การเตรียมพอลิเมอร์คอมพอสิตจากพอลิเอสเทอร์เรซินชนิดไม่อิ่มตัวและฟางข้าว

นางสาวศลิษ์ญา สุริโย

PREPARATION OF POLYMER COMPOSITES FROM UNSATURATED
POLYESTER RESIN AND RICE STRAW

Miss Saliya Suriyo

A Thesis Submitted in Partial Fulfillment of the Requirements

for the Degree of Master of Science in Petrochemistry and Polymer Science

Faculty of Science

Chulalongkorn University

Academic Year 2004

Thesis Title	PREPARATION OF POLYMER COMPOSITES FROM UNSATURATED POLYESTER RESIN AND RICE STRAW
By	Miss Saliya Suriyo
Field of Study	Petrochemistry and Polymer Science
Thesis Advisor	Assistant Professor Surachai Pornpakakul, Ph.D.

Accepted by the Faculty of Science, Chulalongkorn University in Partial Fulfillment of the Requirements for the Master's Degree

....Dean of the Faculty of Science

(Professor Piamsak Menasveta, Ph.D.)

THESIS COMMITTEE

....Chairman

(Associate Professor Supawan Tantayanon, Ph.D.)

....Thesis Advisor

(Assistant Professor Surachai Pornpakakul, Ph.D.)

....Member

(Professor Sophon Roengsumran, Ph.D.)

....Member

(Associate Professor Amorn Petsom, Ph.D.)

....Member

(Associate Professor Wimonrat Trakarnpruk, Ph.D.)

ศลิษญา สุริโย : การเตรียมพอลิเมอร์คอมพอสิตจากพอลิเอสเทอร์เรซินชนิดไม่อิ่มตัวและฟางข้าว (PREPARATION OF POLYMER COMPOSITES FROM UNSATURATED POLYESTER RESIN AND RICE STRAW) อ. ที่ปรึกษา: ผศ.ดร. สุรชัย พรภคกุล, 115 หน้า, ISBN: 974-53-1633-4

งานวิจัยนี้เกี่ยวกับการเตรียมพอลิเมอร์คอมพอสิตจากพอลิเอสเทอร์เรซินชนิดไม่อิ่มตัวและฟางข้าว และจากพอลิเอสเทอร์เรซินชนิดไม่อิ่มตัวและฟางข้าวปรับปรุงสภาพพื้นผิวด้วยมาเลอิกแอนไฮไดรด์และไวนิลไตรเมธอกซิไซเลน ในการปรับปรุงสภาพพื้นผิวบำบัดฟางข้าวด้วยมาเลอิกแอนไฮไดรด์ และไวนิลไตรเมธอกซิไซเลน 10, 30, และ 60 เปอร์เซ็นต์ เพื่อปรับปรุงแรงยึดเกาะระหว่างผิวหน้าและการเข้ากันได้ระหว่างพอลิเมอร์เมทริกซ์และฟางข้าวคอมพอสิตที่เตรียมได้ ทำการศึกษาสมบัติเชิงกลและสัณฐานวิทยาของพื้นผิวจากการแตกหัก พบว่าสมบัติเชิงกลของพอลิเมอร์คอมพอสิตที่มีฟางข้าวที่ผ่านการปรับสภาพพื้นผิวด้วยมาเลอิกแอนไฮไดรด์ แสดงสมบัติการต้านทานแรงดึง การทนแรงดัดโค้ง การทนต่อแรงกระแทกที่ดีกว่าคอมพอสิตที่เตรียมจากฟางข้าวที่ผ่านการปรับสภาพพื้นผิวด้วยไวนิลไตรเมธอกซิไซเลนและที่ไม่ได้ผ่านการปรับสภาพพื้นผิว คอมพอสิตที่ 70:30 พอลิเอสเทอร์เรซินชนิดไม่อิ่มตัวและฟางข้าวที่ปรับสภาพพื้นผิวด้วย 30 เปอร์เซ็นต์มาเลอิกแอนไฮไดรด์ แสดงสมบัติเชิงกลที่สูงที่สุดเมื่อเปรียบเทียบกับคอมพอสิตอื่นๆ สัณฐานวิทยาของพื้นผิวที่แตกหักของคอมพอสิตจากฟางข้าวปรับสภาพพื้นผิวด้วย 30 เปอร์เซ็นต์มาเลอิกแอนไฮไดรด์ แสดงการเข้ากันระหว่างผิวหน้าที่ดีกว่าคอมพอสิตจากฟางข้าวที่ปรับสภาพพื้นผิวด้วยไวนิลไตรเมธอกซิไซเลนและที่ไม่ได้ผ่านการปรับสภาพพื้นผิว นอกจากนั้นพบว่าการดูดซึมน้ำของพอลิเมอร์คอมพอสิตฟางข้าวที่ผ่านการปรับสภาพพื้นผิวด้วยมาเลอิกแอนไฮไดรด์และไวนิลไตรเมธอกซิไซเลนมีค่าลดลง 29 และ 20 เปอร์เซ็นต์ตามลำดับเมื่อเปรียบเทียบกับคอมพอสิตจากฟางข้าวที่ไม่ได้ปรับสภาพพื้นผิว

สาขาวิชา ปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์   ลายมือชื่อนิสิต..

ปีการศึกษา   2547   ลายมือชื่ออาจารย์ที่ปรึกษา.........................

SALIYA SURIYO: PREPARATION OF POLYMER COMPOSITES FROM UNSATURATED POLYESTER RESIN AND RICE STRAW. THESIS ADVISOR: ASST. PROF. SURACHAI PORNPAKAKUL, Ph.D., 115 pp, ISBN: 974-53-1633-4

This research work involved the preparation of the polymer composites made from unsaturated polyester resin and rice straw and from unsaturated polyester resin and surface modified rice straw with maleic anhydride (MA) and vinyltriethoxy silane (VTS). In the process of surface modification, the rice straw was treated with 10, 30, and 60% of MA and of VTS for improvement of the interfacial adhesion and compatibility between rice straw and polymer matrix. The prepared composites were investigated their mechanical properties and fracture surface morphology. It was found that mechanical properties of composite containing MA treated rice straw displayed better tensile strength, flexural strength, and impact strength than VTS treated rice straw and untreated rice straw composites. A 70:30 composite of unsaturated polyester resin and 30% MA treated rice straw showed the highest mechanical properties compared to those composites. The fracture surface morphology of 30% MA treated rice straw composites showed better compatibility and interfacial adhesion than untreated and VTS treated rice straw composites. Moreover, the study found that the water absorption of MA and VTS treated rice straw composites can be reduced compared to the untreated rice straw composite, approximately 29% and 20% respectively.

Field of study Petrochemistry and Polymer Science Student's signature…………………

Academic year   2004   Advisor's signature…………………

ACKNOWLEDGEMENTS

CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABBREVIATIONS

ASTM	American Society for Testing and Material
cm.	centimeter
cm^{-1}	per centimeter
^{o}C	degree centigrade
FRP	fiber reinforce polyester
FTIR	fourier transform infrared spectrophotometer
g.	gram
h	hour
in	inch
J/cm.	joule per centimeter
$kgf/mm.^{2}$	kilogram force per square millimeter
KBr	potassium bromide
kV	kilovolt
l	length
MA	malice anhydride
ml	milliliter
mm.	millimeter
%LOI	percentage loss on ignition
%	percentage
% wt	percentage by weight
Psi	pound per square inch
SEM	scanning electron microscopy
SEM/EDX	scanning electron microscopy/electron diffraction X-ray
VTS	vinyltriethoxy silane
XRD	X-ray diffraction spectroscopy
XRF	X-ray fluorescence spectroscopy

CHAPTER I

INTRODUCTION

1.1 The purpose of the investigation

The polymer composite material has been growing steadily, since the Second World War. The definition of the polymer composite is a material in which strong, high modulus fiber with diameter of the order of 10-100 mm, are embedded in a polymer matrix material. Moreover, polymer composite is mixture of polymer material and other material as filler. The mechanical properties of polymer composites are improved better than synthesized polymer such as stiffness, light weight, and thermal stability, which makes them a competitive substitute for metal and wood in many applications. Both thermosetting and thermoplastic polymers can be used as the matrix for polymer-based composites. Today, glass fiber is the most widely used fiber for reinforcing in polyester composite materials. The general purpose of polyester are usually in liquid form that can be handle easily, exhibit good mechanical properties and no high pressure requirement are necessary for molding. However, there were many reports that studied the natural fibers used as reinforcement or filler in polymer composite such as sisal, coir, jute, and flex that has the advantage over inorganic filler and glass fiber in being of lower cost, density, abrasion and using less energy in production. There are two major problems in the application of natural cellulose as filler. One is the lack of adequate interaction between the cellulose and the polymer matrix. The other is hygroscopy nature of the natural fibers, which results in low durability. This problem can be overcome by treating these fibers with suitable chemicals to improve the interfacial adhesion between the filler and polymer matrix.

Thailand is known as an agricultural developing country and the world largest rice exporter, the manufacturing sector is now leading agriculture. The major agricultural residues are the rice straw, which is estimated that there are 0.32-1.6 tones of rice straw per a season. The rice straw have a principal chemical composition namely cellulose, lignin, and crude fiber that it is possible to tailor the end properties of the composites and morphological composition. In this study, the interest in using rice straw as a filler to

preparation of unsaturated polyester composites. Thus the present study investigates the modification surface by chemical coupling of rice straw with different coupling agent by using maleic anhydride (MA) and vinyltriethoxy silane (VTS) before to preparation composite to improve the interfacial adhesion and compatibility between the filler and polymer matrix. The effects of surface modification or coupling agent on the physical and mechanical properties are observed.

1.2   Objective of the research work

1.   To study the preparation of composites from unsaturated polyester resin and rice straw.
2.   To study the physical and mechanical properties of unsaturated polyester resin and rice straw composites obtained under various conditions.
3.   To study the effect of chemical treatment on the physical and mechanical properties of polymer composite.

1.3   Scopes of the research work

In this research, the unsaturated polyester resin-rice straw composites were prepared for the study of their properties. The rice straw, which was used in the study as filler in the polyester matrix, was treated through the process of surface modification with maleic anhydride and vinyltriethoxy silane coupling agent at various concentrations. To determine the optimum condition to make the unsaturated polyester resin-rice straw composites, the physical and mechanical properties of the composites such as water absorption, and morphology of the fracture surface of the polyester resin-rice straw composites would be studied.

CHAPTER II

THEORY AND LITERATURE REVIEW

2.1 Composite material [1]

The word "composite" in composite material signifies that two or more materials are combined on a macroscopic scale to form a useful material, having a recognizable interface between them. The advantage of composites is that they usually exhibit the best qualities of their constituents and often some qualities that neither constituent process. The characteristics of composite materials can be classified three ways. There are three commonly accepted types of composite materials:

1) Filled porous system composites, which consist of porous materials, penetrated by matrix.

2) Particulate composites, which are composed of particles in a matrix.

3) Fibrous composites, which are composed of fiber in a matrix.

2.1.1 Classification of composite materials [1-4]

Composites can be divided into classes in many manners. One simple classification scheme is to separate them according to reinforcement forms.

1) Particulate-reinforced composites

Particulate-reinforced composites consist of particle dispersed in a matrix. This type is considered to be a "particle" if all of its dimensions are roughly equal. Particulate may have any shape, configuration, or size. They may be powdered, beads, rods, crystalline, or amorphic. They may be metallic, ceramic, manmade, or natural materials. Concrete and wood particleboards are two familiar examples of particulate composites. Pieces of ceramic particles are placed in a metallic matrix and used as tough, abrasion resistant cutting tools. Metallic flakes have been added to improve electrical properties and provide some degree of radiation shielding in polymer composites. Generally, particles in polymer usually extend rather then reinforce the material. These are usually referred to as "filled" systems. Because filler particles are included for the aim of cost

reduction rather than reinforcement, these composites are not generally considered to be particulate composites. Nonetheless, in some cases the filler will also reinforce the matrix material.

2) Fiber-reinforced composites

Fiber-reinforced composites are composed of reinforced fibers in a matrix. They contain reinforcements having lengths greater than their cross-sectional dimensions. They can be further divided into those containing discontinuous and continuous fibers. In discontinuous fiber case, the bond between the fibers and the matrix is broken at the fiber's ends, which thus carry less stress than at the fiber's ends, which thus carry less stress than at the middle part of the fiber. Therefore, continuous fibers can carry stresses along its length.

3) Laminar composites

Laminar composites are composed of layers of materials held together by matrix binder, sandwich and honeycomb component as well as the term high-pressure laminates are included. Complicating the definition of a composite as having both continuous and discontinuous phases is the fact that in a laminar composite, neither of these phases may be regarded as truly continuous in these dimensions.

2.2 Polymer composites [5]

Polymer composites are mixtures of polymer with inorganic or organic additives having certain geometries such as fiber, flakes, spheres, and particulate. Thus, they consist of two or more components and two or more phases. The additives may be continuous, e.g. long fiber or ribbon; these are embedded in the polymer in regular geometric arrangement that extend throughout the dimensions of the product. Familiar examples are the well-know fiber based thermoset laminates that are usually classified as high performance polymer composite.

Polymer composites were first developed during the 1940's, for military and aerospace applications. Polymers offer many advantages over conventional materials

including lightness, resistance to corrosion and ease of processing. They can be combined with fibers to form composites, which have enhanced properties, enabling them to be used as structural members and units. Polymer composites can be used in many different forms ranging from structural composites in the construction industry to the high technology composites of the aerospace and space satellite. Considerable advances have been made since then in the use of this material and applications developed in the construction sector. Load bearing and in fill panels have been manufactured using composites. Complete structures have been fabricated where units manufactured from glass-reinforced polyester are connected together to form the complete system in which the shape provides the rigidity. Both thermosetting and thermoplastic polymers can be used as the matrix for polymer-based composites.

The matrix generally performs the function of a binder to transfer stress to the reinforcing fibers and ensure their cooperative interaction. Reinforcing fibers can either be man-made fibers or vegetable fibers, although glass fiber is the most widely used reinforcing agent at present. The strength of the composite is the combination of the strength of the reinforcing fiber and the matrix.

2.2.1 Properties of polymer composite [5]

Composites typically made up of the continuous matrix phase in which fiber and additives are embedded: 1) a three-dimensional distribution of randomly reinforcing elements, e.g., a particulate filled composite. 2) a two-dimensional distribution of randomly oriented elements, e.g., a chopped fiber mat. 3) an ordered to-dimensional structure of high symmetry in the plane of the structure, e.g., an impregnated cloth structure and 4) a highly-aligned array of parallel fibrous randomly distribution normal to the fiber directions, e.g., a filament-wound structure or a prepared sheet consisting of parallel rows of fiber impregnated with a matrix. These properties can be measured. Since composites in turn are often built up by laminating layers of composite sheets, these properties are needed to predict the overall response of the laminated structure. The fiber in each layer can be oriented differently from adjacent layers. If proper attention is not available, peculiar effects can occur, such as, a composite part twisting when a simple

tensile load is applied. With an isotropic material, this would merely stretch the body. However, for purpose of designing optimum materials, it would be desirable to compute the properties of the constituent matrix and reinforcements. The present state of analytical skills allows such predictions to be made with reasonable confidence in specialized cases. However, for many other situations, only upper and lower property bounds can be stated.

The study detail of the stresses and strains within a composite considered as a true heterogeneous system. This approach allows the effective average properties of the composite to be computed when the reinforcement has a simple geometric shape and is located in regular arrays. Such idealized models can be used to provide a semi quantitative framework for the behavior of real composite materials. Modeling of the properties of composites as a function of temperature, pressure, or other environments requires a corresponding knowledge of the behavior of the separate constituents plus of their interactions, such as, result from differences in thermal expansion.

2.2.2 Role of the matrix and interface [6]

While it is true that the fiber in fibrous composites is the major fraction of the load, the matrix and the nature of the fiber-matrix adhesion are often extruding important. In fact, the matrix is the strength-limiting variable in the following cases: aligned continuous fiber tested off-axis; cross-plied continuous fiber, test at all angles; and incompletely dispersed random-in-a-plane discontinuous fiber, test at all angles. Thus, in many common cases, matrix strength is desired-a combination is not often achieved. While the incorporation of rubbery materials in a brittle matrix can increase toughness substantially, the modulus tends are reduced to some extent. Possibly better approaches to achieve both a high modulus and improved toughness might include the addition of small glass spheres, especially if interfacial adhesion is minimized; the use of ductile or crack-stopping filler; the use of high modulus organic fiber; the structural modification of epoxy and other resins; and the use of other matrixes altogether.

In all cases, the nature of the interface must be carefully controlled. For example, in contrast to the case of particulate composites, good adhesion between a ductile matrix and short glass fiber may enhance crazing and hence energy dissipation at the fiber tips.

As with particulate fillers, then, the fracture energy of a fiber reinforced composite is a complex function of the properties of the reinforcement, the matrix, and the interface. Several modes of dissipation may be involved as follows:

1)   Debonding of the fiber from the matrix as a crack impinges upon a fiber. Such a debonding may occur when the fracture strain of the fiber is greater than the fracture strain of the matrix.

2)   Pulling of broken filaments out of the matrix following fracture.

3)   Redistribution of strain energy from fiber to the matrix after fracture of the fiber.

4)   Fracture of the fiber and matrix themselves.

Clearly, all these modes may participate in determining the overall toughness of a composite, though to degrees dependent on the system. Broutman and Agareval have critically analyzed this question, and developed more or less generalized treatments of the overall work required for fracture. Such analysis based on fracture mechanics concepts should be of reasonable level of toughness without sacrificing strengths of the order predicted by the rule of mixtures. In general, an intermediate value of the interfacial bond strength is desired to optimize toughness, an optimum fiber length also exists, the value depending on the nature of the fiber.

The interface may also be "tailored" to obtain a gradation in modulus from the fiber to the matrix, and hence a more efficient transfer of stress; such a tailoring was effective with annealed graphite and glass fiber-reinforced polycarbonate.

2.2.3 Thermoset molding history [7]

Thermosetting molding compounds originated with Dr. Leo H. Baekeland's 1907 discovery of a technique for reaching, under heat and pressure, an acid and an aldehyde to form an (A) stage synthetic resin. A caustic, crystalline acidic compound (phenol) reacting under applied heat and pressure with a pungent, colorless gas (formaldehyde) produced a hot yellowish brown liquid. When cooled, it became a frangible solid that could be pulverized into a free-flowing, granular material. Combining this granular resin

with fillers or reinforcements, colorants, catalysts, and lubricant resulted in a "molding compound" that became identified as a "phenolic" molding compound.

This was the first molding compound capable of further chemical reaction (B stage) when subjected to pressure, heat, and time in a "molding" process that cured the resin into an soluble, infusible mass (C stage). This cured mass exhibited mechanical, electrical, thermal, and chemical properties that made it suitable for numerous industrial and commercial applications. This unique property profile, coupled with its easy moldability, low cost, and adaptability to mass production techniques, provided the basis for what has since become known as the "plastic molding industry."

This phenolic molding compound was the first member of a family of thermosetting molding compounds that grew with the additional several other resin systems, all of which shared the ability to undergo that (B) stage chemical advancement or "curing" when subjected to elevated temperature under pressure over a time span. This chemical reaction, sometimes called polymerization, took place under controlled conditions in a "molding" process.

2.3 Thermosetting molding compounds [7]

Thermosetting molding compounds consist of resin and filler; the resin-usually condensation resins based on phenol or melamine have undergone preliminary condensation i.e., they have already formed moderately long macromolecules. During the subsequent heating of primary processing, the reaction continues: The resin cross-link to form a nonmelting or insoluble thermosetting plastic. All thermosetting resins are characterized by this curing reaction. Each individual resin system posses its own particular rheological flow or gelation properties. The following elements of the compound formulation define the compound reaction profile:

1)   Type and content of resin
2)   Type and content of catalyst
3)   Type and content of reinforcement
4)   Type and content of lubricant

In addition to the above, molding compounds based on unsaturated polyester and epoxy resins are being employed increasingly today. Usually organic or inorganic fillers comprising 35 to 65 vol. % are added to the resins, along with up to 1 % of lubricants and regulators. Condensation resins are prepared in the following manner: After the preliminary condensation, the resulting product is ground and mixed with the additives on a roll mill. After solidification, this product is ground once again.

In contrast to the thermoplastic, thermosetting resins become solid not as a result of cooling, i.e., solidification, but rather as a result of curing. Once a certain degree of cross-linking has been achieved, the material can no longer be formed. Depending on the extent of preliminary condensation, a molding compound that cures more or less rapidly can be produced.

2.3.1 Thermosetting molding process [7-10]

The thermosetting molding process subjects the molding compound to the physical elements of temperature, pressure, and time. These enable the compound to undergo an irreversible chemical reaction, which produces molded articles with rigid, highly cross-link molecular structure. This reaction, with its attendant property value enhancement, is what differentiates molded thermosetting polymers from molded thermoplastic polymers.

Although this same molding process will subject a thermoplastic compound to very similar conditions, the thermoplastic compound will undergo only a physical change-going from a granular solid to a low viscosity melt and then back to a solid as it cools off. There will be little, in any, chemical cross-linking or attendant property enhancement.

In any consideration of thermosetting molding process, it is vitally important that there be a recognition and understanding of the relationship among the molding compound, the molding process, the molding machinery, the molded part design, and the mold design. Each of these affects the others, and they are all directly influenced by the irreversible chemical reaction that occurs during the molding process. Thus the product designer, mold designer, molder, and molding compound supplier must each bring their

individual expertise to the planning stages as early as possible-certainly prior to any mold construction. The principal processing methods are used for thermoset resin for good performance in the various molding processes.

1) Hand lay-up

The hand lay-up process is the oldest and simplest method for making glass fiber-reinforced parts. Male or female molds can be made of easily worked materials such as wood, plaster, or reinforced plastics. In hand lay-up, resin and reinforcements in the from of fabric, woven roving or mat are simply placed in the mold manually. Entrapped air is then removed with squeezer or serrate metal rollers. Successive layers of reinforcement and resin can be added to build the part to the desired thickness. If a smooth colored surface is required, a pigmented material (called a gel coat) can be sprayed on the mold before lay-up.

2) Spray lay-up

The most recent innovation in contact molding is the use of dual spray guns for the application of the resin. Separate mixes of catalyzed and accelerated resin are combined in the gun, or just after they leave it, to yield very fast gel times on the work with no concern for short pot life. Some of these resin guns are used with glass choppers for simultaneous or separate application of glass fibers directly to the mold surface in a random pattern. This method of applying glass offers considerable possibilities for saving money by using glass roving (considerably cheaper than glass mat); by eliminating the wastage of time and material required to tailor preformed mat to fit contoured sections; and by reducing the loss of room temperature curing resin mixes which gel before they can all be used.

