	Typical medical examination glove properties	19
2.5	Development of composites incorporating waste tyres	24
2.6	Development of composites incorporating waste rubber gloves	28
2.7	Development of composites incorporating other types of waste rubber	30
2.8	Properties of rubber matrix composites that incorporating waste rubber	35
2.9	Thermoset attributes	38
2.10	Properties and applications of unsaturated polyester resin	40
2.11	Process of downsizing the waste rubber	45
2.12	Surface treatments on waste rubber crumbs	52
2.13	Tensile strength of polymer matrix composites filled with waste rubber	56
2.14	Aspects involved in the development of polymer composites incorporating waste rubber	58
5.1	Compositions, tensile strength, elastic modulus and materials cost of the composites	102
5.2	Tensile strength of rubber-polymer composites reported by different authors	103

LIST OF FIGURES

Figure	Page	
2.1	General scheme of rubber-sulphur vulcanisation	8
2.2	Vulcanised rubber 3D network	9
2.3	Rubber tree (<i>Hevea Brasiliensis</i>)	11
2.4	World rubber production and consumption in 2012-2019 (million tonnes)	12
2.5	Rubber products exports by Malaysia in year 2012 – 2019 (RM million)	12
2.6	Estimated annual tonnage of waste tyres generated in Peninsular Malaysia for 2010 – 2015	14
2.7	Medical examination gloves	17
2.8	Schematic diagram of glove manufacturing process	18
2.9	Global demand of rubber gloves in billion pieces	21
2.10	SEM micrograph of tensile fracture surface for (a) ENR 50/NBR blend and (b) NR/NBR blend	25
2.11	SEM micrograph of tensile fractured surface (a) PP/NBR with epoxy (b) PP/NBR without epoxy	26
2.12	SEM micrograph of (a) CR/ SMR L blends (b) CR / ENR 50 blends at 150x magnification	27
2.13	Impact strength of polymer composites with a different ratio of NRG/PSF (a) 30/70 (b) 50/50 (c)70/30	37
2.14	Various sizes of crumb rubber used in rubberised concrete	48
2.15	(a) Waste tyre rubber chips (b) Waste tyre rubber crumbs	49
3.1	The overall structure of the research methodology	69
3.2	Preparation of rubber crumbs via cryogenic grinding (a) gloves were washed and dried (b) gloves were cut into smaller pieces (c) gloves pieces were soak with liquid nitrogen until frozen (d) frozen rubber pieces (e) frozen rubber were ground and (f) rubber crumbs	71

3.3	Fabrication of rubber- unsaturated polyester composite filled with crumbs from waste rubber gloves (a) rubber gloves crumbs (b) mixed rubber crumbs with polyester (c) transferred the mixture into mould allowed composite to cure for 24 hours and (d) removed the hardened composite from mould	72
3.4	Different rubber crumbs content of waste rubber- unsaturated polyester composites	73
3.5	Rubber crumbs surface treatment with (a) NaOH (b) H ₂ SO ₄ and (c) KMnO ₄	74
3.6	Unsaturated polyester composites incorporated with rubber crumbs from different treatment (a) NaOH treated rubber (b) H ₂ SO ₄ treated rubber and (c) KMnO ₄ treated rubber	74
3.7	Rubber crumbs surface treatment using different NaOH concentration (a) 1% (b) 4% (c) 7% and (d) 10%	75
3.8	(a) Rubber crumbs (b) sieving process (c) shape and surface characterisation of rubber crumbs using SEM (d) of rubber crumbs thermal analysis using thermal analyser (e) FTIR analysis of treated rubber crumbs and (f) contact angle analysis of treated rubber crumbs	76
3.9	Droplet image captured using contact angle measurement equipment	77
3.10	Measurement of water droplet contact angle	78
3.11	(a) Tensile and flexural testing (b) Impact testing (c) morphological characteristics of the fractured samples using SEM	78
3.12	Tensile samples dimensions	79
4.1	Rubber crumbs preparation (a) waste rubber gloves were washed and dried (b) gloves were soaked with liquid nitrogen until frozen (c) frozen gloves were crushed immediately (d) rubber crumbs	84
4.2	Auto sieve shaker	84
4.3	Morphology analysis (a) Sputter coater (b) Scanning Electron Microscope (SEM)	85
4.4	(a) Thermal analyser (b) Sample loading for TGA analysis	86
4.5	Different sizes of rubber crumbs (a) 300 to 450 μm (b) 450 to 600 μm (c) 600 to 850 μm (d) 850 to 2 mm (e) above 2 mm	87
4.6	SEM image of rubber crumbs sizes distribution	88
4.7	Thermogravimetric curves of fresh rubber crumbs and waste rubber crumbs	90

