THE EFFECTS OF FIBER ARCHITECTURE, FIBER CONTENT AND FIBER TREATMENT ON PHYSICAL

PROPERTIES OF SISAL FIBER/EPOXY

COMPOSITES

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ผลการจัดเรียงเส้นใย ปริมาณเส้นใย และการดัดแปรเส้นใยต่อ สมบัติทางกายภาพของพอลิเมอร์คอมโพสิทระหว่าง เส้นใยป่านศรนารายณ์กับอีพอกซี

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ลายมือชื่อนักศึกษา <u> </u>	
ลายมือชื่ออาจารย์ที่ปรึกษา_	

SAWITRI SRISUWAN : THE EFFECTS OF FIBER ARCHITECTURE, FIBER CONTENT AND FIBER TREATMENT ON PHYSICAL PROPERTIES OF SISAL FIBER/EPOXY COMPOSITES. THESIS ADVISOR : ASST. PROF. PRANEE CHUMSAMRONG, Ph.D., 95 PP.

SISALFIBER/ FIBER ARCHITECTURE/ FIBER CONTENT/ EPOXY COMPOSITES/ ALKALIZATION/SILANE TREATMENT

In this thesis, the effects of fiber architecture, fiber content and fiber treatment on the physical properties of woven sisal fiber/epoxy composites were studied. Woven sisal fiber with three different weave types including plain, twill, and satin weave were used as reinforcement for epoxy resin. Woven sisal fiber contents used in composite preparation were 5, 10, 15 %wt. Flexural strength and flexural modulus of untreated woven sisal fiber composite were higher than pure epoxy resin and increased with increasing fiber content. 15 %wt. satin woven sisal fiber/epoxy composite showed the highest flexural strength and flexural modulus. The maximum impact strength value of 15.45 kJ/m² was found for the composite with 15 %wt. satin woven sisal fiber. Owing to its high mechanical properties, epoxy composite with 15 %wt. satin woven sisal fiber was selected to study the effect of fiber surface treatment.

Alkali, silane and alkali-silane treatment process were applied to satin woven sisal fiber. The maximum load and average diameters of fiber cut from treated satin woven sisal fiber were decreased. The maximum load of treated satin woven sisal fiber was in the order of silane treated woven sisal fiber (STWSF)> alkali-silane treated woven sisal fiber (ASTWSF)> alkali treated woven sisal fiber (ATWSF). The result from FTIR and TGA illustrated that hemicelluloses was removed from the fiber surface. Moreover, FTIR results also confirmed the attachment of silane on the fiber surface that subjected to silane treatment.

ATWSF composites possessed the highest flexural modulus. The flexural strength of treated satin woven sisal fiber/epoxy composites was lower than untreated satin woven sisal fiber composites. The impact strength of ASTWSF composites was the lowest whereas the impact strength of STWSF was not significantly different from UTWSF composite.



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SYMBOLS AND ABBREVIATIONS

A-174	=	γ -methacryloxypropyl trimethoxy silane
A-187	=	γ -glycidoxypropyltrimethoxy silane
GPa	=	Gigapascal
hrs	=	Hours
ILSS	=	Interlaminar shear strength
kN	=	Kilo newton
kV	=	Kilo volt
MPa	=	Megapascal
m ²	=	Square meter
min	=	Minute
mm	=	Millimeter
mol	=	Mole
phr	=	Parts per hundred resin
rpm	=	Revolution per minute
V _f	=	Fiber volume fraction
W	=	Fiber weight fraction
w _m	=	matrix weight fraction
%wt.	=	Percentage weight
wt/wt	=	Weight by weight
$ ho_{\rm f}$	=	Fiber density
$ ho_m$	=	Matrix density

SYMBOLS AND ABBREVIATIONS (Continued)

$ ho_c$	=	Composite density		
2D	=	Two-dimensional		
3D	=	Three-dimensional		
AEP	=	Aminoethylpiperazine		
AHEW	=	Amine hydrogen equivalent weight		
ASTSF	=	Alkali-silane treated sisal fiber cut from alkali-silane treated		
		woven sisal fiber		
ASTWSF	=	Alkali-silane treated woven sisal fiber		
ATR-FTIR	=	Attenuated total reflectance-Fourier transform infrared		
ATSF	=	Alkali treated sisal fiber cut from alkali treated woven sisal		
		fiber		
ATWSF	=	Alkali treated woven sisal fiber		
DDS	=	Diaminodiphenylsulfone		
DETA	=	Diethylenetriamine		
DETDA	=	Diethyltoluenediamine		
DGEBA	=	Diglycidyl ether of bisphenol A		
DSC	=	Differential Scanning Calorimetric		
DTG	=	Derivative thermogravimetry		
EEW	=	Epoxide equivalent weight		
SEM	=	Scanning electron microscope		
STSF	=	Silane treated sisal fiber cut from silane treated woven sisal fiber		
STWSF	=	Silane treated woven sisal fiber		

SYMBOLS AND ABBREVIATIONS (Continued)

TGMDA	=	Tetraglycidylmethylene dianiline
TETA	=	Triethylene tertramine
UTSF	=	Untreated sisal fiber cut from untreated woven sisal fiber
UTPWSF composite	=	Untreated plain woven sisal fiber composite
UTTWSF composite	=	Untreated twill woven sisal fiber composite
UTSWSF composite	=	Untreated satin woven sisal fiber composite
UTWSF composite	=	Untreated woven sisal fiber
XPS	=	X-ray photoelectron spectroscopy
4, 4´ DDS	=	4, 4'-Diamino-diphenylsulfone



CHAPTER I

INTRODUCTION

1.1 General introduction

Polymer composite materials are a combination of two or more chemical different materials. The two major parts in polymer composites are matrix and reinforcements. The reinforcement for polymer composites can be fibers or particulates that are added to the matrix to improve the matrix properties.

For polymer matrix, the matrix material can be either thermoplastics or thermosets. Most of thermoset resins used as composite matrix are unsaturated polyester, vinyl ester and epoxy resin. These resins have low viscosity prior to cure facilitating reinforcement wetout and fabrication of the structure. On the other hand, the high modulus and solvent insensitivity of the crosslink network after curing process results in a strong and durable part. Therefore, thermoset resin is widely used in industrial, aerospace and structural applications. Epoxy is one of thermoset resins that popularly used because it has a number of interesting properties including high modulus, high strength, high stiffness, thermal stability and chemical resistance. Moreover, epoxy has higher fracture toughness than polyester and vinyl ester resin. In addition, the cure shrinkage of epoxy is lower than that of vinyl ester resulted in lower cracking during the cure of thick and high curvature parts.