3) Filament winding

The pipes, tubes, and cylindrical tanks are made by a process known as filament winding. A mandrel or form is rotated about one axis as continuous yarn or roving which passes through a bath or liquid resin is wet-wound on it, usually in complex patterns controlled by automated machinery. Once the required wall thickness is reached, heat

curing in autoclaves, ovens or by heating the mandrel is begun. Because the reinforcement is continuous and its orientation subject to close control, very efficient structures can be produced consistently by this method, and in the case of smooth-bore pipes, the process can be continuous. Once cured, the pipe is removed from the metal mandrel and cut into proper lengths. Mandrels for tanks and pressure bottles often are inflatable, soluble or collapsible, so they can be removed through end ports. Pipe production for oil fields, the chemical processing industry, water distribution, and sewage disposal has reached very large volumes, in diameters from several centimeters to as long as 6 meters; fittings also can be filament wounded or die-molded.

4) Pultrusion.

The pultrusion process makes structural profiles of any shape continuously from composite materials, just as they would be extruded in aluminum. It is a one-step process that converts raw stock into finished product at rates 4.5 m/min up. Reinforcements are drawn into the system, impregnated with resin, and simultaneously formed and cured in a heated die; radio frequency or induction energy can be used to supplement curing. Reinforcements are impregnated by one of the available devices, in this case a pass-through-type wet-out tank, before passing through squeeze-out bushings. The process is run fast enough to bring the material into the forming die just before gelation. The cured product leaving the die proceeds downstream through the pullers, which gives rise to the term pultrusion. Unlike extrusion, in which the material is forced through a die with pressure, pultrusion pulls material through the die.

5) Die-molding

Most automotive parts and general hardware are made in heated matched metal dies or molds mounted in hydraulic presses operating semi-automatically. A charge of BMC (bulk-molding compound) or SMC (sheet-molding compound) is placed in the molds and cured under moderate heat and pressure (120-175°C, 3.5-13.8 MPa (500-1200 psi)) for cycles ranging from 15-90 seconds. The molded piece is removed hot and allowed to air-cool before undergoing further operations such as trimming, assembly or painting. The quality of the product is fairly consistent, much subassembly work normal

to metal practice can be avoided, and die costs are much lower than for sheet-metal stamping or metal die casting. Thus shorter production runs are economical and more design flexibility and variety can be achieved.

6) Injection molding

Injection-molded, fiber-reinforced thermoplastics compete directly with die-cast metals in many hardware and automotive parts. Higher production rates, lighter weight, and assembly simplification are their principle advantages. The fiber stiffen and allow higher operating temperatures. Often, however, the flow patterns of the molten material into the mold cavity cause significant orientation of the fibers and the resulted localized anisotropy can adversely affect strength and thermal-expansion properties. Considerable skills are required of the mold designer and the machine operator. Also the reuse of fiber-reinforced thermoplastic scrap material from runners, sprues, gates, and flash presents problems that can impair the overall efficiency of material utilization. Despite these difficulties, however, this is the fastest growing segment of the entire composites family.

7) Resin injection molding (RIM)

In unreinforced RIM technology, no reinforcing fiber is used in the mold. Two or more reactive component streams are rapidly mixed and then injected into a closed mold where polymerization and cross-linking occur (time scale 3-5 minutes). The disadvantage of this system is the need for special impingement mix heads to rapidly combine and inject the reacting mixture before it cures. A wide variety of starting materials allows tailoring of properties, from rigid to flexible. Characteristics are low finished product density, which is good for light weight parts. Reinforcement fibers can also be used which improves strength and stiffness properties. Applications of RIM are mainly in automotive parts and insulation with some use in aerospace.

8) Resin transfer molding (RTM)

Resin transfer molding or RTM literally means the transfer of the resin mix from one system (the RTM machine) to another system (the closed mold containing the

reinforcement) while molding a product. It bridges the gap between labor intensive hand lay-up process and capital intensive compression molding process. In the RTM process a combination of continuous mat is laid in the mold cavity. A matching mold half is mated to the first half and the two are clamped together tightly. A pressurized resin system mixed with a free radical initiator is then pumped from one or more ports into the closed mold containing the preplaced reinforcement. The resin and fiber remain in the mold until crosslinking occurs, then the composite can be removed. The material can be cured at room temperature or in heated mold by proper choice of initiator. It is important to balance the speed of gelation with the time needed to till the mold and wet out the reinforcement fiber.

9) Miscellaneous

The four processing techniques that have been mentioned are the dominant ones in use today: hand lay-up/spray-up; matched metal die molding (including flat-press laminating); filament winding; and injection molding. However, there are many others that may be variations of the above or are themselves distinctive. For example, vacuum-bag techniques can be used to help remove air from hand lay-up and to provide modest positive pressure on large, low cost molds too bulky for mechanical pressing. Better quality composites result from the reduction in void content and from the superior impregnation of fiber bundles by liquid-matrix resin.

10) Compressing molding

The compression molding process of partially polymerized thermosetting polymer, usually in a preheated, preformed shape vaguely corresponding to that of the cavity, is placed in heat cavity; the mold begins to close, and pressure is applied to the perform, forcing it to further heat up close to the mold temperature and flow to fill the mold cavity. While under pressure, the polymer undergoes complete polymerization (crosslinking), aided by the heat transferred from the mold. After that the mold is opened, the part is ejected, and the cycle starts again. Only occasionally is the process used for thermoplastics forming (PVC phonograph records), its primary use being either with elastomeric or hard thermosetting polymers. This process wastes very little material (no

runners and squre) and can produce large part. It is difficult to produce parts with very closed tolerances, however, because the final size of compression molded article-that is, the degree to which the mold closes-depends on the exact amount of the perform. Furthermore, the process does not easily lend itself to molding of intricate parts with deep undercuts.

2.3.2 Release agents [10]

Molding compounds contain release agents to prevent parts from sticking to hot metal. When a new mold is placed in operation, it may contain minute tool marks that cause sticking until "broken in." Special break - in compounds containing high amounts of release agents are available. Products molded from these materials are discarded.

Release agents used in the normal course of molding come in many forms. Pure carnauba wax or temperature - resistant synthetic waxes are wiped directly on the mold. Excess is blown away to prevent surface smear. Aerosols containing silicone, fluorocarbon, or lecithin are popular for releasing epoxy, DAP, and alkyd resins. Silicone and other synthetic greases, stearates, and petroleum compounds are also used. In general, release agents must not interfere with curing, must not cause crazing or discoloration, and must be easily removable when parts are to be painted, plated, or glued.

2.4 Polyester resin [11-12]

By chemical definition a polyester is formed by the reaction of polybasic acid and polyhydric alcohol to form a series (poly = many) of ester linkages. The particular types of acids and alcohols selected and other modification of the polyester structure determine into which of the following categories the resulting polyester will fall:

1) Unsaturated polyester resin: linear polyester resins based on dibasic acids and dihydric alcohols capable of crossling with vinyl monomer to from thermoset copolymers.

2) Alkyds: generally used to refer to oil-modified polyesters used for coatings (some crosslinking polyester systems are referred to as alkyd molding compounds).

3) Plasticizer: certain completely saturated polyesters used to plasticize other plastic compositions are referred to as polymeric plasticizer.

4) Fiber and film: high molecular weight highly oriented polyesters base on rather specific acids and alcohols are used to from fiber and film.

5) Polyester foams: polyester with a high concentration of hydroxyl groups, which are cross-linked with isocyanates to from foam, coatings, elastomers, etc.

2.4.1 Saturated polyester

The saturated polyester prepared principally from acids and alcohols by condensation polymerization have been highly developed on a commercial basis in the form of fiber and films. In the fiber and woven form these polyesters are among the most amazing of the miracle fabrics. As films, they are displacing other plastic film and pioneering in new application because of their strength, toughness and electrical properties. The repeat unit of saturated linear polyester as shown in Fig.2.1.
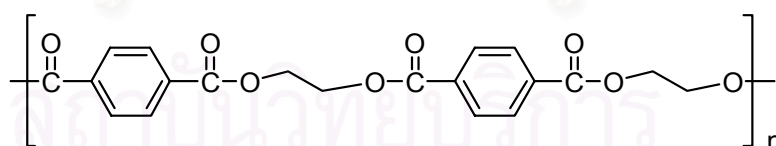
Figure 2.1   Segment of the saturated linear polyester.

The saturated linear polyester cannot be cross-linked with cross-linking monomer. Other saturated polyester of different composition were reacted with diisocyanate

compounds through terminal hydroxyl groups to produce foams, coating, adhesives and rubbers.

2.4.2 Unsaturated polyester

Unsaturated polyester resins are polymers formed by condensation polymerization of dibasic acids and dihydric alcohols, with ester bonds linking the repeating units. The resins are able to form crosslinks to thermoset copolymer because each repeat unit contains an active carbon-carbon double bond that can react with vinyl monomer usually styrene by the addition polymerization mechanism. Reaction of carbon -carbon double bonds by addition polymerization requires that a free radical be formed and attack the double bond to initiate the reaction sequence.

Usually heat-activated or time-activated peroxide or some other free radical source is added to the resin as an initiator at a concentration of 1-2 % to initiate the crosslinking reaction. The resin is normally cured at room temperature followed by a post cure at higher temperature to complete the reaction. A repeat unit of unsaturated polyester resin containing a carbon-carbon double bond and crosslink of unsaturated linear polyester with styrene as shown in Figs.2.2-2.3.
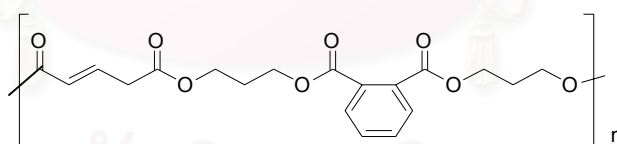
Figure 2.2 Segment of an unsaturated polyester resin chain, showing the active C = C site.
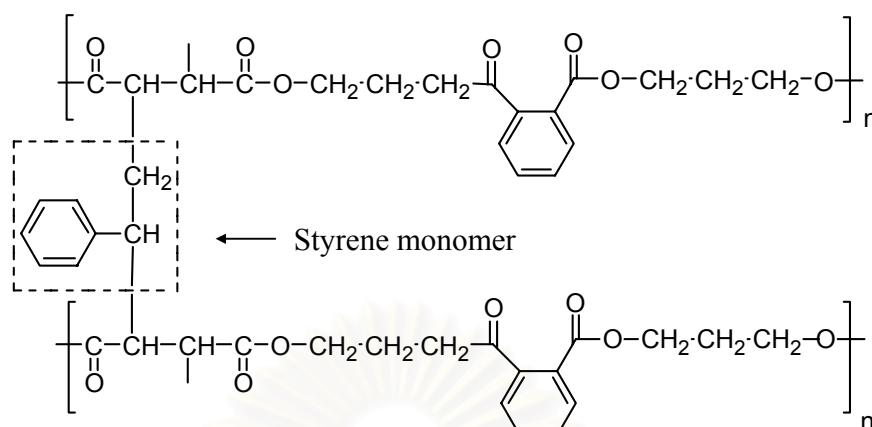
Figure 2.3   Crosslink of unsaturated linear polyester with styrene monomer.

Innumerable variations can be made in the composition of the base polyester and the type and amount of cross-linking monomer, to yield resin with range of properties be fore, during and after polymerization. The main variables which control these properties are:

1) The kind and amount of dibasic acids used.
2) The kind of alcohols used.
3) The type and amount of cross-linking monomer.
4) The molecular weight of the polyester resin.
5) The inhibitor system.

Material for synthesis of unsaturated polyester

1) Acid Constituents

A large number of dibasic acids, which could be used in the synthesis of a polyester molecule, are available on a commercial basis. The type of acid selected with respect to presence of unsaturated bonds available for cross-linking contribute to the properties of the final product. Since unsaturated dihydric alcohols are not variable on a commercial basis, the majority of commercial resins have their unsaturated groups in the acid component of the polyester chain.   The dibasic acid are used for polyester synthesized show in Table 2.1.

Table 2.1   Types of dibasic acid for synthesized polyester.

Saturated acids	Unsaturated acids
- Succinic acid	- Maleic anhydride
- Glutaric acid	- Fumaric acid
- Adipic acid	- Chloromaleic acid
- Pimelic acid	- Citraconic acid
- Azelaric acid	- Mesaconic acid
- Sebasic acid	

2) Alcohol component

The alcohol component of the polyester structure has as important an influence on the cross-linked copolymer as any of the acid used. Flexibility, crystallinity, water and heat sensitivity are properties which are particularly affected by the choice of alcohol. The principal alcohol used in polyester synthesis are glycols (two hydroxyl groups) as shown in Table 2.2. Polyhydric alcohol (more than two hydroxyl groups) gives branching in the polyester chain and rapid build-up in molecular weight which is difficult to control.

Table 2.2   Types of glycols for polyester synthesis.

Type	Molecular weight
Ethylene glycol	62
Propylene glycol	76
Butylene glycol	90
Diethylene glycol	106
Dipropylene glycol	134
Triethylene glycol	150
Isopropylidene bis-(phenyleneoxy-propanol-2)	344

3) Crosslinking monomer

Crosslinking monomer effect to polyester resin formed thermosetting plastic. As indicated earlier, monomers are used to copolymerize with the point of unsaturation in the linear polyester and thus develop the cross-linked or thermoset structure. Before this cross-linked or curing stage the monomer serves as a solvent and for the high viscosity polyester molecule. The number of characteristics of styrene with respect to cost, strength, compatibility, reactive and volatility are responsible for its being the monomer most frequently used for crosslinking unsaturated polyester. Variation in the ratio of styrene to unsaturated groups in the polymer structure affects the properties of the resulting copolymer with respect to physical strength, chemical resistance and electrical properties. Other crosslinking monomer such as methyl methacrylate, vinyl toluene, and divinyl benzene, etc.

4) Inhibitor system

The solution of unsaturated polyester in monomer are potentially highly reactive, in that heat, light, contamination and other factors can cause the free radical mechanism to start the cross linking reaction, which rapidly develops a gel structure. By the incorporation of the chemical structure which promptly react with free radicals and prevent them from reacting with other double bonds, premature gelation can be avoid extended storage periods under normal condition.

In the broad classification of inhibitor used to provide storage stability and control the polymerization process there are two general types whose reaction mechanical are somewhat different. A true inhibitor can be defined as a substance which provides a measurable introduction period during which essentially no polymerization take place. Such substances react very rapidly with any free radical catalyst from the monomer by heat or other mean. The other type of inhibitor is generally called a retarded. Such a material produces no measurable introduction period, but merely causes the polymerization to proceed at a diminished rate during the entire polymerization.

5) Accelerator

The terms accelerator and promotor are used interchangeably to refer to compounds which are added to a resin system to speed the decomposition of catalyst into free radical at room temperature or at temperature considerably below those required to release free radicals when catalyst alone is used. The quaternary metallic salts are example of the accelerator such as Co-octoate and Co-naphthenate or amine compounds such as diethyl aniline and dimethyl aniline.

6) Catalyst

The usual means of promoting the crosslinking reaction is by the addition of so-called catalyst bodies, which decompose to very active free radicals that become the center for polymer growth. The catalysts are different with accelerator or promotor, those react with free radicals formed by the opening of double bonds of the unsaturated polyester or monomer. The principal material used are the organic peroxide compound such as benzoylperoxide methyl and ethyl ketone peroxide (MEKPO)

The properties of unsaturated polyester resin, which characterize them and are the chief factors in their success are:

(1) Ease of handing in liquid form
(2) Rapid cure
(3) Excellent dimensional stability
(4) Good insulate properties
(5) Good general physical properties
(6) Ease of coloring and modifying for special characteristics.

The general-purpose polyester are usually in liquid form and, hence, are completely reactive when mixed the proper amount of catalyst. Polyester resins are the principal plastics used in this phase of the plastics industry. The polyester resins are used in a variety of applications, which can be broadly classified under the categories of molding, casting, and coating. The compounding of polyester resin for use in various fabricating operations is an important factor in the quality of the finished object. The thermosetting polyester resins have found the widest use in reinforced plastics to date. The populality of polyester is base

on such unique characteristic as their ease of combinding with reinforcement and their ability to cure without giving off volatile by product. The synthesis of unsaturated polyester as show in Fig. 2.4.
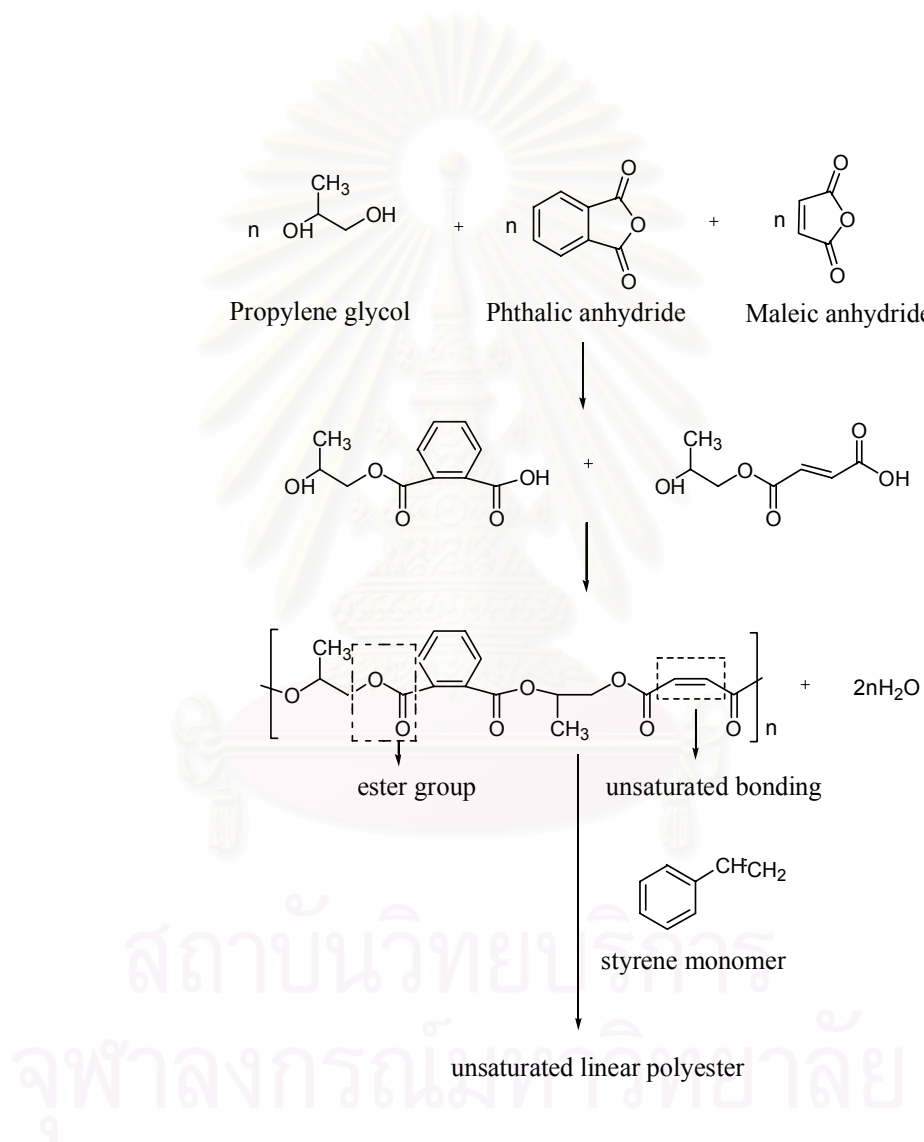
Figure 2.4   Unsaturated linear polyester synthesis.[11]

2.5 Surface modification of natural fibers [13]

Surface modification of a natural fiber may improve wet-out with a polymer, as well as dispersion of fillers in liquid polymer. The surface modifications protect the natural fibers against cleavage and even heal flaws in the surface. The polymer layer may be strengthened by interface modification that promotes alignment of polymer segments and interdiffuses with the polymer, possibly forming interpenetrating polymer network (IPNs). The natural fiber surface, which is generally hydrophilic, can be made hydrophobic, thereby increasing its compatibility, dispersibility, and processibility. The natural fiber modified surface is also less likely to adsorb and deactivate other formulation additives such as plasticizers, heat stabilizers, and antioxidants that are required for optimum performance.

Therefor, the quality of the fiber-matrix interface is significant for the application of natural fibers as reinforcement fibers for plastics. There are two method of surface modification that the physical and chemical methods can be used to optimize this interface. These modification methods are of different efficiency for the adhesion between matrix and fiber.

1) Physical methods

The physical methods, such as stretching, calandering, thermotreatment, and the production of hybrid yarns don't change the chemical composition of the fiber. Physical treatments change structural and surface properties of the fiber and thereby influence the mechanical bonding to polymers. In addition, the electric discharge (corona, cold plasma) is another way of physical treatments. The corona treatment is one of the most interesting techniques for surface oxidation activation. This process changes the surface energy of the cellulose fiber and in case of wood surface activation increase the amount of aldehyde groups. These methods are known to be very effective for "non-active" polymer substrates as polystyrene, polyethylene, polypropylene, etc.

The same effects are reached by cold plasma treatment. Depending on type and nature of the used gases, a variety of surface modifications could be achieved. Surface

crosslinking could be introduced, surface energy could be increase or decrease, reactive free radicals and groups could be produced.

2) Chemical methods

Strongly polarized cellulose fibers are inherently incompatible with hydrophobic polymers. When two materials are incompatible, it is often possible to bring about compatibility by introduction a third material that has properties intermediate between those of the other two. There are several mechanisms of coupling in materials:

- Weak boundary layers, coupling agents eliminate weak boundary layers
- Deformable layers, coupling agents produce a tough, flexible layers
- Restrained layers, coupling agents develop a highly crosslinked interphase region between substrate and polymer
- Wettability, coupling agents improve the wetting between substrate and polymer
- Chemical bonding, coupling agents from covalent bonds with both materials

The important chemical modification method is the chemical coupling method, which improves the interfacial adhesion, consequently, obtain better mechanical properties of composites. The fiber surface is treated with a compound that forms a bridge of chemical bonds between fiber and matrix. The surface modification of natural fibers for chemical method can be chemically treated with various compounds such as methanolamine, diisocynate, and sebacoyl chloride etc. All these reagents are expected to block the hydroxyl group of the cellulose thus making the fiber more hydrophobic and provide chemical bonds across the interface between organic polymer and mineral surfaces.

Inaddition, the effective method of chemical modification of natural fiber is graft copolymerization. This reaction is initiated by free radicals of the cellulose molecule. The cellulose is treated with an aqueous solution with selected ions and is exposed to high-energy radiation. For example, the treatments of cellulose with hot polypropylene-maleic anhydride (MAH-PP) copolymer. Then the cellulose molecule cracks and radicals are formed. Afterwards the radical sites of the cellulose are treated with a suitable solution

(compatible with the polymer matrix), for example vinyl monomer, acrylonitrile, and methyl methacrylate. The resulting co-polymer processes properties characteristic of both, fibrous cellulose and grafted polymer. The development of a definitive theory for the mechanism of bonding by coupling agent in composites is a complex problem. The main chemical bonding theory alone is not sufficient. So the consideration of other concepts appears to be necessary which include the morphology of the interphase, surface energy and the wetting phenomena.

