อิทธิพลของสารคู่ควบไซเลนที่มีผลต่อสมบัติของสารประกอบแต่ง
ระหว่างพอลิไวนิลคลอไรด์กับชานอ้อย

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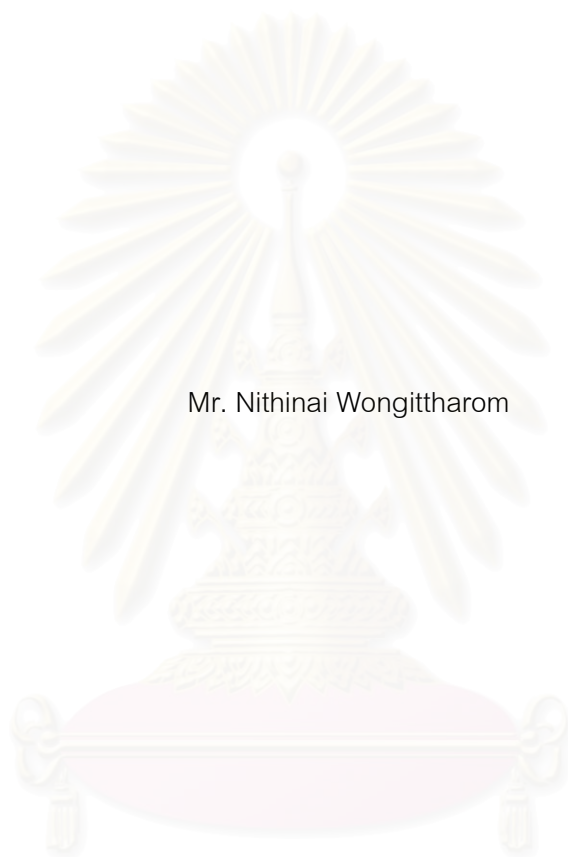
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THE EFFECT OF SILANE COUPLING AGENTS ON PROPERTIES OF

POLY(VINYL CHLORIDE)/BAGASSE COMPOSITES

Mr. Nithinai Wongittharom

A Thesis Submitted in Partial Fulfillment of the Requirements

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อิทธิพลของสารคู่ควบไซเลนที่มีผลต่อสมบัติของสารประกอบแต่งของพอลิไวนิลคลอไรด์กับชานอ้อย

(THE EFFECT OF SILANE COUPLING AGENTS ON PROPERTIES OF

POLY(VINYL CHLORIDE)/BAGASSE COMPOSITES) อ. ที่ปรึกษาวิทยานิพนธ์หลัก :

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      งานวิจัยนี้ศึกษาอิทธิพลของสารคู่ควบไซเลนที่มีผลต่อสมบัติต่างๆของ สารประกอบแต่งของ
พอลิไวนิลคลอไรด์กับชานอ้อยที่มีและไม่มีการปรับสภาพผิวด้วยสารคู่ควบไซเลน 3 ชนิด คือ แกมมา-
อะมิโนโพรพิวไตรเอทอกซีไซเลน (อะมิโน), เบต้า-อะมิโนเอทิล-แกมมา-
อะมิโนโพรพิวไตรเมทอกซีไซเลน (ไดอะมิโน) และไกลซิดรอกซีโพรพิวไตรเมทอกซีไซเลน (อีพอกซี)
และยังทำการศึกษาถึงผลของชนิดและปริมาณของสารคู่ควบที่มีผลต่อสมบัติทางกล ทางความร้อน
และสมบัติการดูดซับน้ำของสารประกอบแต่ง   โดยชานอ้อยแบ่งออกเป็นสองขนาดคือ
ขนาดเล็กอยู่ในช่วง 45-106 ไมโครเมตรและขนาดใหญ่อยู่ในช่วง 106-180 ไมโครเมตร
โดยขึ้นรูปด้วยเครื่องบดผสมชนิดสองลูกกลิ้งและเครื่องกดอัด

จากผลการทดลองพบว่าสารประกอบแต่งที่ชานอ้อยมีการปรับสภาพผิวด้วยสารคู่ควบไซเลนจะให้
สมบัติต้านแรงดึงและสมบัติต้านแรงดัดโค้งมากกว่าสารประกอบแต่งที่ชานอ้อยไม่มีการปรับสภาพผิวแ
ต่มีปริมาณการดูดซับน้ำของวัสดุต่ำกว่า

โดยสารประกอบแต่งที่ชานอ้อยมีการปรับสภาพผิวด้วยสารคู่ควบไซเลนมีค่าต้านแรงดึงเพิ่มขึ้น 24-
27% และค่าต้านแรงดัดโค้งเพิ่มขึ้น 14-17%
เมื่อเทียบกับสารประกอบแต่งที่ชานอ้อยไม่มีการปรับสภาพผิว

อย่างไรก็ตามชนิดและปริมาณของสารคู่ควบไม่ได้ส่งผลเด่นชัดต่อสมบัติต้านแรงกระแทกและสมบัติทา
งความร้อนของสารประกอบแต่ง

นอกจากนั้นเมื่อเปรียบเทียบชนิดของสารคู่ควบไซเลนที่ใช้ทั้งสามชนิด จะพบว่าสารประกอบแต่งที่ชาน

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สาขาวิชา …วิศวกรรมเคมี	ลายมือชื่อ อ. ที่ปรึกษาวิทยานิพนธ์หลัก ....
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NITHINAI WONGITTHAROM: THE EFFECT OF SILANE COUPLING AGENTS ON PROPERTIES OF POLY(VINYL CHLORIDE)/BAGASSE COMPOSITES. ADVISOR: ASST.PROF. ANONGNAT SOMWANGTHANAROJ, Ph.D., CO-ADVISOR: VARUN TAEPAISITPHONGSE, Ph.D., 97 pp.

In this research, the effect of silane coupling agents on properties of Poly (vinyl chloride)/Bagasse composites which were produced from poly (vinyl chloride) and untreated or treated bagasse were evaluated. The surface of bagasse was modified by treating the fiber with γ-aminopropyltriethoxysilane (amino), N-(β-aminoethyl)-γ-aminopropyltrimethoxysilane (diamino) or glycidoxypropyltrimethoxysilane (epoxy). Particularly, this research studied the effects of various types and concentrations of silane coupling agents on mechanical, thermal, and water absorption properties of composites. Bagasse at 60 phr with fiber sizes of 45-106 μm and 106-180 μm were used. PVC dry blend and bagasse were mixed in a two-roll mill followed by compression molding. The composites with treated bagasse showed the increase in the tensile and flexural properties. It showed that tensile and flexural strengths of these composites increased between 24-27% and 14-17%, respectively, compared to composites with untreated fiber. In addition, these composites with treated bagasse absorbed less amount of water than composites with untreated bagasse. However, types and concentrations of silane coupling agents did not show significant effect on impact strength and thermal properties of PVC/bagasse composites. Among three types of silane coupling agent used, the composites with bagasse treated with amino silane coupling agent at 2% by weight of bagasse showed the best results in tensile and flexural properties and they also showed the lowest amount of water absorption.

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CHAPTER I

INTRODUCTION

1.1 General Introduction

The term "wood-plastic composites" (WPCs) refers to composites that contain wood and either thermoset or thermoplastic polymers (Roger, 2000). Thermoset polymers are able to be cured but cannot be remelted by heating. These include cured resins, such as epoxies. Whilst, thermoplastics are plastics that can soften when heated and harden when cooled such as polyethylene (PE), polypropylene (PP), polystyrene (PS) and poly(vinyl chloride) (PVC) (Whelan, 1997). One of the most attractive thermoplastic polymers is PVC which has the essential benefits of easy processing, high productivity, long term stability and low cost. Furthermore, PVC provides the greatest strength and stiffness for WPCs followed by polypropylene and polyethylene. Typically, the PVC/wood composites are used to produce door/window profiles, decking, siding and railing (Roger, 2000). The greater the wood content is, the better the stiffness properties of the composite is.

Natural fibers are advantageously used as a result of their low density and increasing environmental pressures. There are a large variety of natural fibers such as rice straw, rice husk, palm, bagasse, hemp, flax and other agricultural residues (Bledzki and Gassan, 1999, Georgopoulos et al., 2005, Jongmailuck et al., 2006 and Kamel, 2004). As different natural fibers have different chemical compositions, different physical structures and mechanical properties can be expected from the composites resulted from different natural fibers (Maldas et al., 1989 and 2001). Little effort has been made so far to prepare non-wood fiber (e.g., agricultural fibers) reinforced PVC composites and to improve their properties. These fibers, when used in plastic composites,which can provide significant reinforcement and often contribute greatly to the structural performance.

PVC/fiber composites with several kinds of natural fibers such as bagasse, jute, and rice straw as filler have increasingly attracted a number of researchers because of their several advantages such as cost savings, good mechanical properties, better dimensional stability, low density and renewable raw materials. The mechanical performance of natural fiber reinforced plastics varies greatly depending on the type of natural fibers, fiber treatments, the type of plastic, additives, and processing methods. Natural fibers are added to plastics to improve mechanical performance such as stiffness and strength without increasing the density or cost. The generally low impact performance of natural fiber composites tends to limit their use. Natural fibers are

hydrophilic and do not tend to be easily wetted or to bond well with many matrix materials, particularly the commodity thermoplastics. Blending natural fiber with PVC shows incompatibility problem due to hydrophobic nature of polymer and hydrophilic nature of fiber. This incompatibility problem can usually be solved by the use of coupling agents (Keneer et al., 2004). A wide variety of coupling agents and fiber surface modifications and treatments has been investigated for use in plastic natural fiber composites.

Generally, chemical coupling agents act as bridge to link polar wood fibers and non-polar thermoplastics. This helps transfer the stresses between wood and thermoplastic thus also improve the interfacial bonding strength in WPCs (Haihong and Kamdem, 2004). In recent years, there have been many studies mentioned the necessity of using coupling agents such as poly [methylene(polyphenyl isocyanate)] (PMPPIC) (Haihong and Kamdem, 2004, and Maldas and Kokta, 1991), silane (Matuana et al.,1998, Sombatsompop and Chaochanchaikul, 2005, Zhao et al, 2006, Kotchasaeni et al., 2006 and Jongmailuck et al., 2006), maleated anhydride polypropylene (MAPP) (John et al., 2004 and Kotchasaeni et al., 2006), chitin and chitosan (Shah and Matuana, 2005) to enhance the mechanical properties of PVC/wood composites.

Silane coupling agents have better performance in organic-inorganic coupling agents recently used in PVC/wood composites because the attachment of silane to hydroxy groups of cellulose or lignin is accomplished either directly to the alkoxy group attached to silicon or via its hydrolyzed products (i.e. silanol) by the hydrogen bonds or ether linkage. The functional group in silane also influences the coupling action (Sombatsompop, 2005).

This study contributed further understanding on the effect of the addition of various silane coupling agents on mechanical properties of PVC/bagasse composites. The types and loading of silane coupling agents were examined. The most appropriate silane coupling agent was used to enhance the mechanical properties of PVC/bagasse composites.

1.2 Objective of the Research

This study aimed at generating the technical knowledge and formulations for production of PVC/bagasse composites by mixing PVC resin and natural fiber available locally, i.e. bagasse, as an alternative for natural wood. The main objective was to study and compare the effects of type and loading of various silane coupling agents on properties of PVC/bagasse composites.

1.3 Scopes of the Research

1.   Preparation of materials (PVC resins, bagasse)

2.   Treatment of natural fiber, i.e. washing, drying, sieving and storing

3.   Preparation of PVC matrix and natural fiber (bagasse) composites at the natural fiber contents of 60 phr and average particle sizes of 45-106 μm and 106-180 μm. The silane coupling agents were γ-aminopropyltriethoxysilane (amino), N-(β-aminoethyl)-γ-aminopropyltrimethoxysilane (diamino), and Glycidoxypropyltrimethoxysilane (epoxy) and the loading of silane coupling agent were 0- 3 %wt of bagasse.

4.   Preparation PVC/bagasse composites using two-roll mill and compression molding.

5.   Analysis of the PVC/bagasse products using two-roll mill and compression molding.

6.   Characterizations of the PVC/bagasse composites for tensile properties, flexural properties, impact strength, thermal stability, water absorption and other benefit for physical properties as wood-substituted materials.

7.   Research discussion, conclusion.

8.   Report preparation.

CHAPTER II

THEORY

2.1 Poly(vinyl chloride) (PVC)

Poly(vinyl chloride) (PVC) is a thermoplastic material and is commonly used in the construction and industrial sectors. The basic raw materials for PVC are derived from salt and oil. Chlorine is manufactured by the electrolysis of sodium chloride salt. The electrolysis of salt water produces chlorine. The chlorine is then combined with ethylene (C_2H_4), obtained from oil, and is converted at very high temperature to vinyl chloride monomer (VCM). The VCM is then polymerized into PVC. The molecular structure of PVC is shown in Figure 2.1.
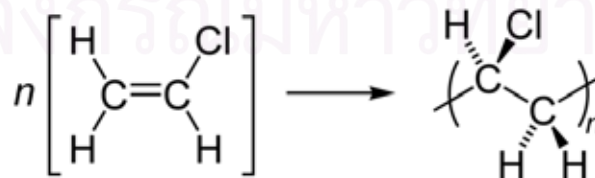
Figure 2.1 Molecular Structure of PVC (Gachter and Muller, 1987)

Due to poor properties of PVC such as stiff, hard, tough and poor heat stability, PVC resins are normally mixed with other additives such as impact modifiers and stabilizers, providing hundreds of PVC based materials with a variety of engineering properties (Gachter and Muller, 1987). General properties of rigid and plasticized PVC shown in Table 2.1.

Table 2.1 Properties of rigid and plasticized PVC (Walten, 1963)

Properties	Rigid PVC	Plasticized PVC
Density (g/cm^3)	1.3-1.4	1.1-1.7
Specific Heat Capacity (cal/g ^{o}C)	0.25	-
Sag Temperature (^{o}C)	78	-
Milling Temperature (^{o}C)	150	20-140
Shore Hardness (D)	-	47
Tear Resistance (kg/m)	-	8500

2.2 Natural Fibers and Their Compositions

The basic components of natural fibers are cellulose, hemicellulose, lignin, pectin, and waxes with regard to the physical properties of the fibers. The percentage composition for each of these components varies for different fibers.

2.2.1. **Cellulose** is one of many polymers found in nature (wood, paper, and cotton). About 33 % wt of all plant matter is cellulose (the cellulose content of cotton is 90 % wt and that of wood is 50 % wt) (Siriarcha, 2005). Cellulose is derived from D-

glucose units, which condenses through β(1→4)-glycosidic bonds. This linkage motif

contrasts with that for α(1→4)-glycosidic bonds present in starch, glycogen, and other

carbohydrates. The multiple hydroxyl groups on the glucose residues from one chain

form hydrogen bonds with oxygen molecules on another chain, holding the chains firmly

together side-by-side and forming microfibrils with high tensile strength. This strength is

important in cell walls, where they are meshed into a carbohydrate matrix, conferring

rigidity to plant cells (Sjostrom, 1993). The structure of cellulose is shown in Figure 2.2.
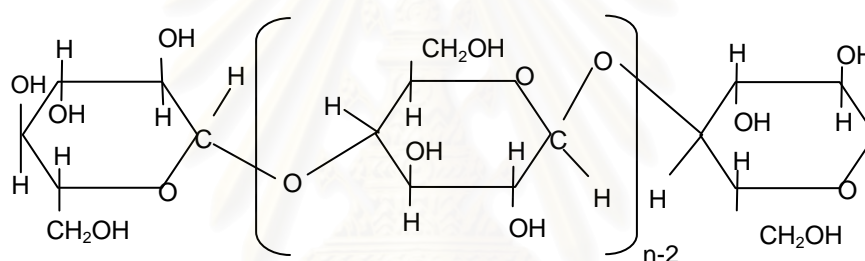
Figure 2.2 Structure of cellulose (www.fibersource.com)

2.2.2. **Hemicellulose** contains many different sugar monomers. In contrast,

cellulose contains only anhydrous glucose. A hemicellulose can be any of several

heteropolymers (matrix polysaccharides) presented in almost all cell walls along with

cellulose. While cellulose is crystalline, strong, and resistant to hydrolysis, hemicellulose

has a random, amorphous structure with little strength. It is easily hydrolyzed by dilute

acid or base as well as myriad hemicellulase enzymes (Sjostrom, 1993). However, fiber

contains the hemicellulose about of 5 to 20 %wt (Siriarcha, 2005). The structure is

illustrated in Figure 2.3.
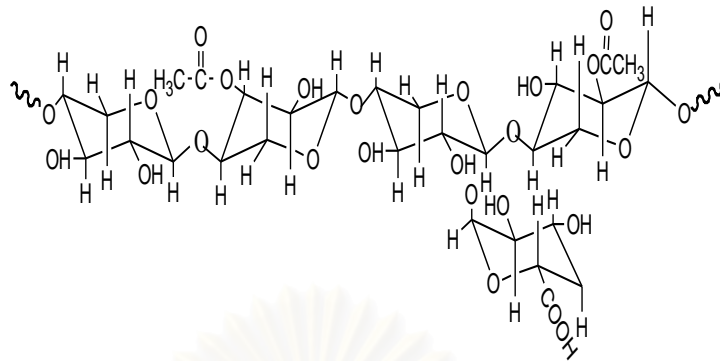
Figure 2.3 Structure of hemicellulose (www.fibersource.com)

2.2.3. **Lignin** is a complex hydrocarbon polymer with both aliphatic and aromatic constituents. Their chief monomer units are various ring-substituted phenyl-propanes with C-O-C and C-C linkages. The mechanical properties of lignin are lower than those of cellulose. Composition of lignin in fiber is about 2 to 14 %wt (Sjostrom, 1993). The structure of lignin is shown in Figure 2.4.
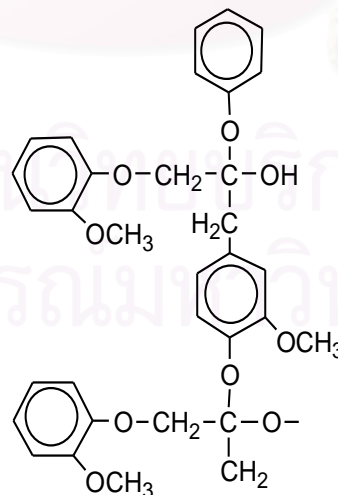
Figure 2.4 Structure of lignin (www.fibersource.com)

2.2.4. **Pectin** is a white to light brown powder and is a heteropolysaccharide derived from the cell wall of plants. The characteristic structure of pectin is a linear chain of α-(1-4)-linked D-galacturonic acid that forms the pectin-backbone, a homogalacturonan. Pectin is soluble in water only after a partial neutralization with alkali or ammonium hydroxide (Sjostrom, 1993). The structure of pectin is illustrated in Figure 2.5.
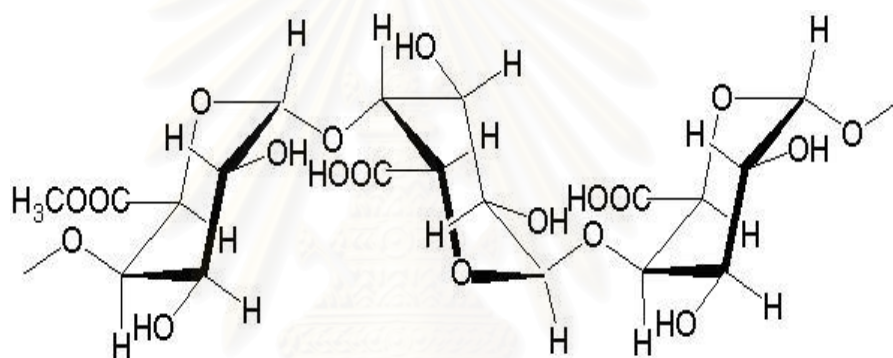
Figure 2.5 Structure of pectin (www.fibersource.com)

2.2.5. **Wax** is a general term used to refer to the mixture of polar long-chain lipids forming a protective coating (cutin in the cuticle) on plant leaves and fruits and also in animals. All waxes are water-resistant materials made up of various substances including hydrocarbons (normal or branched alkanes and alkenes), ketones, diketones, primary and secondary alcohols, aldehydes, sterol esters, alkanoic acids, terpenes (squalenes) and monoesters (wax esters) (Siriarcha, 2005). The wax components are

demonstrated in Table 2.2. Commonly, waxes are esters of an alcohol other than glycerol (long chain alcohol, sterol, hydroxycarotenoids and vitamin A) and a long **chain** acid (wax esters). Wax esters are saponified by hot alkaline solutions and the reaction gives a fatty acid and an alcohol. They are soluble in aromatic solvents, chloroform, ethers, esters and ketones (Sjostrom, 1993).