4.8	Derivative curves of fresh rubber crumbs and waste rubber crumbs	90
5.1	Particle size distribution of rubber crumbs	97
5.2	Preparation of rubber crumbs via cryogenic grinding; (a) Gloves were washed and dried (b) gloves were cut into smaller pieces (c) gloves pieces were soak with liquid nitrogen until frozen (d) frozen rubber pieces (e) frozen rubber were ground and (f) rubber crumbs	98
5.3	Fabrication of rubber-polyester composite filled with crumbs from waste rubber gloves; (a) rubber gloves crumbs (b) mixed rubber crumbs with polyester (c) transferred the mixture into mould allowed composite to cure for 24 hours and (d) removed the hardened composite from mould	99
5.4	Different rubber crumbs contents of waste rubber-polyester composites	99
5.5	Tensile strength of the composites vs rubber crumbs contents	102
5.6	SEM micrographs of tensile fracture surface of rubber-polyester composites; (a) pure polyester (b) 5 % rubber (c) 10 % rubber (d) 20 % rubber (e) 30 % rubber and (f) 40 % rubber	104
5.7	SEM micrographs of void and bubbles within the rubber filled polyester composites	105
5.8	SEM micrographs of composites with (a) 5% rubber and (b) 40% rubber	105
5.9	Flexural strength of the composites vs rubber crumbs contents	107
5.10	Impact strength vs rubber crumbs contents	108
5.11	SEM micrograph of impact surface fracture of (a)10% of rubber (b) 30% of rubber	108
5.12	SEM micrograph of impact surface fracture of rubber-polyester composites	109
6.1	Measurement of water droplet contact angle	120
6.2	FTIR spectra for untreated and treated rubber crumbs	122
6.3	Contact angles of rubber crumbs	125
6.4	Water droplet profiles on rubber surfaces of (a) untreated, (b) NaOH treated, (c) H ₂ SO ₄ treated and (d) KMnO ₄ treated	125
6.5	Tensile strength of composites	127

6.6	SEM surface morphology of (a) untreated rubber and (b) NaOH treated rubber at 3000 x magnification respectively	128
6.7	SEM micrographs of composite tensile fractured surfaces of (a) untreated rubber and (b) NaOH treated rubber at 300 x magnification respectively	129
6.8	SEM micrographs of composite tensile fractured surface of (a) H ₂ SO ₄ treatment and (b) KMnO ₄ treatment at 100 x magnification respectively	130
7.1	Rubber crumbs surface treatment using NaOH solution of (a) 1%, (b) 4%, (c) 7%, and (d) 10%	136
7.2	Tensile samples dimensions	137
7.3	Contact angle measurement equipment.	138
7.4	Image of the (a) water droplet and (b) contact angle measurement with Image J software	138
7.5	FTIR spectra for untreated rubber crumbs and treated rubber crumbs with different NaOH concentrations	139
7.6	Tensile strength and elongation at break of the composites with different NaOH concentration	141
7.7	SEM of 7% NaOH treated rubber surface morphology	142
7.8	SEM of 10% NaOH treated rubber surface morphology	143
7.9	SEM of 1% NaOH treated rubber surface morphology	143
7.10	SEM of 4% NaOH treated rubber surface morphology	144
7.11	Tensile fracture micrograph of composites with 7% NaOH treated rubber crumbs	145
7.12	Tensile fracture micrograph of composites with 4% NaOH treated rubber crumbs	145
7.13	Modulus of elasticity of the rubber polyester composites.	146
7.14	Contact angles of treated rubber crumbs	147
7.15	Water droplet profiles on NaOH treated rubber crumbs surface: (a) 1%, (b) 4%, (c) 7%, and (d) 10%	148
7.16	Effect of rubber contact angle on tensile strengths	149