Fibers are generally used as reinforcements in polymer composites because they can improve impact resistance and other mechanical properties. Mostly, synthetic fiber such as glass, carbon and aramid fibers have been used in polymer composites (Gilchrist, Kinloch, Matthews, and Osiyemi, 1996; Zhao and Takeda, 2000; Alvarez, Valdez, and Vázquez, 2003; Xu and Hoa, 2008; Pihtili, 2009) because they possess high strength and high stiffness and lightweight. However, their applications are limited because of high cost of production. On the other side, the synthetic fiber reinforcements cause many environmental problems. Fore examples, they come from non-renewable resources and they emit high pollutant and greenhouse gas at the end of life time.

The growing of environmental awareness, environmental agreement and regulations for decrease in non-renewable resources throughout the world has triggered a paradigm shift towards designing materials compatible with the environment. These reason force industries to produce goods based on economic and ecological design (eco-design) for response to market demand. Then, the newer materials and composites that have both economic and environmental benefits are considered for many applications such as automotive, building, furniture, and sporting goods. Many researchers in the field of polymer science and engineering have focused on developing natural fibers for polymer composites.

Nowadays many polymer composite industrials are increasing in the use of natural fibers for composites. The advantages of natural fibers over traditional reinforcing materials such as glass fibers, talc and mica are acceptable specific strength properties, low cost, low density, high toughness, good thermal properties, reduced tool wear, enhanced energy recovery, and biodegradability (Karnani, Krishnan, and Narayan, 1997).

The several types of natural fiber such as hemp, flex, jute, bamboo, banana, oil palm, and sisal fiber have been studied in order to use as reinforcement in polymer

composites. Some of mechanical properties of natural fibers are shown in Table 1.1.

Table 1.1 The specific mechanical performance of the principal natural fibers used as reinforcement in composites materials (Wambua, Ivens, and Verpoest, 2003).

Fiber	Density (g/cm ³)	Strain at break (%)	Specific stress at break (MPa)	Specific Young's modulus (GPa)
Alfa	1.4	1.5-2.4	134-220	13-17.8
Agave	1.4	20	350	4.2
Cotton	1.5	7-8	191-398	3.6-8.4
Jute	1.3	1.5-1.8	300-600	20
Flax	1.5	2.7-3.2	230-690	18
Hemp	1.5	1.6	460	17
Ramie	1.5	3.6-3.8	266-630	42-86
Sisal	1.5	2-2.5	340-423	6-14

้าว_{อักยาลัยเทคโนโลยีส์}

Natural fiber can be used in the form of continuous or discontinuous fiber. Discontinuous fibers which also referred to as chopped, staple or short fibers are used mostly in randomly oriented form as in sheet moulding compound composites and injection moulded plastic composites. Continuous fibers are in the form of stands, tows, roving, yarn, mats, textile etc. They are used in filamentwound, pultruded or laminated structure in which the fiber orientation can be precisely controlled. Therefore, mechanical properties of continuous fibers reinforced composites are higher than those of discontinuous fibers reinforced polymer composites (Shibata, Cao, and Fukumoto, 2005). Apart from fiber orientation, fiber architecture which refers to the arrangement of fibers also has influence on the properties of polymer composites (Pearce, Guild, and Summerscales, 1998; Pearce, Summerscales, and Guild, 2000; Asi, 2009). Nowadays, woven fiber reinforced composites are used in various high performance applications. The woven fiber architecture has been popularly used as reinforcement due to simple processing and acceptable mechanical properties (Jekabsons and Bystrom, 2002). In addition, woven fabric composites provide more balanced properties in the fabric plane than unidirectional laminas (Jang, 1994). This special feature can lead to a significant improvement in mechanical properties of the composites. Moreover, woven fiber architecture composites show more damage tolerant in the case of a delamination (Naik, 1994). Then the formation of different woven fiber architecture is an important stage in polymer composites technology.

Although natural fibers have many interesting properties, the adhesion between the fiber and matrix is generally insufficient because of hydrophilic properties of natural fibers. The interfacial adhesion between reinforcements and matrix can be enhanced by chemical or physical treatments. Chemical treatment methods include alkalization, acetylation, cyanoethylation and silane treatment (Sreekumar et al.,2009; Bessadok, Roudesli, Marais, Follain, and Lebrun, 2009).

In this work, woven sisal fibers prepared by hand weaving process were used as reinforcement for epoxy composites. Alkalization and silane treatment were applied for improving interfacial bounding between woven sisal fiber and epoxy matrix.

1.2 Research objectives

The objectives of this study are:

- to study the effects of fiber content and fiber architecture on the physical properties of woven sisal fiber/epoxy composites.
- (ii) to study the effects of alkalization, silanization and alkali-silane treatment on physical properties of woven sisal fiber/epoxy composites.

1.3 Scope and limitation of the study

In this study, woven sisal fibers with plain weave, twill weave and satin weave were prepared by hand weaving process. Woven sisal fiber/epoxy composites were prepared by hand lay-up process. The fiber contents in the composites were 5, 10 and 15 % wt. The stoichiometric ratio of epoxy and hardener was 100: 40 by weight for all composites. The flexural and impact properties of pure epoxy and untreated woven sisal fiber/epoxy composites with three weave types were determined by using a universal testing machine and a basic pendulum impact tester respectively. The composites with optimum flexural and impact properties were selected to study the effect of fiber treatments on physical properties of woven sisal fiber/epoxy composites.

The selected woven type was subjected to alkalization, silanization and alkalisilane treatment. The functional groups of untreated and treated woven sisal fibers were studied by Fourier transform infrared spectrometer (FTIR). The thermal properties of untreated and treated woven sisal fiber were investigated by a thermogravimetric analyzer (TGA). The universal testing machine was used to study the tensile properties of untreated and treated woven sisal fiber. The flexural and impact properties of treated woven sisal fiber/epoxy composites were determined by using a universal testing machine and a basic pendulum impact tester respectively. In addition, the morphologies of sisal fiber and fractured surfaces of neat epoxy and the composites were analyzed using scanning electron microscopy (SEM).



CHAPTER II

LITERATURE REVIEW

As mentioned in chapter I, The mechanical properties of natural fiber/epoxy composite depend on each part of composite. The characteristics of epoxy resin and curing agent in composites are important as well as natural fiber. Epoxy resin and curing agent selection controls process ability, upper use temperature, flammability characteristics, and corrosion resistance of the composites, whereas the loads are carried by the fiber. The development of fiber design such as fiber length, fiber orentation, fiber architecture have resulted in the improvment of mechanical properties of the composites, interfacial adhesion between the matrix and fiber also plays important role. However, it has been known that interfacial adhesion between a hydrophilic natural fiber surface treatment has been studied in many research works (Mwaikambo and Ansell, 2001; Gomes, Matsuo, Goda, and Ohgi, 2007; Threepopnatkul, Kaerkitcha, and Athipongarporn, 2009). In this chapter, the resin system, properties of natural fiber and fiber surface treatment method are reviewed.

2.1 Epoxy resin

Epoxy resins are chemical mixtures containing two main parts, the epoxy resin and the curing agent or hardener. Some commercial epoxy resins and examples of important hardener are discussed below.