Table 2.2 Wax components (Chevreul, 2000)

Compound	General structure
n-Alkanes	$H_3C(CH_2)_nCH_3$
Ketones	R^1COR^2
Secondary alcohols	$R^1CH(OH)R^2$
β-Diketones	$R^1COH_2COR^2$
Monoesters	R^1COOR^2
Primary alcohols	RCH_2OH
Aldehydes	$RCHO$
Alkanoic acids	$RCOOH$
Dicarboxylic acids	$HOOC(CH_2)_nCOOH$
Ω-Hydroxy acids	$HOOCH_2(CH_2)_nCOOH$

2.3 Composite Material

Composites are combinations of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and is embedded in the other material called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer. Typically, reinforcing materials are

strong with low densities while the matrix is usually a flexible, or tough, material. If the composite is designed and fabricated correctly, it combines the **strength of the reinforcement** with the toughness of the matrix to achieve a combination of desirable properties not available in any single conventional material. The drawback is that such composites are often more expensive than conventional materials (Hull and Clyne, 1996).

2.4 Fiber Composite Matrices

A role of a matrix in a plastic/fiber composite is to transfer stresses between the fibers, to provide a barrier against an adverse environment, and to protect the surface of the fibers from mechanical abrasion. Generally, the matrix plays a minor role in the tensile load-carrying capacity of a composite structure. However, selection of a matrix has a major influence on the interlaminar shear as well as on in-plane shear properties of the composite material. The interlaminar shear strength is an important design consideration for structures under bending loads, whereas the in-plane shear strength is important for structures under torsional loads. The matrix provides lateral support against the possibility of fiber buckling under compression loading, thus influencing to some extent the compressive strength of the composite material. The interaction between fibers and matrix is also essential in designing damage-tolerant structure.

Finally, the processability and defects in a composite material depend strongly on the physical and thermal characteristics such as viscosity, melting point, and curing temperature of the matrix. Other desirable properties of a matrix are to minimize moisture absorption, to wet and bond to fiber and to eliminate voids during the compacting and curing processes. Moreover, a matrix should show high strength at elevated temperature (depending on the application), low shrinkage, and dimensional stability.

2.5 Coupling Agent

Coupling agent is primarily defined as materials that enhance the adhesive bonds of dissimilar surfaces. It is usually used for eliminating weak boundary layers, developing a highly crosslinked interphase region or altering the acidity of the fiber substrate surface. The mechanism of bonding by coupling agents in fiber-plastic composites appears to be very complex as different polymer systems and different coupling agents tend to show different types of interfacial adhesion (Maldas and Kokta, 1989 and 1991, Matuana et al., 1998, John et al., 2004, Keneer, 2004, Sombatsompop and Chaochanchaikul, 2005, Zhao et al, 2006, Kotchasaeni et al., 2006, and Jongmailuck et al., 2006).

2.5.1 Classification of Coupling Agent (Dalväg et al., 1985)

Coupling agent is classified into organic, inorganic, and organic-inorganic groups. Organic agents include isocyanates, anhydrides, amides, imides, acrylates, chlorotriazines, epoxides, organic acids, monomers, polymers, and copolymers. Only a few inorganic coupling agents, such as silicates, are used in wood-plastic composites. Organic-inorganic agents include silanes and titanates.

Organic coupling agents in plastic/fiber composites normally have bi- or multifunctional groups in their molecular structure. These functional groups, such as (-N=C=O) of isocyanates, $[-(CO)_2-]$ of maleic anhydrides, and (-Cl-) of diclorotriazine derivatives, interact with the polar groups [mainly hydroxyl groups (-OH)] of cellulose and lignin to form covalent or hydrogen bonding. On the other hand, organic coupling agents can modify the polymer matrix by graft copolymerization, thus resulting in strong adhesion, even crosslinking at the interface.

Inorganic coupling agents possibly act as dispersing agents to counteract the surface polarity of wood fiber and improve the compatibility between wood fiber and polymer.

Organic-inorganic agents are hybrid compounds in structure. For example, titanates usually contain a titanium center and an organic part surrounding this inorganic atom. The functionality of the organic part in these agents determines their coupling

effectiveness in plastic/fiber composites. Organic-inorganic coupling agents are between organic and inorganic agents in function.

2.5.2 Silane Coupling Agent (www.dowcorning.com)

Silicon is in the same family of elements as carbon in the periodic table. In their most stable state, silicon and carbon will both conveniently bond to four other atoms; but silicon-based chemicals exhibit significant physical and chemical differences compared to analogous carbon-based chemicals. Silicon which is more electropositive than carbon, does not form stable double bonds, and is capable of very special and useful chemical reactions. Silicon-based chemicals include several types of monomeric and polymeric materials. Monomeric silicon chemicals are known as silanes. The four substituents have been chosen to demonstrate differences and similarities in physical and chemical properties between silicon- and carbon-based chemicals. A silane that contains at least one carbon-silicon bond (CH_3-Si-) structure is known as an organosilane. The carbon-silicon bond is very stable, very non-polar and gives rise to low surface energy, non-polar, hydrophobic effects. Similar effects can be obtained from carbon-based compounds, although these effects are often enhanced with silanes. The silicon hydride (–Si-H) structure is very reactive. It reacts with water to yield reactive silanol (-Si-OH) species and, additionally, will act across carbon-carbon double bonds

to form new carbon-silicon-based materials. The methoxy group on the carbon compound gives stable methyl ether, while its attachment to silicon gives a very reactive and hydrolyzable methoxysilyl structure. The organofunctional group, the aminopropyl substituent, will act chemically the same in the organosilicon compound as it does in the carbon-based compound. The distance of the amine, or other organofunctional group, from silicon will determine whether the silicon atom affects the chemistry of the organofunctional group. If the organic spacer group is a propylene linkage (e.g., -$CH_2CH_2CH_2$-), then the organic reactivity in the organofunctional silane will be similar to organic analogs in carbon chemistry. Certain reactive silanes, particularly vinyl silanes (-Si-CH=CH_2) and silicon hydrides (-Si-H), are useful reactive groups in silicon chemistry, even though the reactive group is attached directly to the silicon atom. Attachment of chlorine, nitrogen, methoxy, ethoxy or acetoxy directly to silicon yields chlorosilanes, silylamines (silazanes) and alkoxysilanes respectively, that are very reactive and exhibit unique inorganic reactivity. Such molecules will react readily with water, even moisture adsorbed on a surface, to form silanols. These silanols then can react with other silanols to form a siloxane bond (-Si-O-Si-), a very stable structure; or in the presence of metal hydroxyl groups on the surface of glass, minerals or metals, silanols will form very stable –Si-O-metal bonds to the surface. This is the key chemistry that allows silanes to function as valuable surface-treating and coupling agents.

Silane coupling agents are silicon-based chemicals that contain two types of reactivity – inorganic and organic – in the same molecule. A typical general structure is $(RO)_3SiCH_2CH_2CH_2$-X, where RO is a hydrolyzable group, such as methoxy, ethoxy, or acetoxy, and X is an organofunctional group, such as amino, methacryloxy and epoxy . A silane coupling agent will act at an interface between an inorganic substrate (such as glass, metal or mineral) and an organic material (such as an organic polymer, coating or adhesive) to bond, or couple, the two dissimilar materials. A simplified picture of the coupling mechanism is shown in Figure 2.6.
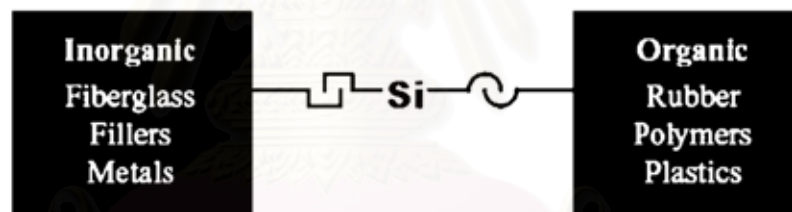
Figure 2.6 The silane coupling mechanism (www.dowcorning.com)

When organic polymers are reinforced with glass fibers or minerals, the interface, or interphase region, between the polymer and the inorganic substrate is involved in a complex interplay of physical and chemical factors. These factors are related to adhesion, physical strength, coefficient of expansion, concentration gradients and retention of product properties. A very destructive force affecting adhesion is

migration of water to the hydrophilic surface of the inorganic reinforcement. Water attacks the interface, destroying the bond between the polymer and reinforcement, but a "true" coupling agent creates a water-resistant bond at the interface between the inorganic and organic materials. Silane coupling agents have the unique chemical and physical properties not only to enhance bond strength but also, more importantly, to prevent de-bonding at the interface during composite aging and use. Silane coupling agents that contain three inorganic reactive groups on silicon (usually methoxy, ethoxy or acetoxy) will bond well to the metal hydroxyl groups on most inorganic substrates, especially if the substrate contains silicon, aluminum or heavy metal in its structure. The alkoxy groups on silicon hydrolyze to silanols, either through the addition of water or from residual water on the inorganic surface. Then the silanols coordinate with metal hydroxyl groups on the inorganic surface to form an oxane bond and eliminate water. As can be seen in Figures 2.7 and 2.8.
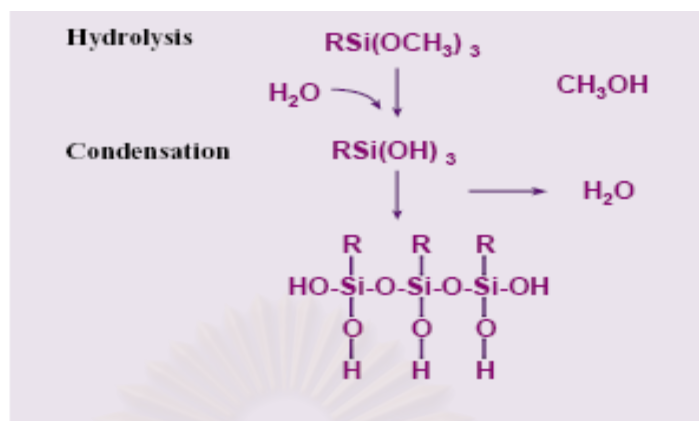
Figure 2.7 Hydrolysis of alkoxysilanes (www.dowcorning.com)
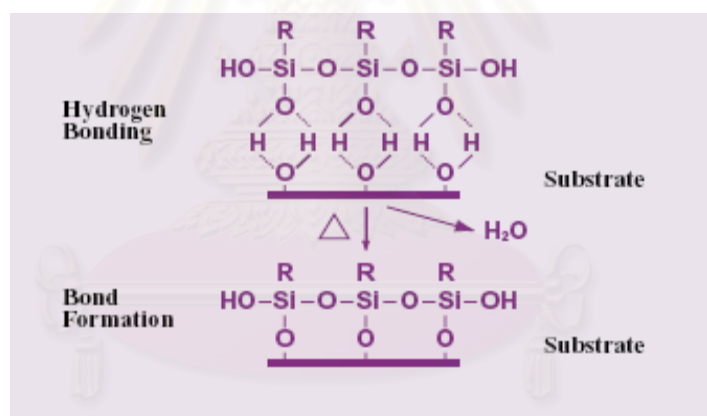
Figure 2.8 Bonding to an inorganic surface (www.dowcorning.com)

CHAPTER III

LITERATURE REVIEW

There were many researches on the effect of parameters such as size of fibers, loading of fibers, and coupling agents on the mechanical and other properties of PVC/natural fiber composites.

3.1 The Effect of Size of Fibers

The size of natural fiber is one of the significant factors on the mechanical properties of PVC/fiber composites. It was reported that the tensile and flexural strength of PVC/wood composite with Aspen sawdust mesh size 60 was 50% higher than that with aspen sawdust mesh size 40 (Maldas et al., 1989).

The reasons were that a short fiber (mesh size 60) were homogeneous, had critical length and prevents entanglement while all fiber characteristics were maintained. Short fiber length also provided better dimensional stability as well as resistance to break during fabrication. Therefore, sawdust (Aspen) with a smaller particle size is more compatible with polymer than those with larger one. However, the tensile strengths of

PVC/sawdust (Spruce), and PVC/sawdust (Birch) composites with sawdust mesh 60 were not much different from those with sawdust mesh 40.

3.2 The Effect of Coupling Agents

Composite manufactured from blends of thermoplastics and fibers have increasingly attracted a numbers of researchers and manufactures because of cost savings, good mechanical properties, and better dimensional stability (Roger, 2000). There are several coupling agents that can affect on mechanical properties for PVC/wood composites, i.e., poly [methylene(polyphenyl isocyanate)] (PMPPIC) (Haihong and Kamdem, 2004, and Maldas and Kokta, 1991), silane (Matuana et al.,1998, Sombatsompop and Chaochanchaikul, 2005, Zhao et al, 2006, Kotchasaeni et al., 2006 and Jongmailuck et al., 2006), maleated anhydride polypropylene (MAPP) (John et al., 2004 and Kotchasaeni et al., 2006), chitin and chitosan (Shah and Matuana, 2005).

Some polymeric coupling agents have been widely examined for their effectiveness in the wood-plastic composites (WPCs) area, and they serve more like compatibilizers between the wood phase and the plastic matrix. Poly [methylene(polyphenyl isocyanate)] (PMPPIC) has been proved to be very effective polymeric coupling agent for polyvinyl chloride (PVC)/wood fiber (WF) composites (Haihong and Kamdem, 2004). There were an approximately 89.6% increased in

tensile strength, 33.8% increased in impact strength and 68.5% increased in elongation at break for PVC/WF composites containing 25%wt wood sawdust and 8%wt the loading of PMPPIC compared with composites without PMPPIC (Haihong and Kamdem, 2004).

Besides, Maldas et al. also used PMPPIC as a coupling agent in PVC/bagasse composites. The experimental result showed that there were 19.04% raise in tensile modulus, 33.12% raise in tensile strength, 37.5% raise in elongation at break and 19.6% raise in impact strength with PVC/bagasse composites containing 32%wt bagasse and 3%wt loading of PMPPIC compared wth composite without PMPPIC (Maldas and Kokta, 1991).

Silane is an important coupling agent in the filled-plastics industry since it has the –OH side group that can connect to the wood surface through hydrogen bonds formed between the hydroxyl groups of silanols and wood (Matuana et al., 1998, Sombatsompop and Chaochanchaikul, 2005, Zhao et al, 2006, Kotchasaeni et al., 2006 and Jongmailuck et al., 2006). It was found that aminopropyltriethoxysilane is the essential adhesion promoter for PVC/newsprint composite and tensile strength of the composites was improved significantly up to 34% from PVC/untreated fiber composites (Matuana et al., 1998).

The effect of molecular structure of silane coupling agent that was used as property promoters for PVC/wood sawdust composites. In this study, *N*-2(aminoethyl)3-

aminopropytrimethoxysilane is used as coupling agent whose structure was shown in

Figure 3.1 and was recommended for the optimization of the tensile properties

(Sombatsompop and Chaochanchaikul, 2005). There were about 14% increased in

tensile modulus, 89.6% increased in tensile strength, 68.75% increased in elongation at

break and 33.8% increased in impact strength with PVC/sawdust composites containing

9.1 %wt sawdust and concentrations of 1.0 %wt of *N*-2(aminoethyl)3-

aminopropytrimethoxysilane compared with non-treated composite. This might be

because *N*-2(aminoethyl)3-aminopropytrimethoxysilane coupling agent had three

hydroxyl groups and it had the formed linkages between the coupling agent and the

wood sawdust (Sombatsompop and Chaochanchaikul, 2005).

$$CH_3CH_2O—Si—CH_2CH_2CH_2—N—CH_2CH_2NH_2$$

with OCH_2CH_3 above Si, and $O\|CH_2CH_3$ below Si, and H below N.
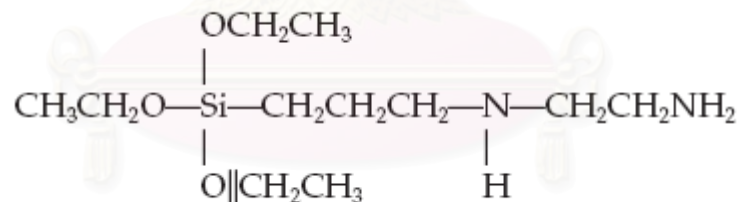
Figure 3.1 Structure of *N*-2(aminoethyl)3-aminopropytrimethoxysilane

(Sombatsompop and Chaochanchaikul, 2005)

Zhao et al. (2006) studied the PVC/WF composites containing 9.1%wt sawdust

with silane γ-aminopropyldiethoxysilane at the loading level of 1.5%wt. The tensile

strength increased 21.95% when the treated PVC/WF was filled with silane at the loading

level of 1.5%wt. The impact strength increased from 16.08 kJ/m^2 to 19.34 kJ/m^2 until

silane was loaded up to 2.5%wt. After the highest optimum loading levels, its tensile strength and impact strength droped with further addition of silane. The interface was critical in improving the mechanical properties of PVC/WF composites (Zhao et al, 2006).