8.1	SEM micrograph of tensile fractured surface of composite with 30% rubber	154
8.2	SEM micrograph of tensile fractured surface of composite with (a) 20% rubber and (b) 40% rubber	154
8.3	Composite flexural strength vs different amount of rubber	155
8.4	Mechanical interlocking adhesion between the unsaturated polyester matrix and rubber rough surface	156
8.5	Schematic diagram of sodium stearate	157
8.6	Oxidation reaction happened during the surface modification	158
8.7	Effects of different NaOH concentrations of rubber surface treatment on composite tensile strength	159

LIST OF ABBREVIATIONS

3D	Three-Dimensional
ATR	Attenuated Total Reflectance
BR	Butadiene Rubber/ Polybutadiene
C=C	Carbon-carbon bond
C-H	Carbon-hydrogen bond
CH ₂	Methylene group
CH ₃	Methyl group
CO	Carbon-oxygen bond
CR	Polychloroprene/ Neoprene
CR	Polychloroprene
C-S	Carbon-sulphur bond
CSM	Chlorosulphonated Polyethylene rubber
EOC	Ethylene- 1-octene copolymers
EPDM	Ethylene-propylene-diene monomer
FDA	US Food and Drug Administration
FTIR	Fourier Transform Infrared Spectrometry
H ₂ O ₂	Hydrogen peroxide
H ₂ SO ₄	Sulphuric acid
HAF	High Abrasion Furnace
HDPE	High Density Polyethylene
HNO ₃	Nitric acid
K ₂ Cr ₂ O ₇	Potassium dichromate
KMnO ₄	Potassium permanganate
LDPE	Low Density Polyethylene
MA	Maleic Anhydride
MAPE	Maleic Anhydride Grafted Polyethylene

MARGMA	Malaysian Rubber Glove Manufacturers Association
MEKP	Methyl Ethyl Ketone Peroxide
NaHSO ₃	Sodium bisulfite
NaOH	Sodium hydroxide
NBR	Acrylonitrile butadiene rubber /Nitrile rubber
NR	Natural rubber
NRG	Natural rubber glove
NRP	Natural rubber prophylactic
OH	Hydroxyl group
PE	Polyethylene
PP	Polypropylene
PSF	Polystyrene foam
PU	Polyurethane
PVA	Polyvinyl acetate
PVC	Polyvinyl chloride
PPE	Personal Protective Equipment
RRII	The Rubber Research Institute of India
S=O	Sulphonic group
SBR	Styrene Butadiene Rubber
SEM	Scanning Electron Microscope
SMG	Standard Malaysian Glove
T_g	Glass transition temperature
TGA	Thermogravimetric Analysis
UPM	Universiti Putra Malaysia
UP	Unsaturated Polyester
WHO	World Health Organization
kg	kilogram
g	gram

mg	milligram
m	metre
cm	centimetre
mm	millimetre
μm	micrometre/micron
mL	millilitre
μl	microliter
cm^{-1}	reciprocal wavelength
cm^3	cubic centimetre
cps	centipoise
h	hour
min	minute
MPa	Megapascal
GPa	Gigapascal
J	Joule
kN	kilo Newton
kV	kilovolts
phr	Parts per hundred rubber
RM	Ringgit Malaysia
$^{\circ}$	Contact angle
$^{\circ}\text{C}$	Temperature
wt.%	Weight percent
%	Percent
vol.%	Volume percent