2.6 Coupling agents [14, 15]

Coupling agents are defined primarily as material that improve the adhesive bond of dissimilar surfaces. The application of coupling agents for surface treatment of fillers and reinforcements in plastics have generally been directed toward improved mechanical strength and chemical resistance of composites related to improved adhesion across the interface. Although adhesion is central to any "coupling" mechanism, it is recognized that many factors are involved in total performance of a composite system. The interface, or interphase region, between polymer and filler involves a complex interplay of physical and chemical factors related to composite performance.

2.6.1 Organofunctional silanes

Silane coupling agents are chemical tools for the plastic compounder, they can increase filler loading, higher reinforcement levels and improve processing and end-product properties. Silane coupling agents, used in a wide range of composite products, enable the plastic compounder to maximize, as economically as possible, the contributions of individual components to the properties of the composites. Organosilanes are the main group of coupling for glass-fiber reinforced polymers. They have been developed to couple virtually any polymer to the minerals, which are used in reinforced composites. When components of differing chemical nature and physical form are combined, problems originate in the interface region where the organic resin phase must wet and spread over the inorganic filler or reinforcement surface. Silane coupling

agents provide significant benefits by modifying the interfacial region. Their unique chemistry permits reaction with organic materials.

2.6.1.1 Structure and chemistry of silane coupling agents

The generic composition of the silane coupling agents has a chemical structure as follows:

$$R - (CH_2)_n - Si(OR')_3$$
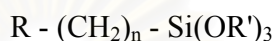
where

n	=	0 - 3
OR'	=	hydrolyzable alkoxy group
R	=	functional organic group
Si	=	silicon atom

The chemistry of preparation of this family of materials has been extentively studied in detail from both the formation of Si-C bond and the modification of Si- OR' groups. The organofunctional (R) in the coupling agent causes the reaction with the polymer. This could be a co-polymerization, and / or the formation of an interpenetrating network. The curing reaction of a silane treated substrate enhances the wetting by the resin.

Silane coupling agent provides chemical bonds between organic polymer and mineral surfaces. Methoxy silane may react directly with hydroxyl group on a mineral surface to form oxane bonds.The silane may also be prehydrolyzed to form silanetriols, which condense with surface hydroxyl groups and with each other to form an organosilicon bonded surface. The general mechanism of how alkoxysilanes form bonds with the fiber surface, which contains hydroxyl groups as shown in Figure. 2.5.

The modification of silane coupling agents may be necessary to retard initial bonding during mixing while allowing final bonding during molding. Table 2.7 shows a line of commercially available silane coupling agent, their structures and suitability for use with various matrix resins.
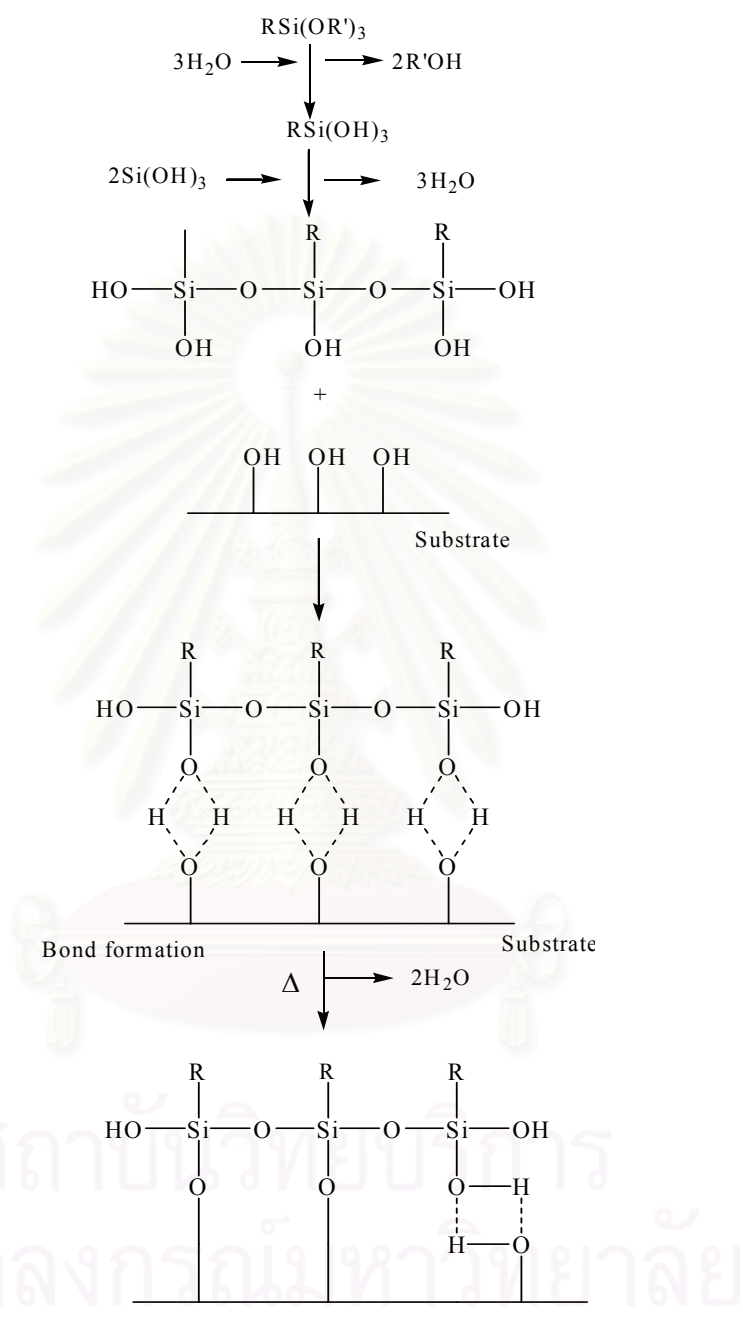
Figure 2.5   Mechanism of alkoxysilanes form bonds with the fiber surface, which
                  contains hydroxyl groups. [13]

Alkoxysilanes undergo hydrolysis, condensation (catalysts for alkoxysilane hydrolysis are usually catalysts for condensation) and the bond formation stage, under base as well as under acid catalyzed mechanisms. In addition to this reaction of silanols with hydroxyls of the fiber surface, the formation of polysiloxane structures can take place. The final reaction of silane treated substrate in a composites enhances the adhesion of the polymer matrix to be fiber substrate. The filler surface interacts with the polymer through catalytic activity, orientation of molecular segment, and other modification of polymer morphology. One important function of filler treatment by silane is to reduce the inhibitory action of filler on cure of thermosetting resin, and to promote a ligament of molecular segment of thermoplastic polymer. Mechanical stress across the interface may cause fracture in the filler particle, or under conditions of high temperature shear, may tear off polymer segments that have become chemically bonded during mixing operations.

Analogous to glass - fibers, silanes are used as coupling agents for natural-fiber-polymer composites. For example, the treatment of wood-fibers with methacrylate improves the dimensional stability of wood. In contrast, a decrease of mechanical properties was observed for coir-unsaturated-polyester composites after a fiber modification with dichloromethylvinyl silane. The treatment of mercerized sisal-fiber with aminosilanes before forming sisal-epoxy composites markedly improves moisture repellence of the composite. These examples show that theories used for the silane treatment of natural fibers are contradictory, therefore further studies are necessary.

2.7 Dispersibility [14]

Dispersibility of a filler is established primarily by 1) the chemical nature of the filler. 2) its surface characteristics. 3) its particle size, and 4) the process by which it is made. The chemical nature of the filler defines the bond strengths and many of the special surface characteristics that will be encountered in any given material. Filler manufacturers have generally gone to great lengths to define the chemical and surface characteristics of their materials. The physical and chemical properties of the filler surface will establish its tendency to aggregate and the strength of the clusters formed.

Particle size of the filler is also important to its ease of dispersion. Particle size of the filler is also important to its ease of dispersion. In general, the surface energies of particles greater than 1 μm is such that the driving force for aggregation is minimal and the aggregates that do form are generally weak. As the particle size drops below 1 μm, and particularly as it enters the colloidal region below 0.1 μm, the tendency of the particles to aggregate in large clusters increases progressively and substantially. In this region, protective colloids or surfactants must also be employed to stabilize the particles from reagglomerating once the initial aggregates are broken during the dispersion process.

Finally, the closely guarded processes by which the fillers are produced have an all-important effect on the ultimate dispersibility of the product. It is in this area that substantial effort is expanded to maintain the optimum filler performance at a minimum manufacturing cost. An incredibly extensive patent literature has grown out of an almost universal need by filler manufacturers to improve the ease of dispersion of their product. Favorite techniques include 1) the use of various surface treatments to change the surface chemistry and reduce the forces of particle - particle attraction and bonding and 2) methods to circumvent the drying process or reduce the forces of agglomeration that occur during drying. Clearly, techniques to improve pigment dispensability will continue to receive a great deal of attention from filler manufacturers and users.

In compounding, three basic dispersion processes are commonly used: dry blending, melt shear, and liquid dispersion. In practice, a combination of these techniques is often employed, although there are few hard and fast rules. The experience of the compounder is still the primary factor determining the dispersion processes used. The dry-blending technique achieves dispersion primarily by impact and attrition grinding. Unless followed by another dispersion process, there is little opportunity for effective wetting of the pigments by the resin. Melt shear dispersion is most commonly commonly employed in thermoplastics compounding and works well in follow-up combination with dry blending. The effectiveness of melt shear depends on the ease of wetting of the filler by the polymer and a high enough viscosity to allow high shear forces to be effectively transmitted to the filler aggregates. Liquid dispersion processes for plastisols involve predispersion of the filler in the plasticizer. Equipment type can vary, but the type of

dispersion forces is generally determined by viscosity, which is most often controlled by the filler concentration. The choice of dispersion technique usually is dictated by cost, the ultimate property requirements of the filled composite, and, finally, the dispersibility of the fillers required to provided those properties.

2.8 Fillers [16]

Fillers are defined as a variety of solid particulate materials that may be irregular, acicular, fibrous or plate-like in shape and which are used in reasonably large volume loading in plastic. Pigments and elastomeric materials are not normally included in this definition. Fillers can be classified based on application for three way: 1) reinforce that polymer and improve its mechanical performance. 2) use the take-up space and so reduce the amount of resin to produce a part sometime referred to as extender. 3) Less common, that are dispersed through the polymer to improve its electric conductivity.

There is significant diversity in the chemical structure, forms, shapes, sizes, and inherent properties of the various inorganic and organic compounds that are used as filler. They have usually rigid materials, immiscible with the matrix in both the molten and solid states, and, as such, form distinct dispersed morphologies. Their common characteristic is that they are used at relatively high concentration (>5% by volume), although some surface modifiers and processing aid are used at lower concentrations. Fillers may be classified as inorganic or organic substances, and further subdivided according to chemical family as shown in Table 2.3.

The most common filler used to reinforce polymeric material is glass fiber. However, wood fiber, which is commonly used as an extender, also increases the stiffness and mechanical performance of some thermoplastics. To improve the bonding between the polymer matrix and reinforcement, coupling agents such as silane and titanate are often added. Extenders, used to reduce the cost of the component, are often particulate fillers.

Table 2.3   Types of fillers for polymer.[16]

A. Glass	F. Silica products
1. Solid spheres	1. Minerals
2. Hollow spheres	(a) Sand
3. Flakes	(b) Quartz
4. Granules	(c) Novaculife
B. Carbon	(d) Diatomaceous earth
1. Carbon black	2. Synthetic amorphous silica
(a) Channel black	(a) Wet process silica
(b) Furnace black	(b) Fumed colloidal
2. Ground petroleum coke	(c) Silica aerogel
3. Pyrolyzed product	G. Silicates
C. Cellulosic fillers	1. Mineral
1. Wood flour	(a) Kaolin (China clay)
2. Shell flour	(b) Mica
3. Cellulose fiber	(c) Talc
D. Metal Powder	(d) Wollastonite
1. Aluminum	(e) Asbestos
2. Bronze	2. Synthetic products
3. Lead	(a) Calcium silicate
4. Zinc	(b) Aluminum silicate
E. Metallic oxides	H. Calcium carbonate
1. Zinc oxide	1. Barium sulfate
2. Alumina	2. Silicon carbide
3. Titanium dioxide	3 Molybdenum disulfide
4. Zirconia	4. Barium ferrite
F. Calcium carbonate	5. Potassium titanate
1. Chalk	J. Polymer
2. Limestone	1. Comminute polymer

2.9 Cellulose [16]

The existence of cellulose as the common material of plant cell walls.In 1838, Anselm Payen suggested that the cell walls of large numbers of plants consist of the same substance, to which he gave the name cellulose. It occurs in almost pure form in cotton fiber and in combination with other material, such as lignin and hemicellulose, in wood, plant leaves and stalks, etc.

It is generally accepted that cellulose is a linear condensation polymer consisting of D-anhydroglucopyranose units (often abbreviated as anhydroglucose units or even as glucose units for convenience) joined together by β-1, 4-glycosidic bonds. It is thus a 1, 4-β-D-glucan. The pyranose rings are in the $^{4}C_{1}$ conformation, which means that the - CH_2OH and -OH groups, as well as the glycosidic bonds, are equatorial with respect to the mean planes of the rings. The Haworth projection formula of cellulose as show in Figure 2.6.
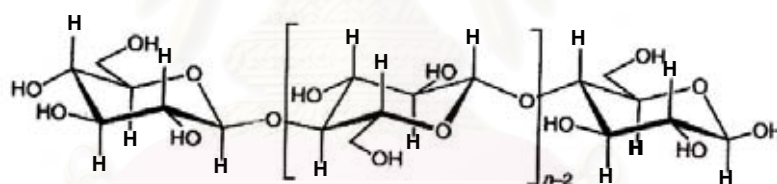
Figure 2.6   The molecular structure of cellulose.[13]

The molecular structure of cellulose is responsible for its supermolecular structure and this, in turn, determines many of its chemical and physical properties. In the fully extended molecule, adjacent chain units are orientated by their mean planes at an angle of 180° to each other. Thus, the repeating unit in cellulose is the anhydrocellubiose unit and the number of repeating units per molecule is half the degree of polymerization.

The chemistry of cellulose is primarily the chemistry of alcohol; and it from many of the common derivatives of alcohol, such as ester, ethers, etc. These derivatives form the basic for much of the industrial technology of cellulose in use today. Cellulose

derivatives are used commercially in two ways, as transient intermediates or as permanent products. Because of the strong hydrogen bonds that occur between cellulose chains, cellulose dose not melt or dissolve in common solvents. The mechanical properties of natural fibers depend on its cellulose type, because each type of cellulose has its own cell geometry and the geometrical conditions determine the mechanical.

2.10 Rice straw

Thailand is a tropical country with the agriculture based economy and the rice straws are the major agricultural residues, which are estimated that there are 0.32-1.6 tones of rice straw per a season. The average chemical composition of the rice straw is show in Table 2.4.

Table 2.4   The average chemical composition of the rice straw. [18]

Chemical composition	Value (%)
Crude protein	2.5
Crude fiber	28.0
Lignin	4.5
Cellulose	34.0
Ash	18.3
Silica	9.0
Nitrogen	0.75
Other	2.95

The rice straws were obtained in the fabrication from the stem plant of rice. However, there is an extensive literature dealing with utilization of the agricultural residues such as husk, cob and other waste part derived from plants are occasionally used in plastic molding.

2.11 Literature survey

During the last decade, the natural fiber as reinforcement in polymer composites has been the matter of a large number of published papers. There are many reports detailing studies of the modification of natural fiber surface to improve compatibility of fiber with polymer matrix are listed below.

Lexuthai [19] synthesized 2-diallyamino-4,6-dichloro-1,3,5-triazine as a coupling agent for coir fiber-unsaturated polyester composites. It was found that the modified coir fiber composites had better mechanical properties than the unmodified fiber composites. However, in comparison with glass-fiber reinforced composites, only the impact strength of modified coir-fiber composites was comparable to the glass fiber reinforced composites while the other mechanical properties were lower.

Nouwezen, *et al.* [20] studied the production and properties of thermosetting composites based on pine wood and unsaturated polyester mixtures by esterified pine wood with maleic anhydride. They found that the use of wood esterified with malelic anhydride lead to an increase of the constraint to flexion rupture, Rockwell toughness. This was more significant as the wood content increase.

Devi, *et al.* [21] studied the mechanical properties of pineapple-leaf fiber-unsaturated polyester composites by analyzing the influence of fiber length, fiber loading, and types of coupling agent on the in mechanical properties of the composites. Composites with good strength were obtained by using vinyltris(2-ethoxy methoxy) silane treated pineapple-leaf fiber with unsaturated polyester. Maximum strength was obtained at 30 %wt fiber loading and 30 mm.

Sreekala, *et al.* [22] studied the morphology and mechanical properties of oil palm fiber by analyzing the influence of chemical treatment such as alkali treatment and silane treatment. They found that the modulus and hydrophobicity of the fiber increase upon treatment using vinyltriethoxy silane a coupling agent and the morphological changes of fiber upon alkali and silane treatment are evident from the respective SEM photographs.

Kasemsook [23] studied the mechanical properties of kenaf fiber-unsaturated polyester composites by fiber surface were treated by coating with vinyltriethoxy silane (VTS) and γ-methacryloxypropyltri ethoxysilane (γ-MPA), which containing different organofunctional groups. It was found that the silane with methacrylate as the organofunctional group can improve the mechanical and water absorption properties of the composite better than a vinyl functional group.

Morcovich, *et al.* [24] studied the effect of modified wood flour and the filler content as thermoset filler on mechanical properties of unsaturated polyester composites. The wood flour was chemically modified with malelic anhydride by using different time of reaction. They found that the improve dispersion/adhesion with polymer matrix and maximum strength was obtained at 30% wt modified fiber.

Pothan, *et al.* [25] studied the dynamic mechanical behavior of chemically modified banana fiber rein forced polyester composites. Of the various silane coupling agent, vinyltriethoxy silane, and γ-methacryloxypropyl trimethoxysilane were used to modify the fiber surface. They found that the storage modulus of the fiber treated with the γ-methacryloxypropyl trimethoxysilane was increased and fiber-matrix adhesion in banana polyester system better than treated with vinyltriethoxy silane.

Valadezet, *et al.* [26] studied the mechanical properties of continuous henequen fibers- high density polyethylene composites by fiber surface modification using an alkaline treatment and a matrix preimpregnation together with a vinyltris(2-methoxy-ethoxy) silane. They found that the surface modification of henequen fiber with a silane coupling agent was carried out and the changes in their surface physicochemical properties were measured. The adsorption isotherm of silane treated henequen fiber was higher than untreated. The resulting strength and stiffness of the composites depended on the amount of silane deposited on the fiber. A maximum value for the tensile strength was obtained for a certain silane concentration.

According to the literature review, all the reports showed significant improvement in hydrophobicity of modified fibers. The mechanical strength of the composites which was also improved indicates that there was an improvement in the compatibility between the fiber and polymer matrix.

However, the methods used in most of the study to modify the fiber are rather complicate with the need of either synthesizing a new coupling agent or carrying out complicated reaction. It is; therefore, unlikely that they would find effective application of fiber modification. Moreover, in all the studies there have been no attempts to study the nature of the interactions between fiber and polymer matrix, which is the most important factor, determining the strength of a composite.

CHAPTER III

EXPERIMENTAL

3.1 Materials and Reagents

3.1.1 Unsaturated polyester resin

The unsaturated polyester resin used in this study was all liquid, commercially available material: POLYLITE SMF-8111, ortho phthalic type. It was supplied by Siam chemical Industry Co., Ltd. Typical characteristics and physical properties of POLYLITE SMF-8111 were shown in Table 3.1 and 3.2 respectively.

Table 3.1   Typical characteristic of POLYLITE SMF-8111.

Specification:	
Appearance	Clear translucent slight yellow
Viscosity (Ps, 25°C)	8.0-12.0
Color (Gardner)	2 max.
Gel time (min., 25°C)	5-10

Curing condition:

POLYLITE SMF-8111	=	100.0 g.
MEKPO (Butanox M-60)	=	2.0 g.

Post cured at 100 ^{o}C, 2 h.

Table 3.2   Physical properties of POLYLITE SMF-8111.

Water absorption (24 h)	ASTM D 570	0.2	%
Flexural strength	ASTM D 790	12.1	kgf./mm^2
Flexural modulus	ASTM D 790	450	kgf./mm^2
Tensile strength	ASTM D 638	6.3	kgf./mm^2
Tensile modulus	ASTM D 638	480	kgf./mm^2
Charpy impact strength	JIS K 6911	2.5	kgf./cm/cm^2

Table 3.3   The composition of POLYLITE SMF-8111.

Substances	Percent
Polyester resin	70
Styrene monomer	30

Applications:

POLYLITE SMF-8111 is suitable for FRP products i.e. FRP panels, water tank, cooling tower, bath tube, etc.

3.1.2 Rice straw

Rice straw was a material of natural cellulose used in this research was obtained from Phitsanulok province. The particle size of rice straw was 120-mesh (<75 μm).

3.1.3 Reagents

Table 3.4   The reagents

Chemicals	Grade	Source
Vinyltriethoxy silane (VTS)	AR	Fluka
Maleic anhydride (MA)	AR	Fluka
Triethylamine	AR	Fluka
Acetic acid	AR	Merck
Ethanol	AR	Merck
Hexane	AR	Merck
Toluene	AR	Fluka
Releasing agent	Commercial	TR, USA
Methyl ethyl ketone peroxide (MEKPO)	Commercial	Siam Chemical Industry

3.2 Apparatus and Instruments

1)   FTIR spectroscopy, Nicolet Impact, model 410

2)   Scanning electron microscopy, JOEL, model JSM T6400

3)   X-ray fluorescence spectroscopy, Philip, model PW 2400.

4)   Grinder machine, Retsh, model Type S1

5)   Size sieving machine 120 mesh

6)   Compression molding machine, LABTECH model V50h.

7)   Tensile testing machine, Shimabzu Autograph model AG1

8)   Flexural testing machine, Shimabzu Autograph model AG1

9)   Impact testing machine, Zwick-Materialprufung model GT 7045.

3.3 Procedures

3.3.1 Preparation of rice straw

Rice straw, which was a natural material composing of cellulose, was used in this research. It was obtained from Phitsanuloke province, and was chopped at the length of 10-15 cm. lengths. The chopped rice straw were dried at the temperature of 80 ^{o}C in the oven for 24 h. Then, the fine particles of rice straw were obtained by using grinding machine and size separated by using 120-mesh sieve.
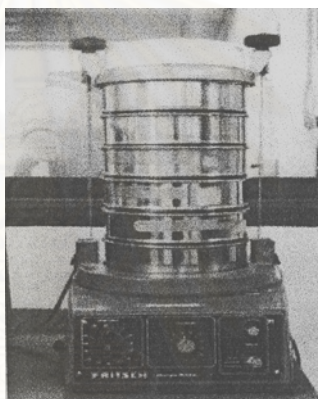
Figure 3.1   Size sieving machine.