Maleic Anhydride (MA) is an α, β-unsaturated carbonyl compound, containing one carbon-carbon double bond (C=C) and two carboxylate groups (-COO-) whose structure is shown in Figure 3.2. This conjugated structure greatly increases the graft reactivity of the carbon-carbon double bond on the heterocyclic ring with the polymer matrix through the conjugate addition under a radical initiator, resulting in crosslinking or strong adhesion at the interface (Kotchasaeni et al., 2006).
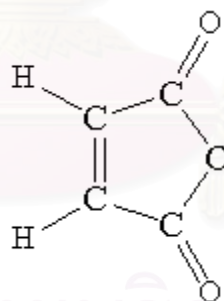
Figure 3.2 Structure of maleic anhydride (John et al., 2004)

In the maleation method, MA is used to modify the polymer matrix such as polyethylene (PE) and polypropylene (PP) in the presence of a free radical initiator. It is then grafted on to wood fibers by a succinic half-ester bridge. Maleic anhydride modified polypropylene or maleated polypropylene (MAPP) is a coupling agent used for

wood-plastic composites (WPCs) (John et al., 2004 and Kotchasaeni et al., 2006). The

grafting of maleic anhydride onto polypropylene is shown in Figure 3.3.
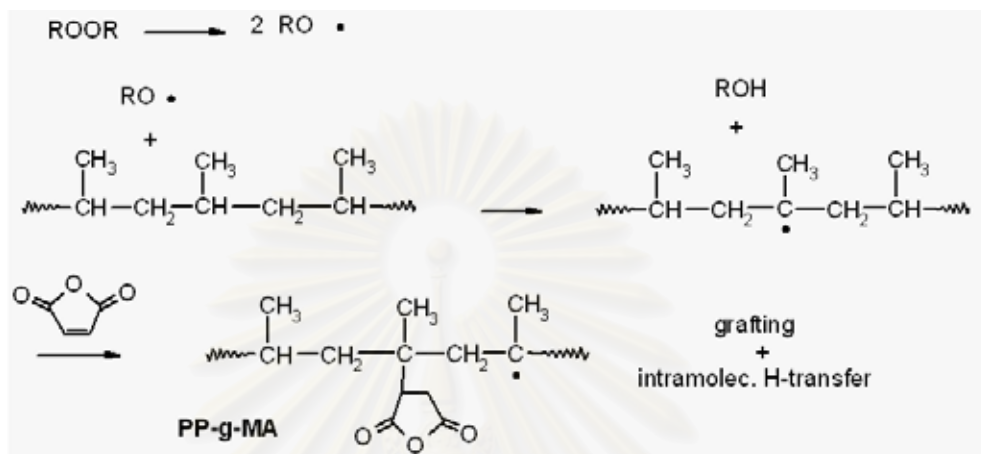
Figure 3.3 Grafting of maleic anhydride onto polypropylene

(John et al., 2004)

The acid number, which represents the amount of functionality in a coupling

agent, and molecular weight of polymer backbone chain are two important factors

influencing the coupling effectiveness of MAPP in WPCs (John et al., 2004 and

Kotchasaeni et al., 2006). John et al investigated maleation that affected thermal

behavior of maleated wood and resultant wood-polymer composites (John et al., 2004).

In this study, two MAPPs were used as coupling agent ; one had an average weight

molar mass was 9,100 g/mol and its acid number was between 40 and 45 and the other

had a high molecular weight of 47,000 g/mol but had low acid number which was

between 12 and 18. The experimented results showed that the latter one is an

approximate 20% increased in storage modulus and 18% increased in loss modulus at

25°C. Due to MAPP with a high molecular weight effectively improves the mechanical properties of WPCs. It probably acts as a dispersing agent instead of a true coupling agent in melt-blending formation because of its low molecular weight (John et al., 2004).

Chitin is the second most abundant natural polymer after cellulose and is extracted from the shells of crustaceans. Chitosan is the deacetylated form of chitin. These polymers are widely available, nontoxic, bio-compatible and lower in cost than many synthetic coupling agents. The acetyl amine functionality of the chitin and the amine functionality of the chitosan should permit these polymers to interact with wood and PVC in a manner similar to aminosilane and to enhance the interfacial adhesion between PVC and wood fibers, while to potentially improve other properties and also to be more cost-effective (Shah and Matuana, 2005).

Shah and Matuana used two natural polymers: chitin and chitosan as novel coupling agents for PVC/WF composites. Chitin and chitosan had a positive effect on the flexural strength of PVC/WF composites. Flexural strength of PVC/wood flour composite was increased by 20% when 6.67 %wt chitin or 0.5 %wt chitosan was added to the composite. These could be related with the better interaction between wood flour and matrix (Shah and Matuana, 2005).

CHAPTER IV

EXPERIMENT

4.1 Materials

4.1.1 PVC Resin

Suspension PVC resin (Siamvic 258 RB, K-value 58), stabilizer, processing aid, external lubricant, and internal lubricant were supplied by Vinythai Public Company Limited. Table 4.1 showed characteristics of siamvic 258 RB suspension PVC resin.

Suspension PVC resin with K-value 58 is a low K value resin suitable for rigid PVC processing. It produces very low dust during transportation. It can be processed easily at low temperature. It has good thermal stability, good initial color, no tendency to stick on hot metal surface, and very low level of fish-eyes. (www.vinythai.co.th)

Table 4.1: Characteristics of Suspension PVC Resin (www.vinythai.co.th)

Characteristics	Units	Values (*)	Standards
K-value (cyclohexanone)		58	DIN 53726
Polymerization Degree		680	JIS K6721
Apparent Bulk Density	kg/l	0.56	ISO 60
Volatile Matter	%	0.3	ISO 1269

K – value (cyclohexanone) is PVC Resin viscosity based on cyclohexanone viscosity.

(*) In the case of certain characteristics the values given in this are means based on a large number of individual measurements distributed around the means in a range corresponding to the normal manufacture and measurement tolerances. These values should not be considered as specifications.

4.1.2 Natural Fiber

Figure 4.1: Bagasse

Bagasse was obtained from the biomass remaining after sugarcane stalks were crushed to extract their juices. In this research, bagasse was received from Saraburi Sugar, Co., Ltd., Thailand. The average size of the bagasse fiber used in this work was in the range 45-106 μm with aspect ratio = 3.12 and 106-180 μm with aspect ratio = 3.78. The content of bagasse particles added into the PVC compound was fixed at 60 phr. The composition of bagasse was shown in Table 4.2.

Table 4.2: Average composition of bagasse (Siriarcha, 2005)

Composition	Percentage base on Dry Weight
Ash	1.50
Lignin	18.29
Hemicellulose	35.84
Alphacellulose	41.00
Other	3.37

4.1.3 Silane Coupling Agents (www.dowcorning.com)

Three silane coupling agents which were used in this study

1) γ-aminopropyltriethoxysilane (amino)
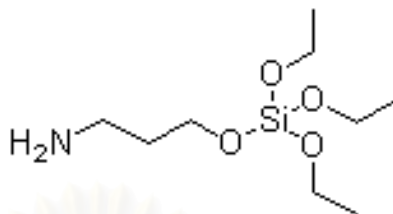
2) N-(β-aminoethyl)-γ-aminopropyltrimethoxysilane (diamino)
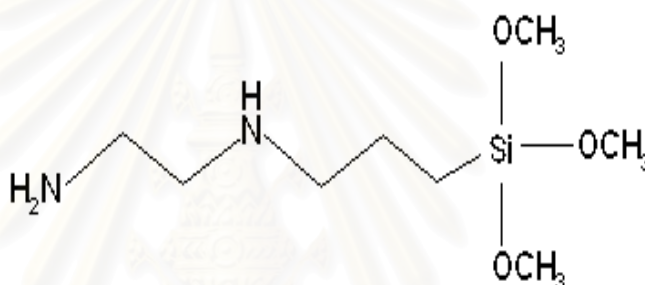
3) Glycidoxypropyltrimethoxysilane (epoxy)
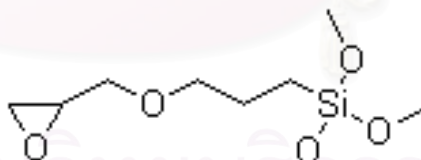
They were supplied by Dow Corning Corporation (Thailand). The loading of silane coupling agent used in this work were 0-3%wt. The maximum 3%wt was used base on the assumption that silane added to the composite can cover all bagasse surface area.

4.2 Preparation of PVC Dry Blend

PVC dry blend for PVC/bagasse composites in this research was prepared from the formula given in Table 4.3 which was received from Vinythai PCL, Thailand. Ingredients of PVC dry blend were expressed in parts per hundred parts of pure PVC resin (phr). PVC and additives were mixed in dry blend machine at the temperature of 130 $^{\circ}$C for 15 minutes and then cooled with the temperature of 30 $^{\circ}$C for 5 minutes.

Table 4.3: PVC Dry Blend Formulation (www.vinythai.co.th)

Ingredients	Concentration (phr)
PVC	100
Stabilizer	4
Processing aid	6
External Lubricant	2
Internal Lubricant	0.5

4.3 Preparation of PVC/Natural Fiber Composites

The fillers were milled to by cutting mill. Before mixing, bagasse with particle sizes 45-106 µm and 106-180 µm and a fixed content of 60 phr were dried in an oven at

105 °C for 12 hours. Then PVC dry blend mixed with untreated fillers by silane coupling agent in a two-roll mill as shown in Figure 4.2 (Labtech) for 5-6 min with temperature of both front and back rolls at 170 $^{\circ}$C and the roll gap was 0.3 mm. But PVC dry blend was mixed with treated fillers by silane coupling agent in a two-roll mill with temperature of both front and back roll at 195 °C mixing for 5-6 min. In the case of treated fiber, the molding temperature was higher because it cannot be molded at 170 °C (Sombatsompop, 2005). After mixing, the sample was compression molded in steel mold. The size of specimen was 200x200x4 mm^3. The temperature in both of upper and lower heaters were 170 °C under pressure of 150 psi for 4 min followed by cooling to room temperature in another press for 4 min. The obtained sheets were cut into appropriate sizes for mechanical and thermal properties testing.

Figure 4.2: Two Rolls Mill Machine (www.vinythai.co.th)

4.4 Surface Treatment Method of the Natural Fiber by Silane Coupling Agents

The bagasse was carefully dried before use through heat treatment in an oven for 24 h at 105 °C until the bagasse weight was constant to minimize the moisture content. A mixture of deionized water and 1% acetic acid was prepared, and the silane coupling agent was slowly dropped into the mixture until the required concentration is met. The silane solution was prepared in the acetic acid mixture because it was more reactive in the acidic condition. The silane concentration were varied from 0.0 to 3.0%wt. The silane solution was then added onto the bagasse particles, with a better mixing obtained with a high-speed mixer for 5 min. Thereafter, the mixtures are filtered to remove the solvent. The treated bagasse was then oven dried for 6 h at 100 °C until a constant weight was achieved (Sombatsompop, 2005).

4.5 Characterizations

4.5.1. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was conducted on a PerkinElmer spectrometer to determine the functional groups at surface of the samples. The spectrum was averaged from 100 scans recorded in absorbance units from 4000 to 650 cm^{-1} wavenumber using attenuated total reflectance (ATR) mode for both treated and untreated composites. In

addition, FTIR spectroscopy of treated and untreated fiber were evaluated by using KBr

mode.

4.5.2. Scanning Electron Microscopy (SEM)

The interfacial interaction or the adhesion between bagasse filler and

poly(vinylchloride) polymer matrix was examined using SEM micrographs. The

micrographs were obtained using Scanning Electron Microscope (Model JSM-5410LV,

Japan) at an acceleration voltage of 15 kV. The fracture surface of each specimen was

coated with thin gold film, of which the thickness was between 10 to 20 nm, prior to

obtaining the micrograph.

4.5.3. Mechanical Properties

Tensile Property

Tensile test measures the force required to break a specimen and the extent to

which the specimen stretches or elongates to that breaking point. Tensile test produces

a stress-strain diagram, which is used to determine tensile modulus.

The dog-bone shape specimens were prepared for tensile testing following

ASTM D638 (or ISO 527).   At least five measurements were taken using Instron

Universal Testing Machine (Instron 5567, NY, USA) and statistically averaged to obtain

an average value and a standard deviation. The crosshead speed was 50 mm/min.

<u>Flexural Property</u>

In engineering mechanics, flexure (also known as bending) characterizes the behavior of a structural element subjected to a lateral load. A structural element subjected to bending is known as a beam. A closet rod sagging under the weight of clothes on clothes hangers is an example of a beam experiencing bending.

The bar shape specimens (10×4×80 mm) were prepared for flexural testing following ASTM D790. At least seven measurements were taken using Instron Universal Testing Machine (Instron 5567, NY, USA) as shown in Figure 4.3 and statistically averaged to obtain an average value and a standard deviation. The set up value for flexural testing according to ASTM D790 were standard speed 1.2 mm/min, span 48 mm, and load cell 1 kN.
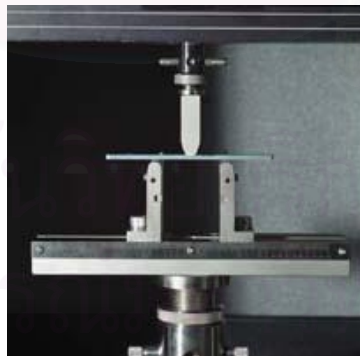
Figure 4.3: Flexural Testing (www.ptli.com)

Izod Impact Property

Notched Izod Impact is a single point test that measures a resistance of material to impact from a swinging pendulum. Izod impact is defined as the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. Izod specimen is notched to prevent deformation of the specimen upon impact. The specimen is clamped into the pendulum impact test fixture with the notched side facing the striking edge of the pendulum. The pendulum is released and allowed to strike through the specimen. If breakage does not occur, a heavier hammer is used until failure occurs. ASTM impact energy is expressed in J/m or ft-lb/in. ISO impact strength is expressed in kJ/m^2. Impact strength is calculated by dividing impact energy in J (or ft-lb) by the thickness of the specimen. The test result is typically the average of 10 specimens. The bar shape specimens were prepared for Izod impact strength testing following ASTM D256 (or ISO 180). The dimension of the standard specimen was 64×12.7×3.2 mm. The depth under notch of the specimen was 10.2 mm.

4.5.4. Thermal Properties

Heat Distortion Temperature

Heat distortion temperature is defined as the temperature at which a standard test bar deflects a specified distance. It is used to determine short-term heat resistance. It distinguishes between materials that are able to sustain light loads at high

temperatures and those that lose their rigidity over a narrow temperature range. Heat distortion temperature testing following A were done ASTM D648. The specimen was placed under the deflection measuring device. The specimen was then lowered into a silicone oil bath where the temperature was raised at 120 °C per hour until they deflected 0.25 mm.

Vicat Softening Temperature

The vicat softening temperature is the temperature at which a flat-ended needle penetrates the specimen to the depth of 1 mm under a specific load. The temperature reflects the point of softening to be expected when a material is used in an elevated temperature application. Vicat softening temperature testing was done following ASTM D-1525. A test specimen was placed in the testing apparatus so that the penetrating needle rested on its surface at least 1 mm from the edge. A load of 10N was applied to the specimen. The specimen was then lowered into a silicon bath at 23 °C. The bath was raised at a rate of 50 °C per hour until the needle penetrated 1 mm.

4.5.5. Water Absorption

Water absorption is used to determine the amount of water absorbed under specified conditions. Factors affecting water absorption include type of plastic, additives used, temperature and length of exposure. The data sheds light on the performance of the materials in water or humid environments. Water absorption testing was done following ASTM D570 for the water absorption test, the specimen is dried in an oven for a specified time and temperature and then placed in a desiccator to cool. Immediately upon cooling, the specimens were weighed. The material was then submerged in water at agreed upon conditions, often 23 °C for 24 hours or until equilibrium. Specimens are removed, patted dry with a lint free cloth, and weighed every week. The dimension of sample was 4 mm thick. Water absorption was expressed as increase in weight percent as given in equation below ,

Percent Water Absorption = [(Wet weight - Dry weight)/ Dry weight] x 100

4.5.6. Aspect Ratio

The aspect ratio is defined as the ratio of length to width of bagasse can be determined by measuring the length and width of the fiber from SEM micrograph of bagasse. One hundred samples of bagasse were measured.

CHAPTER V

RESULTS AND DISCUSSION

The effects of bagasse sizes as well as types and loading of silane coupling agents on the mechanical, thermal, water absorption, and morphological properties of PVC/bagasse composites were investigated. The PVC/bagasse composite samples were prepared by a two-roll mill with both front and back roll at 195 °C for 5-6 min and compression molded at 170 °C and 150 psi for 4 min. The treatment of bagasse with silane coupling agents was done by spraying the silane solution onto the bagasse fibers, and mixing with a high-speed mixer for 5 min. Tension and flexural properties of PVC/bagasse composites were tested by universal testing machine and their impact property was tested by impact tester. Thermal properties such as heat distortion temperature and vicat softening temperature were tested by Vicat/HDT tester. In addition, the microstructure of impact fracture of the composites was observed by scanning electron microscope (SEM).

5.1 Fourier Transform Infrared Spectroscopy (FTIR)

Surface of composites having untreated and treated fibers were examined using FTIR spectroscopy to identify any possible chemical reaction occurring between untreated or treated bagasse and PVC matrix. FTIR spectroscopy is uniquely suited for studies of chemical reaction because absorbance peaks are characteristic of chemical bonds rather than of atoms.

