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### **APPROVAL AND DECLARATION SHEET**

This thesis titled Effect of Chemical Modification of Eggshells Powder Filled Low Density Polyethylene Composites was prepared and submitted by Siti Shuhadah Binti Md Saleh (Matrix Number: 0730410162) and has been found satisfactory in terms of scope, quality and presentation as partial fulfillment of the requirement for the award of degree of Master of Science (Materials Engineering) in University Malaysia Perlis (UniMAP). The members of otionalcot the Supervisory committee are as follows:

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#### ACKNOWLEDGEMENT

بسم الله الرحمن الرحيم

Alhamdulillah and thank to Allah, which has gave me the commitment and the strength to finish this project. First of all I would like to express my sincere gratitude to project supervisor Dr.Supri A.Ghani for his support throughout my time at UniMAP. His talents, dedication, and enthusiasm for research will be a continuing source of inspiration. My thanks also for my co-supervisor Brig. Jen. Dato Prof. Dr. Kamarudin Hussin.

Special thanks to my father Md Saleh bin Abdullah, my mother Rahmah binti Senik, my sister Siti Rodziah and Siti Rosilah & family for their patience, understanding, unfailing love and support through the ups and downs. I also wish to express my appreciation to Dr Salmah Hussiensyah and Prof. San Myint , who made many valuable suggestions and gave constructive advice. I also would like to express thanks to the Dean School of Materials Engineering, Dr. Khairel Rafezi Ahmad with the approval of my master thesis.

A also would like to express my gratitude to all postgraduate colleagues in School of Materials Engineering especially Dahlia, Nurul Izza, Raudah, Radzi Ali, Shahrizan and Hashahrin. Not forgetting for all technicians of School of Materials Engineering, especially Mr Norzaidi, Mr Nasir, Mr Azmi, Mr Rosmawadi and Mr Hazrul for the guidance to operate the lab equipments. My sincere thanks also goes to my friends Nur Hanim Naim, Zubir, Nur Syuhada, Zarimawati, Nur Farhan ,Nurul Husna and Rosyidi for their support.

iv

Not forgotten, I would like to express my gratitude and thanks to our late lecturer Allahyarham Prof Nasir and my best friend Allahyarham Mohd Hafizuddin Abd Hady.

Specials thanks to Ministry of Science, Technology and Innovation (MOSTI) for .EH ERLIS ADD OTIGETAD financial support via National Science Fellowship Scheme (NSF). Finally, would like to thank everyone that has been involved in this project directly or indirectly for their help and

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# LIST OS SYMBOLS, ABBREVIATIONS OR NUMENCLATURE

	μm	Micron meter
	ABS	Acrylonitrile butadiene styrene
	$AI_2O_3$	Aluminium oxide
	ASTM	American society for testing and materials
	C <sub>2</sub> H <sub>4</sub> (NH <sub>2</sub> ) <sub>2</sub>	Ethylene diamine
	$C_2H_4$	Ethylene
	C <sub>6</sub> H <sub>4</sub> (COOH) <sub>2</sub>	Isophthalic acid
	CaCO <sub>3</sub>	Calcium carbonate
	CaO	Calcium oxide
	CI	Chlorine
	cm	Centimeter
	Cr <sub>2</sub> O <sub>3</sub>	Chromium oxide
	CuO	Copper oxide
	DMA	Dynamic mechanical analyzer
	DSC	Differential scanning calorimetry
	ES	Eggshells
	ESCR	Environmental stress crack resistance
	ESP	Eggshells powder
	FDT	Final decomposition temperature (°C)
	g	Gram
	HDPE	High density polyethylene
	HMWHDPE	High molecular weight high density polyethylene

	kg	Kilogram
	LDPE	Low density polyethylene
	LLDPE	Low linear density polyethylene
	LOI	Loss of ignition
	MA	Maleic anhydride
	MFI	Melt flow index
	min	Loss of ignition Maleic anhydride Melt flow index Minute Millimeter Manganese oxide
	mm	Millimeter
	MnO	Manganese oxide
	Mt	Percentage of water absorption
	MW	Molecular weight
	MWD	Molecular weight distribution
	NaCl	Sodium chloride
	NaOH	Sodium hydroxide
	°C	Degree celcius
	ovx	Ovariectomy
	PE . CIT	Polyethylene
	РЕМАН	Polyethylene-grafted-maleic anhydride
<i>(</i>	PR	Polypropylene
$\bigcirc$	PS	Polystyrene
	RM	Residual mass
	rpm	Rotation per minute
	S	Sulfur
	SEM	Scanning electron microscope
	SiO <sub>2</sub>	Silicon dioxide
	T- <sub>50</sub> %	Temperature of 50% weight loss (°C)