2.1.1 Commercial types of epoxy resins

2.1.1.1 Tetraglycidylmethylene dianiline (TGMDA)

TGMDA is the high performance epoxy resin. It is a tetrafunctional epoxy with results in high crosslink after curing process. The high crosslink density gives high value of Young's modulus and high service temperature. Figure 2.1 shows the chemical structure of TGMDA epoxy resins.



Figure 2.1 Chemical structure of TGMDA epoxy resin (Juska and Pucket, 1997).

2.1.1.2 Diglycidyl ether of bisphenol A (DGEBA)

The bisphenol A base epoxy is the most widely used resin. It is used for wet filament winding resins, prepreg, laminating resins, resin transfer moulding (RTM) resins, coating and adhesives. Bisphenol A epoxy is made by reacting biaphenol A with epichlorohydrin. The chemical structure of bisphenol A is shown in Figure 2.2.



Figure 2.2 Chemical structure of bisphenol A epoxy resins (Juska and Pucket, 1997).

2.1.1.3 Novolacs

Novolacs are epoxidized phenol-formaldehyde or substituted phenol-formaldehyde resins epoxies. They have a higher functionality and consequently cure to a higher crosslink density than TGMDA. Addition of novolacs to a formulation increase the resin T_g but decrease the failure strain. Novolacs are used extensively in prepreg formulations. The chemical structure of novolacs is shown in Figure 2.3.

CHCH₂-O OCH₂CH-CH₂ $-CH_2CH-CH_2$ CH-

Figure 2.3 Chemical structure of novolacs (Goodman, 1998).

2.1.1.4 Trifunctional epoxy resins

It has properties intermediate between bisphenol A and TGMDA. Trifunctional epoxies are used predominantly as a modifier in prepreg formulations. For example, it is employed as failure strain enhancement for TGMDA and as T_g improvement for a bisphenol A epoxy resin.

2.1.1.5 Bisphenol F base epoxy resins

Bisphenol F resin have methylene linkage between the aromatic ring instead of the isopropylene linkage in bisphenol A. The advantage of bisphenol F epoxy is lower viscosity than bisphenol A. The chemical structure of bisphenol F is shown in Figure 2.4.



Figure 2.4 Chemical structure of bisphenol F epoxy resins (Juska and Pucket, 1997).

2.1.2 Curing agents or hardener for epoxy resin

Curing agents for epoxy resin are available with a wide range of cure characteristics. The basic types of curing agent are discussed below.

2.1.2.1 Amine curing agents

2.1.2.1.1 Aliphatic amines

Aliphatic amines are highly reactive and fast cure epoxy resin at room temperature. Epoxies cured with aliphatic amines have relatively low service temperature depending on the other components of the formulation. The five types of aliphatic amines are low molecular weight amine adducts, amino amines, polyamides and restricted diamines. The most reactive are low molecular weight molecules such as diethylenetriamine (DETA) and triethylene tertramine (TETA). They have pot life about 30 minutes and they are completely cured in a few days.

2.1.2.1.2 Cycloaliphatic amines

Cycloaliphatic amines can gel an epoxy at room temperature. They give elevated-temperature performance and chemical resistance of cured resins. Cycloaliphatic amines are used in adhesive and coating applications.

2.1.2.1.3 Aromatic amines

Epoxy cured with aromatic amines have the best overall properties. There are two commonly used aromatic amines. One is diaminodiphenylsulfone (DDS), where is both the 3, 3 and 4, 4 isomers are used and diethyltoluenediamine (DETDA) are used to cure epoxy resins at high temperatures and long cure times resulting from the rigid benzene ring in their structure.

Some examples of commercial curing agents are presented in Table 2.1. The aromatic amines are widely used in composite fabrication in both wet and dry lay-up application for filament winding, electrical, piping, and tooling.



Name	AHEW	Pot life
Aliphatic amines $H_2N \xrightarrow{H_1} N \xrightarrow{N} NH_2$ H Triethylenetetramine (TETA)	24.3	30 min
$ \begin{array}{c} \begin{array}{c} \begin{array}{c} H\\ R\\ N\\ H\\ \end{array}\\ \begin{array}{c} \end{array}\\ N\\ H\\ \end{array}\\ \begin{array}{c} H\\ \end{array}$	95	70 min
$H_{2}N \xrightarrow{N} N \xrightarrow{N} R \xrightarrow{H} H_{1} \xrightarrow{N} N \xrightarrow{H} N \xrightarrow{N} 2$ Polyamide	95	2hrs
Cycloaliphatic H^{N} NH 2 Aminoethylpiperazine (AEP)	9 43	15 min
$H_{2N} \xrightarrow{H_{1}} N_{NH_{2}}$ bis (p-aminocyclohexyl)	52	1.6 hrs
Aromatic amine H_2N NH_2 $4,4$ '-Diamino-diphenylsulfone (4,4' DDS)	_	_

 Table 2.1 Chemical structure and characteristics of aliphatic amines (Mallick, 1997).

2.1.3 Characteristics of epoxy resins

2.1.3.1 Viscosity

The viscosity of epoxy resins depends on the temperature, molecular weight, molecular weight distribution and chemical component of the resin and presence of any modifiers or diluents. An increase in temperature reduces the viscosity.

2.1.3.2 Epoxide equivalent weight (EEW)

The epoxide equivalent weight is a measure of amount of epoxy groups. This is the weight of resin containing 1 gram chemical equivalent epoxy. For a pure diglycidyl ether with two epoxy groups per molecule, the epoxide equivalent will be half the molecular weight (EEW = 170). For epoxy resin with one component, EEW is calculated by

$$EEW = \frac{Molecular weight of epoxy resin}{No. of active epoxide group}$$
(2.1)

For a mixture of epoxies, the EEW of the mixture is

$$EEW = \underline{Total weight}$$
(2.2)
$$\frac{Wt.A}{(EEW A)} + \frac{Wt.B}{(EEW B)} + \dots$$

Epoxide equivalent can be determined by reacting a known quantity of resin with hydrochloric acid and measuring the unconsumed acid by back titration.

Example of EEW calculation for DGEBA with two epoxy groups per molecule is described as follows:

Molecular weight of DGEBA:

21 carbons = $21 \times 12 = 252$ 24 hydrogens = $24 \times 1 = 24$ 4 oxygens = $4 \times 16 = 64$

Molecular weight (g/mol) = 340

DGEBA has two active epoxide groups per molecule.

Therefore,

EEW = <u>Molecular weight of epoxy resin</u> No. of active epoxide group

EEW = 170 g/equivalent

2.1.3.3 Amine hydrogen equivalent weight (AHEW)

The hydrogen equivalent is the weight of resin containing one equivalent weight of hydrogen groups that may be determined by reacting the resin with acetyl chloride.