3.3.2 Chemical treatment of the rice straw surface with coupling agents

The rice straw surface (120 mesh < 75 μm) was chemically modified with maleic anhydride and vinyltriethoxy silane as coupling agents as follows:

3.3.2.1 Maleic anhydride treatment

100 g. of rice straw (120 mesh< 75 μm) and a catalytic amount of triethylamine (1% mole MA) were added into the solution of maleic anhydride (MA) 10, 30, 60 g. in toluene 400 ml. and it was heated at reflux temperature for 8 h. The esterified rice straw particles were separated from the toluene solution by passing through a suction filter and

washed with dichloromethane in order to remove the unreacted meleic anhydride. Finally, the treated rice straw was dried at 100 ^{o}C in the oven until it was constant weight.

3.3.2.2 Silane treatment

Silanol solutions were prepared by adding vinyltriethoxy silane 10, 30, and 60 g. into distilled water of 400 ml. The pH of the solutions were adjusted to 3.5 with acetic acid and 100 g. of rice straw (120 mesh < 75 μm) was added into silanol solutions and was heated at reflux temperature for 4 h. The treated rice straw particles were separated from the silanol solution by passing through a suction filter and washed with ethanol and hexane, respectively in order to remove the unreacted silane. Finally, the treated rice straw was dried at 100 ^{o}C in the oven until it was constant weight.

3.3.3 Compounding and molding

The polymer composite sheet samples were prepared by using the hydraulic compression molding machine, LABTECH model V50h as shown in Fig. 3.1.

3.3.3.1 Preparation of neat unsaturated polyester sheets

The preparation was carried out in a glass plate molding, composing of two glass plates with the dimension of 20 cm. x 20 cm. x 3mm. The 100 g of unsaturated polyester resin (POLYLITE SMF 8-111) was mixed with 2 g of methyl ethyl ketone peroxide (MEKPO). One side of each the glass plate was coated with releasing agent and the mixture was placed over it on one of the glass plates. These two glass plates were placed one over the other in the way that compound sheet laying inside and facing one another. The compound was allowed to precure at room temperature for 1 h for curing. After that, it was post-cured at 100 ^{o}C in an oven for 2 h. Finally, the polyester sheet was removed from the glass plate molding.

3.3.3.2 Preparation of the composite sheet

The different types composite sheets were prepared as shown in Table 3.5.

Table 3.5   The different types and compositions of composite sheets.

| Composite | Rice straw (g) | | | | | | | UP (g) | MEKPO (g) |
| | Untreated | MA treated | | | VTS treated | | | | |
		10 %	30 %	60 %	10 %	30 %	60 %		
1	10	-	-	-	-	-	-	90	1.8
2	20	-	-	-	-	-	-	80	1.6
3	30	-	-	-	-	-	-	70	1.4
4	40	-	-	-	-	-	-	60	1.2
5	50	-	-	-	-	-	-	50	1.0
6	-	10	-	-	-	-	-	90	1.8
7	-	20	-	-	-	-	-	80	1.6
8	-	30	-	-	-	-	-	70	1.4
9	-	40	-	-	-	-	-	60	1.2
10	-	50	-	-	-	-	-	50	1.0
11	-	-	10	-	-	-	-	90	1.8
12	-	-	20	-	-	-	-	80	1.6
13	-	-	30	-	-	-	-	70	1.4
14	-	-	40	-	-	-	-	60	1.2
15	-	-	50	-	-	-	-	50	1.0
16	-	-	-	10	-	-	-	90	1.8
17	-	-	-	20	-	-	-	80	1.6
18	-	-	-	30	-	-	-	70	1.4
19	-	-	-	40	-	-	-	60	1.2
20	-	-	-	50	-	-	-	50	1.0
21	-	-	-	-	30	-	-	70	1.4
22	-	-	-	-	-	30	-	70	1.4
23	-	-	-	-	-	-	30	70	1.4

The procedures of the composites preparation are detailed as follows:

1) The compound was placed on metal molding with the dimension of 190 mm. x 190 mm. x 32 mm. that was coated by releasing agent and was compressed at 100 ^{o}C by hydraulic machine at the pressure of 140 psi for 30 min as shown in Fig. 3.2.

2) The compound was post cured at 100 ^{o}C in an oven for 2 h, then, the composite sheet was removed from the metal plate molding.

3) The composite sheets were cut into the standard specimens in according to the ASTM test method.

Figure 3.2   Compression molding machine LABTECH model V50h.

3.3.4 Water absorption test

Water absorption of the neat polyester and composites was determined according to ASTM D570-98. The specimen were heat at 50 $^{\circ}$C, 24 h then cool at room temperature in desicator until constant weight, which the specimen was weighed (m_1). After that time, the samples were immersed in distilled water at room temperature for 24 h, then swiped the sample with dry and clean utility, which the specimen was weighed (m_2) within 1 minutes after taken from water.

The percentage of water absorption can be represented in terms of the following equation 3.1:

$$\text{Water absorption (\%)} \; = \; \frac{(m_2 - m_1)}{m_1} \times 100 \qquad\qquad 3.1$$

where 　　　　m_1　=　weight before immersed in water

m_2　=　weight after taken from water

3.3.5 FTIR spectroscopy

The FTIR analysis was carried out by using a Nicolet Impact FTIR spectroscopy, model 410. The rice straw was then mixed with an analytical grade KBr and pressed into a pellet. The spectra were recorded in transmittance in the 4000-400 cm^{-1}range, with a resolution of 4 cm^{-1}.

3.3.6 Scanning electron microscopy

The surface of the fractured specimens from tensile testing, untreated rice straw, and treated rice straw were examined with a JOEL scanning electron microscope model JSM T6400. Scanning electron microscope operated at 15-25 kV. The specimens were coated with gold to prevent electrical charging.

3.3.7 X-ray fluorescence spectroscopy

The X-ray fluorescence spectroscopy can provide information about chemical composition of the samples. They were examined with a Philip X-ray fluorescence microscope model PW 2400. X-ray fluorescence microscope operated at 24 kV. The specimens were burnt in crucible at 970 $^{\circ}$C for 1.5 h and then compressed it into an aluminum ring to form it into a cylindrical shape.

3.3.8 Measurement of mechanical properties

3.3.8.1 Flexural Strength

Flexural strength of the neat polyester and composites were determined according to ASTM D790-91. Method I (A-three point loading).The testing was performed with the span-to-depth ration of 16:1.

ASTM D790-91: Standard test method for flexural properties.

The test specimen dimension was presented in Fig. 3.3.
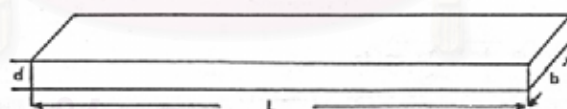
where L = 100 cm.

b = 13 cm.

d = 3 mm.

Figure 3.3 Specimen for flexural testing.

The flexural testing condition were as follows:

Temperature	25	$^{\circ}$C
Relative humidity	50	%
Speed of testing	1.5	mm/min.
Distance between grips	45	mm.

Procedure

Measure the width and depth of the specimen to the nearest 0.02 mm at the center of support span. Set the machine to the specified rate of crosshead motion, or as near as possible. Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading and supports. Apply the load to the specimen at the specified crosshead rate, until the specimen was rupture. Therefore, the flexural strength can be represented in terms of the following equation 3.2.

$$\text{Flexural strength} = 3PL/2bd^2 \qquad 3.2$$

Where   P   = maximum load at rupture, kgf

L   = span length, mm.

b   = width of specimen, mm.

d   = thickness of specimen, mm.

3.3.8.2 Tensile strength

The tensile strength of the neat polyester and composites were determined according to ASTM D 638-77 using the dumbbell-shaped specimens as shown in Fig. 3.4. A T/M Avery testing machine of the constant rate of cross-head movement type.

ASTM D638-77:   Standard test method for tensile properties.

The test specimen dimension was presented in Fig.3.4.
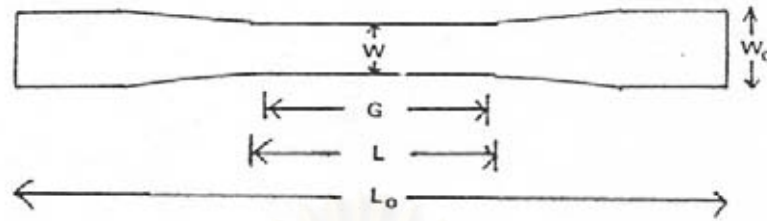
where	W	=	width of narrow section	13	mm.
	L	=	length of narrow section	57	mm.
	Wo	=	width of over-all	19	mm.
	Lo	=	length of over-all	165	mm.
	G	=	gauge length	50	mm.
	R	=	radius	76	mm.

Figure 3.4   Specimen for tensile testing.

The tensile testing condition were as follows:

Temperature	25	^{o}C
Relative humidity	50	%
Speed of testing	5	mm/min.
Distance between grips	115	mm.

Procedure

A specimen was gripped at the ends and pulled by the machine until fracture took place. The ultimate tensile strength was calculated from the applied load at a breaking point and the cross-sectional area of the specimen. Therefore, the tensile strength can be represented in terms of the following equation 3.3.

$$\text{Tensile Strength} \quad = \quad L / A \qquad\qquad 3.3$$

where   L   =   load at break, kgf

A   =   area, square mm.^{2}

3.3.8.3 Impact strength

The impact strength of the neat polyester and composite were determined according to ASTM D 256-78 using the rectangular specimens as shown in Fig. 3.5.

ASTM D638-77:   Standard test method for tensile properties.

The test specimen dimension was presented in Fig. 3.5.
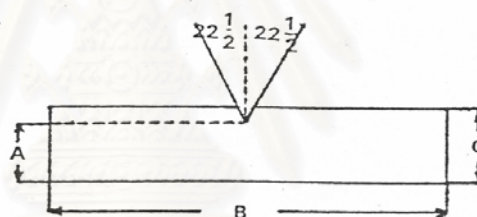
where   A   =   depth of the specimen, 10.16 +/- 0.5 mm.

B   =   length of the specimen, 63 mm.

C   =   width of the specimen, 12.70 +/-0.15 mm.

Figure 3.5   Specimen for impact testing.

The impact testing condition were as follows:

Temperature	25	$^{\circ}$C
Relative humidity	50	%
Pendulum capacity	11.0	J
Depth of specimen	10.16	mm.

Procedure

Position the specimen precisely and rigidly but not too tightly clamped in the vise. Release the pendulum from its normal starting position and record the excess energy in the pendulum after breaking the specimen. Therefore, the impact strength can be represented in terms of the following equation 3.4.

$$\text{Izod impact strength} = IE / b \qquad\qquad 3.4$$

where IE = impact energy, J

     b = width of the specimen, cm.

CHAPTER IV

RESULTS AND DISSCUSSION

4.1 Characterization

The structure of untreated and treated rice straw with different coupling agent were characterized by Fourier transform infrared spectroscopic (FTIR) analysis.

4.1.1 Fourier transform infrared spectroscopic (FTIR) analysis.

FTIR spectra of untreated rice straw and treated rice straw were shown in Figs. 4.1 a-c. All spectra were recorded in transmittance in the 4000-400 cm^{-1}range, with a resolution of 4 cm^{-1}.The corresponding molecular vibration were summarized in Tables 4.1-4.3.

FTIR spectrum of untreated rice straw (Fig.4.1a) indicated that the rice straw comprised mainly cellulose. It revealed the absorption of O-H stretching, C=C stretching, C-H bending and C-O vibration at 3411, 1640, 1432, and 1037 cm^{-1} respectively. The assignment of the absorption wave was summarized in Table 4.1.

Table 4.1   Assignment of the FTIR spectrum of untreated rice straw.

Wave number (cm $^{-1}$)	Characterization
3100-3600	O-H stretching
2800-3000	C-H stretching
1640	C=C stretching
1521	C=C aromatic ring stretching
1432	C-H bending
1060	C-O-C pyranose ring skeletal vibrations
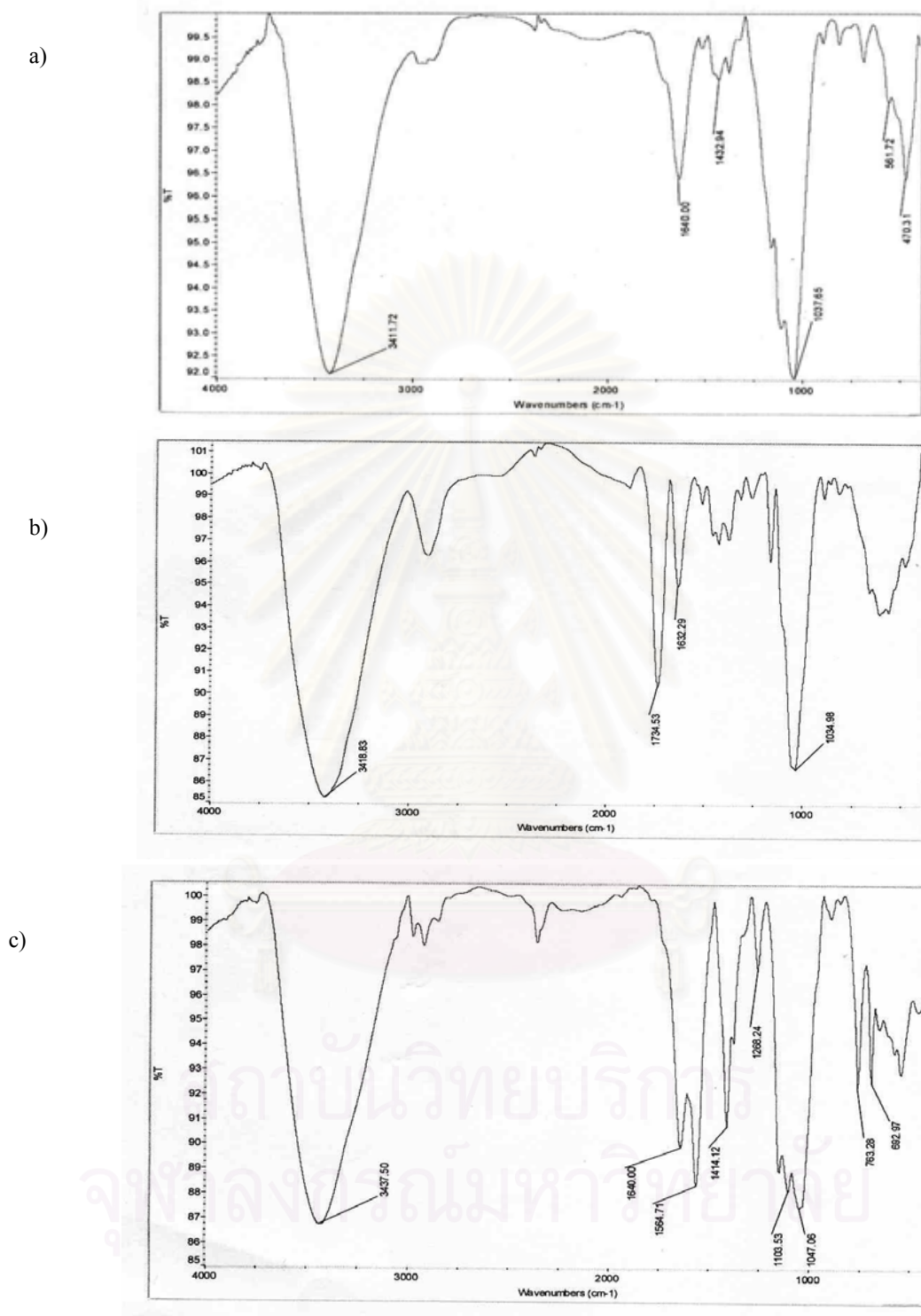
Figure 4.1 The FTIR spectrum of rice straw; a) untreated b) MA treated c) VTS treated.

FTIR spectrum of MA treated rice straw was shown Fig.4.1b. After treatment rice straw with MA, It was found the presence C=O at 1740 cm^{-1} of carbonyl group. This is evidence for the formation of ester linkage. The presence of this peak is an indication of the chemical bond on the rice straw surface that means an esterification reaction between the hydroxyl group of cellulose on the rice straw and MA. The assignment of the absorption wave was summarized in Table 4.2.

Table 4.2   Assignment of the FTIR spectrum of MA treated rice straw.

Wave number (cm^{-1})	Characterization
3100-3600	O-H stretching
2800-3000	C-H stretching
1740	C=O stretching
1640	C=C stretching
1424	C-H bending
1045	C-O-C pyranose ring skeletal vibrations

FTIR spectrum of the rice straws treated with MA (Fig. 4.1b), indicated that some changes have occurred as the result of the reaction between MA and cellulose fiber of the rice straw. The chemical reaction of the MA treated rice straw was shown in Fig.4.4.

$$Fiber\text{-}OH \quad + \quad C_4H_2O_3 \quad \longrightarrow \quad Fiber\text{-}OC_4H_3O_3$$

Figure 4.2   The chemical reaction of the MA treated rice straw.

FTIR spectrum of the rice straws treated with MA was shown in Fig. 4.1c. The appearance of absorption band at 1285 cm^{-1}, was an indication of the presence of the Si-O-C bond on rice straw surface and it mean that the condensation reaction between the silane coupling agent and the cellulose of rice straw may have taken place. [24] Alkoxy

silanes are able to form bonds with hydroxyl groups. Silanes undergo hydrolysis, condensation and the bond formation stage. Silanol can form polysiloxane structures by reaction with hydroxyl groups of the fibers.[36]

Table 4.3   Assignment of FTIR spectrum of VTS treated rice straw.

Wave number (cm^{-1})	Characterization
3100-3600	O-H stretching
2800-3001	C-H stretching
1640	C=C stretching
1414	C-H bending
1285	Si-O-C stretching
1047	C-O-C pyranose ring skeletal vibrations
763	Si-C stretching
692	Si-O-Si stretching

The difference of FTIR spectra of untreated rice straw and rice straw treated with silane coupling agent was shown in Figs. 4.1a and 4.1c., it was used to confirm the chemical reaction between the silane coupling agent and the cellulose on the rice straw. The chemical reactions of silane coupling agent were shown in Fig.4.5.

$$CH_2CHSi(OCH_2CH_3)_3 + 3H_2O \xrightarrow{\text{H}^+} CH_2CHSi(OH)_3 + C_2H_5OH \quad (4.1)$$

$$CH_2CHSi(OH)_3 + \text{Fiber-OH} \xrightarrow{\Delta} CH_2CHSi(OH)_2O\text{-Fiber} + H_2O \quad (4.2)$$
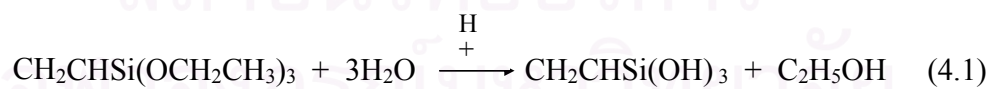
Figure 4.3   The reaction of VTS treated rice straw.

4.1.2 SEM/EDX spectroscopy (SEM/EDX)

The SEM/EDX spectroscopy provides information about the composition of the surface sample. The element analysis of untreated rice straw and rice straw treated with VTS coupling was examined by SEM/EDX spectroscopy models JSM 6400. The samples were previously coated with gold. The spectra of the element on the surface of untreated rice straw and of the treated one with 10% VTS, 30% VTS, and 60% VTS were shown in Figs. 4.7 a-c. According to the shown evidence in the element analysis micrograph, it showed that the amount of the silicon on the surface of the rice straw which was treated with VTS was higher than the untreated rice straw. The amount of the silicon on the surface of the rice straw was increased when the concentration of silane coupling agent was increased. This finding seems to confirm the presence of the silane group deposited on the surface of the rice straw.

4.1.3 X-ray fluorescence spectroscopy (XRF)

X-ray fluorescence was used as a method of quantitative analysis by measuring the wavelength of X-ray emitted from the sample. The sample was burnt in a crucible at 970°C for 1.5 h and was compressed into an aluminum ring to form it into a cylindrical shape. The concentration of silicon and other elements of untreated rice straw particles and rice straw treated with VTS were examined with X-ray fluorescence spectroscope, Phillip, model PW 2400.

Table 4.4   Silicon dioxide content in untreated and VTS treated rice straw at various concentrations.

