

MECHANICAL PROPERTIES OF RICE HUSK FILLED IMPACT MODIFIED
UNPLASTICISED POLY(VINYL CHLORIDE) COMPOSITE

MAZATUSZIHA BINTI AHMAD

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Specially for my beloved parents'

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ABSTRACT

Rice husk (RH) is one of the natural fillers that offer a number of advantages over inorganic fillers since they are biodegradable, low cost, recyclable and renewable. This study investigates the performance of RH as filler for unplasticised poly(vinyl chloride) (PVC-U) composites. In the sample preparation, composites with different RH loadings varied from 10 to 40 phr were prepared using two-roll mill at temperature 165 °C before being hot pressed at temperature 185 °C. Tensile, flexural and izod impact test were conducted in order to investigate the mechanical properties of the composites. Incorporation of RH fillers from 10 to 40 phr has resulted in the increased of flexural modulus indicating an improvement in stiffness. The effects of acrylic impact modifier and LICA 12 coupling agent on the mechanical properties of PVC-U composites were investigated. The acrylic impact modifiers were found to be effective in enhancing the impact strength at all levels of RH content. Effectiveness of the impact modifier in enhancing the impact strength decreased with increasing RH content. LICA 12 was found to be the most effective in increasing impact strength at 20 phr RH loading. The processability of RH filled PVC-U composites was studied by using Brabender Plasticorder. It was found that incorporation of RH has resulted in decreasing the fusion time of the PVC compounds while the heat distortion temperature (HDT) increased at all RH loadings. The degradation temperature ($T_{10\%}$) decreased with increasing RH content. The percentage of water absorption increased slightly with increasing RH content and treated samples exhibited lower percentage of water absorption compared to the untreated samples. The optimum composition which gives balance of properties based on stiffness and toughness of PVC-U composites is PVC-U at 8 phr of acrylic impact modifier and 20 phr of RH treated with LICA 12.

ABSTRAK

Sekam padi merupakan pengisi semulajadi yang mempunyai banyak kelebihan berbanding pengisi tidak organik kerana ia boleh terbiodegradasi, mempunyai kos yang rendah, boleh dikitar semula dan merupakan bahan semulajadi. Penyelidikan ini mengkaji kebolehan sekam padi sebagai pengisi alternatif terhadap komposit pol(ivinil klorida) tidak terplastik (PVC-U). Dalam penyediaan sampel, komposit dengan kandungan sekam padi dari 10 hingga 40 phr disediakan menggunakan penggiling dua pada suhu 165 °C sebelum pengacuan mampatan dilakukan pada suhu 185 °C. Ujian regangan, lenturan dan hentaman Izod dilakukan untuk mengkaji sifat-sifat mekanikal komposit. Kajian menunjukkan, penambahan sekam padi dari 10 hingga 40 phr meningkatkan modulus lenturan disebabkan kekakuan bahan meningkat. Kesan pengubahsuaian hentaman akrilik dan agen perekatan LICA 12 terhadap sifat-sifat mekanikal komposit PVC-U dikaji. Pengubahsuaian hentaman akrilik didapati berkesan meningkatkan kekuatan hentaman pada setiap kandungan sekam padi. Keberkesanan pengubahsuaian hentaman dalam meningkatkan kekuatan hentaman menurun dengan peningkatan kandungan sekam padi. LICA 12 amat berkesan meningkatkan kekuatan hentaman pada kandungan sekam padi 20 phr. Kebolehprosesan komposit berpengisi sekam padi dikaji menggunakan Brabender Plasticorder. Penambahan sekam padi didapati menyebabkan penurunan masa peleburan manakala suhu pembengkokkan haba (HDT) meningkat pada setiap kandungan sekam padi. Suhu degradasi komposit menurun dengan peningkatan kandungan sekam padi. Peratus penyerapan air meningkat sedikit dengan peningkatan kandungan sekam padi dan sampel terawat LICA 12 menunjukkan peratusan yang rendah berbanding sample tidak terawat. Formulasi optimum yang memberikan kombinasi kekuatan hentaman dan kekakuan yang seimbang adalah komposit yang mengandungi 8 phr pengubahsuaian hentaman akrilik dan 20 phr sekam padi yang dirawat dengan LICA 12.

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LIST OF ABBREVIATIONS AND SYMBOLS

A	-	Constant related to the Einstein coefficient
ABS	-	Acrylonitrile butadiene styrene
APE	-	Aminopropyl triethoxysilane
ASTM	-	American Standard of Testing Materials
b	-	Mean width of the specimens (m)
B	-	Related to the relative modulus of filler and polymer,
BRHA	-	Black rice husk ash
CaCO ₃	-	Calcium carbonate
CO ₂	-	Carbon dioxide
CPE	-	Chlorinated polyethylene
d	-	Mean thickness of the specimens (m)
DSC	-	Differential scanning calorimetry
DTG	-	Differential thermogravimetric
EFB	-	Empty fruit bunch
EVA	-	Ethylene/vinyl acetate
FTIR	-	Fourier Transform Infra Red Spectroscopy
G _f	-	Moduli of filler
G _p	-	Moduli of polymer matrix
HCl	-	Hydrogen chloride
HDPE	-	High density polyethylene
HDT	-	Heat Deflection Temperature
H ₂ O	-	Water
KBr	-	Potassium bromide
L	-	Span between the centers of support (m)

