MECHANICAL, THERMAL AND PROCESSING PROPERTIES OF OIL PALM EMPTY FRUIT BUNCH-FILLED IMPACT MODIFIED UNPLASTICISED POLY (VINYL CHLORIDE) COMPOSITES

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ABSTRACT

The main objective of this study is to investigate the effects of EFB filler and two types of impact modifiers on mechanical and thermal properties, and processability of PVC-U composites. Two methods were used to prepare the samples, i.e., compression moulding and single screw extrusion. A torque rheometer was used to determine the fusion characteristics of PVC. EFB increased the flexural modulus of 22 % but decreased the impact strength and flexural strength of 20 % and 18 % of PVC, respectively, as the filler contents increased. Increasing the impact modifiers contents, on the other hand, improved the impact strength but slightly decreased the flexural modulus and flexural strength. Acrylic impact modifier performed better than CPE in producing composites with balanced properties of toughness and stiffness. The use of Prosil 9234 coupling agent for filler surface treatment proved to be more reliable compared to NZ 44 in providing better filler-matrix interaction. The TGA study showed that both the EFB filler and acrylic impact modifier decreased the PVC thermal stability. Oil residues in the EFB did not influence the mechanical properties of PVC-U composites. The fusion time also decreased significantly with the addition of acrylic. Both the temperature and screw speed significantly influenced the fusion level of PVC, hence the mechanical properties of the extrudates. Overall, the study showed that EFB has the potential to be used as filler for PVC-U composites.

ABSTRAK

Objektif utama kajian ini ialah untuk menyelidik kesan pengisi EFB dan pengubahsuai hentaman terhadap sifat mekanikal, terma dan kebolehanproses komposit PVC-U. Dua kaedah telah digunakan untuk penyediaan sampel, iaitu pengacuan mampatan dan penyemperitan berskru tunggal. Reometer tork telah digunakan untuk menentukan ciri-ciri pelakuran PVC. EFB telah meningkatkan modulus lenturan sebanyak 22 % tetapi telah mengurangkan kekuatan hentaman PVC sebanyak 20 % dan kekuatan lenturan sebanyak 18 % apabila meningkatnya kandungan pengisi. Peningkatan kandungan pengubahsuai hentaman sebaliknya telah meningkatkan kekuatan hentaman tetapi mengurangkan sedikit modulus lenturan dan kekuatan lenturan. Pengubahsuai hentaman akrilik telah menunjukkan prestasi yang lebih baik dalam menghasilkan komposit bersifat kekukuhan dan kekakuan yang seimbang berbanding CPE. Penggunaan agen gandingan Prosil 9234 untuk rawatan permukaan pengisi telah menghasilkan interaksi pengisi-matrik yang lebih baik berbanding dengan NZ 44. Kajian TGA menunjukkan bahawa pengisi EFB dan pengubahsuai hentaman akrilik telah mengurangkan kestabilan terma PVC. Sisa minyak pada EFB didapati kurang mempengaruhi sifat-sifat mekanikal komposit PVC-U. Masa pelakuran juga telah menurun dengan penambahan kandungan akrilik. Suhu dan laju skru telah mempengaruhi tahap lakuran PVC begitu juga sifat-sifat mekanikal ekstrudat. Keseluruhannya kajian menunjukkan bahawa EFB berpotensi digunakan sebagai pengisi di dalam komposit PVC.

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LIST OF SYMBOLS

b	-	width
d	-	thickness
CPE	-	chlorinated polyethylene
EFB	-	oil palm empty fruit bunch
E_c	-	flexural modulus of composite
E_p	-	flexural modulus of polymer
G_f	-	shear modulus of filler
G_p	-	shear modulus of polymer
HDT	-	heat deflection temperature
k	-	Boltzmann's constant
L	-	span
M_t	-	water absorption at time t
NPCC	-	nano-precipitated calcium carbonate
NZ 44	-	zirconate based coupling agent
Prosil 9234	-	silane based coupling agent
PVC-U	-	unplasticised poly (vinyl chloride)
S	-	sample
T_g	-	glass transition temperature
TG	-	thermogravimetric curve
DTG	-	differential thermogravimetric curve
v	-	velocity
V_f	-	volume of filler
V_p	-	volume pyknometer
V_w	-	volume of water
W	-	failure load

