UNIVERSITI PUTRA MALAYSIA

EFFECTS OF BANANA PSEUDOSTEM FILLER AND ACRYLIC IMPACT MODIFIER ON THERMO-MECHANICAL PROPERTIES OF UNPLASTISIZED POLYVINYL CHLORIDE COMPOSITES

EDI SYAMS BIN ZAINUDIN

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By

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

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Abstract of the 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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Chairman : Professor Ir. Mohd Sapuan Bin Salit, PhD

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The main objective of this study is to investigate the effects of banana pseudo-stem (BPS) filler and acrylic impact modifier on mechanical and thermal properties of unplastisized poly (vinyl) chloride (UPVC) composites. BPS/UPVC composites with up to 40% by weight filler content were produced using the compression moulding. The use of BPS fibre as filler in the composites contributed to the enhancement of stiffness, but decreased the ductility, tensile and flexural strength of the UPVC composites. It was also revealed that composites with increased impact modulus and hardness could be successfully developed using BPS filler. The thermal stability of acrylic modified and unmodified BPS/UPVC composites has been studied. The results showed that the BPS filler degraded before UPVC matrix and the BPS/UPVC composites are more stable than both components. The thermal stability of acrylic modified BPS/UPVC composites was found to be higher than that of unmodified BPS/UPVC composites. The dynamic mechanical properties

(storage modulus and tan δ) of neat UPVC and BPS filler–UPVC were studied at temperatures ranging from 30 to 140 °C. The gradual addition of 10% filler to the neat UPVC matrix increased the storage modulus of the composites. The highest stiffness has been obtained for 40% BPS filler of BPS/UPVC composites. This could be attributed to the highly restricted movement in the side chain or adjacent atoms in the main chain. The damping properties with the addition of acrylic also decreased with the gradual addition of 10% filler. The glass transition temperature is clearly seen to increase as the filler content increases, which is consistent with the theory that the incorporation of filler has a restricting effect on segmental mobility of the molecular structure. Overall, it can be concluded that BPS, which is agricultural by-product from banana trees, has the potential to be used as filler in UPVC composites as it enhanced the stiffness and reduced the cost of the composites.

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

KESAN-KESAN PENGISI GENTIAN BATANG PISANG DAN PENGUBAHSUAI HENTAMAN AKRILIK TERHADAP SIFAT TERMA-MEKANIK KOMPOSIT POLYVINYL CHLORIDE TIDAK-PLASTIK

Oleh

EDI SYAMS ZAINUDIN

Februari 2009

Pengerusi: Professor Ir. Mohd Sapuan Bin Salit, PhD

Fakulti: Kejuruteraan

Objektif utama kajian ini ialah untuk menyelidik kesan pengisi gentian batang pisang (BPS) dan pengubah suai hentaman akrilik terhadap sifat mekanikal dan terma komposit UPVC. Komposit BPS/UPVC dengan berat sehingga 40% kandungan pengisisi dihasilkan menggunakan pengacuan mampatan. Penggunaan gentian BPS sebagai pengisi bagi komposit ini telah menyumbang kepada penambahbaikan ketegaran komposit UPVC, namun pengurangan nilai berlaku kepada sifat kenyal dan kekuatan tegangan dan lenturannya. Adalah didapati, komposit yang sifat modulus hentaman dan kekerasan yang meningkat berjaya dibangunkan dengan pengisi BPS. Kestabilan terma bagi kedua-dua komposit yang dibaiki dan tidak dibaiki (dengan akrilik) juga telah dikaji. Keputusan menunjukkan bahawa pengisi BPS menjadi meluput (degrade) sebelum matriks UPVC meluput tetapi komposit BPS/UPVC didapati lebih stabil

berbanding kedua-dua komponen jika mereka berasingan. Kestabilan terma untuk komposit yang dibaiki akrilik didapati lebih baik berbanding dengan komposit BPS/UPVC yang tidak dibaiki secara akrilik. Sifat mekanik yang dinamik (modulus storan dan tan δ) dimana UPVC dan BPS pengisi dengan suhu dari 30 to 140 °C. Penambahan 10% pengisi ke dalam UPVC tulen meningkatkan modulus storan komposit. Kekakuan yang paling tinggi dapat dicapai bagi 40% pengisi dalam komposit BPS/UPVC. Ini adalah disebabkan halangan yang tinggi terhadap pergerakan pada rantaian sisi atau atom-atom berdekatan di dalam rantaian utama. Sifat redaman dengan kehadiran akrilik juga menyebabkan berlaku pengurangan dengan setiap penambahan 10% pengisi. Suhu transisi kaca juga jelas nampak pertambahan apabila kandungan pengisi ditambah, yang mana nilai ini konsisten dengan teori bahawa penambahan pengisi mempunyai kesan halangan pada pergerakan 'segmental' dalam struktur molekul. Pada keseluruhannya, dapat disimpulkan bahawa gentian BPS, iaitu sebagai bahan buangan pertanian daripada pokok pisang, mempunyai potensi untuk digunakan sebagai pengisi di dalam komposit UPVC kerana ia menambah sifat kekakuan dan seterusnya menyumbang kepada pengurangan kos penghasilan komposit.