CHAPTER 1

INTRODUCTION

1.1 Background

Recycling and recovery of waste rubber product such as glove, tyres, mattresses, shoe soles, automotive parts have received much attention in recent years. It provides an important implication such as protection to the environment, conservation of energy, as an alternative raw materials, helps in reducing cost, turning the waste to eco-friendly materials and improves the existing rubber related processing and properties (Rajan et al., 2006; Sienkiewicz et al., 2017). Rubber is difficult to degrade due to the highly durable properties which poorly managing the waste rubber products can lead to many environmental issues. As a result, many innovations and initiatives have been taken to recycle rubber goods and continue to evolve. Waste rubber has many valuable features that are worth reusing instead of being discarded and are relatively inexpensive and easy to obtain. The development of composites filled with waste rubber therefore provides a new future in the field of polymer research.

Several studies have shown that researchers are utilising waste rubber by generating new material or new products and incorporating waste rubber into existing product compounds or formulations. The history of using waste rubber as fillers begins with Charles Goodyear, the rubber historian who invented the vulcanisation process. He patented his work in 1853 of which describes the process of making a polymer materials from rubber granulate and natural rubber (Sienkiewicz et al., 2017). The works soon grew with many researchers exploring the mixing of waste rubber products into many mediums such as cements and resins.

Waste rubber has many excellent qualities that can be fully exploited for use in many useful applications. Rubber plays a major role in polymer-based composites, as the soft rubber phase gives flexibility to the composite, while the hard phase in polymers, such as polyester, provides strength. (Esmizadeh et al., 2017). The combination of these two distinct properties of the two components gives the composite some remarkable properties. In this research, waste rubber glove has been chosen as a raw material because of its high elasticity (Eng, 2016; Ikram, 1999; MREPC, 2017). Elastic behaviour of this elastomer can be benefited into functional material such as asphalt modifier, damping components, thermal and noise insulator and others. In addition, waste rubber glove is relatively inexpensive and easy to acquire, as this can lead to low production costs (Ahmad et al., 2016). This unique behaviour of glove has attracts researchers to further explore its potential in developing new sustainable material (Ismail et al., 2009; G. Mathew et al., 2001, 2006; Riyajan et al., 2012).

1.2 Problem Statements

As the standard of life improved and healthcare awareness risen globally, the rubber glove industry has grown rapidly over the years. Moreover, the rubber glove demand at surge due to the latest Covid-19 pandemic that hit the world badly. The pandemic had caused hundreds of thousands of people dead and millions of people more infected. As of 19 July 2020, there were 597,583 deaths and 5,934,936 confirmed cases worldwide, according to the World Health Organization (WHO). Hence, the gloves industry, faced with high global demand as gloves are used by frontliners, industries and individuals in the fight against the global pandemic. Before the pandemic, an average growth of rubber glove was 8%, generally due to the growing population, increasing usage in industrial such as food and electronic industries and development in healthcare systems.

Malaysian Rubber Glove Manufacturers Association (MARGMA) previous estimation of global demand for rubber gloves are 345,000 million pieces in 2020. However, when the COVID-19 pandemic hit and spreads aggressively across the globe, the Economic Intelligence Center estimated that global demand for total rubber gloves in 2020 will be 360,000 million pieces, which is 20% higher than the usual projection. It is the result of a 57% rise in the average demand for medical rubber gloves, which increased to 60,000 million pieces from normal (Kanjavisut, 2020).