M	-	Tetravalent base metal (Ti, Zr and Si)
MACR	-	Butadiene-modified acrylic
MBS	-	Methacrylate butadiene styrene
M_t	-	Water content at any time t
NPDE	-	Non predefined elastomers
OH	-	Hydroxyl
OPE	-	Oxidised polyethylene
PDE	-	Predefined elastomers
PP	-	Polypropylene
phr	-	Per hundred resin
PVC	-	Poly(vinyl chloride)
PVC-U	-	Unplasticised poly(vinyl chloride)
R	-	Organofunctional group
RH	-	Rice husk
RHA	-	Rice husk ash
rpm	-	Rotational per minute
S	-	Increment in deflection
SEM	-	Scanning Electron Microscopy
TG	-	Thermogravimetric
TGA	-	Thermogravimetry analysis
T_g	-	Glass transition temperature
T_m	-	Melting point
TiO_2	-	Titanium dioxide
UV	-	Ultraviolet
VC	-	Vinyl chloride
VCM	-	Vinyl chloride monomer
W	-	Ultimate failure load (N)
WRHA	-	White rice husk ash
W_d	-	Weight after drying
W_o	-	Original weight
wt	-	Weight percent

W	-	Increment in load (N)
X	-	Hydrolysable group
E_R	-	Relative modulus (of composite to polymer)
ϕ	-	Volume fraction of filler
ψ	-	Reduced concentration term
ϕ_m	-	Maximum packing fraction
ν_p	-	Poisson's ratio.
μm	-	Micrometer

CHAPTER 1

INTRODUCTION

1.1 Introduction

Fillers are defined as materials that are added to the formulation to lower the compound cost. This concept of cost reduction by use of fillers has been known throughout the ages. Very significant advances have been made in this area in terms of fine-particle size technology, tailoring particle morphology, beneficiation of natural materials to attain high purity, surface treatments for improved matrix compatibility, and the development of coupling agents to achieve polymer-to-filler bonding for improved mechanical properties. By the appropriate selection and optimization of such materials, not only the economics but other properties such as processing and mechanical behaviour can be improved. The growing interest in environmental friendly materials has produced re-evaluation of organic materials as fillers in plastics. Biodegradable and compostable products, especially those made of renewable materials from the disposable agrowastes, are essential for the applications of environmental materials. The organic materials which are abundantly available in quantity are generally less expensive than the resins with which they are compounded, and this is a

most important reasons for their use. As the field of plastics developed and expanded, a variety of naturally occurring materials were explored such as wood flour, shell fibres, and empty fruit bunch (EFB). This natural fillers consisting mostly of cellulose, lignin and also hemicellulose.

Utilization of EFB as fillers has been reported by Aznizam and Azman Hassan (2002). They investigated on the impact properties of empty fruit bunch (EFB) impact modified unplasticised polyvinyl chloride (PVC-U) composites. The results showed that by incorporation of EFB into unmodified PVC-U and modified PVC-U has resulted in the reduction in the impact strength with increasing EFB content. The effectiveness of impact modifier decreased with increasing filler loadings. They also observed that the unfilled PVC-U samples changed from brittle to ductile mode with increasing impact modifier concentration.

Besides that, other natural filler which has attracted many researchers is rice husk (RH). Interest in the use of entire rice husk for the manufacture of composite products is growing up through the year. Rice husk is a by-product of the rice milling process, and there is abundance of rice husk in Malaysia. However, the applications of this material are limited. These include as fuel in heat generation for drying rice, used in making cement, and used as a fertilizer in agriculture. The rest is burned or used for landfilling. Therefore, more efficient utilization of rice husk is urgently needed. One of the efforts is to produce value-added products such as composite materials from this important bio-resources. Utilization of rice husk offers some economical and environmental advantages too. There has been considerable effort and interest in the addition of rice husk to thermoplastic. Many papers have been published on the study of rice husk ash (RHA) and RH as fillers in thermoplastic composites (Ahmad Fuad *et al.* 1995, Hattotuwa, *et al.* 2002, Costa *et al.* 2000 and 2002, Hanafi Ismail, *et al.* 1999, Siriwardena, *et al.* 2002, Visconte *et al.* 2003; Sae-Oui *et al.* 2002; and Hanafi Ismail *et al.* 2002). Most of these study focused on the mechanical properties of the composites.

They revealed that the flexural modulus increased with filler content while tensile strength, elongation at break and izod impact strength showed a decrease.

Sivaneswaran (2002) investigated the effect of RHA fillers on mechanical properties of ABS impact modified PVC-U. In this study, ABS was used as impact modifier. He found that the flexural modulus increased as the filler content increased from 0 to 40 phr. However, the impact strength decreased around 40% with the similar filler content. The ABS impact modifier and coupling agents used has improved the impact strength. The formulation containing 8 phr ABS and 40 phr RHA treated with LICA 12, as coupling agent was found to be the most effective formulation in terms of flexural modulus, impact strength and cost. This study has provided valuable background information on the use of rice husk for making lignocellulosic fillers-PVC-U composites and prompt us to investigate the performance of rice husk as new filler in thermoplastic composites, especially for polyvinyl chloride (PVC).