Sample	% LOI	Factor	Concentration (%)	SiO_2 (%)
Untreated	88.5	0.11	82.82	9.49
10% VTS	85.4	0.15	91.64	13.56
30% VTS	78.6	0.21	95.12	20.39
60% VTS	70.6	0.29	96.90	28.52

The silicon dioxide (SiO$_2$) content of the untreated and of treated rice straw with silane coupling at various concentrations were shown in Table 4.4 and Fig. 4.6. The percentage of silicon dioxide can be represented in terms of the equation 4.1 shown below.

$$\% \ SiO_2 \quad = \quad Concentration \ (\%) \ x \ Factor \quad\quad 4.1$$

The result of the quantitative analysis corresponded to the SEM/EDX spectroscopy that the amount of the silicon content was increased when the concentration of silane coupling agent increased as shown in Fig. 4.7 and 4.16. The amount of silicon content indicated that silanol groups were deposited on the surface of rice straw, consequently, the morphology and physical properties of rice straw were changed.
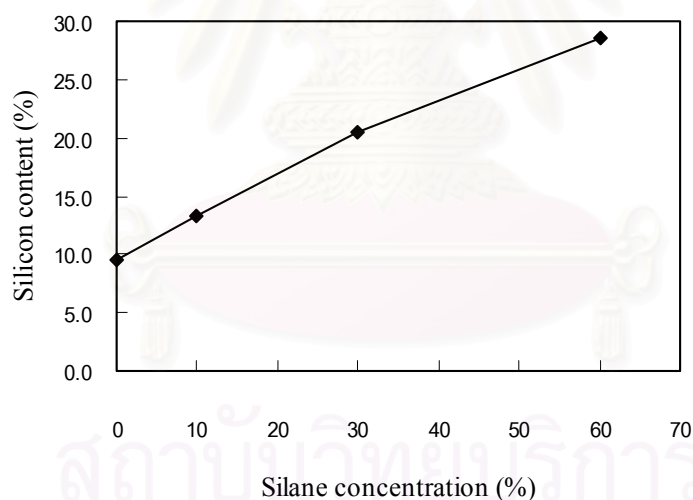
Figure 4.4   Silicon content (%) of untreated and VTS treated rice straw.
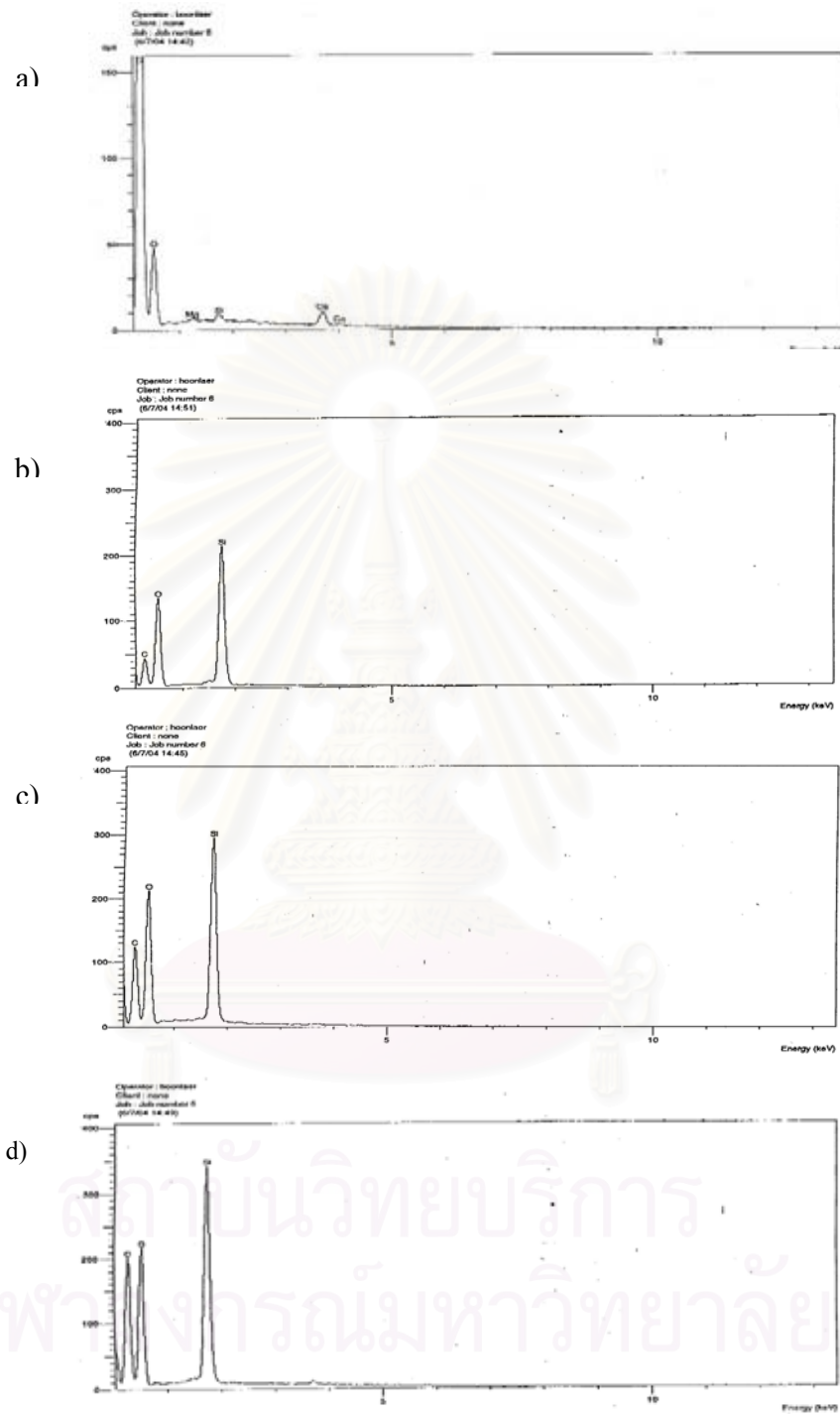
Figure 4.5 Elemental analysis micrographs of rice straw; a) untreated, b) 10% VTS treated, c) 30% VTS treated and d) 60% VTS treated.

4.2 Water absorption of the composites

The water absorption is an important property of the polymer composites; it has many effects on other mechanical properties such as impact strength, tensile strength and flexural strength of the composites. The samples were immersed in distilled water at room temperature for 24 h. At the saturation point, the water absorption depended on the chemical treatment of the surface.

4.2.1 The water absorption of MA treated rice straw composite.

The water absorption of untreated and MA treated rice straw composite were shown in Fig.4.6.
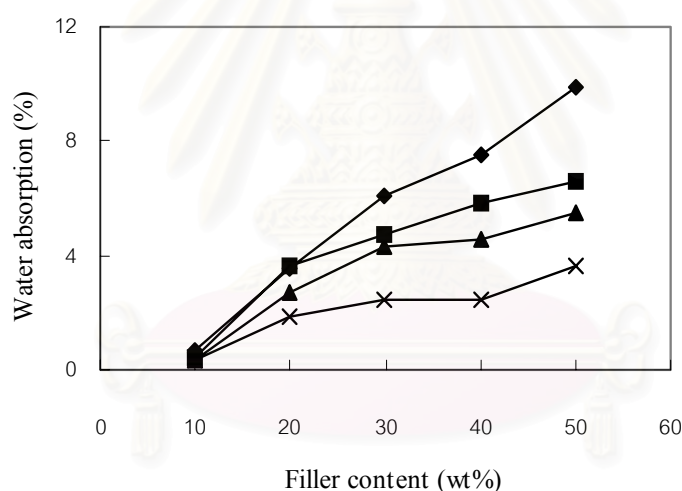
Figure 4.6 The water absorption of untreated and MA treated rice straw composites.; (♦) untreated, (■) 10% MA, (▲) 30% MA, and (x) 60% MA.

The water absorption was increased when the filler content was increased in the untreated rice straw- unsaturated polyester composites. The composites obtained from MA treated rice straw resulted in the water absorption lower than the untreated rice straw. This result indicated that the esterification of the rice straw helped reduce the water absorption of the composites. The rice straw which was treated with 60% maleic

anhydride showed the lowest degree of water absorption. This result might be from the esterification that reduced the number of cellulose hydroxyl groups, which have ability in absorbing moisture. The results are also in agreement with the improved mechanical properties of the composites.

4.2.2 The water absorption of VTS treated rice straw composite.

The results of water absorption of MA and VTS treated rice straw composites at 30% wt of filler content at room temperature were shown in Fig.4.7. The water absorption of MA treated was lower than VTS treated rice straw composite. The water absorption of composites was decreased with an increase of the concentration of MA and VTS. The result indicated that the esterification and silane reaction reduced the water absorption of composites.
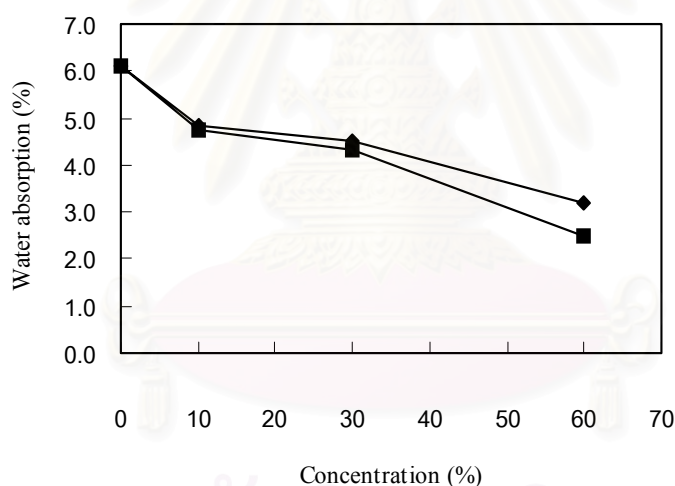
Figure 4.7   The water adsorption of VTS and MA treated rice straw composites at various concentrations; (■) VTS treated and (▲) MA treated.

As silane and MA treatments of rice straw can reduce the hydroxyl groups in the cell wall of molecules, thus the decrease of the water absorption of the rice straw-polyester composite was found. The composites of VTS treated rice straw resulted in the water absorption lower than untreated rice straw but higher than the MA treated rice straw composites. Chemical treatments of rice straw reduced some of the hydroxyl

groups, thus the reduction of the hygroscopic nature of the rice straw composites was found. This result has a benefit as a supplementary material regarding water absorption than the untreated rice straw. Strong intermolecular fiber-matrix bonding also decreased the rate of moisture absorption in the composite. In addition, water absorption of composite is depended on filler loading, permeability of the rice straw, etc. It is necessary to impart hydrophobicity to the natural fibers by chemical reaction with suitable coupling agents or by coating them with appropriate resins. Such surface modification of fiber does not only decrease moisture adsorption, but also concomitantly increases wettability of fibers with resin and improves the interfacial bond strength, which are crucial factors in obtaining better mechanical properties of composites.

4.3 Mechanical properties of the composite

Mechanical properties of the rice straw-unsaturated polyester composite could indirectly indicate the performance behavior of the interfacial adhesion between the filler and the matrix. In this research, three types of the mechanical properties of the composites were tested: tensile strength, flexural strength, and impact strength.

4.3.1 Tensile strength of untreated and MA treated rice straw composite.

The results of tensile strength of the composite samples containing 10-50% wt of untreated and MA treated rice straw at various concentrations were illustrated in Fig. 4.8.
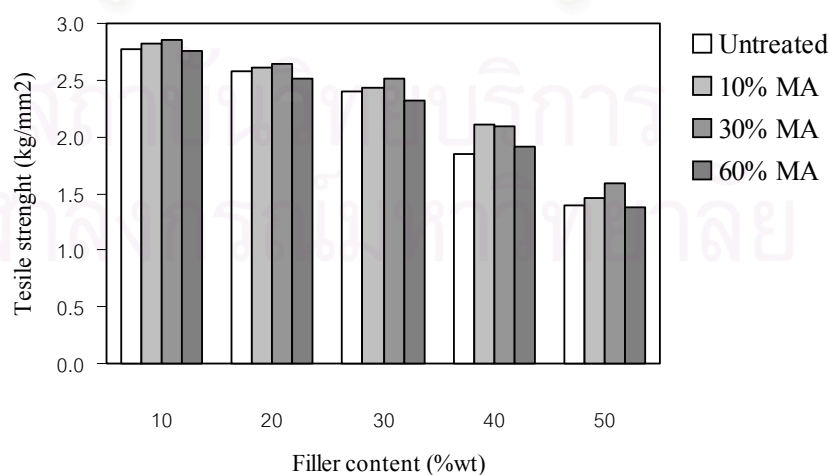
Figure 4.8   Tensile strength of rice straw-unsaturated polyester composite.

The tensile strength was decreased with the increase of filler content in untreated and MA treated rice straw-unsaturated polyester composites. Compared to the untreated rice straw composites, the tensile strength was found higher with the MA treatment. This indicates that the modification of rice straw with MA coupling agent has enhanced the efficiency of dispersion/adhesion of the polymer matrix. Moreover, the maximum strength was obtained at 30% wt modified fiber. However, at a higher concentration of 60% MA showed that the significant decrease of tensile strength due to the plasticizing effect on the surface of rice straw was also supported by SEM micrograph.

4.3.2 Flexural strength of untreated and MA treated rice straw composites.

The results of flexural strength of the composite samples containing 10-50 %wt of untreated rice straw and MA treated at various concentrations were shown in Fig. 4.9.
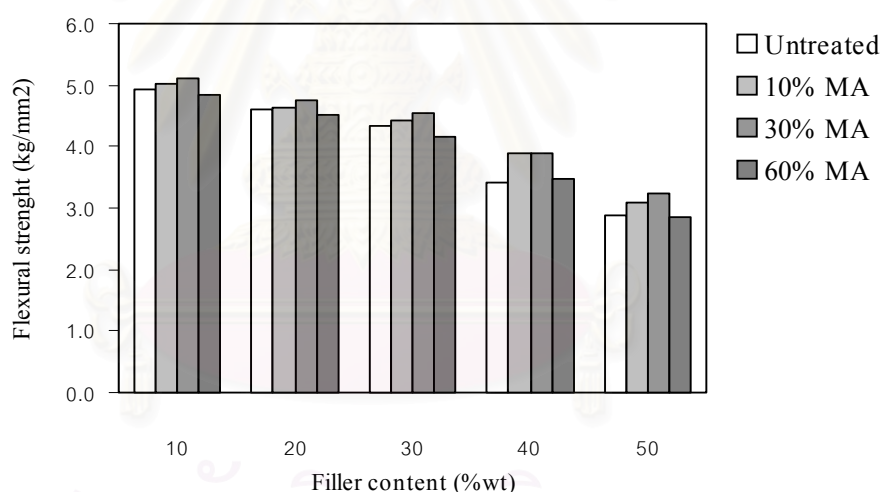
Figure 4.9   Flexural strength of rice straw-unsaturated polyester composites.

The results showed that the flexural strength was decreased with an increase of filler content in untreated and MA treated rice straw-unsaturated polyester composites. There is a slight improvement in flexural property of the composites made from rice straw treated with MA, as compared with the untreated rice straw composites. This may be noted that chemical adhesion between the fiber and the matrix induces the better of

flexural properties. At high concentration of 60% MA, it was found that the important decrease of flexural strength was due to the plasticizing effect. [26]

4.3.3 Impact strength of untreated and MA treated rice straw composite.
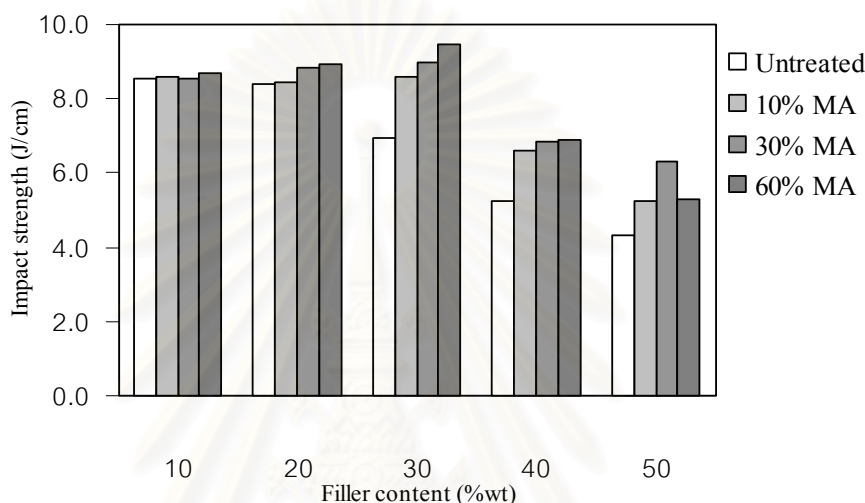
Figure 4.10   Impact strength of rice straw-unsaturated polyester composites.

The result of impact strength from Fig. 4.10 showed that the rice straw treated with MA with a smaller amount of filler can improve more impact strength of composites better than the untreated ones. This effect was due to the increased adhesion brought by the reaction of their reactive group of MA treated rice straw with the unsaturated polyester matrix which help support the absorption of the total energy of the surface fracture.

Thus, an increase of coupling agent concentration to treat the rice straw surface would increase the interfacial tension energy between the filler and polymer matrix. The result is that when the fracture toughness was increased, the impact strength of 10, 30, and 60% MA treated rice straw composites would be also respectively increased.

Consequently, the tensile and flexural properties of the MA treated rice straw composites were higher than those of the untreated rice straw composites at 10% and

30% MA concentration whereas it was found the significant decrease of both properties at 60% MA concentration. However, the mechanical properties of the MA treated rice straws composites are lower than those of the neat polyester resin.

4.3.4 Tensile strength of VTS and MA treated rice straw composite
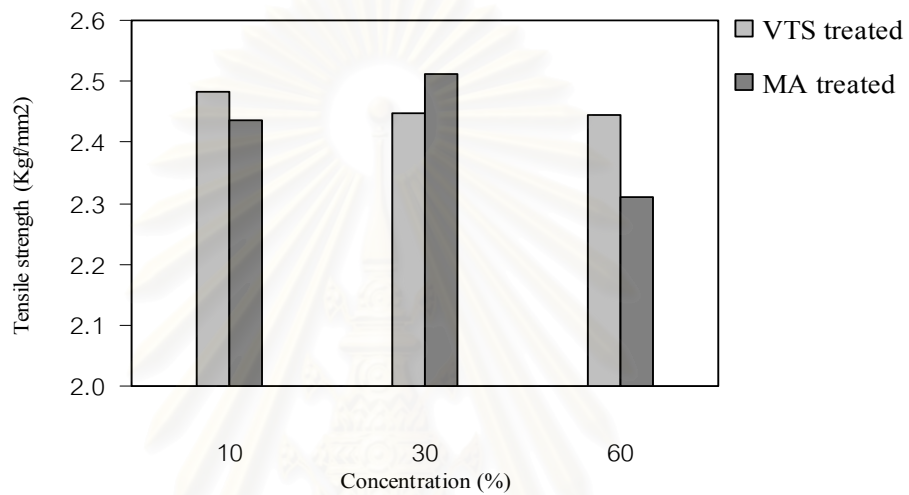
Figure 4.11   Tensile strength of rice straw composite at filler content 30% wt.

4.3.5 Flexural strength of VTS and MA treated rice straw composite
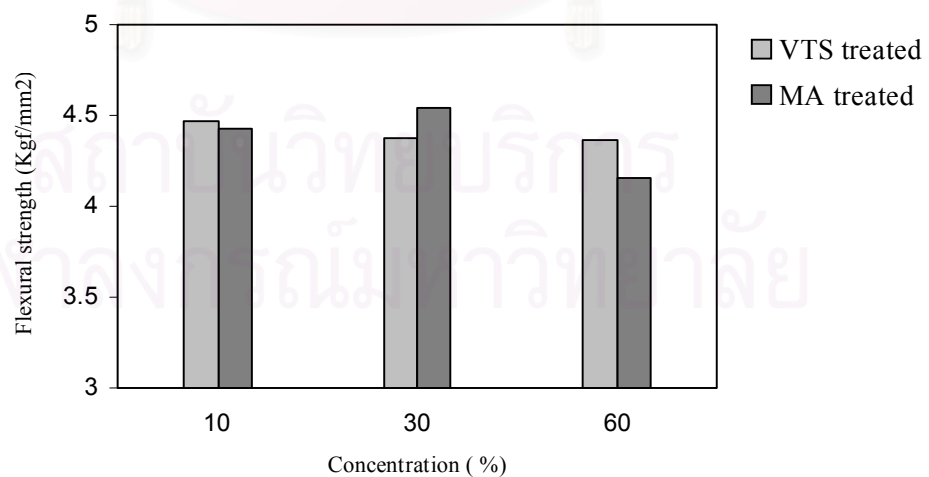
Figure 4.12   Flexural strength of rice straw composite at filler content 30% wt.

4.3.6 Impact strength of VTS and MA treated rice straw composite.
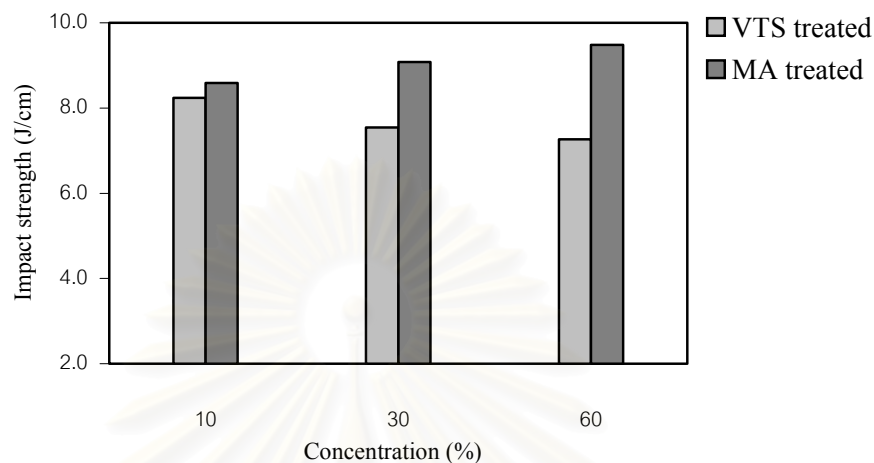
Figure 4.13   Impact strength of rice straw composites at filler content 30% wt.

The mechanical properties of the VTS treated rice straws composites containing 30% wt of filler content at various concentrations were shown in Figs. 4.11-4.13. From the result of the mechanical properties, it was observed that the composites with VTS treated rice straw improved their tensile strength, flexural strength, and impact strength, as compared with the untreated rice straw composites. The maximum strength obtained at 10% silane concentration indicated that the silane coupling agent can improve the adhesion by forming a thin interfacial layer. The interfacial layer was developed through chemical and physical interactions among the coupling layer, filler, and polymer matrix. As it is known that the molecular structure of the silane coupling agent, contains the ethoxy group (CH_3CH_2O) of the silane that can be easily turned into hydroxyl group under proper conditions by the possible hydrolysis reaction shown in Fig.4.3.

The hydroxyl group is hydrophilic in nature, therefore, the fiber surface is easily wetted by the hydrolyzed silane. Moreover, the hydrolyzed silane is able to react with the hydroxyl group on the fiber surface to form chemical bonds.[37] However, all mechanical properties of the composite were slightly decreased at high concentration of silane. This might be concluded that an increasing silane coupling concentration would

increase the strength of interbonding between the fibers and polymer matrix. The higher the strength of interbonding was found, the composite would be more brittle.

This result was confirmed by the elemental analysis that presents the silane group deposited on the surface of the rice straw.

It may be noted that chemical adhesion between the rice straw and the matrix induces better mechanical properties. However, the mechanical properties of the VTS treated rice straw composites were found poorer than those of the MA treated rice straw composites and those of the neat polyester resin.

4.4 Effect of the filler content on mechanical properties of the composite.

It was observed that the mechanical properties of untreated rice straw-unsaturated polyester composite i.e., tensile, flexural, and impact properties were decreased with the increase of filler content as presented in Figs.4.9-4.11. For the untreated rice straw as the filler, the deterioration of the mechanical properties of the composites may be due to the poor adhesion between the two phases of the filler and polymer matrix.

 In the case of chemical modification surface with coupling agent using MA and VTS, the result showed that the mechanical properties were elevated as the composites containing the optimum amount of 30% rice straw by weight. One However, another consideration was that, at the amount over the optimum fiber content, the capability of load-bearing of the composites was decreased. The loss of the capability to bear load of the fiber may be due to the fact that the interfacial adhesion between polymer and filler was obstructed in the way that the matrix cannot penetrate thoroughly the holes among the filler. This results in the increase of fiber density leading to poor resin penetration [26]. However, the mechanical properties of the untreated and treated rice straw composites were also found poorer than those of the polymer without filler.

A crucial observation in the process of sample preparation was that it impossible for the composite to obtain filler load more than 50% wt of rice straw. At this filler load, there is macroscopic decohesion of the composite components which cannot make a sheet of composite.

4.5 Effect of the chemical treatment on mechanical properties of the composites.

The comparison of mechanical properties of the unsaturated polyester resin filled with untreated rice straw, MA treated rice straw and VTS treated rice straw was summarized shown in Table 4.5 and Figs.4.8-4.13.

Table 4.5 The mechanical properties of the composites at 30%wt filler content.