Figure 5.1 shows the FTIR spectrum of the PVC dry blend, untreated bagasse and untreated fiber composite of 45-106 µm. Focusing on the PVC dry blend spectra, there are peaks at $700 - 800$ cm^{-1}, 1270 cm^{-1}, $1500 - 1600$ cm^{-1}, and $2950 - 3000$ cm^{-1} which represented the C–Cl, C–H, C=C, and CH$_2$ bonds, respectively. For untreated bagasse, there are peaks at $950 - 1000$ cm^{-1}, 1100 cm^{-1}, 1250 cm^{-1}, $1700 - 1950$ cm^{-1}, $2950 - 3000$ cm^{-1}, and $3200 - 3500$ cm^{-1} which represented the C–O, C–O–C, C–H, C=O, CH$_2$ and OH bonds. The last spectrum is PVC/bagasse composite showed the combined peaks of PVC dry blend and untreated bagasse.
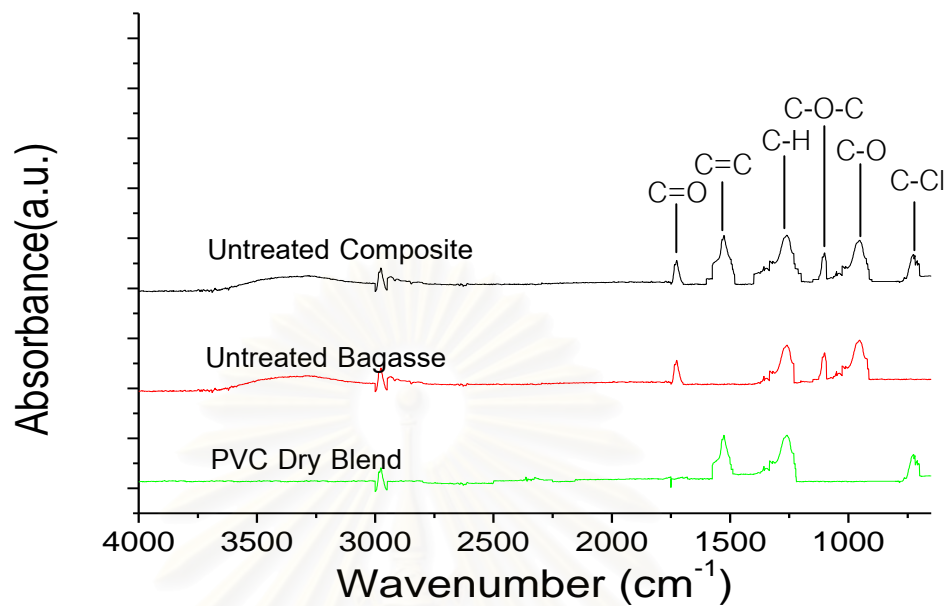
Figure 5.1: FTIR spectrum of PVC dry blend, untreated bagasse

and untreated composite size of bagasse 45-106 µm

When the bagasse was treated with different kinds of silane, amino 2%wt, diamino 2%wt, and epoxy 2%wt, the FTIR spectrum showed the different peaks. Figure 5.2 showed the spectrum of untreated and treated bagasse. The first one, treated bagasse with amino 2%wt showed the peaks that differ from untreated bagasse at 1000 – 1100 cm^{-1}, 1250 – 1350 cm^{-1}, 1400 – 1500 cm^{-1}, and 3100 – 3400 cm^{-1} which represents Si–O–Si or Si–O–C, C–N (amine), Si–C, and N–H(1°) bonds. The second one, treated bagasse with diamino 2%wt showed the peaks that differ from untreated bagasse at 1000 – 1100 cm^{-1}, 1250 – 1350 cm^{-1}, 1400 – 1500 cm^{-1}, 1500 – 1600 cm^{-1},

and 3100 – 3400 cm^{-1} which means Si–O–Si or Si–O–C, C–N (amine), Si–C, N–H(2°),

and N – H (1°) bonds. The last one, treated bagasse with epoxy 2%wt showed the

peaks that differ from untreated bagasse at 850 cm^{-1}, 1050 – 1150 cm^{-1}, 1000 – 1100

cm^{-1}, and 1400 – 1500 cm^{-1} which represents C–O–C (epoxy), C–O–C (ether), Si–O–Si or

Si–O–C, and Si–C bonds.
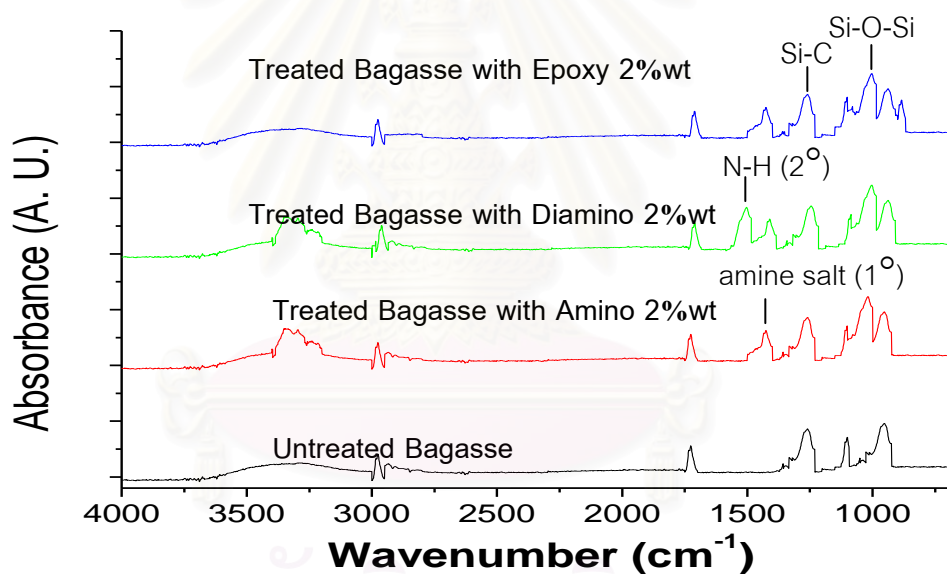
Figure 5.2: FTIR spectrum of untreated bagasse and treated bagasse with

amino, diamino and epoxy silane coupling agent at 2%wt silane

contents on 45-106 μm fiber

Figure 5.3 demonstrated the different peaks of FTIR spectrum of treated

bagasse composite and untreated composite. It can be seen that treated bagasse with

amino 1%wt, 2%wt, and 3%wt composite had different peaks from the untreated

composite at 1000 – 1100 cm^{-1}, 1250 – 1350 cm^{-1}, 1400 – 1500 cm^{-1}, 1600 – 1650 cm^{-1}

and 3100 – 3400 cm^{-1} which represents Si–O–Si or Si–O–C, C–N (amine), Si–C, amine

salt (1°) and N–H (1°) bonds.

Figure 5.3: FTIR spectrum of PVC/bagasse (45-106 μm) composites where

were by treated amino silane coupling agent at various contents

Figure 5.4 revealed the different peaks of FTIR spectrum of treated fiber composite and untreated composite. It can be seen that treated fiber with diamino 1% wt, 2%wt, and 3%wt composite had different peaks from the untreated composite at 1000 – 1100 cm^{-1}, 1250 – 1350 cm^{-1}, 1400 – 1500 cm^{-1}, 1500 – 1600 cm^{-1}, 1600 – 1650 cm^{-1} and 3100 – 3400 cm^{-1} which means Si–O–Si or Si–O–C, C–N (amine), Si–C, N–H (2°), amine salt (1°) and N–H (1°) bonds.
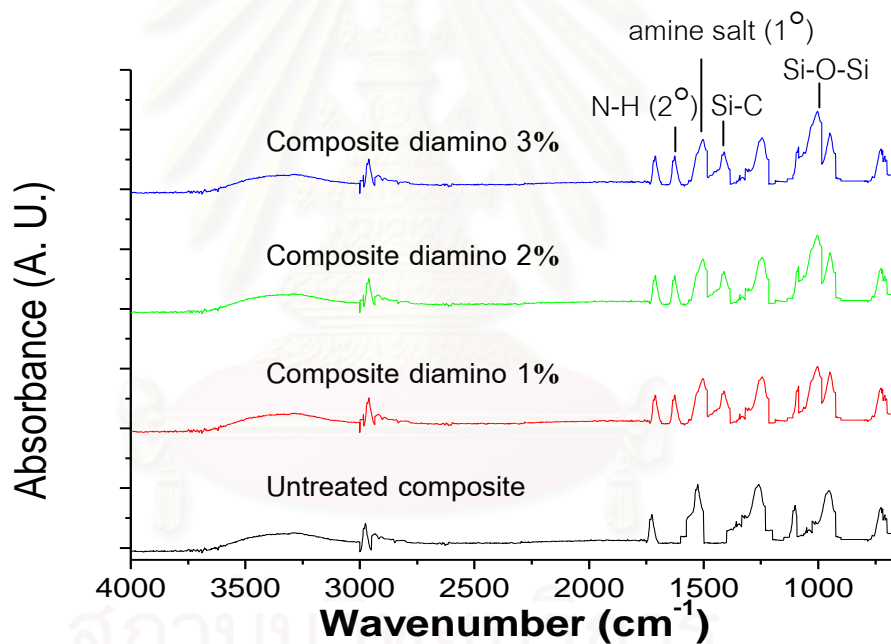
Figure 5.4: FTIR spectrum of PVC/bagasse (45-106 μm) composites where were by treated diamino silane coupling agent at various contents

Figure 5.5 showed the different peaks of FTIR spectrum of treated bagasse composite and untreated composite. It can be seen that treated bagasse with epoxy 1% wt, 2%wt, and 3%wt composite had different peaks from the untreated composite at 850 cm^{-1}, 1050 – 1150 cm^{-1}, 1000 – 1100 cm^{-1}, and 1400 – 1500 cm^{-1} which represents C–O–C (ether), Si–O–Si or Si–O–C, and Si–C bonds.

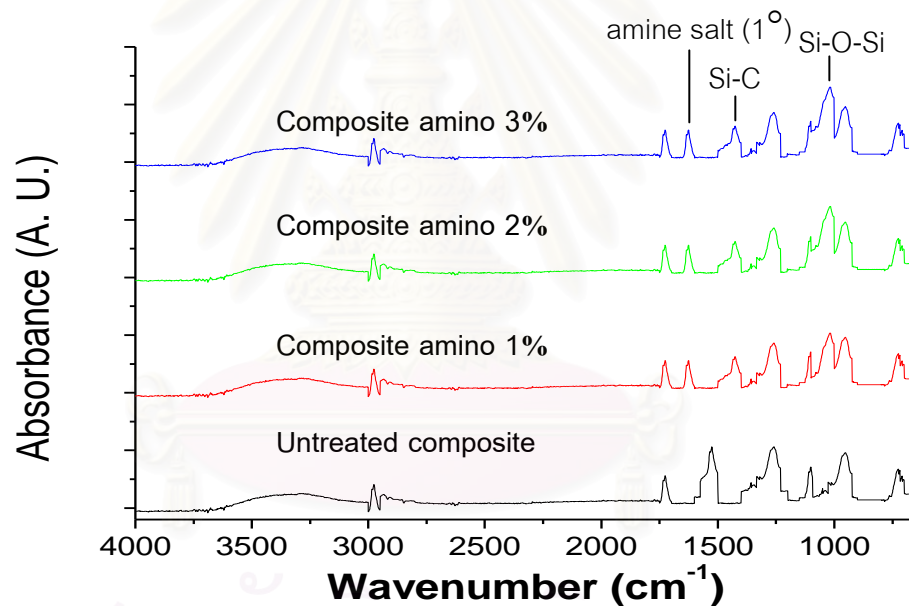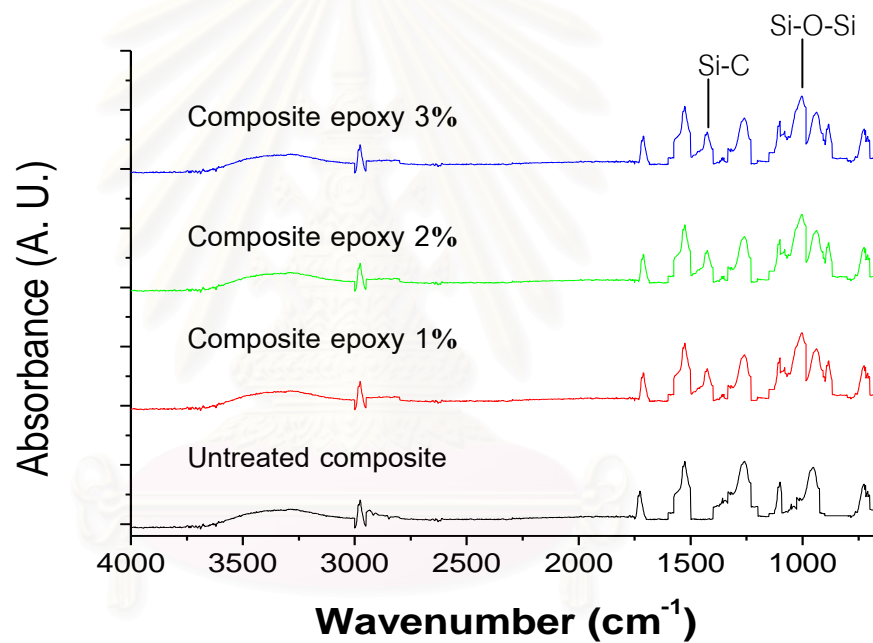**Figure 5.5**: FTIR spectrum of PVC/bagasse (45-106 μm) composites where were by treated epoxy silane coupling agent at various contents

5.2 Effect of Treated Fiber on Change in Surface of PVC/Bagasse Composites

SEM micrographs of the surface of PVC/bagasse composites at bagasse content of 60 phr which showed the best properties, particle size of 45-106 μm and 106-180 μm. The SEM micrographs of Figures 5.6 (a) and 5.6 (e) reveals that the surface of PVC/bagasse composites at 45-106 μm and 106-180 μm and Figure 5.6 (b)-5.6 (d) and Figure 5.6 (f) - 5.6 (h) show that the surface of PVC/treated bagasse with amino, diamino and epoxy silane coupling agent (2% wt of bagasse) composites at 45-106 μm and 106-180 μm, respectively. In PVC/bagasse composites, swelling of bagasse particles after absorbing and desorbing of moisture apparently resulted in voids at the interface between PVC and bagasse in the composites. It can be seen that untreated composite in Figure 5.6 (a) and (e) have void more than treated composite in Figure 5.6 (b) - 5.6 (d) and 5.6 (f) - 5.7 (h) . The suggested that fibers treated with the silane coupling agents could bind with PVC matrix than untreated fiber.

a)

b)

c)

e)

f)
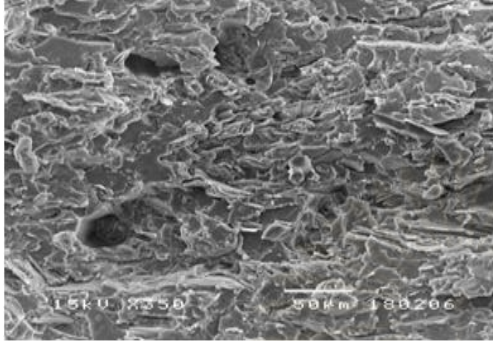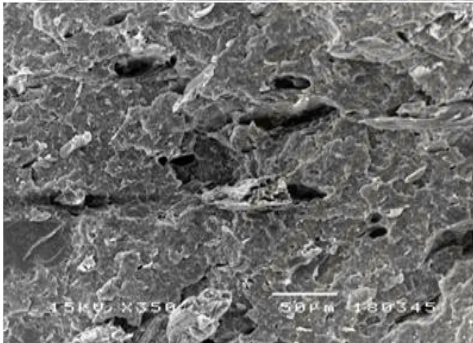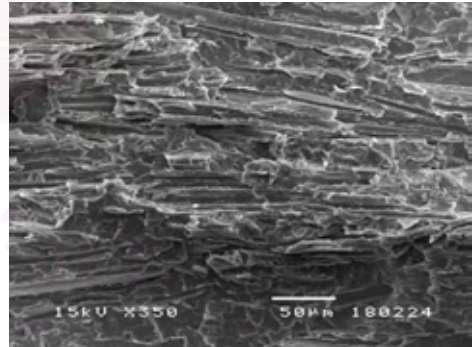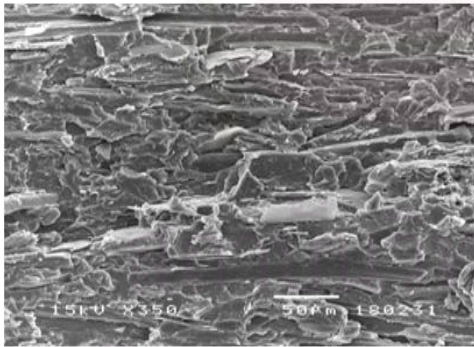
g)

Figure 5.6: SEM micrographs of surface of PVC/bagasse composites:

(a) composite with untreated fiber size 45-106 µm.

(b) composite with treated fiber size 45-106 µm by amino silane coupling

agent at content 2%wt.

(c) composite with treated fiber size 45-106 µm by diamino silane coupling

agent at content 2%wt.

(d) composite with treated fiber size 45-106 µm by epoxy silane coupling

agent at content 2%wt.

(e) composite with untreated fiber size 106-180 µm.

(f) composite with treated fiber size 106-180 µm by amino silane coupling

agent at content 2%wt.

(g) composite with treated fiber size 106-180 µm by diamino silane coupling

agent at content 2%wt.

(h) composite with treated fiber size 106-180 µm by epoxy silane coupling

agent at content 2%wt.

5.3 Mechanical Properties

The measurement of mechanical properties in this research were tensile

properties, flexural properties and impact strength. It can be noted from the results that

bagasse sizes as well as types and loading of silane coupling agent have an effect on

mechanical properties of the PVC/bagasse composites.

In this chapter, there were specific terms which are commonly mentioned

throughout this study. In order to have a mutual understanding and avoid

misconception, these terms were clarified as follows:

● Small Size in this study refered to the bagasse size which is in the range 45-106 µm.

● Large Size in this work refers to the bagasse size which is in the range 106-180µm.

- Amino silane coupling agent was γ-aminopropyltriethoxysilane.

- Diamino silane coupling agent was N-(β-aminoethyl)-γ-aminopropyltrimethoxysilane.

- Epoxy silane coupling agent was glycidoxypropyltrimethoxysilane.

- Composite amino was composite in which bagasse was treated with amino silane coupling agent.

- Composite diamino was composite in which bagasse was treated with diamino silane coupling agent.

- Composite epoxy was composite in which bagasse was treated with epoxy silane coupling agent.