ACKNOWLEDGEMENT

Alhamdulillah, I would like to express my sincere gratefulness and appreciation to my supervisor, Professor Ir. Dr. Mohd Sapuan Salit for his remarkable encouragement and endless support during my study. I am also very grateful to Dr. Khalina Abdan, Dr. Mahmud Tengku Mohamed and Dr. Abdul Maleque, my co-supervisors, for giving so much supports and advices. Special thanks to my research colleagues who provided constructive advice for the completion of this thesis. This include: Riza Wirawan, Firdaus Adam, Zolhilmi, Soo Wen Leong and Afzan Saiful. Special thanks are also due to Mrs. Noraini from Institute of Bioscience, UPM for the help of SEM work. This study will not to be accomplished without finance and therefore I would like to express my appreciation to the Malaysia Government and Universiti Putra Malaysia for the financial support. Finally, I wish to thank to my beloved wife, sons and daughter for their love, support, understanding and patience.

APPROVAL

I certify that an Examination Committee has met on the 11th of February 2009 to conduct the final examination of Edi Syams Zainudin on his Doctor of Philosophy thesis entitled "Thermo-Mechanical Properties of Banana Pseudo-stem Filled Unplasticized Polyvinyl Chloride Composites" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not previously, and is not concurrently, submitted for any other degree at Universiti Putra Malayasia or any other institutions.

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CHAPTER 1

INTRODUCTION

1.1 Overview

Poly (vinyl) chloride commonly abbreviated PVC, is a widely used thermoplastic polymer. In terms of revenue generated, it is one of the most valuable products of the chemical industry. Globally, over 45% of PVC manufactured is used in construction (Anon, 2006). As a building material, PVC is cheap, durable, and easy to assemble. PVC is used in a variety of applications. As a hard plastic, it is used as vinyl siding, magnetic stripe cards, window profiles, pipes and plumbing and conduit fixtures. The material is often used for pipelines in the water and sewer industries because of its inexpensive nature and flexibility. It can be made softer and more flexible by the addition of plasticizers where it is mostly used in clothing and upholstery (Markarian, 2005).

Price reduction is achieved when fillers are incorporated into the PVC matrix due to its versatility. Other typical reasons for using fillers are stiffness enhancement and thermal properties alteration of vinyl products. Some important fillers for PVC studied presently, are inorganic materials such as glass fibre, calcium carbonate, talc (Saad et al., 1999; Xie et al., 2001). These fillers are high-density materials and offer broad property changes in the composites but their use is not cost effective on a volumetric basis (Zhao et al., 2006). In addition, these inorganic fillers cause wear of apparatus during processing. Therefore the use of natural fillers has been suggested as an alternative to mineral materials (Saheb and Jog, 1999).

1.2 Significance of Study

Natural fibres have already established a track record as simple filler material in automobile parts. Natural fibres like sisal, jute, coir, oil palm fibre have all been proved to be good reinforcement in thermoset and thermoplastic matrices (Joseph et al., 1996, Geethamma et al., 1998, Nishinoa et al., 2003). The idea of using natural fibres as reinforcement in composite materials is not a new or recent one. Man has used this idea, since the beginning of our civilization when grass and straw were used to strengthen mud bricks. During the 1970s and 1980s, these cellulose fibres were gradually substituted by newly developed synthetic fibres because of their better performance over conventional reinforcement materials, glass-fibres. The advantages are summarised by Bledzki and Gassan (1999) as :

- Natural fibres are a renewable raw material and that will be available continuously.
- When they are subjected to a combustion process or landfill at the end of their life cycle, the released amount of CO_2 of the fibres is neutral with respect to the assimilated amount during their growth.

- The abrasive nature of natural fibres is much lower compared to that of glass-fibres, which leads to advantages with regard to technical, material recycling or process of composite materials in general.
- The density of natural fibres is much lower compared to glass-fibres. It means there is potential advantage of weight saving.