Composite material	Coupling agent	Tensile strength (kgf/mm^2)	Flexural strength (kgf/mm^2)	Impact strength (J/cm)
UPE	-	5.663	9.821	13.407
Untreated	-	2.405	4.340	7.0703
MA treated	10%	2.436	4.430	8.654
MA treated	30%	2.512	4.542	9.080
MA treated	60%	2.314	4.161	9.395
VTS treated	10%	2.483	4.469	8.206
VTS treated	30%	2.449	4.371	7.534
VTS treated	60%	2.444	4.364	7.266

The mechanical properties of composites that were made from the modification of rice straw with 10, 30% MA concentration coupling agent have enhanced the efficiency of compatibility and dispersion/adhesion between polymer matrix and filler. This has an affect on the improvement of the mechanical properties of composites as compared with the untreated rice straw composites. Since the C=O groups of MA reacted with the active C=C site of the unsaturated polyester resin chain to form chemical bond, this result led to an increase of interfacial adhesion between filler and polymer matrix. However, at a higher concentration of MA, it was found that the decrease of tensile and flexural properties due to the plasticizing effect of MA treatment can also be the factor leading to

the increment in the toughness of the MA treated composites and could suffer larger plastic deformation than untreated rice straw composites. [26]

In the case of surface modification with VTS which slightly increases the mechanical properties of the composites as compared with the untreated rice straw composites, the improvement of a mechanical properties indicates that VTS helps improve the filler-matrix adhesion by the reaction of its reactive groups with the unsaturated polyester matrix. A possible coupling mechanism of polymer, silane coupling agent, and filler was that the silane may also be prehydrolyzed to form silanetriols which can condense with surface hydroxyl groups of the cellulose to develop covalent to form either siloxane bond or hydrogen bond as demonstrated in Fig. 2.5. [9] An increase of silane coupling agent concentration at 30% and 60% of VTS showed the decreasing of the mechanical properties was due to the higher interbonding that caused the composite become brittle. At any concentration of VTS, each VTS reacted with hydroxyl group at different content that made different mechanical properties to be obtained.

In the comparison of the chemical treatment surface of rice straw between MA and silane coupling agent, all mechanical properties of MA treated rice straw composites were better than those of VTS treated rice straw composites as shown in Table 4.5. The results indicated that the MA treatment produced high compatibility of rice straw – unsaturated polyester matrix as shown in Figs 4.15. This may be due to the much smaller size of MA, compared to VTS, that led the higher packing of the MA molecules on the fiber and polymer matrix can be obtained.

An important chemical modification method is the chemical coupling method which improved the interfacial adhesion. The fiber surface was treated with a compound that can form a bridge of chemical bonds between fiber and matrix. The enchanced interfacial adhesion can be confirmed by the result of the fracture surface shown in the SEM study. The scanning electron micrographs of the MA treated and VTS treated rice straw composites showed good dispersing effect and bonding on the interface evidenced by the fact that less rice straw particles were pulled out from the polyester resin matrix. All two coupling agents also acted as dispersing agents which was confirmed by SEM micrographs of fracture surface of the composites.

4.6 Scanning electron microscopy (SEM)

JSM The surface of the rice straw particles and the fracture surface of composites were examined by scanning electron microscopy, JOEL, model T6400.The samples were coated with gold.

The surface of the untreated rice straw particles was shown in Fig. 4.14 a. Uneven surface of the untreated rice straw particles showed certain globular protrusions. The surface of the MA treated rice straw particles were shown in Figs. 4.14 b-d. The MA treated rice straw showed the rather smooth surface. The surface of the VTS treated rice straw particles were shown in Figs. 4.14 e-g. In the case of modification surface with VTS produced a rough surface in comparison to the untreated and MA treated rice straw particles. This may be due to the silicon atom of silane coupling agent deposited on the surface of the rice straw.

Fig.4.15 showed the fracture surface of MA treated and untreated composites that obtained from tensile testing at the containing of 10-50% wt rice straw. The fracture surface morphology of 30% MA treated rice straw composites showed better compatibility and interfacial adhesion than untreated and VTS treated rice straw composites. The fracture surface of untreated composites showed the poor interfacial adhesion between rice straw and unsaturated polyester matrix. At the filler content more than 30%wt, it showed the holes on the matrix.

The fracture surface of MA treated rice straw composites showed the better compatibility and dispersibility than untreated rice straw composites. This result indicated high interfacial adhesion between the treated rice straw and polyester matrix. It can be concluded from this study that the MA act as a compatibilizer. The surface of rice straws treated with coupling agent exhibited interfacial adhesion improvement and resulted to mechanical properties enhancement.
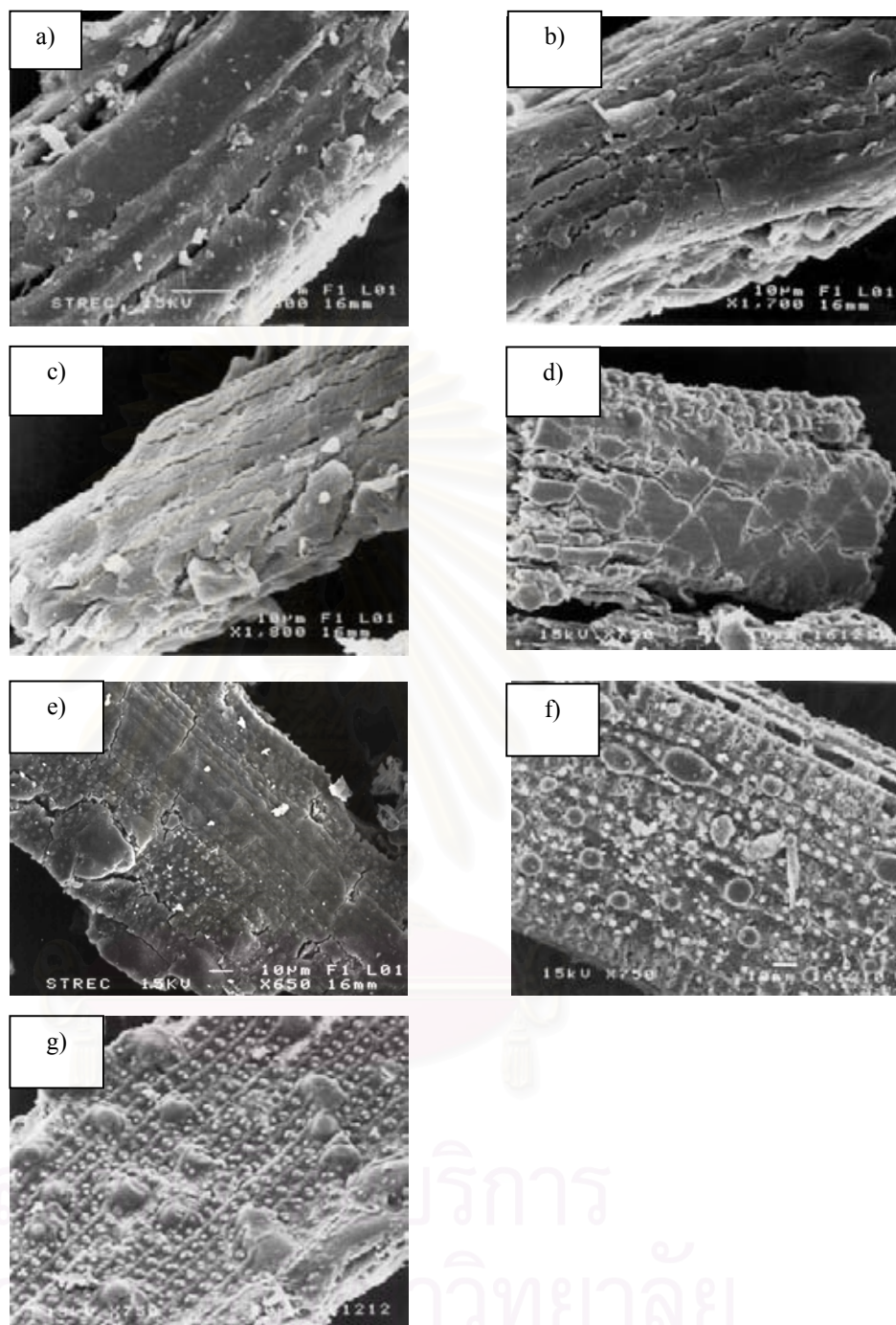
Figure 4.14 SEM micrographs of rice straw particles; (a) Untreated, (b) 10% MA,
(c) 30% MA, (d) 60% MA, (e) 10% VTS, (f) 30% VTS, and
(g) 60% VTS**.**
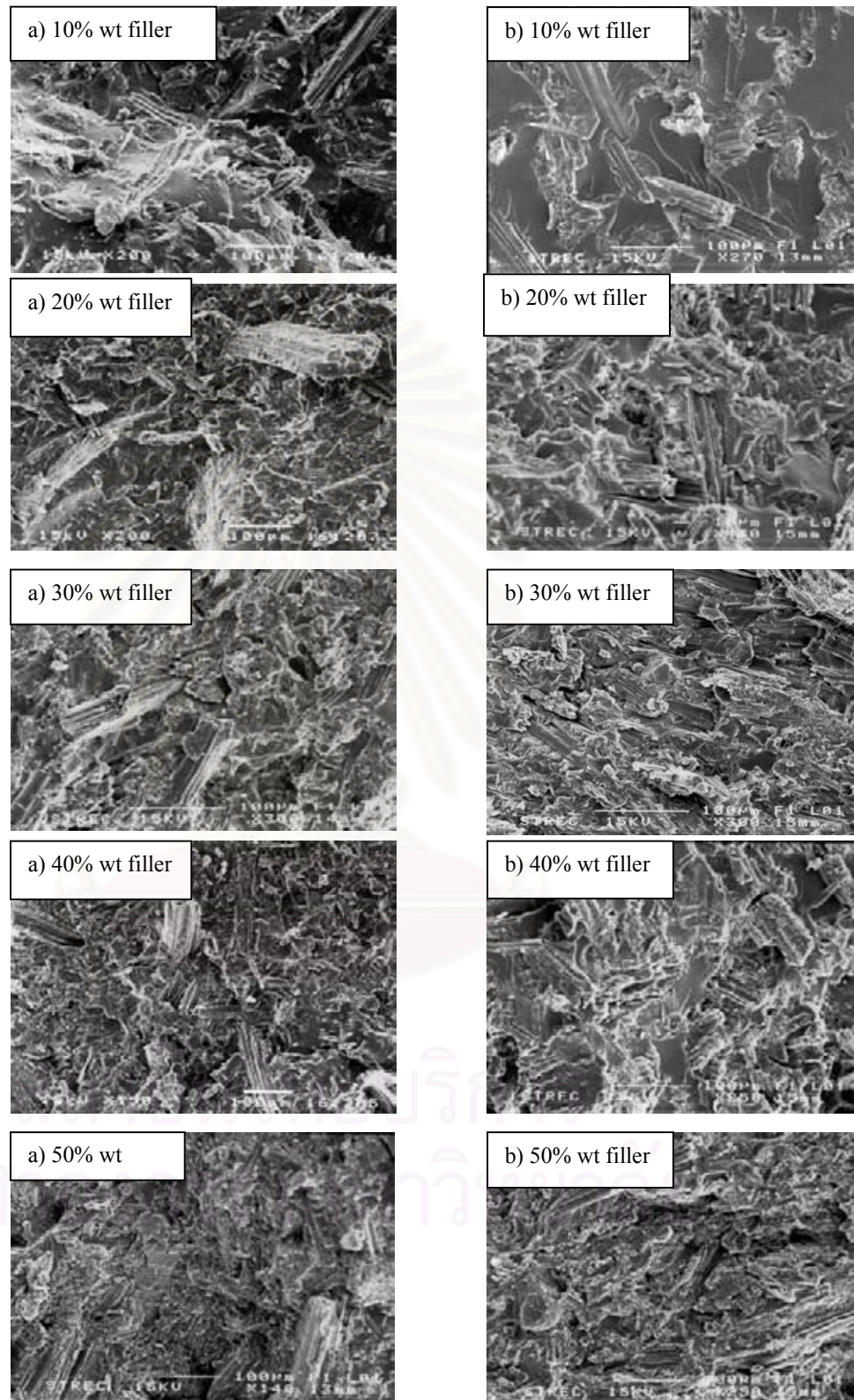
Figure 4.15 SEM micrographs of facture surface; (a) Untreated, b) 30% MA treated.
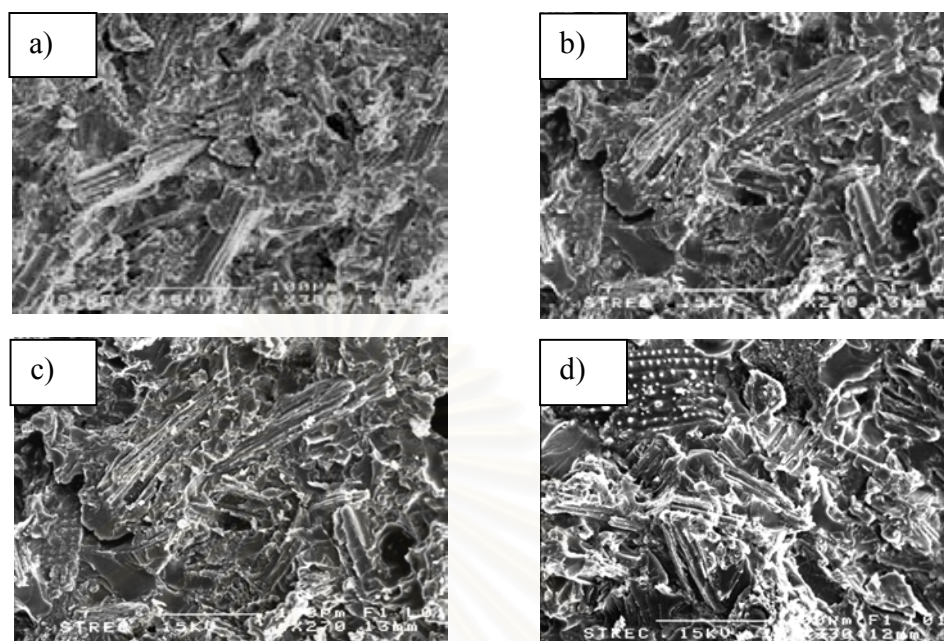
Figure 4.16 SEM micrographs of facture surface of composite at 30%wt filler content; (a) Untreated, (b) 10% VTS treated, (c) 30% VTS treated, and (d) 60% VTS treated.

The fracture surface of the composites from tensile testing at containing 30% wt rice straw were shown Figs. 4.16 a-d. The fracture surface of untreated rice straw composite showed the poor interfacial adhesion between rice straw and unsaturated polyester matrix. The VTS treatment showed the better compatibility than untreated rice straw. This result of silane coupling provided chemical bonds between organic polymer and rice straw surface.

CHAPTER V

CONCLUSIONS

5.1 Conclusion

       This research is the study of the preparation of the composites which were made from unsaturated polyester resin and the rice straws which were chemically modified with maleic anhydride (MA) and vinyltriethoxy silane (VTS) coupling agents at various concentrations. The surface modification of the rice straw with MA and VTS was carried out and the changes in their surface physicochemical properties were measured. Moreover, the influence of this surface treatment on the effective mechanical properties of the rice straw- unsaturated polyester resin composites was also assessed. The composites which were made from the modification of rice straw with 10% and 30% MA concentration displayed better mechanical properties than those of the unmodified rice straw, whereas in the composites made from the higher concentration of 60% MA, the decrease of tensile and flexural properties was found due to the plasticizing effect of MA treatment. This result can also be responsible for the increment in the toughness of this composite. The result indicated that the MA treatment produced high interfacial adhesion and compatibility of rice straw–polyester matrix. The enchanced interfacial adhesion can be confirmed by the result of the fracture surface by SEM study that showed the good dispersing effect and bonding at the interface which was evidenced by the resulted that less fillers were pulled out from the polyester resin matrix.

       For the surface modification with VTS, the mechanical properties of VTS treated rice straw composites were found slightly increased higher than those of unmodified rice straw composites and the mechanical properties were increased at a lower degree of silane solution concentration. It is generally approved that the adhesion between interface of the filler and the matrix can enhance mechanical properties of the composites due to the increment of the ability to transfer load from the matrix to the filler. The better adhesion between the filler surface and polymer matrix was the crucial element to indicate the better mechanical properties.

The comparison between the modified surface with VTS and of the MA modified composites showed the better mechanical properties. A 70:30 composite of unsaturated polyester resin and 30% MA treated rice straw showed the hightest mechanical properties compared to those composites. However, the mechanical properties of treated rice straws composites are lower than those of the pure polyester resin. The optimum filler content for the preparation of polymer composite was recorded at 30% wt. At the filler content higher than 30% wt, it resulted in the decrease of mechanical properties of composites. Moreover, the study found that the water absorption of MA and VTS treated rice straw composites can be reduced, compared to the untreated rice straw composite, approximately 29% and 20%, respectively.

The results of the experiment indicated that the modified rice straw composites had better mechanical properties than unmodified rice straw composites. It is indicated that the coupling agent improved interfacial adhesion and compatibility of the filler-polymer matrix. For the untreated rice straw as the filler, the deterioration of the mechanical properties of composites may be due to the poor adhesion of the two phases between the filler and polymer matrix which was confirmed by SEM micrographs of the tensile surface showing that filler was pulled out from the polymer matrix which indicated poor filler matrix interaction. The rice straw used in this research was obtained from agricultural residues appeared to be a potential filler for unsaturated polyester resin which can be used to reduce resin usage. The application of the modified rice straw surface composites can be used as the component of the products that do not need high strength.

5.2 Suggestion for further work

1. Prepare the unsaturated polyester composite with other materials such as natural rubber to improve the mechanical properties of the composite.
2. Synthesize the unsaturated polyester that has suitable viscosity for preparation of natural fiber composite.

REFERENCES

1. Reinhart, T. J.; Clements, L. L. <u>Engineering materials handbook</u>. Ohio: ASM International, 1989.

2. Hillig, W. B. <u>Encyclopedia of chemical technology</u> 15 (1981): 75-79.

3. Chanda, M. and Roy, S. K. <u>Plastic technology handbook</u>. New York: Marcel Dekker, 1977.

4. Richardson, T. <u>Composites: A design guide.</u> New York: Industrial press, 1987.

5. Mishra, S.; Mohanty, A. K.; Drzal, L. T.; Misra, M. and Parija, S. <u>Composites Science and Technology</u>. 63 (2003): 1377-1385.

6. Nicholls, R. <u>Composite construction material handbook</u>. New Jersey: Prentice-hall, 1976.

7. Ralph, E. <u>Injection / Transfer molding of thermosetting plastics</u>. Cincinnati: Hanser Gardner Publication, 1995.

8. Parratt, N. J. <u>Fiber-Reinforced Material Technology</u>. London: Van Nostrand Reinhold Company, 1972.

9. Tadmor, Z.; and Gogos, C. G. <u>Principal of polymer processing</u>. John Willey & Sons, 1937.

10. Tim, A. O. <u>Polymer processing fundamentals</u>. Cincinnati: Hanser Gardner Publication, 1990.

11. Lawrence, J. R. <u>Plastic application series</u>. London: Van Nostrand Reinhold Company, 1960.

12. Seymour, R. B. <u>Additive for plastic Academic Press</u>. New York: Marcel Dekker, 1978.

13. Satyanayana, K.; Kulkarri, A, G.; and Rohatgi, P, K. Potential of natural fibers as a resource for industrial materials in kerala. <u>J. Sci. and Ind. Res</u>. 40 (1981): 222-237.

14. Odian, G. <u>Principal of polymerization</u>. New York: John Willey & Sons, 1933.

15. Plueddeman, E. P. <u>Silane Coupling Agent</u>. New York: Plenum Press, 1982.

16. Harry, S.; Katz, H.; John, V.; and Mileevski, N. <u>Handbook of fillers and reinforcements for plastic</u>. London: Van Nortrand Reinhold Company, 1982.

17. Sperling, L. H. <u>Introduction to Physical polymer science</u>. New York: John Willey & Sons, 1932.

18. Wollerdorfer, M.; and Bader, H. Influence of natural fibres on the mechanical properties of biodegradable polymers. <u>Industrial Corps and Production</u>. 8 (1998): 105-112.

19. Lekuthai, S. Modification on adhesion properties of coir fiber for reinforced plastics, <u>Mater's Thesis of Petrochemistry and Polymer science</u>, Faculty of Science, Chulalongkorn University, 1990.

20. Nouwezen, R.; Borredon, M. E.; and Gaset, A. Production and properties of thermosetting composites based on wood and unsaturated polyester mixtures. <u>Bioresources technology</u>. 48 (1994): 17-19.

21. Devi, L. U.; Bhagawan, S. S.; and Thomas, S. Mechanical properties of pineapple leaf fiber-reinforced polyester composites. <u>J. Appl Polym Sci</u>. 64 (1997): 1739-1748.

22. Sreekala, M. S.; Kumaran, S.; Joseph, P.; and Jacob, M. Oil plam fiber reinforced phenol formaldehyde composites: Influence of fiber surface modifications on the mechanical performance. <u>Applied composite materials</u>. 7 (2000): 295-329.

23. Kasemsook, N. The use of silane coupling agent in natural fiber-unsaturated polyester composite. <u>Master's Thesis of the Petroleum and Petrochemical college</u>, Chulalongkorn University, 1999.

24. Marcovich, N. E.; Reboredo, M. M.; and Aranguren, M. I. Modified woodflour as thermoset fillers II: Thermo degradation woodflours and composites. <u>Thermochimica acta</u>. 372 (2001): 45-57.

25. Pothan, L. A.; and Thomas, S. Polarity parameter and dynamic mechanical behaviour of chemically modified banana fiber reinforce polyester composites. <u>Composites science and technology</u>. 63 (2003): 1231-1240.

26. Valadez-Gonzalez, A.; Cervantesp, J. M.; Olayo, U. R.; and Herrera-Fanco, P. J. Chemical modification of henequen fibers with an organosilane coupling agent. <u>Composites Part B</u>. 30 (1999): 321-331.

27. Banchorndhevakul, S. Effect of urea and urea gamma treatment on cellulose degradation of Thai rice straw and corn stalk. <u>Radiation Physics and chemistry</u>. 64 (2002): 417-422.

28. Mishra, S.; Naik, J. B.; and Patil, Y. P. The compatibilising effect of maleic anhydride on swelling and mechanical properties of plant-fiber-reinforced novolac composites. <u>Polymer</u>. 60 (2000): 1729-1735.

29. Gowda, T. M.; Naidu, A. C.; and Chhaya, R. Some mechanical properties of unsaturated jute fabric reinforced polyester composites. Composites: Part A. 30 (1999): 277-284.

30. Samal, R. K.; Panda, B. B.; Rout, S. K.; and Mohanty, M. Effect of chemical modification on FTIR spectra I. Physical and chemical behavior of coir. J. Appl Polym Sci. 58 (1995): 745-752.

31. Samal, R. K.; and Ray, M. C. Effect of chemical modification on FTIR spectra. II. Physicochemical behavior of pineapple leaf fiber (PALF). J. Appl Polym Sci. 64 (1997): 2119-2125.

32. Sinsh, B.; Gupta, M.; and Verma, A. Polyester moulding compounds of natural fibres and wollastonite. Composites: Part A. 34 (2003): 1035-1043.