The formulations are desirable to have an equal number of epoxy groups and amine hydrogens. The calculation for the amount of amine to add per hundred part of the epoxy resin (phr), described as follows:

$$phr = AHEW/EEW \times 100 \tag{2.3}$$

AHEW is the amine hydrogen equivalent weight of the amine

which is the molecular weight of the amine divided by number of active hydrogen.

For amine hydrogen equivalent weight, AHEW is calculated by

$$AHEW = \frac{Molecular weight of amine}{No. of active hydrogen}$$
(2.4)

Example of AHEW calculation for triethlenetetramine (TETA) is described as

follows:

Molecular weight of triethlenetetramine (TETA):

6 carbons = 12x6 = 72

18 hydrogens = 18x1 = 18

4 nitrigen = 4x14 = 56

Molecular weight (g/mol) = 146

There are six active hydrogen per molecule.

Therefore,

AHEW = <u>Molecular weight of amine</u> No. of active hydrogen

 $AHEW = \frac{146 \text{ g/mol}}{6 \text{ active hydrogen/mol}}$

AHEW = 24.3 g/equivalent

2.1.3.4 Properties of cured resin

Being cross-linked, the epoxy resin will not dissolve but will be swollen by liquids of similar solubility parameter to the cure resin. The chemical resistance of epoxy resin is not only dependent upon the resin used, but it is also dependent upon the type of curing agent. For example, Randall (2003) reported that
the utilization of different amine curing agent system can significantly change the sulfuric acid resistance.

The thermal properties of the epoxy resin depend on degree of crosslink and flexibility of the epoxy resin and curing agent molecules. For example, the rigid structures obtained from cycloaliphatic resins and pyromellitic dianhydride will raise the heat distortion temperatures.

2.2 Natural fibers

2.2.1 Sources

Natural fibers can be derived from plants (cotton, kapok, jute, hemp, ramie, and sisal), animals (wool and silk), and mineral sources (asbestos). Figure 2.5 shows the classification of natural fibers. However, plant fibers are widely used because they are abundantly available, cheap and have good mechanical properties. Plant fibers used as reinforcement come from different part of plant such as seed-fibers (cotton and kapok), bast fiber (hemp, flax, and jute) and leaf fibers (pineapple, curaua, and sisal).





2.2.2 Chemical composition of natural fiber

Climatic conditions, age and the digestion process influences not only

the structure of fibers but also the chemical composition. Component mean values of some plant fibers are shown in Table 2.2. With the exception of cotton, the components of natural fibers are cellulose, hemi-cellulose, lignin, pectin, waxes and water soluble substances, with cellulose, hemicellulose and lignin as the basic components with regard to the physical properties of the fibers.

 Table 2.2 Composition of different cellulose based natural fibers (Gassan and Bledzki, 1996).

Fiber Composition	Cotton (%wt.)	Jute (%wt.)	Flax (%wt.)	Ramie (%wt.)	Sisal (%wt.)
Cellulose	82.7	64.4	64.1	68.6	65.8
Hemicellulose	5.7	12	16.7	13.1	12
Pectin	5.7	0.2	1.8	1.9	0.8
Lignin		11.8	2	0.6	9.9
Wax	0.6	0.5	1.5	0.3	0.3
Moisture content	10	10	10	10	10

2.2.2.1 Cellulose

Cellulose is the essential component of all plant-fibers. It is generally accepted that cellulose is a linear condensation polymer consisting of Danhydroglucopyranose units (often abbreviated as anhydroglucose units or even as glucose units for convenience). The chemical structure of cellulose is shown in Figure 2.5.



Figure 2.6 Chemical structure of cellulose (Mallick, 1997).

2.2.3.2 Hemicellulose

Hemicellulose comprises a group of polysaccharides (excluding pectin) that remains associated with the cellulose after lignin has been removed. The hemicelluloses differ from cellulose in three important aspects. In the first place they contain several different sugar units whereas cellulose contains only 1,4- β -D-glucopyranose units. Secondly, they exhibit a considerable degree of chain branching, whereas cellulose is a strictly linear polymer. Thirdly, the degree of polymerization of native cellulose is ten to one hundred times higher than that of hemicelluloses.

2.2.3.3 Pectin ยาลัยเทคโนโลยีลิ

Pectin is a collective name for heteropolysaccharides, which consist essentially of polygalacturon acid. Pectin is soluble in water only after a partial neutralization with alkali or ammonium hydroxide (Gassan and Bledzki, 1999).

2.2.3.4 Waxes

Waxes make up the part of the fibers, which can be extracted with organic solutions. These waxy materials consist of different types of alcohols, which are insoluble in water as well as in several acids such as palmitic acid, oleaginous acid and stearic acid (Gassan and Bledzki, 1999).

2.2.3 Mechanical properties of natural fibers

The advantages of natural fibers over synthetic fibers such as glass fiber and carbon fiber are low density, acceptable specific strength, biodegradability and renewability. Some mechanical properties of natural fibers compared to conventional reinforcing fibers are shown in Table 2.3.

Table 2.3Mechanical properties of natural fibers as compared to conventional
reinforcing fibers (Bledzki and Gassan, 1999).

Property Fiber type	Density (g/cm ³)	Elongation (%)	Tensile strength (MPa)	Young's modulus (GPa)
Cotton	1.5-1.6	7.0-8.0	287-597	5.5-12.6
Jute	1.3	1.5-1.8	393-773	26.5
Flax	1.5	2.7-3.2	345-1035	27.6
Hemp	-	1.6	690	-
Ramie	6475	3.6-3.8	400-938	61.4-128
Sisal	1.5	2.0-2.5	511-635	9.4-22.0
Coir	1.2	30	175	4.0-6.0
Viscose (cord)	-	11.4	593	11
Soft wood kraft	1.5	-	1000	40
E-glass	2.5	2.5	2000-3500	70
S-glass	2.5	2.8	4570	86
Aramide (normal)	1.4	3.3-3.7	3000-3150	63.0-67.0
Carbon (standard)	1.4	1.4-1.8	4000	230.0-240.0

2.3 Natural fiber surface treatment by chemical methods

The quality of the fiber-matrix interface is significant for the application of natural fibers as reinforcement fibers for polymer composites. Both physical and chemical treatment methods can be used to optimize natural fiber-matrix interface. However, in this work, only chemical method was used. Therefore, chemical treatment method was discussed in the following section.

2.3.1 Alkalization

The interested in alkali treated natural fibers has increased dramatically in the past decade. Alkali treatment is a common method used to enhance interfacial adhesion of natural fibers and polymer matrices.

Rong, Zhang, Lui, Yang, and Zeng (2001) studied the effect of alkali treatment on the adhesion characteristics of sisal/epoxy composites. The sisal fibers were treated with 2% NaOH solution. The alkali treated sisal fiber showed the improvement of adhesion characteristics due to increase of surface roughness. Consequently, the composites showed improvements in the tensile and flexural strength. The removal of hemicelluloses and lignin and other surface waxy substances by alkali solution increased the chance for mechanical interlocking and chemical bonding between matrix and fiber.