$W_{additives}$	-	weight fraction of additives
W_d	-	dry weight
W_f	-	weight fraction of filler
W_{pvc}	-	weight fraction of poly (vinyl chloride)
W_w	-	weight after immersed into water
ΔH	-	activation energy
ΔS	-	deflection increment
ΔW	-	load increment
ε	-	strain rate
ε_c	-	elongation at break of composite
ε_p	-	elongation at break of polymer
ϕ_f	-	volume fraction of filler
ϕ_m	-	maximum packing fraction
γ	-	stress concentration factor
ν	-	activation volume
ν_p	-	Poisson's ratio of polymer
$\rho_{additives}$	-	density of additives
ρ_c	-	density of composite
ρ_f	-	density of filler
ρ_w	-	density of water
σ_c	-	strength of composite
σ_p	-	strength of polymer
σ_y	-	yield stress
ψ	-	reduced concentration term

CHAPTER 1

INTRODUCTION

1.1 Overview

Poly (vinyl chloride) has grown into one of the major thermoplastics material since it was first produced in the 1930's. More than one-half of the PVC produced is used in the building industry (Mengeloglu, 2001). Because of its blending versatility (ability to be modified through the introduction of various additives) and cost-effectiveness, PVC is one of the most worldwide used thermoplastics and it plays an important role in the plastic industry as a major component of composite manufacturing (Wenguang and La Mantia, 1996).

Cost reduction is achieved when fillers are incorporated into the PVC matrix. In addition to reducing cost, other typical reasons for using fillers are stiffness enhancement and thermal properties alteration of vinyl products. Currently, the most important fillers for PVC are inorganic materials such as glass fibre, calcium carbonate, agalmatolite, talc (Saad *et al.*, 1999; Wee, 2001; Pedro *et al.*, 2001; Xie *et al.*, 2001). These fillers are high-density materials (Mengeloglu, 2001). While they offer wide property changes in the composites, their use is not cost effective on a volumetric basis (Titow and Lanham, 1975). In addition, these inorganic fillers cause wear of equipment during processing. Therefore the use of natural fillers such as wood has been suggested as an alternative to mineral materials (Nabi Saheb and Jog, 1999).

Interest in the use of natural fillers has grown during the last decade and most of the natural fibres used in thermoplastics composites are derived from wood. The use of natural fillers in USA plastic industry in 2000 was estimated 0.4 billion pounds or 7 % (Gurram *et al.*, 2002). In fact, industry experts believe that the demand for natural fillers for plastics composite applications will grow at least six-fold in the next 5 to 7 years (Stokke, 2002). The rationale behind this interest is that the use of natural fibres offers several benefits, including lower cost, less abrasiveness to equipment, high specific properties such as higher specific modulus (modulus/specific gravity), renewable nature and biodegradability (Nabi Saheb and Jog, 1999).

Even though natural fibres offer several advantages, certain drawbacks such as incompatibility of fibre with the thermoplastics matrix, the tendency to form aggregates, and poor resistance to moisture greatly reduce the potential of these fibres to be used as reinforcement. All these drawbacks, especially incompatibility, lead to low fibre-matrix interfacial bonding strength and poor wetting of the fibres by the matrix resin, which can cause reduction in mechanical performance of the composites. Therefore, surface modification by the use of coupling agents is commonly suggested as solution to overcome the drawbacks of lowered strength property. These chemical reagents convert the hydrophilic surface of cellulosic fibres to hydrophobic one. As a result, the fibre-fibre hydrogen bonds is reduced and the adhesion is also improved via the mechanism such as formation of chemical bonds between treated fibres and polymer matrix (Xanthos, 1983; Raj *et al.*, 1989, 1989, 1990; Maldas and Kokta, 1990,1990,1991; Myres *et al.*, 1991; Maiti and Subbarao, 1991; Ahmad Fuad *et al.*, 1994,1997; Sivaneswaran, 2002; Baiardo *et al.*, 2002; Fabbri *et al.*, 2004).

