



**Effect of chemical modification of eggshells powder  
filled low density polyethylene composites**

by

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A thesis submitted in fulfillment of the requirements for the degree of  
Master of Science (Materials Engineering)

**School of Materials Engineering  
UNIVERSITI MALAYSIA PERLIS**

**2010**

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## APPROVAL AND DECLARATION SHEET

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## LIST OS SYMBOLS, ABBREVIATIONS OR NUMENCLATURE

$\mu\text{m}$	Micron meter
ABS	Acrylonitrile butadiene styrene
$\text{Al}_2\text{O}_3$	Aluminium oxide
ASTM	American society for testing and materials
$\text{C}_2\text{H}_4 (\text{NH}_2)_2$	Ethylene diamine
$\text{C}_2\text{H}_4$	Ethylene
$\text{C}_6\text{H}_4 (\text{COOH})_2$	Isophthalic acid
$\text{CaCO}_3$	Calcium carbonate
$\text{CaO}$	Calcium oxide
Cl	Chlorine
cm	Centimeter
$\text{Cr}_2\text{O}_3$	Chromium oxide
$\text{CuO}$	Copper oxide
DMA	Dynamic mechanical analyzer
DSC	Differential scanning calorimetry
ES	Eggshells
ESCR	Environmental stress crack resistance
ESP	Eggshells powder
FDT	Final decomposition temperature ( $^{\circ}\text{C}$ )
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	Minute
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	Polyethylene
PEMAH	Polyethylene-grafted-maleic anhydride
PP	Polypropylene
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
$T_g$	Glass transition temperature
TGA	Thermogravimetry analysis
$T_m$	Melting temperature
UHMWPE	Ultra high molecular weight polyethylene
ULDPE	Ultra low density polyethylene
UTM	Universal testing machine
VLDPE	Very low density polyethylene
w t%	Weight percent
Wd	Original dry weight
WN	Weight after exposure
$\Delta H_f$	Enthalpy of fusion of the composite

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**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>I</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). Ia juga meningkatkan kestabilan terma dan peratusan penghabluran komposit. Rekatan antara muka yang lebih baik di antara kulit telur dan LDPE telah menyebabkan peningkatan sifat-sifat mekanikal komposit LDPE/serbuk kulit telur terubahsuai seperti yang dilihat pada permukaan patah tensil menggunakan SEM.

# EFFECT OF CHEMICAL MODIFICATION OF EGGHELLS POWDER FILLED LOW DENSITY POLYETHYLENE 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°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>I</sub>), ethylene diamine-co-isophthalic acid (LDPE/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.*

## CHAPTER 1

### INTRODUCTION

#### 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 polymer composites. Filling or reinforcement of polymers to enhance some 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

properties, like impact strength, adversely. Due to the combination of more than one material, the properties of composites are influenced 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 reduce 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