5.3.1 Effects of Bagasse on Mechanical Properties of PVC/Bagasse Composites

5.3.1.1 Tensile Property

The effect of bagasse loading and bagasse size on tensile properties of PVC/bagasse composites were studied by using a universal testing machine. The effect of bagasse content at 60 phr on tensile properties of PVC/bagasse composites were illustrated in Figures 5.7 and 5.8. At 60 phr of bagasse, tensile strength of PVC/untreated bagasse composite with small or large fiber sizes were 42 MPa while tensile strength of PVC dry blend was 51 MPa. Tensile strength of the PVC/bagasse composites was found to decrease with adding bagasse content 60 phr which was possibly caused by a

number of reasons including: (i) poor dispersion of the fiber in the matrix, (ii) moisture pick-up causing the reduction in physical bonding between the fiber and the matrix or acting as a lubricant between two phases, and (iii) increase of interfacial defects and debondings (Maldas, 1989). The reduction in tensile strength was generally observed when fibers were added into polymer matrix.

The effect of bagasse size on tensile strength of PVC/bagasse composites was shown in Figures 5.7 and 5.8. From these figures, the mechanical properties of PVC/bagasse composites with small or large fiber sizes were found to be about the same. Although there was a report that the small-sized fiber had higher specific area than that of large-sized fiber. The small-sized bagasse could be better dispersed in PVC matrix, thus, the composites with small-sized bagasse showed less voids between fibers and PVC matrix than composites with large-sized bagasse as can be seen Figure 5.6 . It is consistent with Zurale etal s work (Zurale, 1998). Composite with 60 phr of small-sized bagasse showed 0.6% higher tensile strength than that with large-sized bagasse. Therefore, the small-sized fiber could better reinforce composites. This behavior may attribute to the reinforcing effect of the bagasse particle on the PVC matrix. The added bagasse particles into polar polymer can improve the mechanical properties of composites (Matuana et al., 1998). The bagasse possesses a lot of hydroxyl group on its surface, that forming some hydrogen bonding with hydroxyl group and polar group of PVC matrix. Therefore, the compatibility between bagasse and PVC

was enhanced by the hydrogen bonding; the surface of treated bagasse was also improved by the physical bonding between filler and matrix (Maldas et al., 1989). It can be concluded that sizes of bagasse used in this study were not significantly different enough from each other to have different effect on the tensile strength of composites.

The effect of types and contents of silane coupling on the tensile strength of PVC/bagasse composite were shown in Figures 5.7 and 5.8. For bagasse treated with amino and diamino silane coupling agents, the tensile strength of PVC/bagasse composites increased with increasing content of silane coupling agents up to 2%wt because low concentration of silane coupling agent had high monomeric silanols inwhich the hydroxyl group (-OH) of silanol could react with hydroxyl group on bagasse surface (Dalväg et., 1985). This caused interfacial covalent bonds. And the amine group ($-NH_2$) of silane coupling agent could react with PVC matrix to increase the bondings at the interface and ,thus, the property of composites improved. On the other hand, the tensile strength rapidly decreased when the content of silane coupling agents was greater than 2%wt. this might be because of a self-condensation reaction of silane coupling agents to polysiloxanes on bagasse surface and these polysiloxanes obstructed the bonding of PVC matrix with amine group in the silane coupling agent.

In addition, the increase of tensile strength of PVC/bagasse composites with amino silane coupling agent was greater than that with diamino silane coupling agent because diamino silane coupling agent had longed chain which can cause

strength steric effect. While the tensile strength of PVC/bagasse composites with epoxy silane coupling agent increased only slightly when increasing the content of silane coupling agents in composites was increased up to 1%wt and decreased when the content of silane coupling agents was greater than 1%wt.
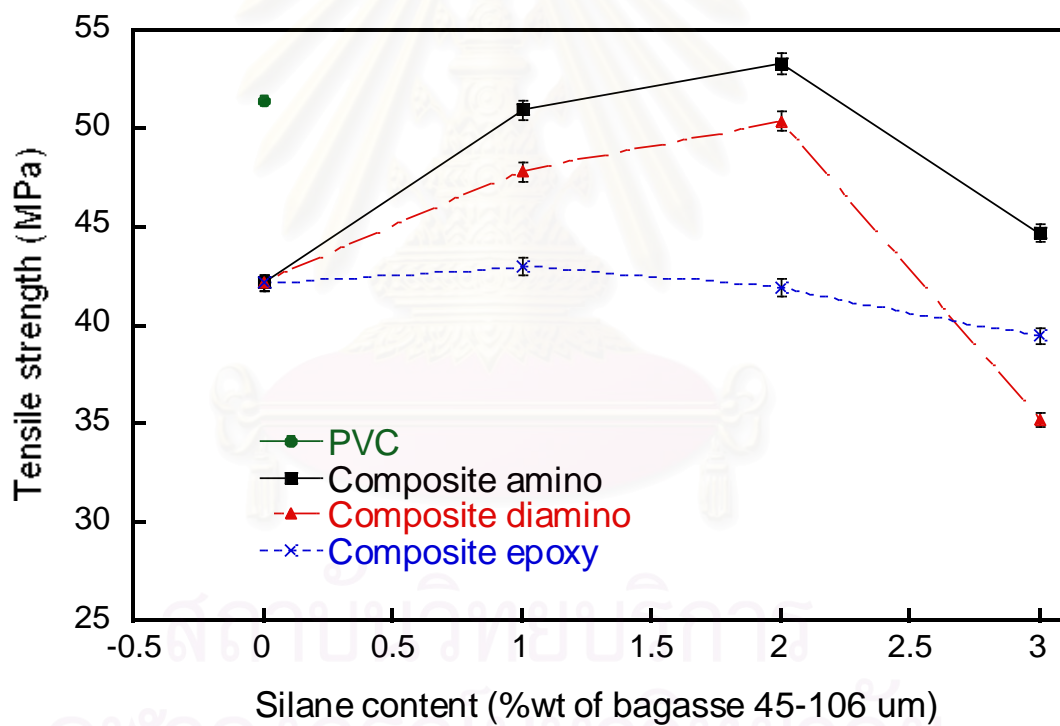
Figure 5.7: The tensile strength of PVC/bagasse composites at various

types and contents of silane treated on 45-106μm fiber
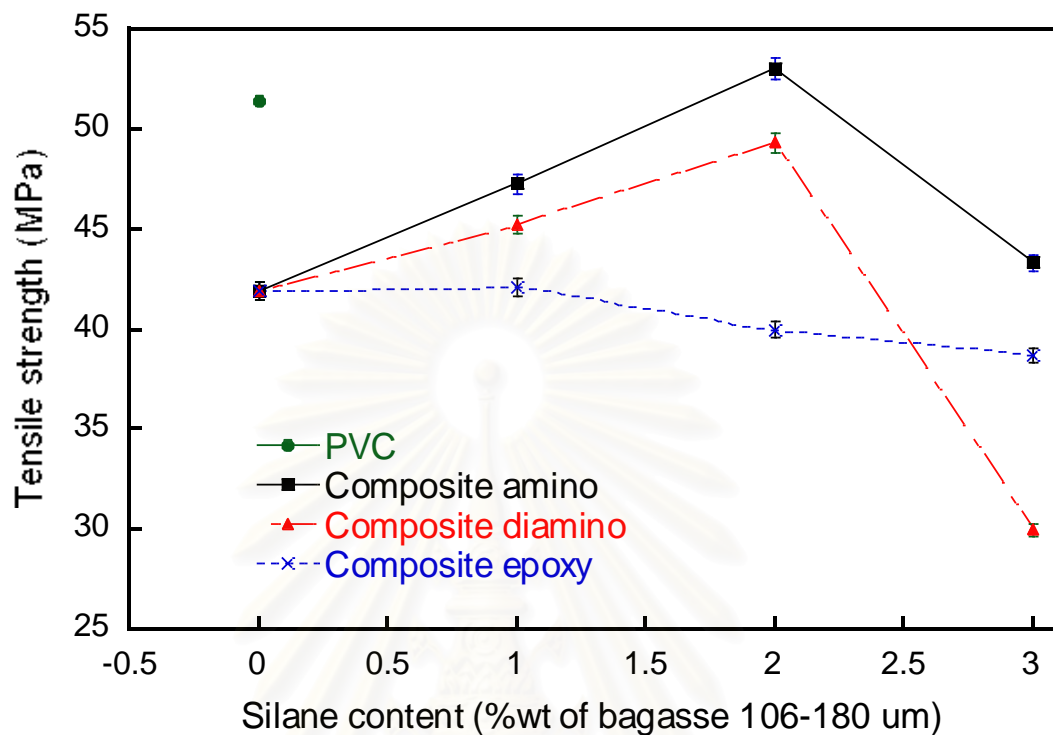
Figure 5.8 The tensile strength of PVC/bagasse composites at various

types and contents of silane treated on 106-180 µm fiber

The addition of bagasse in PVC caused the composite to be stiffer than

PVC dry blend because fiber was reinforcing filler and so the increase of tensile

modulus of composite occurred. These results were shown in Figures 5.9-5.10. They

showed that the addition of bagasse in PVC could raise the tensile modulus. At 60 phr of

PVC/untreated bagasse composite with 106-180 µm fiber, the tensile modulus of

composite was 4.08 GPa with 45-106 µm fiber and 3.83 GPa while tensile modulus of

PVC dry blend was 2.79 GPa. Moreover, the tensile modulus of PVC/bagasse

composites was found to increase with the addition of bagasse content 60 phr owing to the rigid bagasse particles which were able to reinforce the PVC matrix. This was caused by the fact that the natural fiber is more rigid than the polyvinylchloride matrix (Matuana, 2001). It should be noted that , tensile modulus of composites with different fiber size were slightly different as compared in Figure 5.9 and 5.10. For example, tensile modulus of composites with 60 phr of small-sized bagasse was 4.08 GPa while thiat value of composites with 60 phr of large-sized bagasse was 3.83 GPa.

Figures 5.9 and 5.10 also showed the effect of types and content of silane on the tensile modulus of PVC/bagasse composite. For composites with bagasse treated with either amino or diamino silane coupling agent, the modulus increased with increasing content of silane coupling agents up to 2%wt and decreased 5% when the content of silane coupling agents was greater than 2%wt. However, the tensile modulus of PVC/bagasse composites with bagasse treated with epoxy silane coupling agent decreased whith increasing the content of silane coupling agents in composites with fiber size 45-106 μm and slightly increased when increasing the content of silane coupling agents in case of with 106-180 μm fiber up to 1%wt and decreased. The increase in the tensile modulus at small silane coupling agent concentrations (0.0 - 2.0 %wt) was associated with ether linkages formed by the chemical reactions of the silane coupling agent and bagasse particles and N-C linkages between the amino groups in the silane coupling agent and the PVC in the Figures 5.1-5.4 (Maldas and

Kokta, 1991). On the other hand, the decrease in the tensile modulus at higher silane coupling agent concentrations may have been caused by a self-condensation reaction of the hydrolyzed coupling agents on the surfaces of the bagasse. The main product of the self-condensation reaction formed flexible polysiloxynol molecules on the bagasse surfaces (Maldas and Kokta, 1991).
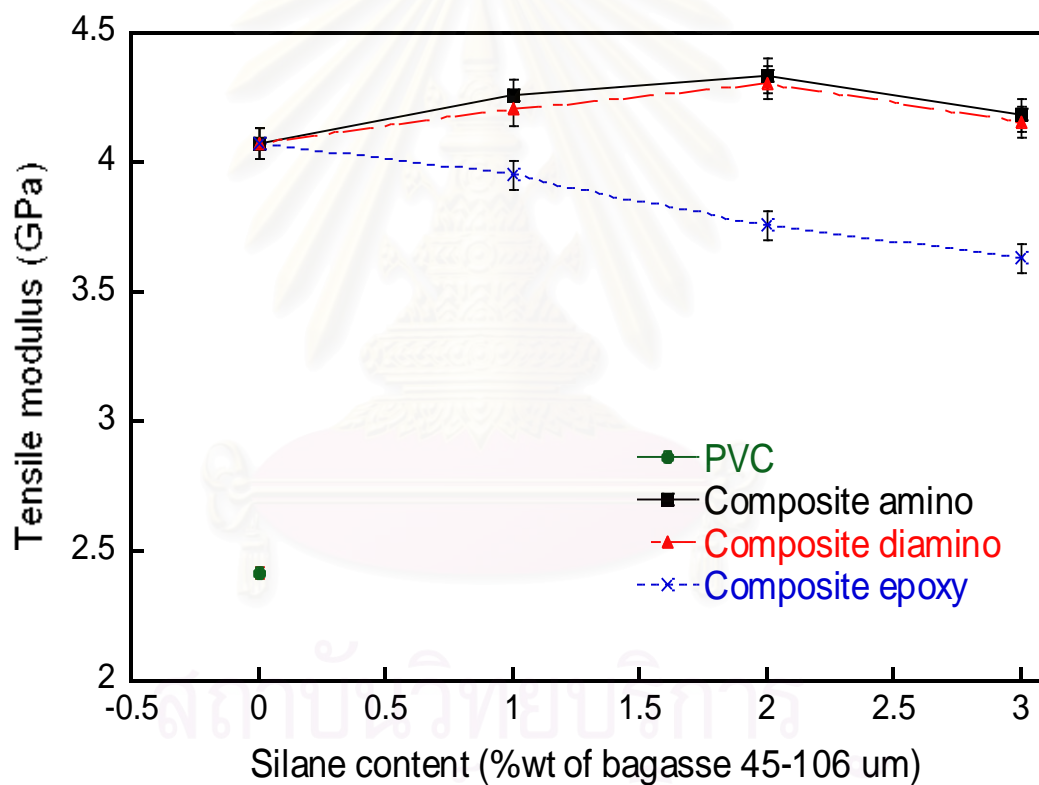
Figure 5.9: The tensile modulus of PVC/bagasse composites at various types and contents of silane treated on 45-106 μm fiber
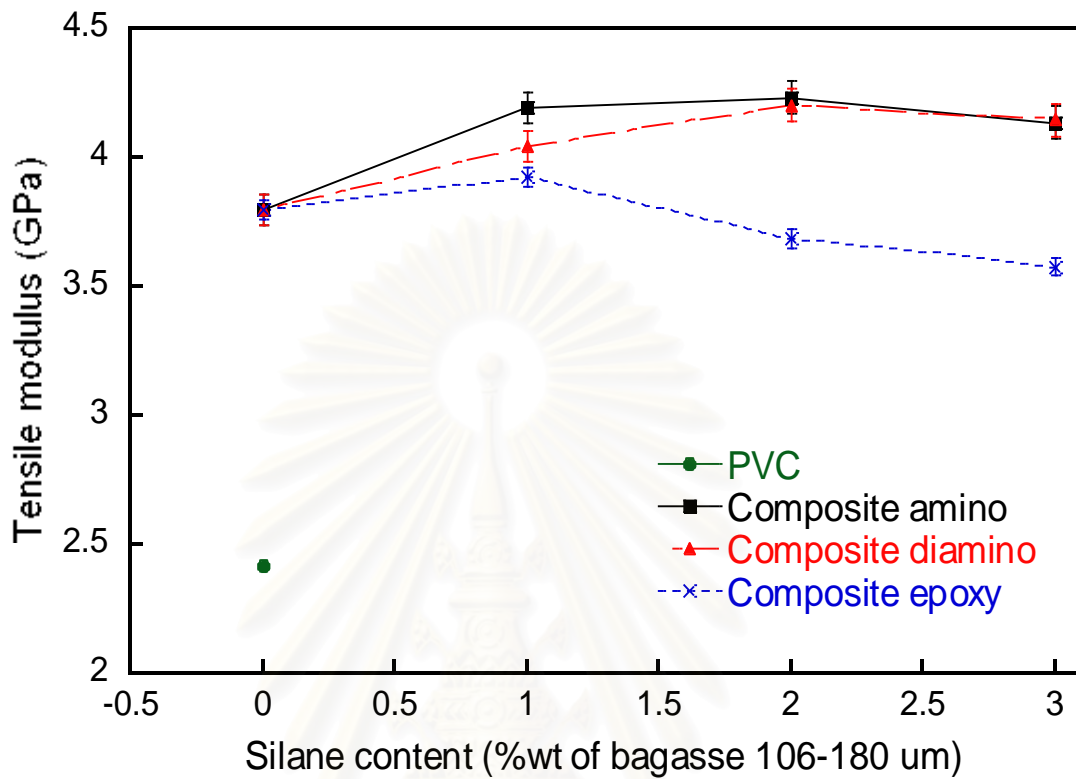
Figure 5.10: The tensile modulus of PVC/bagasse composites at

various types and contents of silane treated on 106-180 μm

fiber

The elongation at break of PVC/bagasse composites was found to

decrease considerably with the presence of bagasse from the neat PVC. These results

were shown in Figures 5.11 and 5.12. This considerable decrease of the elongation at

break of PVC/bagasse composites with the presence of bagasse and slightly decrease

with further adding bagasse was probably caused by the presence of the rigid bagasse

filler in the PVC matrix and the low interfacial interaction between the PVC matrix and the rigid bagasse as discussed in the tensile strength of PVC/bagasse composites. This low interfacial adhesion typically led to the debonding and the initiation of void formation or cracks at the composite interface thus the low elongation at break of the specimens (Matuana, 1998). Figures 5.11 and 5.12 also showed the effect of types and content of silane on the elongation at break of PVC/bagasse composites. The elongation at break of PVC/bagasse composites was lower than that PVC dry blend. The elongation at break of composite with bagasse treated with amino or diamino silane coupling agent reduced with increasing content of silane coupling agents from 0-2%wt. On the other hand, the elongation at break increased 36% when the content of silane coupling agents was greater than 2%wt. For of PVC/bagasse composites with bagasse treated with epoxy silane coupling agent, the elongation at break decreased with increasing content of silane coupling agents in from 0-1%wt and increased when increasing the content of silane coupling agents was greater than 1%wt.
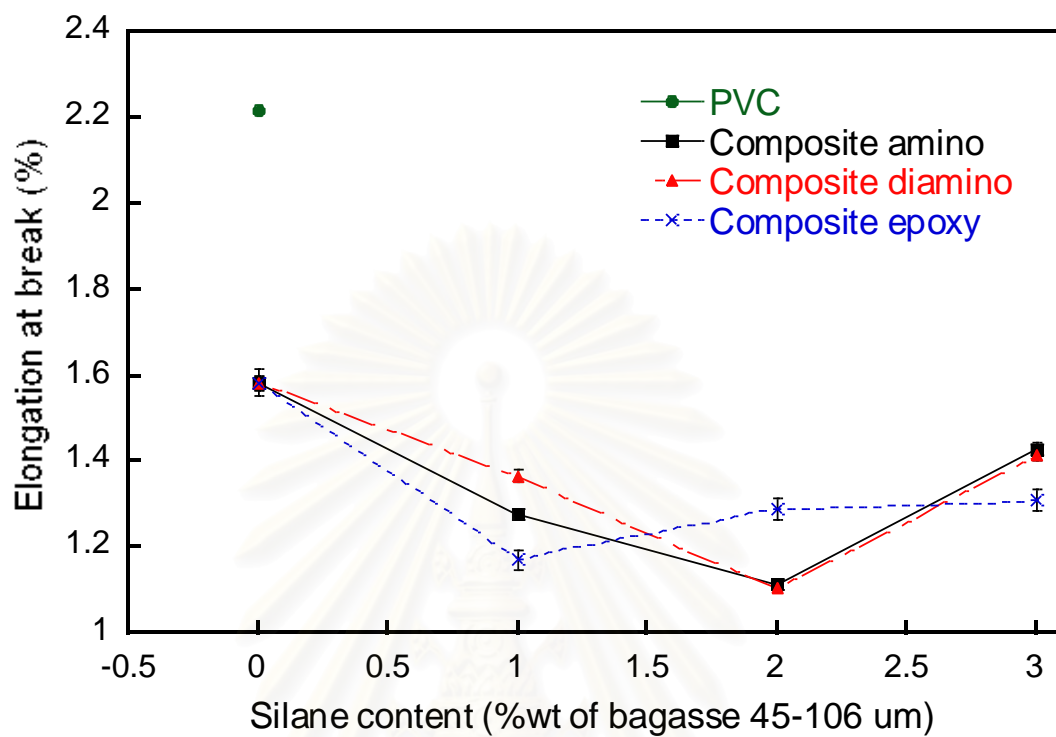
Figure 5.11: The elongation at break of PVC/bagasse composites at