#### TEM Transmission electron microscopy

Τ<sub>α</sub> Glass transition temperature

TGA Thermogravimetry analysis

 $T_{m}$ Melting temperature

e ieinal copyright UHMWPE Ultra high molecular weight polyethylene

ULDPE Ultra low density polyethylene

UTM Universal testing machine

VLDPE Very low density polyethylene

w t% Weight percent

Original dry weight Wd

WN Weight after exposure

,y of this term is protect Enthalpy of fusion of the composite ΔH<sup>o</sup><sub>f</sub>

#### KOMPOSIT POLIETILENA KETUMPATAN RENDAH BERPENGISI SERBUK KULIT

#### TELUR YANG TERUBAHSUAI SECARA KIMIA

ABSTRAK

Komposit polietilena ketumpatan rendah berpengisi serbuk kulit telur (LDPE/ESP) telah dikaji. Komposit disediakan menggunakan pencampur bilah-Z pada suhu 180°C dengan menggunakan kelajuan putaran 50 rpm selama 6 minit. Sampel komposit ditekan pada acuan menggunakan penekan hidraulik panas. Penekanan panas melibatkan prapemanasan pada suhu 180°C selama 4 minit diikuti oleh penekanan selama 2 min pada suhu yang sama dan penyejukan bertekanan selama 2 minit. Didapati bahawa kekuatan tensil dan pemanjangan pada takat putus komposit menurun bila pembebanan pengisi bertambah. Modulus Young untuk komposit bertambah bila pembebanan pengisi bertambah. Komposit kemudiannya diubahsuai menggunakan asid isofetalik (LDPE/ESP<sub>1</sub>), etilena diamina-co-asid isofetalik (LDPE/ESP<sub>M</sub>) dan polietilena-graf-maleic anhydride (LDPE/ESP<sub>PEMAH</sub>). Di dapati bahawa rekatan antara muka telah meningkatkan kekuatan tensil dan rintangan penyerapan air untuk komposit LDPE/serbuk kulit telur terubahsuai berbanding komposit LDPE/serbuk kulit telur yang tidak diubahsuai (LDPE/ESP).la juga meningkatkan kestabilan terma dan peratusan penghabluran komposit. Rekatan antara muka yang lebih baik di antara kulit telur dan LDPE telah menyebabkan peningkatan sifatsifat mekanikal komposit LDPE/serbuk kulit telur terubahsuai seperti yang dilihat pada permukaan patah tensil menggunakan SEM. Thister

#### EFFECT OF CHEMICAL MODIFICATION OF EGGSHELLS POWDER FILLED LOW

#### **DENSITY POLYETYLENE COMPOSITES**

ABSTRACT

Eggshells powder filled low density polyethylene (LDPE/ESP) composites were studied. The composites were prepared by using Z-blade mixer at 180°C using a rotor speed of 50 rpm for 6 minutes. Sample of composites were compression moulded, in an electrically heated Hydraulic press. Hot press procedures involved preheating at  $180^{\circ}$ C for 4 min followed by compression for 2 min at the same temperature and subsequent cooling under pressure for 2 min. It was found that the tensile strength and elongation at break for the composites decreased with the increasing filler loading. Young's modulus for the composites increases with the increasing filler loading. Then the composites were modified with isophthalic acid (LDPE/ESP<sub>1</sub>), ethylene diamine-co-isophthalic acid (LSPE/ESP<sub>M</sub>) and polyethylene-grafted-maleic anhydride (LDPE/ESP<sub>PEMAH</sub>). It was found that improvement in interfacial adhesion has enhanced the tensile strength and water absorption resistance of LDPE/modified eggshells powder composites compare to LDPE/unmodified eggshells powder composites (LDPE/ESP). Its also increased thermal stability and percent of crystallinity of composites. Better interfacial adhesion between LDPE and eggshells powder are responsible for the improvement of mechanical properties of LDPE/modified eggshells powder composites, as evident by scanning electron microscopy (SEM) on tensile fracture surface of the composites. Thister

#### **CHAPTER 1**

INTRODUCTION initial copyright

### 1.1 Research Background

Polymers play an important role in our society. Over the past few decades, polymers have replaced many conventional materials, such as metal and wood, in many applications. This is due to the advantages of polymers over conventional materials (Kuo, Wang, Chen, Hsueh, & Tsai, 2009). Polymer become as important materials that can suited with many application in everyday life. It's was use in house ware to the components of the space shuttle. Polymer industry has the large economic advantage over synthesizing new polymeric materials to fulfill the materials need. The currently available commercial materials cannot satisfy the growing need for new advanced material. Many researchers try to improve the materials properties to fulfill the requirement for application in the new era of technologies.