One of the natural fillers that have been tried was oil palm empty fruit bunch (EFB). EFB is one of the natural fillers obtained from oil palm. The incorporation of

EFB as a reinforcing component in the polymer composites has received much attention (Sareekala *et al*., 1996/97; Rozman *et al.*, 1998, 2001, 2001; Sarani *et al.*, 2001; Abdul Khalil *et al*., 2001). Most of the research works on EFB so far dealt with polymers such as polypropylene, polyester, polystyrene and phenol-formaldehyde resin. The researchers focused on the effect of filler loading, filler particle size distributions and filler treatment on the mechanical properties of the composites. Like other natural fillers, many issues have been identified in the processing of the composites. These include low compatibility between EFB fibre and polymer matrix, higher moisture intake, poor distribution of the fibre in the matrix and effect of oil residue on mechanical properties.

Although EFB has been useful as fillers in various polymers mentioned above, the use of EFB filler in PVC has not been studied extensively. Most of the researchers have used wood flour, wood sawdust and rice husk ash as fillers in PVC matrix (Matuana *et al*., 1998, 2001; Sivaneswaran, 2002; Sombatsompop *et al*., 2003). The use of these fibres in the PVC matrix produces products that are more brittle than neat PVC. The incorporation of the natural fibres alters the ductile mode of failure of the matrix. The brittleness of PVC/wood fibre composite compared to the unfilled PVC may prevent this emerging class of materials from capturing their full market potential in applications such as door and window frames (Mengelollu, 2001).

One of the most important aspects in the development of PVC compound by using fillers 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 (Mohd Ishak *et al*., 2000). The incorporation of filler in thermoplastic such as PVC is associated with an enhancement in stiffness and strength but a decrease in toughness. The incorporation of impact modifiers in PVC, on the other hand will result in a significant improvement in toughness at the expense of stiffness and strength.

The enhancement of impact properties of PVC-U by introduction of a rubbery phase is a well-known phenomenon which has been widely exploited commercially. Among those which have extensively used are chlorinated polyethylene (CPE) and various modified acrylic. Hiltner *et al.* (1984) reported that CPE modifier formed a network enclosing PVC primary particles in order to develop the desired impact strength whereas Robinovic (1983) found that acrylic modifier formed a distinct dispersed phase in the continuous PVC phase. The dispersed rubber particles of acrylic were independent of the processing time and temperature, whereas the CPE network structure was dependent of those factors.

The studies have concluded that the function of the impact modifiers (whether in the form of discrete phase or network structure) was to increase the toughness of PVC by facilitating the ability of PVC matrix to shear yield and/or craze. This means that the PVC would deform rather than fracture when subjected to a sudden load (Petrich, 1973; Lutz, 1993). Meanwhile, Stevenson (1995) has pointed out that the impact modifiers can also be used to correct the detrimental effects of other additives such as fillers by boosting the impact performance of the material.

1.2 Significance of Study

Based on the previous findings mentioned above, no study has been reported on the use of EFB fibre in the PVC matrix. Most studies involved investigation of the mechanical properties of natural fibre polymer composites in terms of natural fibre content, various treatments of the fibre, whereas very few have investigated processability and thermal stability of the composites. According to Sombatsompop *et al*. (2003), the area for PVC/wood fibre composites are still actively pursued, especially in areas of processability and thermal stability-related mechanical properties of the composites.

With the rising environmental issues in the limelight and the need for new applications of agricultural by-products such as EFB to be more useful in order to minimize industrial waste, therefore agricultural by-products seem attractive enough to be use as reinforcement filler alternative for filled thermoplastics composites. Since oil palm has now become a major cash crop and is cultivated commercially in Malaysia, many million tons of EFB on dry weight basis are produced annually throughout the world. EFB is an agricultural waste, which is left unutilized after the removal of the oil seeds for oil extraction. In order to minimize the abundance of this waste and environmental problems, therefore new applications are urgently required for EFB to be more useful (Sreekala *et al.,* 1996/97). The utilization of this EFB as a reinforcement in polymers has some economical as well as ecological importance.