As a consequence, the high use of rubber gloves led to significant gloves being disposed of, which would ultimately contribute to a rise in dumping gloves, either in the landfill, incineration plant or random dumping, such as in the housing area and parks. Rubber gloves are not easy to biodegrade as they have undergone a vulcanisation process that creates highly cross-linked thermoset materials that makes gloves very elastic, robust and incredibly effective for protecting against many harmful substances such as blood, pathogens, microbes, chemicals and gases. Also, the rubber gloves resistance to abrasion, cut, tear and puncture. These features makes rubber gloves resilient to most environmental agents and may take more than 100 years to degrade (McKeen, 2014; Riyajan et al., 2012; Samsuri, 2010; Saxena et al., 1999; Torretta et al., 2015). Because of that, the excessive dumping rubber gloves consequently will create a serious environmental problem such as breeding ground for mosquitoes and rodents as well as dangerous animals like snakes and scorpions. In addition, chemicals additives in the gloves such as zinc and sulphur can leach out and pollute soil and water. Furthermore, illegal gloves burning can release toxic gases, hazardous smoke, particulate matter into the atmosphere and pollute the air (Adhikari et al., 2000; Bailey et al., 2002; Nuzaimah et al., 2018; Ramarad et al., 2015; Thomas & Gupta, 2016). The initiative of this research to explore the feasibility of incorporating waste rubber gloves into polymer composites would help to minimise the amount of discarded waste gloves. With the aim of helping to mitigate the environmental issues caused by rubber products. Furthermore, polymer composites have a large market share which, with only 5% of the waste gloves in the polymer composites it will have a significant impact on the consumption of waste gloves.

1.3 Research Objectives

The objectives of this research are:

1. To characterise the rubber crumbs obtained from waste rubber gloves for the physical, thermal and morphological properties.
2. To evaluate the effect of different contents of rubber crumbs on the mechanical and morphological properties of the waste rubber glove-unsaturated polyester composites.
3. To analyse the effect of sodium hydroxide sulphuric acid and potassium permanganate treatment on the rubber crumbs surface and its effect on the mechanical and morphological properties of waste rubber glove-unsaturated polyester composites.
4. To assess the effect of different concentrations of sodium hydroxide on the rubber crumbs surface and its effect on the mechanical and morphological properties of waste rubber glove-unsaturated polyester composites.

1.4 Significance of the Study

This study adds the following:

1. Successful development of unsaturated polyester composites with waste rubber gloves could benefit by adding more value to the waste rubber and by widening the use of waste rubber gloves in the field of material engineering in general and in the field of polymer composite in particular. To date, most of the recycled waste rubber has been from waste tyres, and is commonly used as fillers in concrete and asphalt.
2. The development of unsaturated polyester composites with waste rubber gloves is expected to widen the source of alternative filler materials as a substitute for conventional materials such as mineral fillers.
3. The study is expected to further explore the potential of waste rubber gloves in the development of recyclable products.
4. The outputs and findings of this research are expected to enhance the knowledge about incorporation of waste rubber in polymer composites.
5. The development of waste rubber gloves as fillers in unsaturated polyester composite is one of the effort to divert the previously known as hard-to-recycle rubber materials into a more beneficial usage.
6. The development of waste rubber gloves unsaturated polyester composite is expected to reduce the rubber gloves disposal issues which eventually reduce the environmental pollutions.
7. The recycling of waste rubber gloves helps to reduce the consumption of fresh raw materials and thus reduces the need for raw materials to be extracted and processed. The recycling of waste rubber gloves will therefore save energy and save natural resources.