Ray, Sarkar, Basak, and Rana (2002) treated jute fibers with 5% NaOH for 2, 4, 6, and 8 hrs. Derivative thermogravimetry (DTG) and Differential scanning calorimetric (DSC) results showed that the moisture desorption was observed at a lower temperature in all treated fibers. The increasing of fiber fineness offered more surface area for moisture evaporation. For all of treated jute fibers, the hemicelluloses content was lowered on alkali treatment because of decreasing in percentage degradation of the hemicelluloses. The decomposition temperature of α -cellulose was decreased to 348°C from 362.2°C for all treated fibers and the residual char formation increased to a significant extent. The enthalpy for the thermal degradation of α -cellulose showed a decreasing trend for 2 and 4 hrs fibers treated, which could be caused by the initial loosening of the structure.

Cao, Shibata, and Fukumoto (2006) improved the mechanical properties of biodegradable composites reinforced with bagasse fiber by alkali treatments. The results showed that mechanical properties of the composites made from alkali treated fibers were higher than those of made from the untreated fibers. Composites of 1 %wt. of NaOH solution treated fibers showed approximately maximum improvement 13% in tensile strength, 14% in flexural strength and 30% in impact strength, respectively. After alkali treatment, increase in strength and aspect ratio of the fiber contributed to the enhancement in the mechanical properties of the composites. The SEM micrograph of composite showed that the surface modification of the fiber occurred and improved fiber-matrix adhesion.

Gomes, Matsuo, Goda, and Ohgi (2007) improved the mechanical properties of high-strength curaua fiber/cornstarch-based composites by alkali treatment. The solution of 15 %wt. NaOH was applied to curaua fibers to improve mechanical properties of composites. Tensile test results showed that the fracture strain of alkali treated fiber composites was higher than that of untreated fiber composites about 2-3 times, without a considerable decrease in strength.

Mathew and Joseph (2007) treated isora fiber with 5 %wt. of NaOH solution for 4 hrs. SEM micrograph showed strong evidence for the physical microcellular structural changes occurring in the fiber surface on alkalization. The

pores of isora fiber became clearer and fibers became thinner. This might be due to the dissolution and leaching out of fatty acids and lignin components of the fiber. This rendered roughness to the fiber and thereby enhanced mechanical interlocking at the interface. The development of a rough surface topography offered better fiber interface adhesion and increased mechanical properties.

Saha, Manna, Chowdhury, Roy, and Adhikari (2010) investigated in physico-chemical properties of jute fibers treated with 0.5% of NaOH solution. The treatments were applied under ambient and elevated temperatures and high pressure steaming conditions. The results from this investigation indicated that a 30 min soaking of the fibers in 0.5% alkali solution followed by 30 min alkali-steam treatment led to an increase in the tensile strength of up to 65%. The increase appeared to be due to fiber separation and removal of non-cellulosic materials resulted in an increased crystallinity.

2.3.2 Acetylation

Acetylations were used to enhance the interfacial adhesion of natural fibers with polymer matrix by making it more hydrophobic. The main principle of the method is to react the hydroxyl group of the fiber with acetyl groups, therefore rendering the fiber surface more hydrophobic.

Hill, Khalil, and Hale (1998) studied on the acetylation of natural fibers. They found that the rate of acetylation was proportional to the lignin content of the fibers, with coir and oil palm empty fruit bunch (EFB) fibers exhibiting the fastest rate of reaction and ultimate substitution. Moreover, the effect of acetylation temperature was also observed. It was found that acetylation at a reaction temperature of 120°C, the fiber structure was damaged resulting in poor mechanical properties,

whereas acetylation at a reaction temperature of 100°C, the modified fibers exhibited improved performance.

Rong et al. (2001) investigated the effect of acetylation on the mechanical properties of unidirectional sisal fiber reinforced epoxy composites. The tensile test showed that acetylation can improve tensile strength of sisal fibers. The improvement of interfacial bonding in sisal fiber reinforced epoxy composites was the results of hydrogen bonds between acetyl groups and hydroxyl or amine groups in epoxy resin and mechanical interlocking between epoxy and the caved fiber surface due to the alkali pretreatment before acetylation.

Tserki, Zaferiropoulos, Simon, and Panayiotou (2005) studied esterification reaction between acetyl groups and hydroxyl groups of natural fiber. Acetylation was applied on flax, hemp and wood fibers. Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) and X-ray photoelectron spectroscopy (XPS) were used to investigate esterification between the acetyl groups and the hydroxyl groups of the fiber and their extent were assessed by titration. The results showed that the highest extent of the esterification reaction was achieved for the wood fibers due to their high lignin/hemicelluloses content. ATR-FTIR and XPS revealed that the fiber surface chemistry was altered after the treatments, as the results indicated that ester bonds are present on the fiber surface. The SEM results revealed that acetylation treatments resulted in a removal of non-crystalline constituents of the fibers, possibly waxy substances, and alter the characteristics of the surface topography. It was also shown that the fiber crystallinity decreased slightly as a result of esterification.

Zhang, Zhang, Liang, and Lu (2008) investigated the effect of surfaceacetylated wood pulp powder on mechanical properties of natural rubber composites. FTIR results showed the spectrum of acetylated wood pulp at 1738 cm⁻¹ assigned to C=O stretching vibration of –COCH₃, included with the increased intensity of C–O ester group stretching at 1244 cm⁻¹, indicating that acetylation between cellulose and acetic anhydride. The result from contact angle measurement showed average water contact angle at equilibrium for the acetylation fibers was 118 degree, which resulted in changing of hydrophilic nature of cellulose to completely hydrophobic of the wood pulp surface. The obtained wood pulp powder with hydrophobic surface showed good compatibility with natural rubber matrix leading to an improvement of mechanical properties.

2.3.3 Silanization

Silanes are recognized as efficient coupling agents extensively used in composites and adhesive formulations. They have been successfully applied in inorganic filler reinforced polymer composites such as glass fiber reinforced polymer composites. The bifunctional structures of silanes have also been of interest in applying them for natural fiber/polymer composites, since both glass fibers and natural fibers have reactive hydroxyl groups. The silane molecule should have bifunctional groups which may react with the two phases thereby forming a bridge between natural fiber and matrix.

Ismail, Shuhelmy, and Edyham (2002) studied the effect of silane coupling agent on mechanical properties of bamboo fiber filled natural rubber composites. The composites were prepared with 3 phr of bis (triethoxysilyl propyl) tetra sulphide (Si69). SEM micrographs of the composites without Si69 showed many holes remaining after the fibers were pulled out from the rubber matrix. In contrast, the composites with Si69 showed a better adhesion between bamboo fibers and rubber matrix. The fibers were well wetted by the rubber matrix and the pulled out of fibers from rubber matrix was minimum. This may be because the silane coupling agent improved the surface functionality of the bamboo fibers and subsequently enabled bamboo fibers chemical bond with the rubber matrix. Consequently, the strength of composite was increased with increasing interfacial adhesion between fiber and matrix.