33. Marcovich, N. E.; Aranguren, M. I.; and Reboredo, M. Modified wood flour as thermoset fillers Part I: Effect of the chemical modification and percentage of filler on the mechanical properties. Polymer. 42 (2001): 815-825.

34. Jane, L.; and Dell, O. Natural fiber in resin transfer molding composite. Composites: Part A. 25 (1995): 115-127.

35. Sreekala, M. S.; Kumaran, G. M.; and Sabu, T. Oil palm fiber: morphology, chemical composition, surface modification, and mechanical. J. Appl Polymer Sci. 66 (1997): 821-835.

36. Martina, W.; and Herbert, B. Influence of natural fiber on the mechanical properties of biodegradable polymer. J. Appl Polym Sci. 8 (1978): 105-112.

37. Gasson, J. A study of fiber and interface parameters affecting the fatigue behavior of natural fibers composites. Composite: Part A. 33 (2002)**:** 369-374.

38. Kongsak, D. On enhancement of interfacial adhesion induced by coupling reaction of s-Triazine in polyester composites. Mater's Thesis of Petrochemistry and Polymer science, Faculty of Science, Culalongkorn University, 1994.

39. Bledzki, A. K.; and Gassan, J. A study of fiber and interface parameters affecting the fatigue behavior of natural fibre composites. Prog. Polym. Sci. 24 (1999): 221-274.

Appendices

APPENDIX A

Tensile strength (ASTM D638)

Date :   5/01/05

Operator :   Jarunee Suriyo

Test Type :   Tensile

V1 :   5 mm/min

Sample : Untreated 10g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.09	11.49	50.00
1 - 2	3.09	11.12	50.00
1 - 3	3.07	10.67	50.00
1 - 4	3.11	11.45	50.00
1 - 5	3.09	10.47	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	99.41681	0.80287	0.69815	2.80015
1 - 2	93.44282	0.77494	0.67386	2.71946
1 - 3	93.18061	0.81753	0.71090	2.84461
1 - 4	96.37676	0.78636	0.68379	2.70649
1 - 5	91.03096	0.83628	0.72720	2.81374
Mean	94.68959	0.80360	0.69878	2.77689

Date :   5/01/05

Operator :   Jarunee Suriyo

Test Type :   Tensile

V1 :   5 mm/min

Sample : Untreated 20g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.04	10.75	50.00
1 - 2	3.09	11.02	50.00
1 - 3	3.12	10.59	50.00
1 - 4	3.08	10.89	50.00
1 - 5	3.17	10.30	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	86.95952	0.78611	0.68357	2.66094
1 - 2	87.81312	0.76921	0.66888	2.57881
1 - 3	84.85274	0.76200	0.66261	2.56812
1 - 4	85.77961	0.77465	0.67361	2.55744
1 - 5	82.27203	0.77643	0.67516	2.51974
Mean	85.53540	0.77368	0.67277	2.57701

Date :   5/01/05   Test Type :   Tensile
Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : Untreated 30g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.12	11.94	50.00
1 - 2	3.07	11.02	50.00
1 - 3	3.05	10.53	50.00
1 - 4	3.06	10.16	50.00
1 - 5	3.10	11.40	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	89.87722	0.75545	0.65691	2.41263
1 - 2	81.04447	0.75561	0.65705	2.39554
1 - 3	77.88701	0.75503	0.65655	2.42514
1 - 4	76.18848	0.75507	0.65658	2.45061
1 - 5	82.76557	0.71738	0.62381	2.34198
Mean	81.55255	0.74771	0.65018	2.40518

Date :   5/01/05   Test Type :   Tensile
Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : Untreated 40g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.12	11.27	50.00
1 - 2	2.97	10.78	50.00
1 - 3	3.09	10.85	50.00
1 - 4	3.16	10.16	50.00
1 - 5	3.21	10.74	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	63.51138	0.55462	0.48228	1.80623
1 - 2	60.80337	0.57226	0.49761	1.89912
1 - 3	63.43415	0.57211	0.49749	1.89206
1 - 4	60.72582	0.57518	0.50015	1.89144
1 - 5	61.80922	0.55074	0.47891	1.79285
Mean	62.05679	0.56498	0.49129	1.85634

Date :   5/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : Untreated 50g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.17	10.87	50.00
1 - 2	3.21	11.05	50.00
1 - 3	3.11	10.97	50.00
1 - 4	3.15	10.16	50.00
1 - 5	2.74	11.50	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	47.29037	0.44369	0.38582	1.37241
1 - 2	50.40926	0.46090	0.40078	1.42116
1 - 3	46.12714	0.44934	0.39073	1.35204
1 - 4	45.28150	0.47520	0.41322	1.41487
1 - 5	43.67142	0.45611	0.39661	1.38722
Mean	46.55594	0.45705	0.39743	1.38954

Date :   6/01/05   Test Type :   Tensile
Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 10%MA 10g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.10	11.13	50.00
1 - 2	3.07	11.54	50.00
1 - 3	3.14	11.25	50.00
1 - 4	3.01	10.98	50.00
1 - 5	3.00	11.05	50.00

Name	Max_Force	Max_Disp	elongation	tensile/strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	97.90088	0.79560	0.69183	2.83746
1 - 2	98.48964	0.78655	0.68395	2.78001
1 - 3	97.69270	0.77739	0.67599	2.76554
1 - 4	94.08386	0.80315	0.69839	2.84673
1 - 5	95.25520	0.80719	0.70190	2.87346
Mean	96.68446	0.79398	0.69041	2.82064

Date :   6/01/05   Test Type :   Tensile
Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 10%MA 20g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.06	11.62	50.00
1 - 2	3.13	11.34	50.00
1 - 3	3.07	11.15	50.00
1 - 4	3.04	10.16	50.00
1 - 5	3.00	10.48	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	91.39267	0.73733	0.64116	2.57030
1 - 2	91.82846	0.75403	0.65568	2.58714
1 - 3	88.97808	0.75365	0.65535	2.59938
1 - 4	83.76515	0.78174	0.67977	2.71204
1 - 5	81.26061	0.74180	0.64504	2.58894
Mean	87.44500	0.75371	0.65540	2.61156

Date :   6/01/05   Test Type :   Tensile
Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 10%MA 30g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.09	12.22	50.00
1 - 2	3.02	11.90	50.00
1 - 3	3.08	12.05	50.00
1 - 4	3.10	10.16	50.00
1 - 5	2.82	11.32	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	88.94170	0.68218	0.59320	2.35546
1 - 2	88.77477	0.71744	0.62386	2.47022
1 - 3	89.31224	0.70171	0.61018	2.40643
1 - 4	77.66126	0.71382	0.62071	2.46575
1 - 5	79.19894	0.72413	0.62968	2.48539
Mean	84.77778	0.70786	0.61553	2.43665

Date :   6/01/05   Test Type :   Tensile
Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 10%MA 40g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.11	11.91	50.00
1 - 2	3.04	12.10	50.00
1 - 3	3.00	12.16	50.00
1 - 4	3.18	10.16	50.00
1 - 5	2.89	12.02	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	76.22075	0.61446	0.53432	2.05779
1 - 2	78.14246	0.64320	0.55930	2.12436
1 - 3	78.25179	0.63746	0.55431	2.14506
1 - 4	66.13579	0.61545	0.53517	2.04699
1 - 5	74.83391	0.64731	0.56287	2.15425
Mean	74.71694	0.63158	0.54920	2.10569

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 10%MA 50g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.05	11.24	50.00
1 - 2	3.06	11.77	50.00
1 - 3	3.10	11.01	50.00
1 - 4	3.09	10.16	50.00
1 - 5	3.09	10.47	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	48.49635	0.44538	0.38729	1.41463
1 - 2	54.10101	0.47391	0.41210	1.50213
1 - 3	47.79535	0.44742	0.38906	1.40035
1 - 4	43.94494	0.44573	0.38759	1.39977
1 - 5	51.08008	0.51204	0.44525	1.57887
Mean	49.08354	0.46490	0.40426	1.45915

Date : 6/01/05   Test Type : Tensile
Operator : Jarunee Suriyo   V1 : 5 mm/min

Sample : 30%MA 10g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.09	10.96	50.00
1 - 2	3.07	10.26	50.00
1 - 3	3.12	10.44	50.00
1 - 4	3.10	10.16	50.00
1 - 5	3.13	10.43	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	101.40277	0.76324	0.66369	2.99420
1 - 2	93.40823	0.75984	0.66073	2.96551
1 - 3	91.41947	0.71731	0.62374	2.80662
1 - 4	87.65400	0.69950	0.60827	2.78302
1 - 5	87.85338	0.69080	0.60070	2.69110
Mean	92.34757	0.72614	0.63142	2.84809

Date : 6/01/05   Test Type : Tensile
Operator : Jarunee Suriyo   V1 : 5 mm/min

Sample : 30%MA 20g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.12	12.10	50.00
1 - 2	3.09	11.97	50.00
1 - 3	3.08	11.89	50.00
1 - 4	3.07	12.09	50.00
1 - 5	3.08	12.00	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	98.27223	0.68577	0.59633	2.60310
1 - 2	100.08541	0.70311	0.61140	2.70594
1 - 3	98.85673	0.70358	0.61181	2.69944
1 - 4	97.08251	0.69037	0.60032	2.61563
1 - 5	96.01986	0.68844	0.59864	2.59794
Mean	98.06335	0.69426	0.60370	2.64441

Date :   6/01/05

Operator :   Jarunee Suriyo

Test Type :   Tensile

V1 :   5 mm/min

Sample : 30%MA 30g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.07	12.26	50.00
1 - 2	3.13	11.65	50.00
1 - 3	3.01	10.85	50.00
1 - 4	3.04	10.16	50.00
1 - 5	3.11	12.63	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	97.87099	0.69991	0.60862	2.60031
1 - 2	90.72404	0.68095	0.59213	2.48801
1 - 3	80.58322	0.66503	0.57828	2.46745
1 - 4	78.80109	0.69199	0.60173	2.55132
1 - 5	96.03486	0.66840	0.58121	2.44886
Mean	88.80284	0.68126	0.59240	2.51119

Date :   6/01/05

Operator :   Jarunee Suriyo

Test Type :   Tensile

V1 :   5 mm/min

Sample : 30%MA 40g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.08	12.26	50.00
1 - 2	3.03	11.65	50.00
1 - 3	3.07	12.16	50.00
1 - 4	3.01	11.72	50.00
1 - 5	2.97	12.11	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	79.41700	0.58261	0.50662	2.10316
1 - 2	74.84765	0.58500	0.50869	2.12036
1 - 3	75.08387	0.56476	0.49109	2.01129
1 - 4	75.74438	0.59557	0.51789	2.14712
1 - 5	73.98354	0.56797	0.49388	2.06047
Mean	75.81529	0.57918	0.50363	2.08848

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 30%MA 50g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.15	12.20	50.00
1 - 2	3.11	12.26	50.00
1 - 3	3.09	11.80	50.00
1 - 4	3.05	10.16	50.00
1 - 5	3.05	11.53	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	57.25763	0.42649	0.37086	1.48992
1 - 2	61.04923	0.44997	0.39128	1.60114
1 - 3	56.66122	0.44624	0.38804	1.55398
1 - 4	50.95047	0.47446	0.41258	1.64420
1 - 5	57.16458	0.47955	0.41700	1.62821
Mean	56.61662	0.45534	0.39595	1.58349

Date : 6/01/05   Test Type :   Tensile

Operator : Jarunee Suriyo   V1 :   5 mm/min

Sample : 60%MA 10g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.17	11.77	50.00
1 - 2	3.13	10.96	50.00
1 - 3	3.05	11.77	50.00
1 - 4	3.08	10.16	50.00
1 - 5	3.07	11.04	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	104.76901	0.86443	0.75168	2.80800
1 - 2	93.35674	0.83963	0.73011	2.72139
1 - 3	100.53375	0.87703	0.76263	2.80050
1 - 4	87.16641	0.86232	0.74984	2.78551
1 - 5	90.13790	0.82882	0.72071	2.65950
Mean	95.19276	0.85445	0.74300	2.75498

Date : 6/01/05   Test Type :   Tensile

Operator : Jarunee Suriyo   V1 :   5 mm/min

Sample : 60%MA 20g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.07	13.18	50.00
1 - 2	3.05	12.93	50.00
1 - 3	3.12	12.43	50.00
1 - 4	3.08	11.68	50.00
1 - 5	3.13	11.33	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	99.41418	0.82096	0.71388	2.45694
1 - 2	97.18337	0.81742	0.71080	2.46430
1 - 3	96.20164	0.83134	0.72290	2.48060
1 - 4	92.84237	0.85251	0.74131	2.58079
1 - 5	92.75570	0.91256	0.79353	2.61557
Mean	95.67945	0.84696	0.73649	2.51964

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 60%MA 30g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.12	10.86	50.00
1 - 2	3.08	11.18	50.00
1 - 3	3.05	11.02	50.00
1 - 4	3.01	10.16	50.00
1 - 5	3.05	11.18	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	79.40799	0.79797	0.69388	2.34358
1 - 2	80.92428	0.80575	0.70065	2.35010
1 - 3	76.84886	0.80168	0.69711	2.28642
1 - 4	70.14899	0.81574	0.70934	2.29383
1 - 5	78.09045	0.81422	0.70802	2.29387
Mean	77.08411	0.80707	0.70180	2.31356

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 60%MA 40g

Units	Thickness mm	Width mm	Gauge Length mm
1 - 1	3.05	11.20	50.00
1 - 2	3.08	11.11	50.00
1 - 3	3.02	10.75	50.00
1 - 4	3.01	10.16	50.00
1 - 5	3.09	10.13	50.00

Name Units	Max_Force kgf	Max_Disp mm	elongation %	tensile strenght kgf/mm2
1 - 1	67.79291	0.75911	0.66009	1.98457
1 - 2	64.42168	0.70932	0.61680	1.88264
1 - 3	59.62976	0.68960	0.59965	1.83674
1 - 4	58.88640	0.74045	0.64387	1.92555
1 - 5	60.97102	0.73071	0.63540	1.94785
Mean	62.34035	0.72584	0.63116	1.91547

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 60%MA 50g

Units	Thickness	Width	Gauge Length
	mm	mm	mm
1 - 1	3.05	11.27	50.00
1 - 2	3.03	10.78	50.00
1 - 3	2.99	10.85	50.00
1 - 4	3.01	10.16	50.00
1 - 5	3.30	10.14	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	48.08921	0.57764	0.50230	1.39902
1 - 2	43.93423	0.51578	0.44850	1.34506
1 - 3	43.21727	0.52142	0.45341	1.33216
1 - 4	44.19439	0.59065	0.51361	1.44513
1 - 5	45.48590	0.54673	0.47542	1.35933
Mean	44.98420	0.55044	0.47865	1.37614

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 10%VTS 30g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.09	12.01	50.00
1 - 2	3.12	11.89	50.00
1 - 3	3.08	12.05	50.00
1 - 4	3.10	11.35	50.00
1 - 5	3.16	11.10	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	90.23849	0.72296	0.62866	2.43159
1 - 2	92.74126	0.72778	0.63285	2.49998
1 - 3	93.20179	0.74610	0.64879	2.51123
1 - 4	88.04096	0.72436	0.62988	2.50223
1 - 5	86.67525	0.73097	0.63563	2.47107
Mean	90.17955	0.73043	0.63516	2.48322

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 30%VTS 30g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.19	11.53	50.00
1 - 2	3.07	11.27	50.00
1 - 3	3.10	11.85	50.00
1 - 4	3.20	11.43	50.00
1 - 5	3.09	11.02	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	94.83572	0.70674	0.61456	2.57841
1 - 2	81.33302	0.65574	0.57021	2.35074
1 - 3	85.96431	0.64435	0.56030	2.34012
1 - 4	90.56913	0.69564	0.60490	2.47619
1 - 5	85.13256	0.68665	0.59709	2.50009
Mean	87.56695	0.67782	0.58941	2.44911

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : 60%VTS 30g

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.09	11.94	50.00
1 - 2	3.02	11.98	50.00
1 - 3	3.08	11.84	50.00
1 - 4	3.10	11.56	50.00
1 - 5	3.11	11.93	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	90.37554	0.90825	0.78978	2.44956
1 - 2	88.84299	0.91327	0.79414	2.45561
1 - 3	87.53550	0.86467	0.75189	2.40039
1 - 4	89.58534	0.93596	0.81388	2.49987
1 - 5	89.64546	0.87719	0.76278	2.41617
Mean	89.19697	0.89987	0.78249	2.44432

Date :   6/01/05   Test Type :   Tensile

Operator :   Jarunee Suriyo   V1 :   5 mm/min

Sample : neat polyester

	Thickness	Width	Gauge Length
Units	mm	mm	mm
1 - 1	3.01	12.03	50.00
1 - 2	3.05	11.96	50.00
1 - 3	3.03	11.91	50.00
1 - 4	3.10	11.56	50.00
1 - 5	3.21	11.79	50.00

Name	Max_Force	Max_Disp	elongation	tensile strenght
Units	kgf	mm	%	kgf/mm2
1 - 1	210.59910	1.11354	0.96829	5.81600
1 - 2	208.07050	1.08486	0.94335	5.70400
1 - 3	202.01670	1.05988	0.92164	5.59800
1 - 4	200.71740	1.08981	0.94766	5.60100
1 - 5	211.78570	1.05859	0.92051	5.59600
Mean	206.63790	1.08134	0.94029	5.66300

Flexural strength (ASTM D790)

Date :   5/01/05   Test Type :   3 Point
Operator : Jarunee Suriyo   V1 :   1.5 mm/min

Sample: 10g Untreated

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	3.05	14.82	45.00
1 - 2	3.09	14.30	45.00
1 - 3	3.07	14.23	45.00
1 - 4	3.11	14.98	45.00
1 - 5	3.13	14.47	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	10.21091	1.39683	4.99943
1 - 2	9.83343	1.32556	4.86134
1 - 3	9.89002	1.37633	4.97759
1 - 4	10.61549	1.35596	4.94551
1 - 5	10.25716	1.29020	4.88398
Mean	10.16140	1.34898	4.93357

Date :   5/01/05   Test Type :   3 Point
Operator : Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 20g Untreated

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	3.08	14.89	45.00
1 - 2	3.09	15.02	45.00
1 - 3	3.12	15.56	45.00
1 - 4	3.15	15.86	45.00
1 - 5	3.16	14.82	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	9.90439	1.34005	4.73299
1 - 2	9.94331	1.34818	4.68002
1 - 3	10.25088	1.31075	4.56821
1 - 4	10.87586	1.30833	4.66491
1 - 5	9.71586	1.23870	4.43162
Mean	10.13806	1.30920	4.61555

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 30g Untreated

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.02	14.31	45.00
1 - 2	3.07	13.93	45.00
1 - 3	3.05	14.06	45.00
1 - 4	3.06	14.89	45.00
1 - 5	2.90	15.41	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	8.41456	1.31438	4.35193
1 - 2	8.37092	1.26117	4.30377
1 - 3	8.45703	1.27667	4.36452
1 - 4	8.94726	1.29420	4.33168
1 - 5	8.34881	1.34670	4.34840
Mean	8.50772	1.29862	4.34006

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 40g Untreated

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.12	15.36	45.00
1 - 2	3.26	14.96	45.00
1 - 3	3.09	14.28	45.00
1 - 4	3.03	15.89	45.00
1 - 5	3.25	14.61	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	7.49759	1.01927	3.38474
1 - 2	8.05824	0.99596	3.42119
1 - 3	6.95654	1.02297	3.44391
1 - 4	7.37177	1.07673	3.41088
1 - 5	7.77199	0.96499	3.39953
Mean	7.53123	1.01598	3.41205

Date : 5/01/05   Test Type : 3 Point

Operator : Jarunee Suriyo   V1 : 1.5 mm/min

Sample : 50 g Untreated

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	2.96	15.01	45.00
1 - 2	3.02	14.52	45.00
1 - 3	3.11	14.31	45.00
1 - 4	3.26	14.77	45.00
1 - 5	3.00	13.94	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	5.83448	1.00862	2.99462
1 - 2	5.64017	0.93325	2.87485
1 - 3	5.98805	0.93397	2.92031
1 - 4	6.87560	0.89960	2.95664
1 - 5	5.02533	0.85893	2.70373
Mean	5.87273	0.92687	2.89003

Date : 5/01/05 Test Type : 3 Point

Operator : Jarunee Suriyo V1 : 1.5 mm/min

Sample: 10% MA 10g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.10	14.13	45.00
1 - 2	3.25	13.56	45.00
1 - 3	3.14	14.26	45.00
1 - 4	3.16	14.95	45.00
1 - 5	3.35	13.55	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	10.10264	1.35546	5.02196
1 - 2	10.60624	1.31038	4.99849
1 - 3	10.20630	1.29317	4.89997
1 - 4	11.30149	1.33162	5.11004
1 - 5	11.27970	1.23148	5.00694
Mean	10.69928	1.30442	5.00748

Date : 5/01/05 Test Type : 3 Point

Operator : Jarunee Suriyo V1 : 1.5 mm/min

Ssample : 10% MA 20g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.06	15.00	45.00
1 - 2	3.13	14.94	45.00
1 - 3	3.07	14.21	45.00
1 - 4	3.20	13.98	45.00
1 - 5	3.19	14.97	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	9.98376	1.29367	4.79804
1 - 2	9.76042	1.20194	4.50125
1 - 3	9.26965	1.27128	4.67193
1 - 4	9.80571	1.22458	4.62355
1 - 5	10.39459	1.23918	4.60583
Mean	9.84283	1.24613	4.64012

Date :   5/01/05

Operator :   Jarunee Suriyo

Test Type :   3 Point

V1 :   1.5 mm/min

Sample : 10%MA 30g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	2.95	15.21	45.00
1 - 2	3.02	14.22	45.00
1 - 3	3.08	15.01	45.00
1 - 4	2.86	14.85	45.00
1 - 5	3.14	15.71	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	8.62873	1.26089	4.40025
1 - 2	8.63680	1.25548	4.49514
1 - 3	9.49580	1.22719	4.50146
1 - 4	8.01085	1.34953	4.45168
1 - 5	9.87404	1.18175	4.30292
Mean	8.92925	1.25497	4.43029

Date :   5/01/05

Operator :   Jarunee Suriyo

Test Type :   3 Point

V1 :   1.5 mm/min

Sample : 10%MA 40g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	3.11	14.99	45.00
1 - 2	3.02	15.73	45.00
1 - 3	3.08	15.26	45.00
1 - 4	3.04	14.95	45.00
1 - 5	3.10	15.57	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	8.51548	1.10602	3.96452
1 - 2	8.40696	1.16862	3.95549
1 - 3	8.21552	1.11389	3.83074
1 - 4	7.79155	1.10844	3.80662
1 - 5	8.70906	1.10030	3.92883
Mean	8.32772	1.11946	3.89724

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 10%MA 50g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.05	12.85	45.00
1 - 2	2.95	13.94	45.00
1 - 3	2.87	14.52	45.00
1 - 4	3.09	14.61	45.00
1 - 5	3.04	14.03	45.00