In this study, PVC was used as a base polymer as it offers certain advantages. As the second largest volume plastic used worldwide, PVC plays an important role in the plastic industry (Ma Wenguang *et al.* 1996). PVC-U is a tough and durable material with many applications where its basic properties effectively meet the demands of service and use (Calvert *et al.* 1991). Major application for PVC-U is successfully in extrusion products such as pipe, gutters, conduit, sheet and a wide range of complicated profiles such as window frames.

The usefulness of PVC-U can be increased by physically blending various modifiers with the polymer prior to use for plastics objects. These additives such as lubricants, impact modifier, processing aids, pigments and stabilizer are incorporated to modify the service properties of the material and these will in turn influence the processing behaviour (Vinyls Group ICI Petrochemicals, 1981). PVC-U window profile

compositions contain a number of components, and the formulations evolve as new materials are developed. Such modifiers, in the context of weatherable compositions suitable for window profiles, include chlorinated polyethylene (CPE), ethylene/vinyl acetate (EVA) copolymers, and polyacrylate rubbers. It is usual to incorporate a rubbery impact modifier into the PVC to improve its robustness in order to use over a wide temperature range. The addition of impact modifiers extends the limits of ductility usually associated with conventional, rigid PVC compounds (Calvert *et al.* 1991). Thus, it gives more impact efficient and better properties to meet the requirements of extruded profiles.

On this account, the research was focused on the effect of filler loading, impact modifier content, and coupling agent on the mechanical properties of RH filled PVC-U composites, besides investigating the effect of accelerated weathering condition on the mechanical properties of PVC-U composites.

1.2 Problem Statement

One of the most important aspects in the materials development of engineering thermoplastics is to achieve a good combination of properties and processability at a moderate cost. As far as mechanical properties are concerned, the main target is to strike a balance of stiffness, strength and toughness.

So far, not much study has been reported on RH filled impact modified PVC-U composites. Therefore, in this research where RH was used as a filler in PVC-U to achieve good combination of mechanical properties and processability, several factors

that influence and affects the properties of the composites need to be considered. Thus the optimum formulation of the composite was investigated. The questions that need to be answered in this area of research are:

- i. What is the effect of RH content on the mechanical, weathering, thermal stability and water absorption properties of unmodified and impact modified PVC-U composites?
- ii. What is the effect of acrylic impact modifier content on the mechanical, weathering and water absorption properties of RH filled PVC-U composites?
- iii. What is the effect of coupling agent on the mechanical and water absorption properties of RH filled -acrylic impact modified PVC-U composites?
- iv. What is the effect of RH and acrylic impact modifier content on the fusion behaviour (torque and fusion time) of the PVC-U composites?

1.3 Objectives of the Study

The objective of this study is to investigate the mechanical, water absorption and weathering properties of RH filled-impact modified PVC-U composites. The objective can be further divided as follows:

- i. To study the effect of RH content on the mechanical, weathering and water absorption properties of acrylic modified PVC-U composites

- ii. To study the effect of acrylic impact modifier on the mechanical, weathering and water absorption properties of RH filled-acrylic modified PVC-U composites.
- iii. To study the effect of coupling agent on the mechanical and water absorption properties of RH filled acrylic impact modified PVC-U composites.
- iv. To study the effect of RH content and acrylic impact modifier on fusion behaviour (torque and fusion time), and thermal stability of the RH filled PVC-U composites.

1.4 Significance of the Study

This research developed a PVC-U composite formulation which has a good mechanical properties based on stiffness and toughness, water absorption and weathering properties. Therefore, the use of thermoplastic composite for the development of exterior building applications such as window profile, siding and cladding structure from PVC-U composites at a premium cost, high impact strength and good weathering performance can be achieved.

1.5 Scopes of the Study

- i. The samples preparation involved the following stages:
 - (a) Dry blending
 - (b) Two roll milling
 - (c) Compression moulding.
- ii. The sizes of fillers of $\leq 75\mu\text{m}$, PVC-U and acrylic impact modifier were used in this research to study the effect on mechanical properties of RH filled acrylic impact modified PVC-U composites. The filler loading was at 10, 20, 30 and 40 phr.
- iii. Flexural test, tensile test and izod impact test were carried out to determine the mechanical properties of the composites.
- iv. Water absorption and accelerated weathering test were carried out to study the effect of RH content on the mechanical properties of PVC-U composites.
- v. SEM (Scanning Electron Microscopy) was used to analyze microstructure and morphology of the composites.
- vi. Brabender plasticorder was used to study the fusion torque and time of unfilled impact modified PVC-U and RH filled unmodified PVC-U composites.
- vii. Fourier Transform Infra Red Spectroscopy (FTIR) analysis was used to study the effect of weathering on the degradation of PVC-U composites.
- viii. Thermogravimetry analysis (TGA) was used to study the thermal stability of the PVC-U composites.

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