Then, the use of cellulose fibres has been limited to the production of rope, string, clothing, carpets and other decorative products (Joseph et al., 1999). However, over the past few years, there has been a renewed interest in using these fibres as reinforcement materials to some extent in the plastics industry. This resurgence of interest is due to the increasing cost of plastics, and also because of the environmental aspects of using renewable and biodegradable materials.

1.3 Problem statements

Based on the earlier findings, no study has been reported on the use of banana pseudo-stem (BPS) fibre in the PVC matrix. There were some studies on BPS with other polymer composites as well as PVC with other natural fibre composites were carried out (Kamel, 2004, Abu Bakar et al., 2005, Djidjelli et al., 2007, Zheng et al., 2007, Crespo et al., 2008). Most studies involved investigation of the mechanical properties of natural fibre polymer composites in terms of natural fibre content, various treatments of the fibre; with very few have investigated thermal properties of the composites. Furthermore, because of the highly temperature-dependent mechanical properties of such composites, the application of a method that monitors property changes over a range of temperatures is critical (Kurvilla et al., 1993; Kim et al., 1997; Manikandan Nair et al., 2001; Abu Bakar et al., 2004; Geethamma et al., 2005). With the rising environmental issues and the need for new applications of agricultural by-products such as BPS to be more useful in order to minimize industrial waste, therefore agricultural by-products seem attractive enough to be used as reinforcement filler alternative for filled thermoplastics composites. Since banana trees has now cultivated as the third largest in Malaysia commercially and most of them are for food industries in Malaysia (Anon, 2007), tons of banana pseudo-stem (BPS) fiber on dry weight basis are at large unexploited. BPS is an agricultural waste, which is left unutilized after the removal of the fruits and its leaves. If the properties of BPS fiber composite can be determined, the applications of the composite can be firmed and engineers would be able to develop new product or replace some materials using this material. By determining the applications, the utilization of the BPS fiber is increased and the waste in banana cultivation can be reduced. In order to minimize the abundance of this waste and to reduce environmental problems, therefore new applications are required for BPS to be more useful. Since most polymers are processed at high temperature, thermal stability of natural fibers or fillers is one of the factors to be considered in developing natural fiber composites (Bilba et al., 2007). Therefore, in order to understand the properties of the newly developed of BPS/UPVC composites, the effects of filler loading and impact modifier treatment on the mechanical and thermal properties of the composites are investigated.

1.4 The aim and objectives of study

The aim of the study is to produce and investigate the effect of the BPS filler and acrylic impact modifier on the thermo-mechanical properties of BPS/UPVC composites. To accomplish this, the following objectives have been carried out:

(a) To determine the effect of BPS filler and acrylic content on the tensile, flexural and impact properties of the compression moulded composites.

(b) To determine the effect of BPS filler and acrylic content on the thermal properties such as thermal stability, degradation temperature and glass transition temperature of the composites.

1.5 Scope and limitation of the study

The scope of the present research is to study the perspectives of using lignocellulosic fibrous plant residues as fillers of thermoplastic polymer. Those natural fibres are low-cost by-products, environmental-friendly and practically sustainable raw materials. The natural fibers used in this study were both unmodified and acrylic modified residues from BPS fibre of banana plant. Polymer matrices of unplastisized PVC (UPVC) were used, to prepare composite specimens with different amounts (10-40% by weight) of the BPS fillers by the mixer and compression molding machine. These specimens were tested for their mechanical and thermal properties, and additional assessment was made via measurements of their surface fracture observations by scanning electron microscopy (SEM).

1.6 The outline of the thesis

Chapter 1 presents the overview of the previous studies that has been conducted by other researchers who used either BPS fibres as reinforcement or PVC as matrix in their studies. Briefing on the advantages of natural fibres is also reported as well as the role of impact modifier in the PVC. The significance of the study, problem statement, the objectives of study and the scope of research are presented.

Chapter 2 begins with some definitions and general feature properties of PVC. The definitions and categories of composites and the properties of BPS 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 BPS as a reinforcing agent in the thermoplastics composites systems. Description on the effect of filler content, acrylic modifier content the impact strength, flexural properties and water absorption of composites are reviewed.

Chapter 3 reports on the methodology used in this study from the preparing of the materials, testing procedure and data collecting of the study.