Pothan, Thomas, and Groeninck (2006) examined the role of fibermatrix interactions in chemically modified banana fiber/polyester composites. Banana fibers were treated with the solution of 0.6% γ -methacryloxypropyl trimethoxy silane (A-174). Dynamic mechanical analysis was employed for dynamic mechanical property evaluation of the composites. The results showed that dynamic modulus values were increased and low damping values showed the improved interactions between the fiber and the matrix. The damping peaks were found to be dependent on the nature of chemical treatment. Both storage modulus and damping values attributed to the effectiveness of silane A-174 in improving fiber matrix adhesion.

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2.4 Factors affecting mechanical properties of natural fiber/ polymer composites

2.4.1 Fiber volume fractions

Fiber volume fraction is an important parameter controlling the properties of a fiber reinforced composites such as modulus, strength, and other properties.

The volume fibers fraction can be calculated from the fiber weight fraction. The fiber volume fraction is given by:

$$v_{f} = (w_{f}/\rho_{f})/(w_{f}/\rho_{f} + w_{m}/\rho_{m})$$
(2.5)

Where w = fiber weight fraction $w_m = matrix$ weight fraction $\rho_f = fiber$ density

 ρ_m = matrix density

In term of the fiber volume fraction, the composite density (ρ_c) can be calculated using

$$\rho_{\rm c} = \rho_{\rm f} \, v_{\rm f} + \rho_{\rm m} \, (1 \text{-} v_{\rm f}) \tag{2.6}$$

Shibata, Cao, and Fukumoto (2005) studied the effect of fiber volume fraction on flexural properties of short natural fiber reinforced composites. The results showed that the flexural modulus increased with increase of maximum fiber volume fraction up to 66%. Above the maximum fiber volume fraction, the flexural modulus decreased owing to insufficient resin.

2.4.2 Fiber orientations

Another important parameter controlling the properties of a fiber reinforced composites was the fiber orientation angle, which defined the orientation of fibers with respect to the loading directions. For example, in uniaxial tensile loading, fibers were most effective when the fiber orientation angle was 0° when they were oriented parallel to load direction. Meanwhile, in pure shear loading, the fibers were most effective when they were oriented at angles with +45 and -45 degree with respect to loading axis. Liu and Hughes (2008) investigated the effects of varying textile yarn linear density, textile weave configuration and the stacking sequence of the textiles on fracture properties of woven flax fiber reinforced epoxy composites. It was found that the addition of woven textile resulted in a 2-4 times improvement in K_{Ic} over that of the unreinforced epoxy, with a value of around 6 MPa/m² being obtained for a crossply laminate. Both fracture behavior and K_{Ic} were found to be strongly dependent upon the linear density of the weft yarn and the direction of crack propagation with respect to the orientation of the textile.

2.4.3 Fiber architecture

Fiber architecture refers to the arrangement of fiber, which can be either linear, two-dimensional and three dimensional. The fiber architecture is shown in Table 2.4.

Pearcea, Guildb, and Summerscalesaf (1998) investigated the effects of fabric architecture on the processing and properties of fiber reinforced composites produced by resin transfer molding. 5-harness satin weave (satin), 5-harness satin injectex weave (injectex) and 2×2 twill weave fabrics (twill) were used as reinforcement. The results showed the permeability increased in the order: satin<injectex<twill. Whereas the interlaminar shear strength (ILSS) increased in order: twill<injectex<satin. From the results obtained, the composites made from twill type fabric showed the minimum processing times but lowest ILSS. On the other hand, the composites made from satin type fabric showed the maximum processing time but possessed highest ILSS. Therefore, a choice must be made between maximum properties or minimum processing time. To compromise between processing time and properties, the development of new fabrics is necessary.

Linear	Two-dimensional (planar or 2D)	Three-dimensional (3D)
1. Continuous a. Monofilament b. Multifilament	1. Woven a. Biaxial b. Triaxial	1. Woven a. Angleinterlock b. Triaxial
- Untwisted	2. Knitted	 Knitted a. Multiaxial warf knit
2. Discontinuous	a. Weft b. Warp or fill	b. Fully fashioned weft knit
	3. Braided a. Biaxial	3. Braided a. Two-step
	b. Triaxial	b. Three-dimensional4. Nonwoven
	้ ^{อกยา} ลัยเทคโนโลยี ^{อุร} ั	a. Multiaxial b. x, y, z

Table 2.4 Fiber architecture (Mallick, 1997).

Pothan et al. (2008) investigated the effects of three different weave architectures including plain, twill, and matt, on tensile and flexural properties of sisal fabric/polyester composites. The woven sisal/polyester composites were prepared using resin transfer moulding technique. From the results obtained, matt-weave composites with a fiber content of 32 %vol gave a higher tensile and flexural property than plain and twill weave composites. The tensile strength and tensile modulus showed an improvement of 52% and 100% respectively. Composites which the maximum fibers are in the loading direction, combined with lower interlace point, were found to give the highest properties.



CHAPTER III

EXPERIMENTAL

3.1 Materials

Sisal fibers were (*Agava sisalana*) purchased from Sisal-Handicraft OTOP Group, Tambon Ban Kao, Amphur Dan Khun Thod, Nakhon Ratchasima, Thailand. Sodium hydroxide (NaOH, Carlo Erba, 97%) and γ -glycidoxypropyltrimethoxy silane (A-187) were used in treatment of sisal fiber. Bisphenol-A based epoxy resin (Epiclon 850) with epoxide equivalent weight 184-194 g/equivalent and polyamide hardener (Luckamide GL-120) were supplied by Siam Chemical Industry Co., Ltd. The chemical structures of Bisphenol-A epoxy resin and polyamide are shown in Figure 3.1 and 3.2 respectively.



Figure 3.1 Chemical structure of bisphenol-A epoxy resin.



Figure 3.2 Chemical structure of polyamide hardener.

3.2 Preparation of untreated woven fiber

The woven sisal fibers with woven space of 0.5 cm×0.5 cm were manufactured by hand weaving process. Three different weave types as shown in Figure 3.3 were prepared.

x		X		Х
	X		x	
х		X		Х
	X		X	
х		x		х

(a) Plain weave

Х			X	X
		X	X	
	X	X		
Х	X			x
х			x	x

(b) Twill weave

X X X X X X (c) Satin weave

Х

XX

XX

x x

X X X X

XX

Figure 3.3 Weave types of woven sisal fiber.

3.3 Fiber treatment

3.3.1. Alkalization

In order to eliminate hemicelluloses and waxes substances, the untreated woven sisal fiber (UTWSF) were soaked in 2% NaOH solution for 2 hrs at room temperature. Fiber to solution weight per volume ratio was 1 gram: 10 ml. Then woven sisal fibers were washed thoroughly with water and air dry at 70°C overnight. This woven sisal fiber was called alkali treated woven sisal fiber (ATWSF).