Name Parameter	Max_Force	Max_Disp	flexural strenght
Units	kgf	mm	kgf/mm2
1 - 1	5.32084	0.92213	3.00456
1 - 2	5.46751	0.95503	3.04219
1 - 3	5.41380	0.99827	3.05545
1 - 4	6.16894	0.91764	2.98502
1 - 5	6.45394	1.03175	3.35988
Mean	5.76500	0.96497	3.08942

Date :   5/01/05

Operator :   Jarunee Suriyo

Test Type :   3 Point

V1 :   1.5 mm/min

Sample : 30% MA 10g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.23	15.22	45.00
1 - 2	3.04	14.23	45.00
1 - 3	3.12	14.56	45.00
1 - 4	3.10	15.08	45.00
1 - 5	3.11	14.96	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	11.78434	1.16424	5.00944
1 - 2	10.13418	1.27538	5.20164
1 - 3	10.39838	1.18602	4.95221
1 - 4	10.78267	1.20316	5.02233
1 - 5	11.46159	1.33287	5.34683
Mean	10.91223	1.23233	5.10649

Date :   5/01/05

Operator :   Jarunee Suriyo

Test Type :   3 Point

V1 :   1.5 mm/min

Sample : 30% MA 20g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.05	14.03	45.00
1 - 2	3.09	15.11	45.00
1 - 3	2.84	14.32	45.00
1 - 4	3.07	14.15	45.00
1 - 5	3.00	14.99	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	9.05963	1.19917	4.68551
1 - 2	10.31538	1.19844	4.82622
1 - 3	8.21064	1.31325	4.79845
1 - 4	9.38535	1.19040	4.75030
1 - 5	9.53757	1.19330	4.77197
Mean	9.30171	1.21891	4.76649

Date : 5/01/05 Test Type : 3 Point

Operator : Jarunee Suriyo V1 : 1.5 mm/min

Sample : 30%MA 30g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	2.95	13.86	45.00
1 - 2	3.08	14.18	45.00
1 - 3	3.05	14.02	45.00
1 - 4	3.01	14.16	45.00
1 - 5	3.16	13.78	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	7.34881	1.46482	4.11257
1 - 2	8.46065	1.41310	4.24551
1 - 3	7.96853	1.37844	4.12415
1 - 4	7.97432	1.40099	4.19567
1 - 5	8.41571	1.36653	4.12830
Mean	8.03360	1.40477	4.16124

Date : 5/01/05 Test Type : 3 Point

Operator : Jarunee Suriyo V1 : 1.5 mm/min

Sample : 30%MA 40g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	3.08	14.62	45.00
1 - 2	3.03	14.33	45.00
1 - 3	3.07	13.45	45.00
1 - 4	3.01	13.06	45.00
1 - 5	3.26	13.84	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	8.06490	1.04930	3.92513
1 - 2	7.59440	1.03804	3.89642
1 - 3	7.42082	1.03585	3.95145
1 - 4	6.98010	1.09631	3.98189
1 - 5	8.16384	0.97608	3.74651
Mean	7.64481	1.03912	3.90028

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 30%MA 50g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.15	14.02	45.00
1 - 2	3.11	14.60	45.00
1 - 3	3.30	13.90	45.00
1 - 4	3.29	15.00	45.00
1 - 5	3.35	14.08	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	6.70139	0.85243	3.25162
1 - 2	6.70810	0.86233	3.20649
1 - 3	7.22035	0.78995	3.21973
1 - 4	7.70188	0.81579	3.20197
1 - 5	7.59840	0.83927	3.24589
Mean	7.18603	0.83195	3.22514

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 60%MA 10g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.17	15.02	45.00
1 - 2	3.13	14.33	45.00
1 - 3	3.05	14.08	45.00
1 - 4	3.08	14.31	45.00
1 - 5	3.02	15.06	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	10.92334	1.44998	4.88507
1 - 2	10.01159	1.42077	4.81362
1 - 3	9.52100	1.46259	4.90664
1 - 4	9.62470	1.43378	4.78575
1 - 5	9.76830	1.43388	4.80047
Mean	9.96979	1.44020	4.83831

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 60%MA 20g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.07	15.18	45.00
1 - 2	3.05	14.93	45.00
1 - 3	3.01	14.43	45.00
1 - 4	3.08	14.68	45.00
1 - 5	3.14	15.08	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	9.61526	1.41997	4.53645
1 - 2	9.25921	1.42577	4.50006
1 - 3	8.68229	1.45131	4.48269
1 - 4	9.40880	1.44063	4.56048
1 - 5	9.95816	1.45466	4.52087
Mean	9.38474	1.43847	4.52011

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample: 60%MA 30g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	2.95	13.86	45.00
1 - 2	3.08	14.18	45.00
1 - 3	3.05	14.02	45.00
1 - 4	3.01	14.16	45.00
1 - 5	3.16	13.78	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	7.34881	1.46482	4.11257
1 - 2	8.46065	1.41310	4.24551
1 - 3	7.96853	1.37844	4.12415
1 - 4	7.97432	1.40099	4.19567
1 - 5	8.41571	1.36653	4.12830
Mean	8.03360	1.40477	4.16124

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 60%MA 40g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	3.09	14.20	45.00
1 - 2	3.16	14.11	45.00
1 - 3	3.11	13.75	45.00
1 - 4	3.01	14.16	45.00
1 - 5	3.18	14.53	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	6.80958	1.20527	3.39015
1 - 2	7.29649	1.25175	3.49556
1 - 3	6.80997	1.24510	3.45641
1 - 4	6.59659	1.30482	3.47078
1 - 5	7.60298	1.22546	3.49275
Mean	7.02312	1.24648	3.46113

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 60%MA 50g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.41	15.45	45.00
1 - 2	3.25	16.01	45.00
1 - 3	3.31	15.03	45.00
1 - 4	3.29	14.98	45.00
1 - 5	3.44	14.13	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	7.86922	0.98623	2.95664
1 - 2	7.32713	1.06482	2.92469
1 - 3	6.78790	0.98324	2.78243
1 - 4	6.82681	1.01035	2.84196
1 - 5	6.87496	0.94712	2.77533
Mean	7.13720	0.99835	2.85621

Date : 5/01/05 Test Type : 3 Point

Operator : Jarunee Suriyo V1 : 1.5 mm/min

Sample : 10% VTS 30g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.15	14.89	45.00
1 - 2	3.16	14.36	45.00
1 - 3	3.19	15.56	45.00
1 - 4	3.27	15.85	45.00
1 - 5	3.23	15.39	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	9.78416	1.26616	4.47004
1 - 2	9.61501	1.27169	4.52611
1 - 3	10.34442	1.21834	4.40980
1 - 4	11.07306	1.21291	4.41008
1 - 5	10.77449	1.23099	4.52957
Mean	10.31823	1.24002	4.46912

Date : 5/01/05 Test Type : 3 Point

Operator : Jarunee Suriyo V1 : 1.5 mm/min

Sample : 30% VTS 30g

	Thickness	Width	Lower Support
Units	mm	mm	mm
1 - 1	3.20	15.21	45.00
1 - 2	3.22	14.65	45.00
1 - 3	3.15	15.01	45.00
1 - 4	3.16	14.99	45.00
1 - 5	3.17	15.74	45.00

Name	Max_Force	Max_Disp	flexural strenght
Parameter			
Units	kgf	mm	kgf/mm2
1 - 1	10.56629	1.17989	4.42041
1 - 2	8.81040	1.24543	4.38009
1 - 3	9.47430	1.18816	4.27591
1 - 4	9.79281	1.16679	4.31991
1 - 5	9.53694	1.28109	4.45858
Mean	9.63615	1.21227	4.37098

Date :   5/01/05   Test Type :   3 Point

Operator :   Jarunee Suriyo   V1 :   1.5 mm/min

Sample : 60% VTS 30g

Units	Thickness mm	Width mm	Lower Support mm
1 - 1	3.20	15.21	45.00
1 - 2	3.22	14.65	45.00
1 - 3	3.15	15.01	45.00
1 - 4	3.16	14.99	45.00
1 - 5	3.17	15.74	45.00

Name Parameter Units	Max_Force kgf	Max_Disp mm	flexural strenght kgf/mm2
1 - 1	10.15617	1.48668	4.40154
1 - 2	9.89721	1.44450	4.39812
1 - 3	9.28999	1.44612	4.21034
1 - 4	9.53413	1.43826	4.29941
1 - 5	10.57473	1.52838	4.51284
Mean	9.89045	1.46879	4.36445

Impact resistance (ASTM D256)

Specimen	Thickness (mm.)	Impact strength (J/cm.)
Untreated 10g		
1	3.13	7.793
2	3.08	9.450
3	3.01	8.878
4	3.03	8.343
5	3.05	7.998
mean		8.511
SD.		0.671
Untreated 20g		
1	2.98	10.172
2	3.01	7.723
3	3.08	8.297
4	3.06	8.351
5	3.05	7.622
mean		8.433
SD.		1.026
Untreated 30g		
1	3.05	7.622
2	3.08	8.297
3	3.12	6.022
4	2.95	7.116
5	2.98	6.305
mean		7.073
SD.		0.935
Untreated 40 g		
1	3.05	4.447
2	3.07	5.425
3	2.95	4.259
4	2.97	4.909
5	2.99	6.284
mean		5.065
SD.		0.817
Untreated 50 g		
1	3.07	3.823
2	3.03	4.147
3	3.00	5.551
4	3.04	4.462
5	3.01	4.174
mean		4.431
SD.		0.666

Specimen	Thickness (mm.)	Impact strength (J/cm.)
10% MA 10g		
1	2.80	7.897
2	3.00	8.907
3	2.96	8.634
4	3.03	9.212
5	2.97	8.213
mean		8.573
SD.		0.527
10% MA 20g		
1	3.15	7.380
2	2.99	9.335
3	3.05	8.761
4	3.08	8.297
5	3.12	8.565
mean		8.467
SD.		0.718
10% MA 30g		
1	3.12	8.946
2	3.01	8.490
3	3.07	8.704
4	3.09	8.648
6	3.15	8.483
mean		8.654
SD.		0.190
10% MA 40 g		
1	2.88	6.903
2	3.05	6.160
3	3.07	6.838
4	2.98	6.305
5	2.99	7.021
mean		6.646
SD.		0.386
10% MA 50 g		
1	3.04	6.539
2	3.03	5.846
3	2.97	5.607
4	2.95	4.942
5	2.98	4.892
mean		5.565
SD.		0.684

Specimen	Thickness (mm.)	Impact strength (J/cm.)
30%MA 10g		
1	2.95	7.880
2	3.02	8.462
3	2.98	8.186
4	3.02	9.242
5	2.96	8.634
mean		8.481
SD.		0.513
30%MA 20g		
1	2.85	8.156
2	3.10	9.778
3	3.00	8.518
4	3.03	9.212
5	2.97	8.604
mean		8.854
SD.		0.641
30%MA 30g		
1	3.05	9.938
2	3.13	9.684
3	3.15	7.380
4	3.17	8.805
5	3.16	9.593
mean		9.080
SD.		1.041
30%MA 40 g		
1	3.10	7.744
2	3.13	6.707
3	3.03	6.560
4	3.02	6.952
5	3.05	6.634
mean		6.919
SD.		0.484
30%MA 50 g		
1	2.85	5.743
2	2.88	7.264
3	3.04	6.181
4	3.08	6.816
5	3.06	6.711
mean		6.543
SD.		0.590

Specimen	Thickness (mm.)	Impact strength (J/cm.)
60%MA 10g		
1	3.13	8.543
2	3.09	7.897
3	3.05	8.761
4	3.01	9.270
5	3.07	8.704
mean		8.635
SD.		0.494
60%MA 20g		
1	2.87	9.725
2	2.80	8.302
3	3.08	9.062
4	3.03	8.434
5	3.01	8.878
mean		8.880
SD.		0.566
60%MA 30g		
1	2.98	10.172
2	3.17	9.181
3	2.80	8.302
4	3.05	9.543
5	3.10	9.778
mean		9.395
SD.		0.709
60%MA 40 g		
1	3.09	7.156
2	3.13	8.165
3	3.08	6.559
4	3.16	6.644
5	3.11	6.750
mean		7.057
SD.		0.660
60%MA 50 g		
1	3.14	4.643
2	3.07	6.108
3	3.20	5.204
4	3.08	4.733
5	3.12	5.338
mean		5.205
SD.		0.585

Specimen	Thickness (mm.)	Impact strength (J/cm.)
10% VTS 30g		
1	3.01	8.102
2	2.99	8.365
3	2.97	7.968
4	2.98	8.186
5	2.95	8.409
mean		8.206
SD.		0.183
30% VTS 30g		
1	3.12	7.669
2	3.12	8.002
3	3.14	7.520
4	3.09	6.938
5	3.04	7.542
mean		7.534
SD.		0.385
60% VTS 30g		
1	2.94	7.300
2	3.05	6.652
3	3.1	7.499
4	3.12	7.245
5	3.07	7.634
mean		7.266
SD.		0.377
nest polyester		
1	3.12	12.988
2	3.10	14.015
3	3.09	13.211
4	3.09	13.414
5	3.07	13.407
mean		13.407
SD.		0.382

APPENDIX B

Water absorption (ASTM D570)

Untreated rice straw : UP	Weight (m_1)	Weight (m_2)	% Water Absorption
10 : 90	1.2161	1.2244	0.682
20 : 80	1.0779	1.1159	3.552
30 : 70	1.0474	1.1113	6.104
40 : 60	0.8794	0.9454	7.505
50 : 50	1.1594	1.2744	9.919
10% MA treated : UP			
10 : 90	1.828	1.8362	0.45
20 : 80	1.2409	1.2859	3.63
30 : 70	1.0537	1.1037	4.742
40 : 60	1.3875	1.4683	5.827
50 : 50	1.1508	1.2267	6.599
30% MA treated : UP			
10 : 90	1.7612	1.7674	0.35
20 : 80	1.441	1.4796	2.678
30 : 70	1.0946	1.142	4.331
40 : 60	1.0897	1.1398	4.596
50 : 50	1.1467	1.2099	5.51
60% MA treated : UP			
10 : 90	1.6878	1.6932	0.32
20 : 80	1.1789	1.2005	1.836

30 : 70	1.1195	1.1473	2.483
40 : 60	1.1328	1.1602	2.417
50 : 50	1.3039	1.3518	3.673
10 % VTS treated : UP			
30 : 70	1.2491	1.3097	4.854
30 % VTS treated : UP			
30 : 70	1.3025	1.3614	4.523
60 % VTS treated : UP			
30 : 70	1.1553	1.1924	3.211
UP	1.2085	1.2106	0.182

APPENDIX C

XRF spectroscopy analysis

Chulalongkorn University
Quantified peaks in c:\superq\data\480757_a.pks

Summary of results

Sample:	480757 A	No treat
Measured:	3/6/05 18:53	
Quantified:	3/7/05 09:37	
Sum:	100. %	
RMS:	0.000	
Used lines:	13	
Traces:	Ba	
Qual. program:	SemlQ2004	

Concentrations

Analyte	Type	Calibration status	Compound	Concentration (%)	Calculation method
Mg	Sample	Calibrated	MgO	0.591	Calculate
Al	Sample	Calibrated	Al2O3	0.301	Calculate
Si	Sample	Calibrated	SiO2	82.822	Calculate
P	Sample	Calibrated	P2O5	0.78	Calculate
S	Sample	Calibrated	SO3	1.091	Calculate
K	Sample	Calibrated	K2O	2.65	Calculate
Ca	Sample	Calibrated	CaO	9.592	Calculate
Ti	Sample	Calibrated	TiO2	0.043	Calculate
Mn	Sample	Calibrated	MnO2	0.864	Calculate
Fe	Sample	Calibrated	Fe2O3	1.19	Calculate
Cu	Sample	Calibrated	CuO	0.02	Calculate
Zn	Sample	Calibrated	ZnO	0.023	Calculate
Sr	Sample	Calibrated	SrO	0.032	Calculate

Sample preparation

Sample type:	solid
Area ratio:	1.61
Additive/sample:	0.
Normalise:	to 100.0%
X-ray path:	Helium

Quantify parameters

Recipe:	c:\superq\data\new.rep
Spectrometer resp. file:	c:\superq\data\semiq.sti
Use compound list:	c:\superq\example\semiq.cpl
Apply drift corr.:	No
Apply medium corr.:	No
Apply film corr.:	Yes
Disall. elem. set:	None
Disall. lines set:	None
Error weighting:	Root
Max. Flow detector intensity:	2000 kcps
Max. Sealed detector intensity:	1000 kcps
Max. Scint detector intensity:	1000 kcps
Max. Duplex detector intensity:	3000 kcps

Disallowed elements

No elements disallowed

Chulalongkorn University

Quantified peaks in c:\superq\data\480757_b.pks

Summary of results

Sample:	480757 B	10%
Measured:	3/6/05 19:13	
Quantified:	3/7/05 09:42	
Sum:	100. %	
RMS:	0.000	
Used lines:	14	
Traces:	Ni Zr	
Qual. program:	SemIQ2004	

Concentrations

Analyte	Type	Calibration status	Compound	Concentration (%)	Calculation method
Mg	Sample	Calibrated	MgO	0.22	Calculate
Al	Sample	Calibrated	Al2O3	0.318	Calculate
Si	Sample	Calibrated	SiO2	91.638	Calculate
P	Sample	Calibrated	P2O5	0.232	Calculate
S	Sample	Calibrated	SO3	0.479	Calculate
K	Sample	Calibrated	K2O	0.484	Calculate
Ca	Sample	Calibrated	CaO	5.643	Calculate
Ti	Sample	Calibrated	TiO2	0.036	Calculate
Mn	Sample	Calibrated	MnO2	0.146	Calculate
Fe	Sample	Calibrated	Fe2O3	0.706	Calculate
Cu	Sample	Calibrated	CuO	0.008	Calculate
Zn	Sample	Calibrated	ZnO	0.009	Calculate
Sr	Sample	Calibrated	SrO	0.012	Calculate
Ba	Sample	Calibrated	BaO	0.069	Calculate

Sample preparation

Sample type:	solid
Area ratio:	1.56
Additive/sample:	0.
Normalise:	to 100.0%
X-ray path:	Helium

Quantify parameters

Recipe:	c:\superq\data\new.rcp
Spectrometer resp. file:	c:\superq\data\semiq.sti
Use compound list:	c:\superq\example\semiq.cpl
Apply drift corr.:	No
Apply medium corr.:	No
Apply film corr.:	Yes
Disall. elem. set:	None
Disall. lines set:	None
Error weighting:	Root
Max. Flow detector intensity:	2000 kcps
Max. Sealed detector intensity:	1000 kcps
Max. Scint detector intensity:	1000 kcps
Max. Duplex detector intensity:	3000 kcps

Disallowed elements

Chulalongkorn University

Quantified peaks in c:\superq\data\480757_c.pks

Summary of results

Sample:	480757 C	30%
Measured:	3/6/05 19:36	
Quantified:	3/7/05 09:46	
Sum:	100. %	
RMS:	0.001	
Used lines:	13	
Traces:	Zr	
Qual. program:	SemIQ2004	

Concentrations

Analyte	Type	Calibration status	Compound	Concentration (%)	Calculation method
Mg	Sample	Calibrated	MgO	0.105	Calculate
Al	Sample	Calibrated	Al2O3	0.125	Calculate
Si	Sample	Calibrated	SiO2	95.118	Calculate
P	Sample	Calibrated	P2O5	0.15	Calculate
S	Sample	Calibrated	SO3	0.422	Calculate
K	Sample	Calibrated	K2O	0.153	Calculate
Ca	Sample	Calibrated	CaO	3.359	Calculate
Ti	Sample	Calibrated	TiO2	0.029	Calculate
Mn	Sample	Calibrated	MnO2	0.072	Calculate
Fe	Sample	Calibrated	Fe2O3	0.446	Calculate
Cu	Sample	Calibrated	CuO	0.008	Calculate
Zn	Sample	Calibrated	ZnO	0.007	Calculate
Sr	Sample	Calibrated	SrO	0.007	Calculate

Sample preparation

Sample type:	solid
Area ratio:	1.59
Additive/sample:	0.
Normalise:	to 100.0%
X-ray path:	Helium

Quantify parameters

Recipe:	c:\superq\data\new.rcp
Spectrometer resp. file:	c:\superq\data\semiq.sti
Use compound list:	c:\superq\example\semiq.cpl
Apply drift corr.:	No
Apply medium corr.:	No
Apply film corr.:	Yes
Disall. elem. set:	None
Disall. lines set:	None
Error weighting:	Root
Max. Flow detector intensity:	2000 kcps
Max. Sealed detector intensity:	1000 kcps
Max. Scint detector intensity:	1000 kcps
Max. Duplex detector intensity:	3000 kcps

Disallowed elements

No elements disallowed

Chulalongkorn University

Quantified peaks in c:\superq\data\480757_d.pks

Summary of results

Sample:	480757 D	60%
Measured:	3/6/05 19:56	
Quantified:	3/7/05 09:49	
Sum:	100. %	
RMS:	0.000	
Used lines:	11	
Traces:	Ni Cu Zr	
Qual. program:	SemIQ2004	

Concentrations

Analyte	Type	Calibration status	Compound	Concentration (%)	Calculation method
Mg	Sample	Calibrated	MgO	0.049	Calculate
Al	Sample	Calibrated	Al2O3	0.112	Calculate
Si	Sample	Calibrated	SiO2	96.904	Calculate
P	Sample	Calibrated	P2O5	0.088	Calculate
S	Sample	Calibrated	SO3	0.272	Calculate
K	Sample	Calibrated	K2O	0.144	Calculate
Ca	Sample	Calibrated	CaO	2.1	Calculate
Mn	Sample	Calibrated	MnO2	0.072	Calculate
Fe	Sample	Calibrated	Fe2O3	0.244	Calculate
Zn	Sample	Calibrated	ZnO	0.007	Calculate
Sr	Sample	Calibrated	SrO	0.007	Calculate

Sample preparation

Sample type:	solid
Area ratio:	1.63
Additive/sample:	0.
Normalise:	to 100.0%
X-ray path:	Helium

Quantify parameters

Recipe:	c:\superq\data\new.rcp
Spectrometer resp. file:	c:\superq\data\semiq.sti
Use compound list:	c:\superq\example\semiq.cpl
Apply drift corr.:	No
Apply medium corr.:	No
Apply film corr.:	Yes
Disall. elem. set:	None
Disall. lines set:	None
Error weighting:	Root
Max. Flow detector intensity:	2000 kcps
Max. Sealed detector intensity:	1000 kcps
Max. Scint detector intensity:	1000 kcps
Max. Duplex detector intensity:	3000 kcps

Disallowed elements

No elements disallowed

VITA

Miss Saliya Suriyo was born on September 6, 1980 in Phitsanulok province. She received Bachelor Degree in Chemistry from Department of Chemistry, Faculty of Science, Naresuan University in 2000. She began her Master Degree study in the Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University, 2002 and completed the program in 2004.