Chapter 4 and 5 reported the mechanical properties results (i.e. tensile, flexural, impact and hardness) extracted from two accepted papers in citation index Journals (proof of is in the appendix -A); (i) E.S. Zainudin, S.M. Sapuan, K. Abdan and M.T.M. Mohamad, The mechanical performance of banana pseudo-stem reinforced unplastisized polyvinyl chloride composites, Polymer Plastics Technology and Engineering (indexed in ‘Science citation index expanded’), accepted, 2008 and (ii) E.S. Zainudin and S.M.

Sapuan, Impact strength and hardness properties of banana pseudo-stem filled unplastisized PVC composites, Multidiscipline Modelling in Materials and Structures (indexed in Scopus), accepted, 2008.

Chapter 6 and 7 reported the thermal properties (i.e. thermal degradation and dynamic mechanical analysis) results extracted from other two accepted papers in citation index Journals i.e. (i) E.S. Zainudin, S.M. Sapuan, K. Abdan and M.T.M. Mohamad, Thermal degradation of banana pseudo-stem fibre reinforced unplastisized polyvinyl chloride composites, Materials and Design (indexed in 'Science citation index expanded'), accepted, 2008 and (ii) E.S Zainudin, S.M Sapuan, K. Abdan and M.T.M.Mohamad, Dynamic mechanical behaviour of banana pseudo-stem filled unplastisized polyvinyl chloride composites, Polymer and Polymer Composites (indexed in 'Science citation index expanded'), accepted, 2008.

Chapter 8 presents the overall conclusions and recommendations for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Natural fiber

Even though natural fillers have several advantages, certain weaknesses such as poor surface adhesion to hydrophobic polymers, the tendency to form aggregates, not suitable for high temperature application, vulnerability to fungal and pest attack and degradation by moisture greatly reduce the potential of these fillers to be used as reinforcement. These drawbacks, especially incompatibility of fillers with the thermoplastics matrix, lead to low fibre-matrix interfacial bonding strength and poor wetting of the fibres by the matrix resin which can lead to reduction in mechanical performance of the composites. Therefore, surface modification by the use of coupling or modifier agents is commonly suggested as solution to overcome the drawbacks of lowered strength property (Geethamma et al., 1998).

One of the natural fillers that have been tried was banana pseudo-stem (BPS) fibre. BPS is one of the natural fillers obtained from banana trunk. The incorporation of BPS as a reinforcing component in the polymer composites has received much attention (Joseph et al., 2002; Idicula et al., 2005; Pothan et al., 2006). Most of the research works on BPS so far dealt with polymers such as, 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. Although BPS has been useful as fillers in various polymers mentioned above, the use of BPS filler in PVC has not been studied extensively. Most of the researchers have used wood flour, wood sawdust, rice husk ash and bagasse as fillers in PVC matrix (Matuana et al., 2001; Hassan et al., 2001; Sombatsompop et al., 2003; Zheng et al., 2007). The utilization 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 panels (Mengeloglu, 2003). One of the most important aspects in the development of PVC compound by using fillers is to achieve a good combination of properties and process-ability at a moderate cost.

Cellulosic fibres like BPS, sisal, palms, bamboo, wood in their natural condition, as well as several waste cellulosic products such as shell flour, wood flour and pulp have been used as reinforcement agents of different thermosetting and thermoplastic resins (Varghese et al., 1994; Mi et al., 1997; George et al, 2001). During the transformation of the raw fibres into cordage, approximately 10% of waste fibres are produced. These waste fibres profitably used in the manufacture of fibre polymer reinforced composites because they posses attractive physical and mechanical properties (Coutinho et al., 1997).

Unlike the traditional engineering fibres, e.g. glass and carbon fibres, along with mineral fillers, these lignocellulosic fibres are able to impart to the composite certain benefits such as: low density; less machine wear than that produced by mineral reinforcements; are readily available from natural sources; biodegradable; minimal health hazards; and a high degree of flexibility (Jayaraman and Bhattacharyya, 2004; Georgopoulos et al., 2005; Goda et al., 2006). The latter is especially true because these fibres, unlike glass fibres will bend rather than fracture during processing. Whole natural fibres undergo some breakage while being intensively mixed with the polymeric matrix, but this is not as notorious as with brittle or mineral fibres (Wambua et al., 2003; Panthapulakkal and Sain, 2007).