various types and contents of silane treated on 45-106 μm
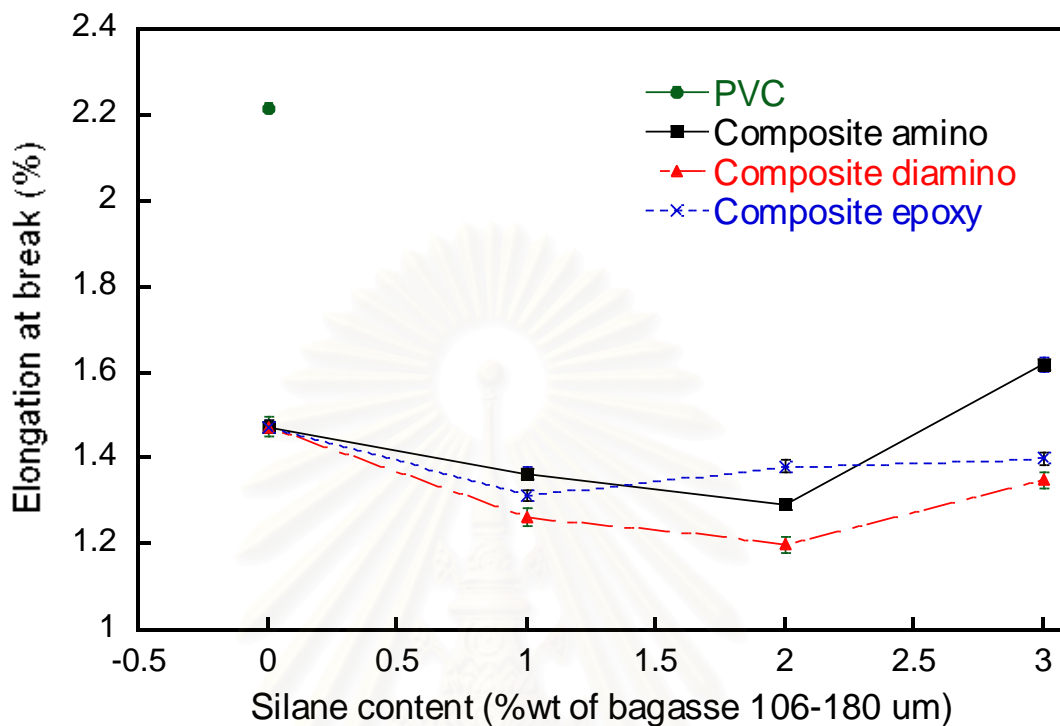
fiber

Figure 5.12: The elongation at break of PVC/bagasse composites at

various types and contents of silane treated on 106-180 µm

fiber

5.3.1.2 Flexural Properties

The effect of bagasse on the flexural strength of the composites tended

to be similar to that of tensile strength as discussed earlier. The composites showed the

decrease of flexural strength as shown in Figures 5.13 and 5.14. Figures 5.15 and 5.16

showed the increment of flexural modulus. From Figures 5.13 and 5.14, at 60 phr of

bagasse, flexural strength of PVC/bagasse composite was 66 MPa with fiber size 45-106 μm and 65.2 MPa with fiber size 106-180 μm while flexural strength of PVC dry blend was 79.3 MPa. The flexural strength of the PVC/bagasse composites was found to decrease with adding the bagasse content 60 phr. The reasons of decrease in strength of PVC/bagasse composites included moisture pick-up in bagasse, poor dispersion of the bagasse in PVC matrix, and increase of interfacial defects or debonding between polymer and bagasse (Sannadi et al., 1999). The flexural strength of the PVC/bagasse composites was found to decrease with increasing the content of silane coupling agent greater than 2%wt as can be seen in Figures 5.13 and 5.14. The effect of silane coupling agent content on the flexural strength of the PVC/bagasse composites tended to be similar to that of tensile strength as discussed earlier. However, the flexural modulus of the PVC/bagasse composites was found to slightly increase with adding the bagasse. The bagasse particles, with greater rigidity, were able to reinforce the PVC matrix with improvement in the modulus value. As expected, the addition of the bagasse has similar effect on enhancement of both the flexural modulus and the tensile modulus of PVC/bagasse composites due to the reasons described previously. The PVC/bagasse composites were stiffened by filled bagasse in PVC matrix because filler is more rigid than the PVC matrix (Maldas and Kokta, 1991). From Figure 5.15 and 5.16, the flexural modulus of the PVC/bagasse composites rose with increasing the content of silane coupling agents up to 2%wt and decreased rapidly when the content of silane coupling

agents was greater than 2%wt because of the self-condensation reaction of silane coupling agent. This may be caused by polysiloxane on bagasse surface as obstruct of bonding of PVC matrix reacted with amine group of silane coupling agent (Sombatsompop and Chaochanchaikul,2005). Surface modification composite properties did not different from the non–surface modified composite because organofunctional group of silane coupling agent did not react with the polymer matrix.

The effects of bagasse size on flexural property of PVC/bagasse composites were shown in Figuress 5.13-5.16. It could be noticed from Figures 5.13 and 5.14 that flexural strength of composites with small-sized bagasse has higher than that with large-sized bagasse. Composite with 60 phr of small-sized bagasse shows 1.6% higher flexural strength than that with large-sized bagasse. Therefore, the small sized fiber could better reinforce composites. This effect was supported by SEM micrographs as shown in Figure 5.7. As a result, the tensile and flexural modulus of PVC/bagasse composites was decreased with increasing bagasse size. However, size of bagasse employed in this study were not significantly affect the flexural modulus of composites.

Figure 5.13: The flexural strength of PVC/bagasse composites at

various types and contents of silane treated on 45-106 μm

fiber
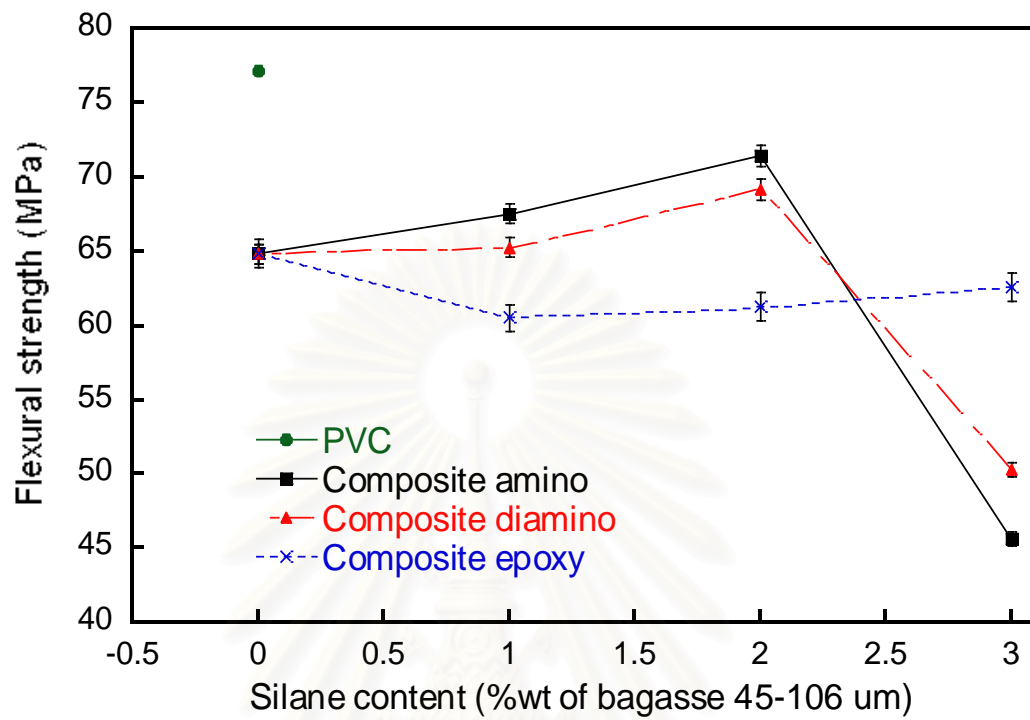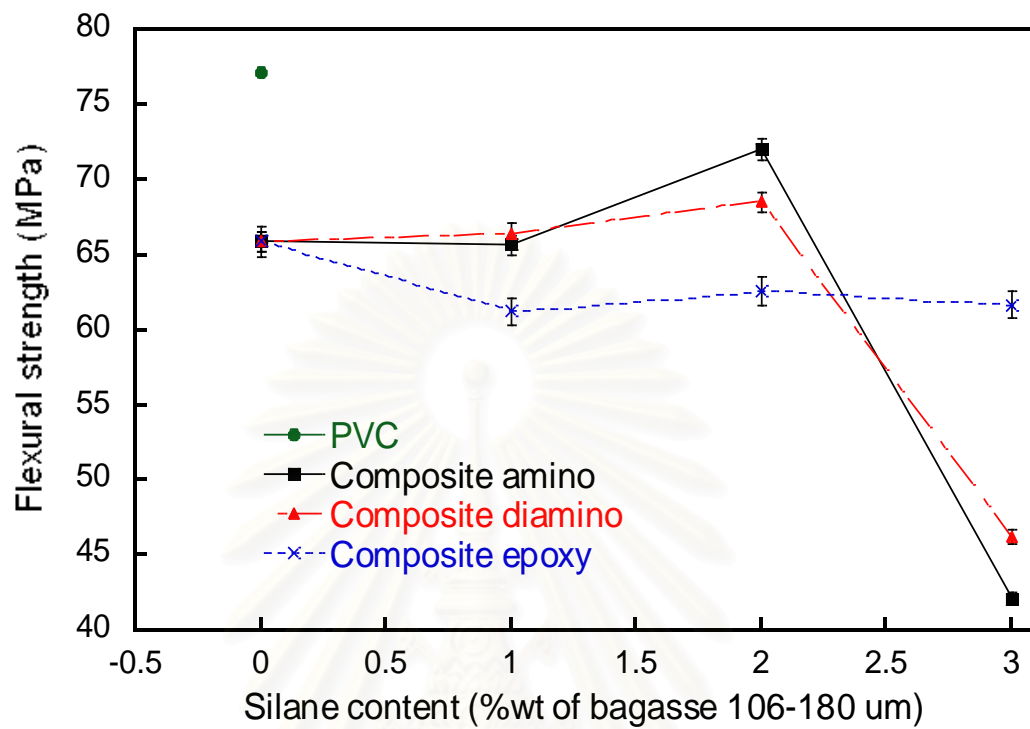
Figure 5.14: The flexural strength of PVC/bagasse composites at

various types and contents of silane treated on 106-180 μm

fiber

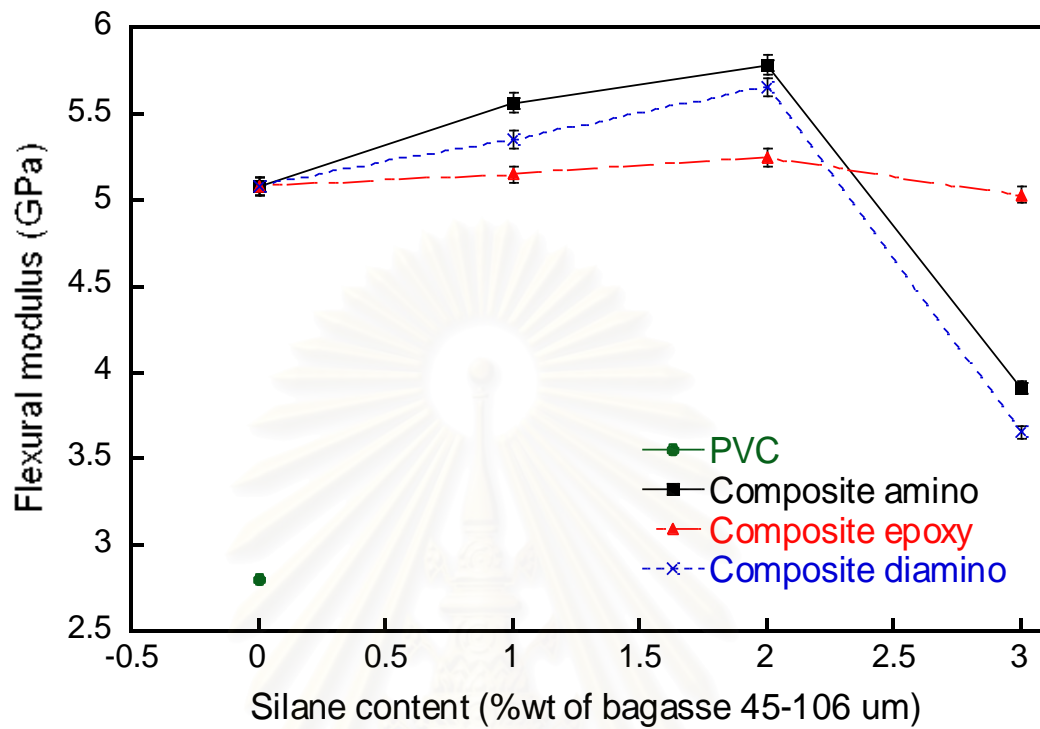**Figure 5.15:** The flexural modulus of PVC/bagasse composites at

various types and contents of silane treated on 45-106 µm
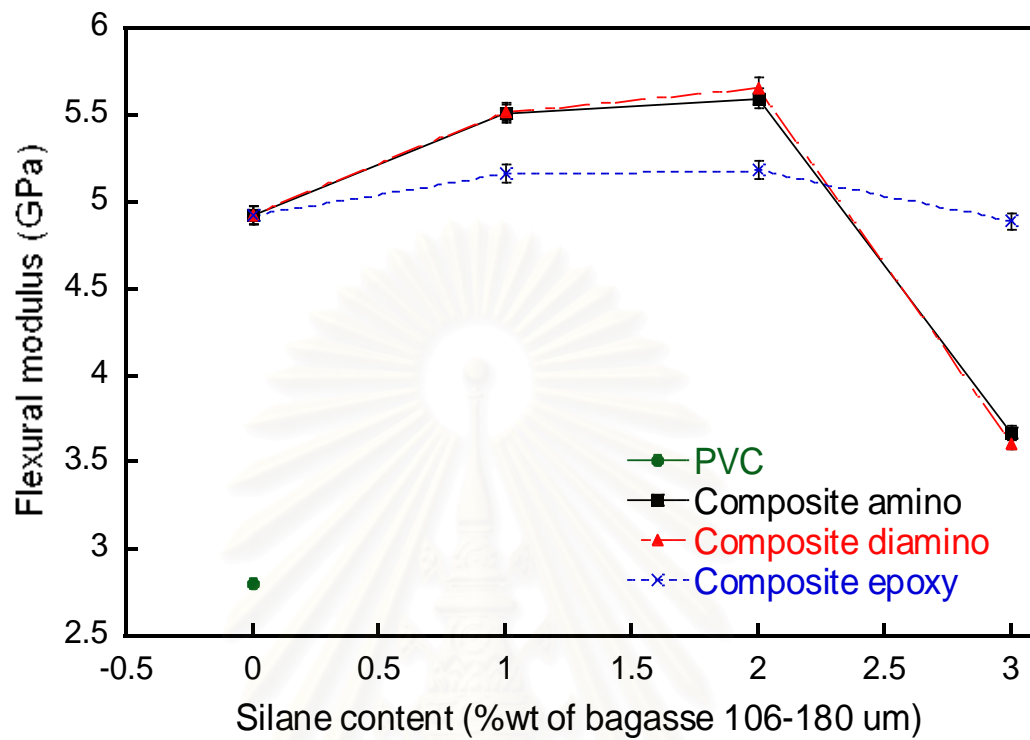
fiber

Figure 5.16: The flexural modulus of PVC/bagasse composites at

various types and contents of silane treated on 106-180 µm

fiber

5.3.1.3 Impact Properties

The Izod impact strength of the PVC/bagasse composites was found to decrease with adding the bagasse. The notch acts as a crack initiator; therefore, the notched Izod impact test is a measure of crack propagation. From Figure 5.17 and 5.18, the impact strength of PVC/bagasse composites were lower than that of PVC dry blend. At 60 phr of bagasse, impact strength of PVC/bagasse composite was 3.90 kJ/m^2 with fiber size 45-106 μm and 3.92 kJ/m^2 with fiber size 106-180 μm while impact strength of PVC dry blend was 7.16 kJ/m^2. As the fiber reinforced the matrix causing the composites to be stiffer, the ductile portion of PVC was reduced, thus, the toughness of composite was decreased (Yu-Tao, 2007). Moreover, the composites showed more pores than pure PVC so it took less energy to break composites. The decrease in impact strength was probably due to the dilution effect. Because the bagasse content was increased, the ductile portion (PVC matrix) automatically reduced, thus decreasing the composite's toughness (Zadorecki, P., and P. Flodin, 1985). The decrease of impact strength of PVC/bagasse composites was usually derived from debonding and friction effects, especially for the composites when no fiber treatments were applied. It can be seen that the presence of bagasse greatly reduced the toughness of the PVC/bagasse composites with an increase in the composite hardness. The results on Izod impact

strength of PVC/bagasse composites corresponded to these on tensile and flexural strengths. The same reasons of strength involve increases of interfacial defects and poor dispersion between PVC and bagasse.