1.5 Scope and Limitation of the Study

This research focuses on the method of producing rubber crumbs from waste gloves and on assessing the potential for the crumbs to be incorporated into an unsaturated polyester matrix. The waste rubber gloves were disposable glove collected from laboratories within Universiti Putra Malaysia (UPM). The waste gloves were grounded into crumbs using cryogenic grinding and were characterised for sizes, shapes, surface morphology using SEM and thermal analysis using TGA. The results of the analyses were then compared to the crumbs obtained from fresh rubber gloves. Toxicity level was not being analyse in this research as this research focus on assessing the potential incorporation of waste gloves into composite. Subsequently, this research was to determine the optimum rubber content in the unsaturated polyester composites by evaluating the effect of addition of 5, 10, 15, 20, 25, 30, 35 and 40 wt. % of rubber content in the composites. The composites were characterised for tensile, flexural and impact strength, elastic modulus, and fracture surface morphology. Composites with rubber content that gives optimum properties was used to analyse the effect of rubber crumbs treatment with sodium hydroxide, sulphuric acid and potassium permanganate. The rubber crumbs that treated with these chemicals underwent FTIR analysis, SEM surface morphology analysis and contact angle measurement and the composites prepared with addition of the treated rubber crumbs underwent tensile testing and fracture morphology analysis. The sodium hydroxide treatment had sufficiently modified the rubber surface which enhances the interfacial rubber-polyester adhesion thus improved the mechanical properties of the composite. Thus, in the final phase of this research was evaluates the effects of different sodium hydroxide concentration on the rubber surface. The rubber crumbs were treated with 1%, 4%, 7%, and 10% by volume of sodium hydroxide and being analysed for surface morphology using SEM, FTIR analysis and contact angle measurement. Composites were prepared with addition of rubber crumbs from each treatment respectively. The tensile test was performed to determine the treatment that gives composites with good tensile strength and the SEM analysis was performed to study the fracture behaviour of the composites. Besides that, the level of toxicity of recycled waste gloves has not been determined in this research as it is not the focus of the study.

1.6 Structure of Thesis

The structure of this thesis is in accordance with the alternative thesis format of Universiti Putra Malaysia which is based on the publications of this study. Each research chapter represents a separate study that has its own 'Introduction', 'Materials and Methodology', 'Results and Discussion', and 'Conclusions'. The details of the structure are presented as follows:

Chapter 1

Background of the research with problem statement that initiates this research is explained in this chapter. Research objectives, significant of the research, scope and limitation of the research also highlighted in this chapter.

Chapter 2

A comprehensive literature review on the generation of waste rubber worldwide, the environmental impact of waste rubber, waste rubber management and waste rubber recycling efforts. This chapter also includes general information on rubber, applications and global rubber production and consumption. In addition, the chapter briefly explains about rubber gloves, the manufacturing process and their properties. Finally, this chapter presents the development of various composites incorporating different types of waste rubber.

Chapter 3

A detail methodology of preparation of materials, testing procedures, and data collection are presented in this chapter.

Chapter 4

This chapter presents the first article entitled **“Physical and Thermal Properties of Rubber Glove Waste as Potential Filler for Polymer Composites”**. In this article, the properties of waste rubber gloves were evaluated to discover the potential of the waste glove to potential to be used as a filler for the polymer composites.

Chapter 5

This chapter presents the second article entitled **“Microstructure and Mechanical Properties of Unsaturated Polyester Composites Filled with Waste Rubber Glove Crumbs”**. In this article, the focus is on studying the effect of different rubber crumbs loading on microstructure and mechanical properties of unsaturated polyester composite filled with waste rubber gloves crumbs.

Chapter 6

This chapter presents the third article entitled **“Effect of Surface Treatment On the Performance of Polyester Composite Filled with Waste Glove Rubber Crumbs”**. In this article, the study conducted is to investigate the effect of different rubber crumbs treatments on the composite properties.

Chapter 7

This chapter presents the fourth article entitled **“Sodium Hydroxide Treatment of Waste Rubber Crumb and Its Effects On Properties of Unsaturated Polyester Composites”**. In this article, presented the investigation of finding the optimum NaOH concentration treatment for rubber crumbs that improves adhesion between the polymer matrix and rubber filler in rubber polyester composites.

Chapter 8

This chapter presents a further discussion of the findings of this research.

Chapter 9

This chapter presents the overall conclusions from the whole study as well as future recommendations for further improvement of this research.



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LIST OF PUBLICATIONS

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Awards and Achievements

Best presenter in Seminar Enau Kebangsaan 2019, 1st-2nd April 2019, Bahau, Negeri Sembilan, Malaysia.

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