PVC is basically a rigid thermoplastic. It is inherently ductile because of the polarity and low-temperature molecular relaxation. However, it is brittle at high deformation rate and under concentrated stress, and PVC is known as a notch-sensitive polymer (Wimolmala *et al.,* 2001). Previous studies have also shown that the impact strength reduces drastically with addition of natural fillers (Nabi Saheb and Jog, 1999; Mengeloglu, 2001). Impact modifier is therefore added to increase the ductility and toughness of PVC. Although ductility increases, the impact modifier used is costly and tends to decrease the PVC-U stiffness. In this study, the strategies to use EFB fillers together with impact modifier in PVC-U are deliberately done to develop PVC-U composites with good stiffness without sacrificing both desirable toughness and cost effectiveness.

Therefore, in order to understand the properties of the newly developed of PVC-U composites, the effects of filler and impact modifier contents, and filler treatment on the mechanical and thermal properties, processability and extrusion process on the PVC-U composites are investigated.

1.3 Objectives of Study

The main objective of the study is to produce EFB-filled PVC-U composites. To achieve the objectives, the following stages have been carried out:

(a) To determine the effect of EFB filler, acrylic and CPE content on the impact strength and flexural properties of the compression moulded composites. The influence of EFB filler, acrylic and CPE content on the thermal properties such as heat deflection temperature, thermal degradation temperature and glass transition temperature are also investigated. In addition, composites accelerated weathering and water absorption properties rare also determined.

(b) To study the effect of silane (Prosil 9234) and zirconate (NZ 44) coupling agents on the impact and flexural properties and water absorption of the compression moulded composites. From this study, the most suitable coupling agent is determined.

(c) To determine the effect of EFB filler, acrylic and CPE content on the fusion characteristics of PVC-U compounds. The effect of different processing temperatures on the composites is also studied.

(d) To determine the effect of processing temperatures and screw speeds on the impact, tensile and flexural properties of extrudates. The influence of processing temperatures on fusion level of extrudates is also determined.

1.4 Outline of Thesis

Chapter 1 presents the overview of the previous research works that has been conducted by other researchers who used either EFB fibres as reinforcement or PVC as matrix in their studies. Both the advantages and disadvantages of natural fibres are also reported as well as the role of impact modifier in the PVC. The significance of the study, the objectives of study and the layout of research are presented.

Chapter 2 begins with some definitions and general feature properties of PVC. Several theories on PVC such as impact modification and fusion mechanism, and the function of additives are described. The definitions and categories of composites and the properties of EFB filler are also described. Literature survey on PVC filled composites is also reported, followed by reviews on the application of various coupling agents and the application of EFB as a reinforcement filler or fibre in the thermoplastics composites systems. Several theories that have been used to predict the relative modulus of filled composites are also reviewed.

Chapter 3 mainly describes the effect of filler content, impact modifier content and coupling agents on the impact strength, flexural properties and water absorption of composites. The effect of filler content on the density of composites, the application of models to predict the relative modulus, and the impact strength and flexural properties of weathered composites are also described. The morphological studies on the impact-fractured surface and the presence of functional groups on the samples investigated using Scanning Electron Microscope (SEM) and Fourier Transform Infra-Red (FTIR), respectively, are also reported.

Chapter 4 reports the thermal properties of the composites. A wide array of thermal analysis techniques such as thermogravimetric analyser (TGA), heat deflection temperature analyser (HDT) and differential scanning calorimetry (DSC) are used to investigate the thermal degradation temperature, heat deflection temperature and glass transition temperature, respectively.

Chapter 5 reports on the processability of PVC-U compounds. The effects of EFB filler content, extracted EFB filler, CPE and acrylic impact modifier content and mixing temperature are reported. The effect of extracted EFB filler content on the processability is also investigated as a minor part of this study. FTIR is used to identify the related functional groups present in the extractives and the changes in EFB filler surface, followed by morphological study on the extracted EFB filler surface using SEM.

Chapter 6 reports the effect of screw speed and processing temperature of extrusion on the impact strength, flexural properties, tensile properties and fusion level of the composites. The effect of crosshead speed on the tensile properties is studied to predict the yield stress of extrudates. The fusion level of extrudates at different temperatures is also investigated using DSC.

Chapter 7 presents the overall conclusions and suggestions for further work.

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