Currently, research to enhance polymer properties with addition of filler to form composite materials has increased and widely practised in industry. Composites represent an important class of engineering materials (Gonzalez, Albano, Ichazo, & Diaz, 2002). It's used in various applications as decks and docks, packaging film, pipes, tubes, window frames or, sporting goods, office equipment and also as materials in the automobile industry and ect. (Suwanprateeb, 2000). Composite materials are those that are formed by the combination of two or more materials to achieve properties that are superior to those of its constituents (Yeh, Feng, Sun, Hsun, & Hsiao, 2003). Polymers filled with solid particulate or fibrous fillers of organic and inorganic nature are classified as polymeric composite materials. Polymer composite consists of a polymer resin as the matrix, with filler as the reinforcement medium (Callister, 2003).

Fillers often increase the performance of polymeric products. The degree of improvement on the judicious choice of filler, particle size and shape, the fraction of filler, and the surface treatment promoting interaction between the polymer matrix and filler (Yeh & Jyun-Jye, 1999). The addition of fillers to polymers is fast and cheap methods to modify the properties of the base materials. The right combination matrix and filler can result in new composite materials with enhanced properties. This reinforcing effect is primarily due to hydrodynamic interaction between the polymer and filler surfaces (Shokri & Bakhshandeh, 2006). It has long been known that the incorporation of filler into polymer matrices lead to a significant improvement in the physical, mechanical and electrical properties of the material is one of the most important and popular methods of production of plastics, rubbers, coatings, adhesives, etc., which must possess the necessary mechanical and physical properties for any given practical application.

Considerable interests have been generated in the manufacture of thermoplastic composites due to their unique properties, such as good mechanical properties and thermal stability and reduce product cost (Kwon et al., 2002; Luyt, Molefi, & Krump, 2006). The introduction of particulate mineral fillers into a thermoplastic polymer can improve some mechanical properties such as Young's modulus, but it effects some others

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properties, like impact strength, adversely. Due to the combination of more than one material, the properties of composites are influence by many factors such as filler characteristic, filler content, interfacial adhesion, etc. (Liang, 2006; Osman, Atallah, & Suter, 2004). This can cause the behaviour of filled polymer to be more complex and different from its unfilled counterpart (Suwanprateeb, 2000). A variety of inorganic and organic reinforcing fillers may be incorporated into thermoplastic in order to improve specific properties or reduces cost (Ferreira, Errajhi, & Richardson, 2006). The addition of inorganic filler to polymers has received considerable attention lately (Fu, Feng, Lauke, & Mai, 2008; Jancar, 1999; Kwon et al., 2002; Luyt & Geethamma, 2007; Mareri, Bastide, Binda, & Crespy, 1998; Mittal, 2007; Osman et al., 2004; Pinchuk et al., 2000; Siti Rohana, Salmah, & Kamarudin, 2008).

Polyolefin's has been widely used owing to their high performance such as a high modulus, high tensile stiffness, high chemical resistance, and low cost for processing (Miyagawa, Tokumitsu, Tanaka, & Nitta, 2007). Polyethylene is used extensively in many fields, including agricultural, automotive and food-packing films. Polyethylene is commonly used in the polymer industry to its abrasion resistance, flexibility, excellent electric insulation properties, low toxicity and easy processing (Bellayer, Tavard, Duquesne, Piechaczyk, & Bourbigot, 2009). Low density polyethylenes (LDPE) are among the most common used plastics and often used as blends for balanced mechanical properties and process ability (Fu, Men, & Strobl, 2003; Huang, Roan, Kuo, & Lu, 2005; Pospisil, Forteln, Michalkova, Krulis, & Slouf, 2005; Yeh et al., 2003). LDPE is usually filled with starch, calcium carbonate, wood flour and others filler to lower the price and improve properties or provide reinforcement (Garg & Jana, 2007).

Calcium carbonate is one of the most widely used filler or extender pigment in the plastic industry. Calcium carbonate has been extensively employed as filler in polymer composites, because of its several remarkable benefits such as abundant raw material

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