Mohanty et al. (2005) classified natural fibres in two broad categories i.e. non-wood fibres and wood fibres. Non-wood fibres are divided into:

- Straw, examples: corn, wheat, and rice straw;
- Bast, examples: kenaf (*Hibiscus cannabicus)*, flax (*Linum usitatissimum*), jute (*Corchorus*) , ramie (*Boehmeria nivea*) , and hemp (*Cannabis sativa*);
- seed/fruit, examples: sisal (*Agave sisalana*), pineapple (*Ananas comosus*) leaf, and henequen (*Agave fourcroydes*) fibre;
- grass fibres, example: bamboo fibre, switch grass (*Panicum virgatum*), and elephant grass (*Erianthus elephantinus*);

Natural fibres in the form of wood flour have also been often used for preparation of natural fibre composites (Yuan et al., 2004; Augier et al., 2007). The characteristic values of natural fibres vary from one fibre to another. Some of the characteristic values are the cellulose content in the fibre, the degree of polymerization of the cellulose and the microfibrils angle fibres. Higher cellulose content, higher degree of polymerization and a lower microfibrillar angle will affect higher tensile strength and modulus. The variations in the characteristic value exhibit the variations in mechanical properties both along the length of an individual fibre and between fibres (Kalaparasad et al., 1997; Jayaraman, 2003; Nechwatal et al., 2003).

Natural fibres also have non-uniformity and variation of dimensions, even between individual plants in the same cultivation. To generate fibres suitable for specific end products, the various types of raw material are separated. Bast or stem fibres, for example, are mainly used in the textile or rope industries because of the length of the fibres (Facca et al., 2006). Bast straw is not separated into single fibres but into fibre bundles, which may contain thousands of single fibres. In contrast with it, wood is usually separated into single fibres or very small fibre bundles suiting the particular needs of the pulp, paper or board industries (Coutts and Warden, 1992). Thus, there are a great number of challenges for selecting fibres in different dimensions and properties (Olesen and Plackett 1999).

Fibre is classified into synthetic and natural. The natural fibre is still in research for being commercialized. Fibre used in the polymeric composite is aimed to add strength to the polymer. Different fibre has different properties in terms of thermal and mechanical for instance. There are many types of natural and synthetic fibres can be used in the composite manufacturing. The examples of natural fibres are BPS, sisal, kenaf, and coconut husk fibre. Synthetic fibres could be glass fibre, carbon and boron fibre.

The fibre which serves as a reinforcement in reinforced plastics may be synthetic or natural. Although glass and other synthetic fibre-reinforced plastics possess high specific strength, their fields of application are very limited because of their inherent higher cost of production. Some of these natural fibres are not only strong and lightweight but also relatively very cheap (Saheb and Jog, 1999, Gurram et al., 2002, Nishinoa et al., 2003). The specific properties of the natural fibre composites were in some cases better than those of glass (Table 2.1). This suggests that natural fibre composites have a potential to replace glass in many applications that do not require very high load bearing capabilities.

Table 2.1. A comparison between natural and glass fibres (Wambua et al., 2003)

	Natural fibres	Glass fibres
Density	Low	Twice that of natural fibres
Cost	Low	Low, but higher than NF
Renewability	Yes	No
Recyclability	Yes	No
Energy consumption	Low	High
Distribution	Wide	Wide
CO2 neutral	Yes	No
Abrasion to machines	No	Yes
Health risk when inhaled	No	Yes
Disposal	Biodegradable	Not biodegradable

Some properties of selected natural fibres and glass fibre are shown in Table 2.2 below. From this table we can see the banana fibre has the highest mechanical properties among others but lower than glass fibre.

Table 2.2. Properties of banana, natural and glass fibers (Biswas et al., 2006)

Fiber	Banana	Coir	Cotton	Glass
Density (kgm^{-3})	1350	1450	1500	2540
Cellulose/lignin (Weight %)	64/5	37-42/42-45	90/-	Nil
Initial Modulus (GPa)	27-32	3-6	1.1	68-96
Ultimate Tensile Strength (MPa)	529-914	106-175	350	827.6-1224
Water absorption (%)	10-11	10-12	7-8	0-3

2.2 Mechanical properties

Generally, the density of natural fibres is much less than that of E-glass fibre (Joshi et al., 2004). Many natural fibres have higher specific modulus compare to E-glass fibres. Hence, there is an opportunity for using the natural fibres to replace the E-glass fibre (Drzal, et al. 2004). The physical properties of the natural fibre composites tested were found to compare favourably with the corresponding properties of glass fiber polypropylene composites.

O'Donnell et al. (2004) found that natural composites were found to have mechanical strength suitable for applications such as housing and automotive. It is also suggested that this composites study still need further investigation in order to monitor the post-curing behaviour and degradation over a long period of time (aging). Another study lead by Pothan et al. (2006) found that the dynamic mechanical properties of short