3.3.2 Silane treatment

The solution of γ -glycidoxy propyltrimethoxy silane was prepared by dissolving 2 gram of γ -glycidoxy propyltrimethoxy silane in 98 gram of water. The 2% solution of γ -glycidoxy propyltrimethoxy silane was adjusted to a pH value of 3.5 by acetic acids. The fiber to solution weight per volume ratio was 1 gram: 10 ml.

Х

X X

Then, woven sisal fiber was soaked in 2% solution of γ -glycidoxy propyltrimethoxy silane for 2 hrs at room temperature. After that, woven sisal fiber was washed thoroughly with water and air dry at 70°C overnight. This woven sisal fiber was called silane treated woven sisal fiber (STWSF).

3.3.3 Alkali-silane treatment

Beside the above methods of treatment, a combination of alkali and silane treatment was also used. In this treatment, ATWSF was further modified using silane treatment method as explained in section 3.3.2. This woven sisal fibers were called alkali-silane treated woven sisal fiber (ASTWSF).

3.4 Preparation of woven sisal fiber/epoxy composites

Woven sisal fibers were dried at 110°C for 3 hrs in a mechanical convection oven before use. The epoxy resin was mixed with 40 phr of polyamide (Luckamide GL 120) and stirred at a speed of 200 rpm for 5 min at room temperature. The mixture was poured into the mould previously coated with releasing agent about a half of mould depth. After that the woven sisal fibers were slowly embedded to avoid the formation of voids. After all the remaining resin was poured into the mould cavities, the mould covers were tightened. The samples were allowed to cure at room temperatures for 24 hrs in the mould. After that, each specimen was cut and polished with sandpaper. The photographs of cut and polished specimen are shown in Figure 3.4. Finally, all specimens were post-cured at 120°C for 2 hrs in a mechanical convection oven.



(a) Plain woven sisal fiber/epoxy composite



(b) Twill woven sisal fiber/epoxy composite



(c) Satin woven sisal fiber/epoxy composite

Figure 3.4 Photographs of sisal fiber/epoxy composites specimens.

The composition and designation of untreated woven sisal fiber/epoxy composites are shown in Table 3.1.

Designation	Epoxy resin	Hardener	Untreated woven sisal fiber (%wt.)		
	(phr) (p	(phr)	Plain weave	Twill weave	Satin weave
	100	40	5	-	-
UTPWSF composite	100	40	10	-	-
	100	40	15	-	-
UTTWSF composite	100	40	4 -	5	-
	100	40	٦-	10	-
	100	40	5	15	-
UTSWSF composite	100	40	-	-	5
	100	40	- 15	-	10
	100	ยาลั40าคโน	โลยีส <u>ุร</u> ั		15

 Table 3.1 Composition and designation of untreated woven sisal fiber/epoxy composites.

The selected woven type was subjected to alkalization, silanization and alkalisilane treatment. The composition and designation of treated woven sisal fiber/epoxy composites are shown in Table 3.2.

Designation	Epoxy resin Hardener		Trea	Treated woven sisal fiber (%wt.)		
Designation	(phr)	(phr)	Alkali	Silane	Alkali-silane	
			treatment	treatment	treatment	
ATWSF composite	100	40	15	-	-	
STWSF composite	100	40	-	15	-	
ASTWSF composite	100	40	-	-	15	

Table 3.2 Composition and designation of treated woven sisal fiber/epoxy composites.

3.5 Material characterization

3.5.1 Fiber characterization

Untreated and treated woven sisal fibers were cut into single fibers for characterization. Sisal fibers that cut from woven fiber fabric were designed in Table 3.3.

 Table 3.3 Designation of single sisal fibers cut from woven fiber fabric.

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Sisal fiber	Designation			
Untreated sisal fiber	UTSF			
Alkali treated sisal fiber	ATSF			
Silane treated sisal fiber	STSF			
Alkali-silane treated sisal fiber	ASTSF			

3.5.1.1 Thermal properties

Thermal degradation temperature and weight loss of untreated and treated sisal fibers were analyzed by a thermogravimetric analyzer (Perkin Elmer, model TGA7). The temperature range was from 30 to 700°C with a heating rate of 10°C/min under a nitrogen atmosphere.

3.5.1.2 Morphological properties

The fiber surface morphologies of untreated and treated sisal fibers were examined using a scanning electron microscope, SEM (JEOL, model JSM6400) at 10 kV. The sample surfaces were coated with gold before SEM analysis.

3.5.1.3 Measurement of fiber dimension

Average diameters of untreated and treated sisal fibers were measured using an optical microscope (Nikon, model Eclipes E600 POL) with a 10x objective lens.

3.5.1.4 Functional groups analysis

Functional groups of untreated and treated sisal fibers were characterized using Fourier transform infrared microspectrometer (FTIR, BRUKER) in the wavenumber range from 4000 cm^{-1} to 400 cm^{-1} , operating in ATR (attenuated total reflectance) mode.

3.5.1.5 Tensile properties

Tensile properties of untreated and treated woven sisal fibers were tested following ASTM D5034 using a tensile testing machine (Instron model 5565) with a load cell of 5 kN, a cross head speed of 300 ± 10 mm/min, and a gauge length of 75 ± 1 mm.

3.5.2 Composite characterization

3.5.2.1 Mechanical properties

Notched Izod impact strength of the pure epoxy resin, woven sisal fiber/epoxy composites was tested according to the ASTM D256, method A, using a basic pendulum impact tester (Atlas model BPI). The total striking impact energy of 2.7 J was assigned at room temperature. The geometry of specimens was 5 mm in thickness, 63.5 ± 2.0 mm in length and 12.7 ± 0.2 mm in width. Ten specimens were tested on each composite. The impact strength (kJ/m²) was calculated and reported.

In order to determine flexural properties of the pure epoxy resin and woven sisal fiber/epoxy composites, five specimens of each material were examined according to ASTM D790 using a universal testing machine (Instron model 5569) with a load cell of 50 kN, a crosshead speed of 2.13 mm/min and span length of 80 mm. The specimens were 5 mm in thickness, 10 mm in overall length and 20 mm in width. The measurements of flexural modulus, flexural strength and flexural strain were recorded. The support span of machine was 16 times the depth of the sample beam. The crosshead speed of machine calculated by the following equation:

$$R = \frac{ZL^2}{6d}$$
(2.7)

Where R = rate of crosshead motion, mm/min

L = support span, mm

d =depth of specimen, mm

Z = rate of straining of the outer fiber, Z shall be equal to 0.01, mm/mm/min

3.5.2.2 Morphological properties

Morphologies of the impact fractured surfaces of the pure epoxy and woven sisal fiber/epoxy composites were examined by scanning electron microscope, SEM (JEOL model JSM 6400), employing an accelerating voltage of 10 kV. The sample surfaces were coated with gold before SEM analysis.