The effect of bagasse size on impact property of PVC/bagasse composites was shown in Figures 5.17 and 5.18. They showed that size of bagasse did not have significant effect on this property. Impact strength of composites with 60 phr of small-sized bagasse was 3.90 kJ/m^2 while this value of composites with 60 phr of large-sized bagasse was 3.92 kJ/m^2. This might be due to sizes of fiber were not different enough to change the energy that composites absorbed before breaking (Maldas et al., 1989). From Figures 5.17 and 5.18, the impact strength of the PVC/bagasse composites was not significantly affected the types and contents of silane coupling agents.
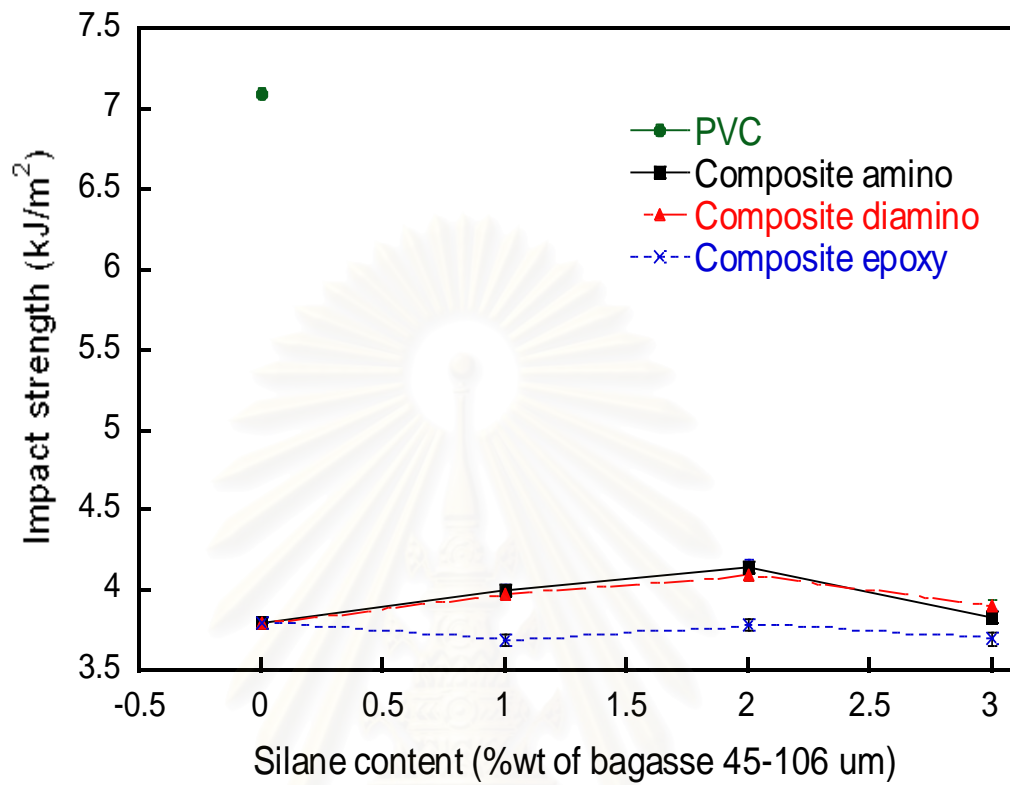
Figure 5.17: The Izod impact strength of PVC/bagasse composites at

various types and contents of silane treated on 45-106μm
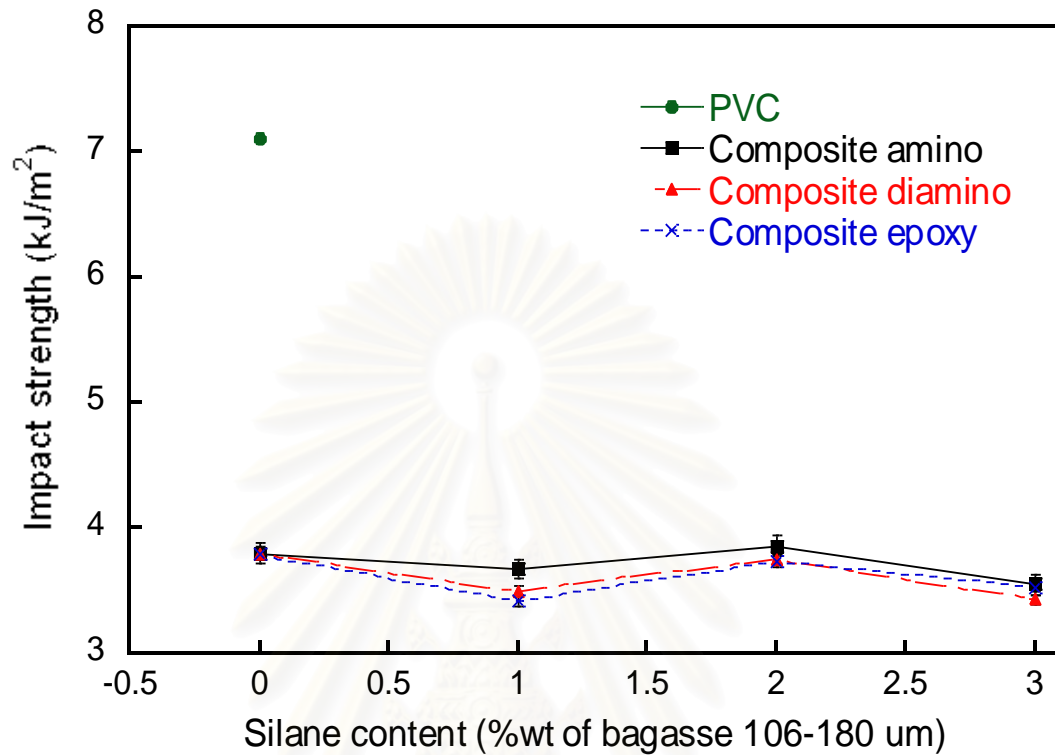
fiber

Figure 5.18: The Izod impact strength of PVC/bagasse composites at

various types and contents of silane treated on 106-180 μm

fiber

5.4 Thermal Properties

In this research, the measurement of the thermal properties consisted of heat distortion temperature and vicat softening temperature. It can be observed from the results that bagasse size, types and loading of silane coupling agent influenced these properties of the composites.

5.4.1 Effects of Fiber Size, Types and Loading of Silane Coupling Agents on Heat Distortion Temperature (HDT) of PVC/Bagasse Composites

The effect of bagasse contents is 60 phr on heat distortion temperature (HDT) of PVC/bagasse composites are shown in Figures 5.19 and 5.20. From these figures, the heat distortion temperature of the PVC dry blend was determined to be 71°C and also slightly increased with the bagasse content to approximately 74°C at the 60 phr of the bagasse. Moreover, bagasse is reinforcing filler so it makes composites stiffer than PVC dry blend (Yu-Tao et al., 2007). Therefore, the more fiber content in PVC matrix is, the better the composites can withstand heat before distortion. From the Figures 5.19 and 5.20, the heat distortion temperature of the PVC/bagasse composites was not significantly affect the types, contents of silane coupling agent and bagasse sizes.

Figure 5.19: The heat distortion temperature of PVC/bagasse composites

at various types and contents of silane treated on 45-106 μm

fiber

Figure 5.20: The heat distortion temperature of PVC/bagasse composites

at various types and contents of silane treated on 106-180 μm

fiber

5.4.2 Effects of Fiber Size, Types and Loading of Silane Coupling Agents on Vicat Softening Temperature of PVC/Bagasse Composites

The effect of bagasse contents is 60 phr on vicat softening temperature of PVC/bagasse composites are shown in Figures 5.21 and 5.22. From these Figures, the vicat softening temperature of PVC/bagasse composites were found to increase with adding bagasse content. As shown in this figure, the vicat softening temperature of the PVC dry blend was determined to be about 74°C and the value was slightly increased to about 79°C at the bagasse content of 60 phr. Moreover, fiber added in PVC matrix slightly increased vicat softening temperature of composites. With the addition of bagasse, it seems that the enhanced rigidity of PVC/bagasse composites, which affected on vicat softening temperature. These effects could be explained that the addition of fiber increased the stiffness of composites due to cellulose and hemicellulose. From the Figures 5.21 and 5.22, the vicat softening temperature of the PVC/bagasse composites was not significantly affect the types, contents of silane coupling agent and bagasse sizes.
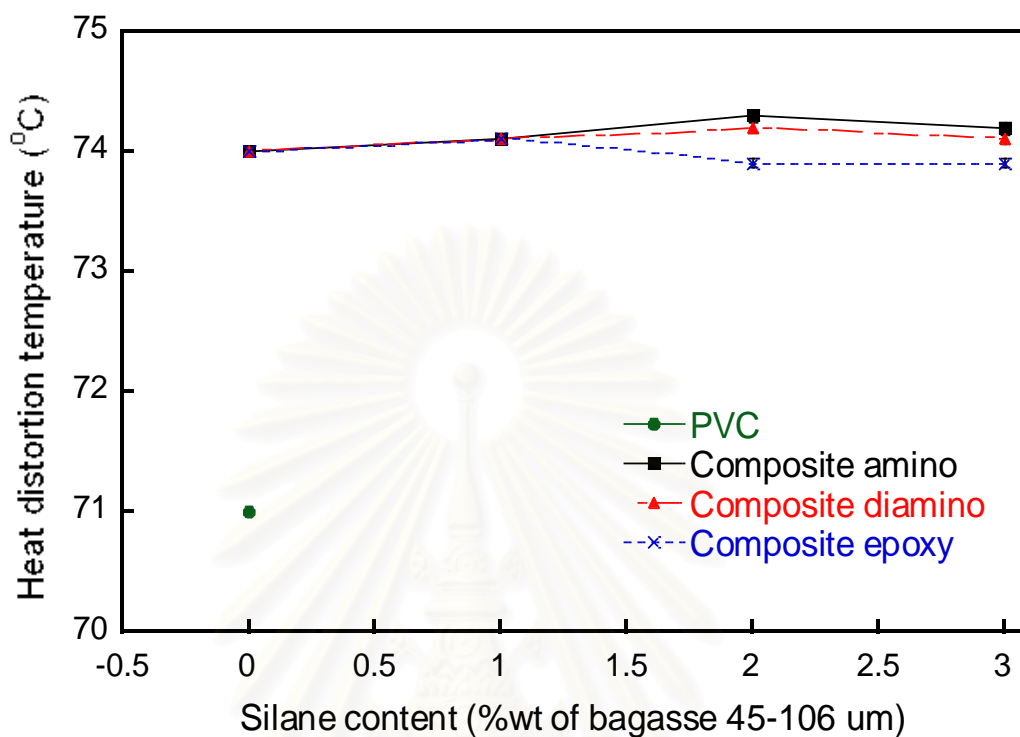
Figure 5.21: The vicat softening temperature of PVC/bagasse composites

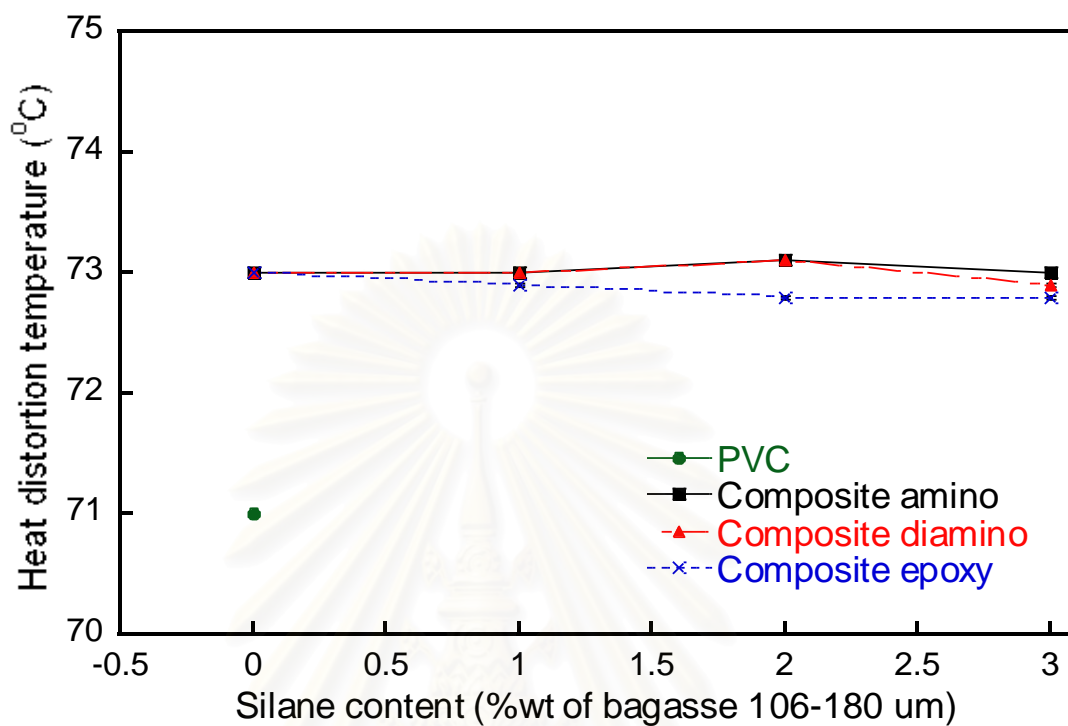at various types and contents of silane treated on 45-106 μm

fiber

Figure 5.22: The vicat softening temperature of PVC/bagasse composites

at various types and contents of silane treated on 106-180 μm

fiber

5.5 Water Absorption

The PVC/bagasse composites were shown in Figures 5.23 and 5.24. They showed that the addition of bagasse increases water absorption of composites. This occurred because fiber is a hydrophilic material. Moreover, with an addition of bagasse into the PVC matrix, the gaps between the two phases would raise owing to the poor interaction between matrix and bagasse. At bagasse size 45-106 µm, water absorption of PVC/bagasse composite was 6.59% and 6.96% at bagasse size 106-180 µm while water absorption of PVC dry blend was 0.32%. The results show that increase particle sizes of bagasse are being made to increase water absorption of PVC/bagasse composites. It is possible to observe that the water absorption is always slightly higher for larger particle size (i.e. 106-180 µm). This is contradiction, because lager surface area of particles should be expected at lower particle sizes (i.e. 45-106 µm) and consequently, higher availability of OH groups coming from cellulose that could absorb water. This discrepancy could be attributed to the general higher tendency of agglomeration of particles of small size. The water absorption of PVC/bagasse composites decreased with increasing the content of silane coupling agents up to 2%wt and decreased slightly when the content of silane coupling agents was greater than 2%wt. This may cause an interfacial covalent bonds. While amine group ($-NH_2$) of silane coupling agent react with PVC matrix can improve the property of composites.
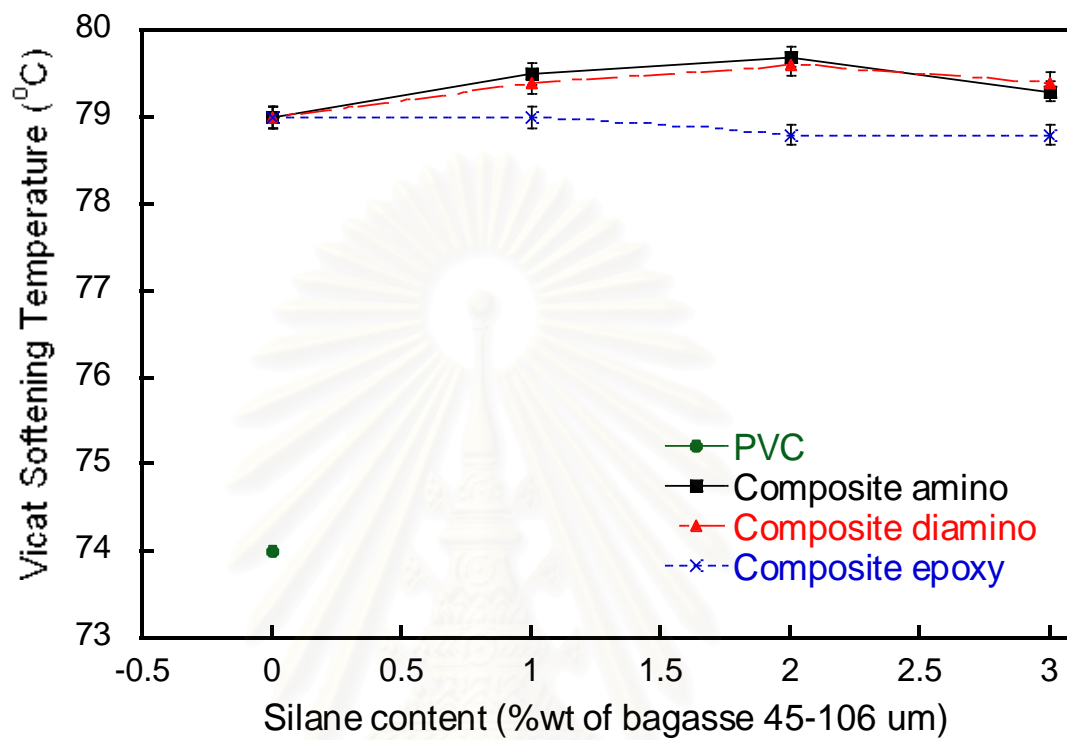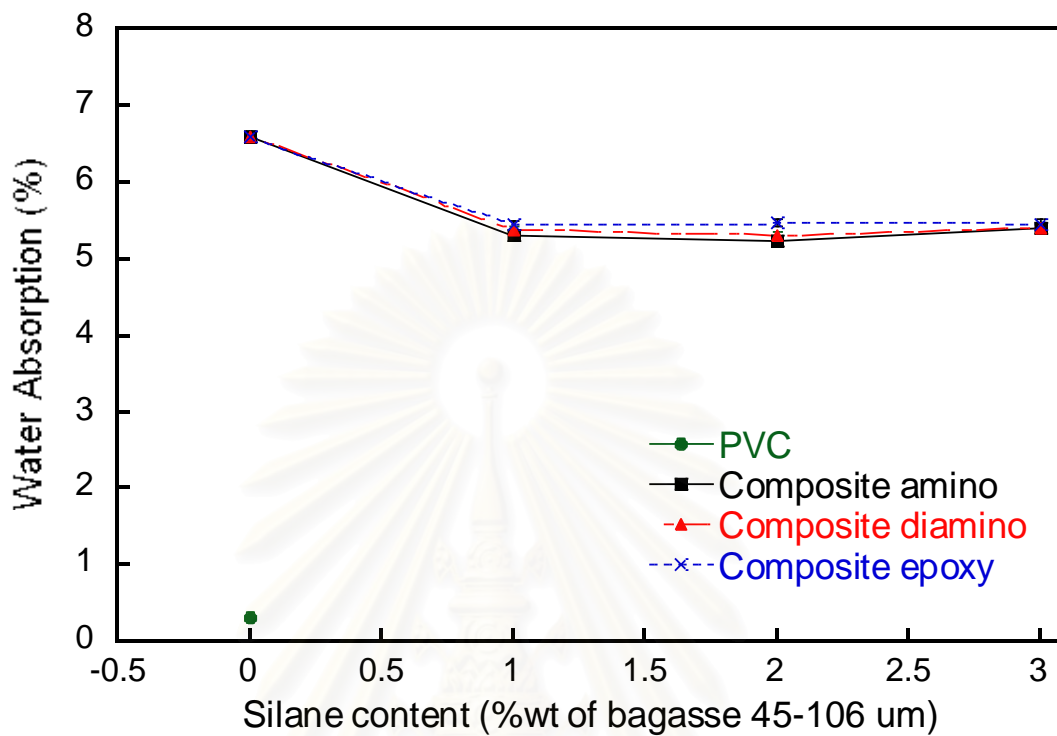
Figure 5.23: Water absorption of PVC/bagasse composites at various
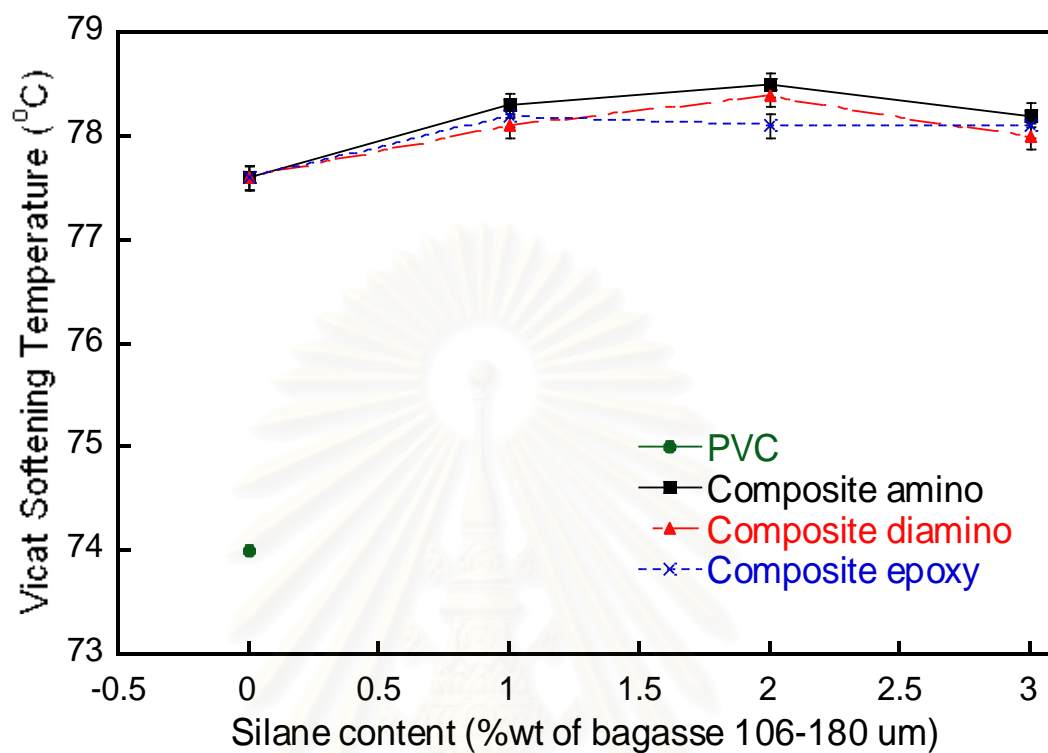
types and contents of silane treated on 45-106 μm fiber
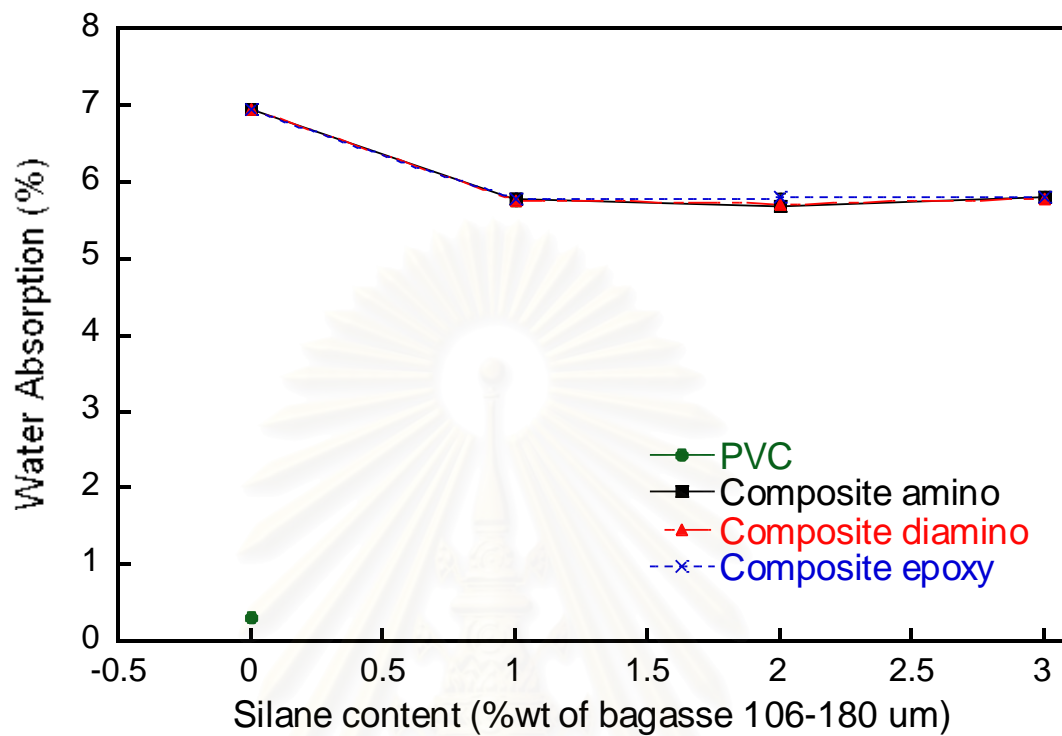
Figure 5.24: Water absorption of PVC/bagasse composites at various

types and contents of silane treated on 106-180 µm

fiber

CHAPTER VI

CONCLUSIONS

Surface of composites were examined using FTIR spectroscopy which was performed on composites having untreated and treated bagasse to identify any possible chemical reaction occurring between treated bagasse and PVC matrix. FTIR spectroscopy results suggested that treated fiber can react with PVC to form strong Si-O-Si or Si-O-C peak of bagasse at 1000 - 1100 cm^{-1}. Thus, it confirms the silane treatment on bagasse. Finally, among three types of silane coupling agent, the composites with treated bagasse with amino silane coupling agent showed the best results in tensile and flexural properties and they also showed the lowest amount of water absorption.

The addition of natural fibers into PVC dry blend resulted in stiff and brittle composites compared to PVC dry blend as noticed from an increase in tensile modulus and flexural modulus of composites. However, both tensile and flexural strength of composite decreased and absorbed more water than PVC dry blend. Small-sized bagasse increased the stiffness of composites more than large-sized bagasse but

bagasse sizes did not show significant effect on thermal properties of PVC/bagasse composite

The composites with treated bagasse showed tensile and flexural strength and tensile and flexural modulus of these composites increased between 24-27%, 9-15%, 8-12% and 14-17%, respectively compared to composite with untreated bagasse. However, these composites with treated bagasse absorb less amount of water than composites with untreated bagasse. However, types and concentrations of silane coupling agents did not show significant effect on impact strength and thermal properties of PVC/bagasse composites.

RECOMMENDATIONS

Recommendations in this work can be concluded as following.

For SEM analysis, the SEM micrographs of composite were quite unclear and very difficult to determine the dispersibility of silane coupling agent and fiber in Poly(vinyl chloride) (PVC) matrix. Therefore, Energy Dispersive X-ray spectroscopy (EDX) should be studied to obtain the clearer results.

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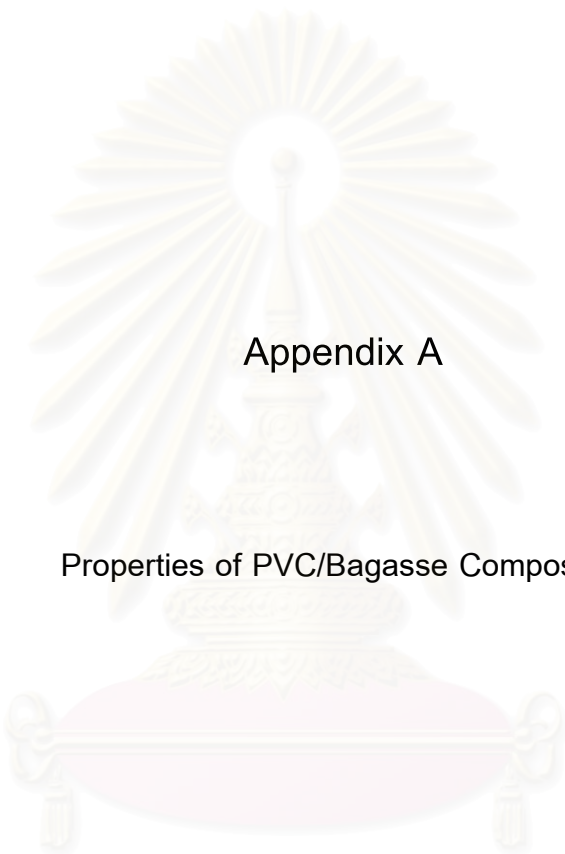Zhao, Y., Wang, K., Zhu, F., Xue, P. and Jia., M. Properties of poly(vinyl chloride)/wood flour/montmorillonite composites: Effects of coupling agents and layered silicate. Polymer Degradation and Stability 91 (2006): 2874-2883.

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APPENDICES

Appendix A

Properties of PVC/Bagasse Composites

Appendix A-1 Mechanical properties of PVC/Bagasse (45-106 µm) Composites at Various Silane Contents in Fiber.

Properties	PVC Dry blend	PVC/ bagasse	PVC/treated bagasse with amino(%wt)			PVC/treated bagasse with diamino(%wt)			PVC/treated bagasse with epoxy(%wt)		
			1	2	3	1	2	3	1	2	3
Tensile Strength (MPa)	51.47±1.32	42.71±0.74	50.07±0.55	53.71±1.3	45.16±0.62	48.35±0.78	50.98±0.98	35.59±1.07	43.33±0.84	41.43±0.97	40.12±1.07
Tensile Modulus (GPa)	2.79±0.11	4.08±0.29	4.25±0.47	4.31±0.88	4.21±0.39	4.13±0.58	4.23±0.82	4.15±0.77	3.97±0.45	3.74±0.64	3.62±0.40
Elongation at break(%)	2.24±0.20	1.89±1.03	1.30±0.14	1.18±0.34	1.4±0.37	1.4±0.41	1.17±0.59	1.39±0.43	1.2±0.28	1.3±0.61	1.32±0.27
Flexural Strength (MPa)	79.30±1.28	66.0±0.44	66.84±1.04	71.21±1.0	45.22±0.42	65.85±1.2	68.5±0.11	50.39±0.39	61.30±0.85	61.5±0.14	62.02±1.02
Flexural Modulus (GPa)	2.79±0.46	5.10±0.52	5.53±0.54	5.75±0.23	3.92±0.47	5.24±0.79	5.54±0.80	3.78±0.44	5.11±0.25	5.20±0.41	5.12±0.35
Impact Strength (kJ/m²)	7.16±0.98	3.90±0.98	3.98±1.01	4.11±1.06	3.78±0.89	3.95±0.46	4.09±0.53	3.80±0.77	3.69±0.55	3.74±0.44	3.72±0.73

Appendix A-2 Mechanical properties of PVC/bagasse (106-180 µm) Composites at Various Silane Contents.

Properties	PVC Dry blend	PVC/bagasse	PV/treated bagasse with amino(%wt)			PVC/treated bagasse with diamino(%wt)			PVC/treated bagasse with epoxy(%wt)		
			1	2	3	1	2	3	1	2	3
Tensile Strength (MPa)	51.47±1.32	41.94±0.02	47.28±1.19	53.05±1.13	43.33±0.95	45.27±0.25	49.35±1.05	30.01±0.79	42.13±1.19	39.98±1.67	38.72±1.30
Tensile Modulus (GPa)	2.79±0.11	3.83±0.11	4.20±0.10	4.24±0.08	4.14±0.09	4.05±0.12	4.20±0.07	4.15±0.05	3.93±0.11	3.69±0.04	3.58±0.10
Elongation at break(%)	2.24±0.20	1.48±1.09	1.37±0.03	1.29±0.03	1.62±0.02	1.264±0.03	1.2±0.05	1.35±0.03	1.314±0.01	1.382±0.05	1.4±0.02
Flexural Strength (MPa)	79.30±1.28	65.20±0.50	65.66±0.74	72.04±0.64	42.10±0.55	66.42±4.42	68.51±0.52	46.22±0.62	61.22±0.73	62.56±0.52	61.66±1.27
Flexural Modulus (GPa)	2.79±0.46	4.95±0.12	5.51±0.23	5.60±0.12	3.67±0.26	5.52±0.18	5.66±0.22	3.61±0.30	5.17±0.07	5.19±0.06	4.89±0.07
Impact Strength (kJ/m^2)	7.16±0.98	3.92±1.23	3.68±2.78	3.86±1.56	3.56±0.75	3.5±0.81	3.75±1.01	3.43±0.98	3.42±1.12	3.74±1.09	3.52±0.98

Appendix A-3 Thermal Properties of PVC/bagasse (45-106 µm) Composites at Various Silane Contents.

Properties	PVC Dry blend	PVC/ bagasse	PVC/treated bagasse with amino(%wt)			PVC/treated bagasse with diamino(%wt)			PVC/treated bagasse with epoxy(%wt)		
			1	2	3	1	2	3	1	2	3
Vicat Softening Temperature (°C)	74±0.0	79±0.2	79.5±0.1	79.7±0.1	79.3±0.0	79.4±0.3	79.6±0.2	79.4±0.1	79.0±0.2	78.8±02	78±0.0
Heat Distortion Temperature (°C)	71±0.0	74±0.1	74.1±0.1	74.3±0.1	74.2±0.2	73.9±0.0	74.0±0.1	74.0±0.1	73.9±0.1	73.9±0.2	73.8±0.1

Appendix A-4 Thermal Properties of PVC/bagasse (106-180 µm) Composites at Various Silane Contents.

Properties	PVC Dry blend	PVC/ bagasse	PVC/treated bagasse with amino(%wt)			PVC/treated bagasse with diamino(%wt)			PVC/treated bagasse with epoxy(%wt)		
			1	2	3	1	2	3	1	2	3
Vicat Softening Temperature (°C)	74±0.0	77.6±0.1	78.3±0.2	78.5±0.1	78.2±0.1	78.1±0.0	78.4±0.2	78.0±0.1	78.2±0.1	78.1±0.0	78.1±0.2
Heat Distortion Temperature (°C)	71±0.0	73±0.1	73±0.1	73.1±0.2	73±0.2	72.8±0.0	73.0±0.1	72.7±0.0	72.9±0.0	72.8±0.0	72.8±0.0

Appendix A-5: Water Absorption of PVC/bagasse (45-106 µm) Composites at
Various Silane Contents.

Properties	PVC Dry blend	PVC/ bagasse	PVC/treated bagasse with amino (%wt)			PVC/treated bagasse with diamino (%wt)			PVC/treated bagasse with epoxy (%wt)		
			1	2	3	1	2	3	1	2	3
Water Absorption (%)*	0.32	6.59	6.30	6.23	6.39	6.37	6.30	6.40	6.44	6.47	6.46

* The amounts of water absorption were measured after being submerged in water for 4

months.

Appendix A-6: Water Absorption of PVC/bagasse (106 -180 µm) Composites
at Various Silane Contents.

Properties	PVC Dry blend	PVC/ bagasse	PVC/treated bagasse with amino (%wt)			PVC/treated bagasse with diamino (%wt)			PVC/treated bagasse with epoxy (%wt)		
			1	2	3	1	2	3	1	2	3
Water Absorption (%)*	0.32	6.96	6.79	6.69	6.8	6.75	6.70	6.79	6.79	6.80	6.81

* The amounts of water absorption were measured after being submerged in water for 4

months.

APPENDIX B

The Calculation for Silane Solution

The calculation for silane solution

    1.Preparation of 1% wt/v of acetic acid (5000 ml) from acetic acid 99.8% v/v

Needed acetic acid $=$ $\dfrac{1\ g}{100\ ml}$ x   5000 ml $=$ 50 g

Acetic acid 99.8% v/v, d = 1.05 g/ml

Acetic acid solution   100   ml has acetic acid 99.8 ml x 1.05 g/ ml = 104.79 g

Acetic acid 104.79 g   come from acetic acid solution   100   ml

Acetic acid 50 g   come from acetic acid solution $\dfrac{50\ g \times 100\ ml}{104.79\ g}$ = 47.71 ml

$\therefore$ 47.71 ml of acetic acid 99.8% v/v was used to prepare 5000 ml of acetic acid 1% wt/v

2. Preparation of silane content with respect to bagasse in composite

Silane content: 1%wt of bagasse

In this experiment use 60 phr bagasse

Bagasse   100   g   used silane   1 g

Bagasse   60   g   used silane $\dfrac{1 \times 60}{100}$ = 0.6 g

$\therefore$ Silane used in this experiment was 0.6 g

3. Preparation of silane solution in acetic acid 1% wt/v

Silane 1 ml (d = 0.475 g/ml) was added in 1% wt/v acetic acid 99 ml

Silane solution 1 + 99 = 100 ml has silane 1 ml x 0.475 g/ml = 0.475 g

Silane 0.475 g come from silane solution   100 ml

Silane 0.6 g   come from silane solution $\underline{100 \times 0.6}$ = 126.32 ml

                                                                 0.475

$\therefore$ 126.32 ml of silane solution in 1% wt/v acetic acid was added to the composite to

make 60 phr bagasse

VITA

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