CHAPTER IV

RESULTS AND DISCUSSION

4.1 The characteristics of woven sisal fiber

In this study, plain weave, twill weave and satin weave were employed as reinforcements in making woven sisal fiber/epoxy composites. The photographs of plain, twill and satin weave woven sisal fiber are shown in Figure 4.1.



Figure 4.1 Photographs of woven sisal fibers used as reinforcement in epoxy composite.

The woven characteristics such as interlace point, crimp angle and unit volume can affect the mechanical properties of composites (Mariatti et al., 2000; Houshyar and Shanks 2004; Pothan et al. 2008). Therefore, the woven characteristics are explained as follows.

4.1.1 Interlace point

A woven fiber is produced by interlacing in the warp and fill yarns. The interlace point is refered to the warp yarns connecting with fill yearns (Mallick, 1997). Orthogonal 2-D woven fibers were used in this study. Unit cell of plain, twill and satin weave are shown in Figure 4.2. From Figure 4.2 (a) unit cell of plain weave shows the highest interlace point and shortest float length. Float length is the length of yarn in between adjacent interlace. Interlace point of twill weave is lower than plain weave but higher than satin weave as shown in Figure 4.2 (b) and (c) respectively. These result in a longer float length for twill and satin weave than plain weave.



(c) Satin weave

Figure 4.2 Unit cell of plain, twill and satin weave.

4.1.2 Woven crimp angle

Woven crimp angle (β) is defined as the angle between the undulated yarn and the fabric plane. A woven fabric with a lower crimp angle has a lower number of over-under undulations and hence a longer flat segment in the yarns. Plain weave has the highest crimp angle whereas twill weave and satin weave have lower crimp angle. The fabric crimp angle is shown in Figure 4.3.



Figure 4.3 Example of woven crimp angle in plain weave (Atas and Liu, 2008).

4.1.2 Density of fiber per unit volume

The density of fiber per unit volume is referred to fiber volume in unit volume of composites. An increase of density of fiber per unit volume was caused by adding more woven ply into the composites with the same thickness as seen in Figure 4.4. When this density increase, woven fiber can connect to each other ply. Thus stress can be transfer along each woven ply.



Figure 4.4 Number of woven ply in unit volume of composites (Mallick, 1997)

It can be seen that each weave style has its own characteristic. The role of woven characteristics in mechanical properties of woven sisal fiber/epoxy composites are discussed in the next section.

4.2 Effects of fiber architecture and fiber content on physical properties of untreated woven sisal fiber/epoxy composites

4.2.1 Tensile properties of untreated woven sisal fiber

The tensile properties of untreated woven sisal fiber were summarized in Table 4.1.

Woven type	en type Rang of Average maximum load (N) (N)		Extension (mm)
Plain weave	928.04-948.45	935.80±11.05	3.43±0.23
Twill weave	935.15-975.01	956.55±20.09	3.42±0.09
Satin weave	1029.05-1077.18	1054.60±24.02	3.39±0.10

Table 4.1 Tensile properties of woven sisal fiber with different weave type.

It can be seen that satin weave showed the highest maximum load whereas twill weave and plain weave had a similar value of maximum load. This might be because satin weave had the longest float length and lowest in number of crimp angles. Therefore, satin weave had the highest fiber orientation in load direction and withstood the highest load. There was no different in fiber extension for woven fiber with different woven type.

4.2.2 Flexural properties of untreated woven sisal fiber/epoxy composites

The mechanical properties of untreated sisal fiber/epoxy composites are summarized in Table 4.2. The results from flexural tests are plotted in Figure 4.5 and Figure 4.6. The flexural strength of all composites was higher than that of pure epoxy resin as seen in Figure 4.5. This might be because the fibers act as load carrier

and stress is transferred from the matrix along the fibers leading to stress distribution. Figure 4.5 also shows that the flexural strength of untreated sisal fiber/epoxy composites was increased with an increase of fiber content. At 15 % wt., woven sisal fibers/epoxy composite showed the highest flexural strength for all woven type. These might be because flexural strength of woven sisal fiber composite depends on density of fiber per unit volume. The density of fibers in unit volume of woven sisal fiber composites was increased with an increase of fiber content. The higher fiber in unit volume of composites helped in crack bridging and crack arresting. This led to effective stress transfer (Mallick, 1997). On the observation of the effect of woven fiber architectures, it was found that the flexural strength of composites containing either plain weave or twill weave sisal fiber was lower than that of the composites containing satin weave sisal fiber at all fiber contents. 15 %wt. satin weave sisal fibers/epoxy composite showed the highest flexural strength (95.73 MPa). These might be because satin weave could withstand the highest load (1054.60 MPa) as presented in Table 4.1. Moreover, a number of interlace point has been identified as one of the weakest point in most woven fabric composite systems because it has the tendency for more voids and fiber distortion at the interlace gap (Ishikawa and Chou, 1982; Houshyar, Shanks, and Hodzic, 2004). Plain and twill weave woven sisal fiber have more interlace points and number of crimp angles than satin weave woven sisal fiber. These increased interstitial positions served as crack initiation points (Pothan et al., 2008). On the other hand, the close arrangement of fibers in satin weave gave rise to lesser interstitial positions and lesser chances of crack initiation compared to the other two weave patterns.

Samples	Fiber content (%wt.)	Flexural strength (MPa)	Flexural modulus (GPa)	Flexural strain (%)	Notched Izod Impact strength (kJ/m ²)
Pure epoxy	0	80.00±1.61	2.16±0.05	5.00±0.00	0.83±0.04
	5	82.09±3.69	2.52±0.20	3.85±0.71	5.68±0.66
UTPWSF Composite	10	85.89±5.65	3.10±0.28	3.34±0.33	10.43±0.41
Composite	15	88.01±3.66	3.23±0.12	3.13±0.14	13.20±0.32
UTTWSF Composite	5	86.18±2.97	2.48±0.18	4.72±0.49	6.17±0.78
	10	90.10±2.08	2.96±0.10	4.18±0.10	11.29±0.74
	15	91.08±3.44	3.29±1.48	3.87±0.20	13.77±0.67
	5	89.31±3.11	2.61±0.20	4.51±0.51	7.35±0.72
UTSWSF Composite	10	92.18±4.83	3.41±0.07	3.58±0.23	11.79±0.53
	15	95.73±1.55	3.44±0.03	3.38±0.16	15.45±1.11

Table 4.2 Mechanical properties of untreated woven sisal fibers/epoxy composites.



 Figure 4.5
 Flexural strength of pure epoxy and untreated woven sisal fiber/epoxy composites.

The flexural moduli of pure epoxy and woven sisal fiber/epoxy composites are shown in Figure 4.6. The flexural modulus of all composites was higher than that of pure epoxy resin. Moreover, flexural modulus also increased with increasing fiber content. This is as expected because the modulus of sisal fiber is high (Chand, Tiwary, and Rohatgi, 1998). The 15 %wt. satin woven sisal fiber/epoxy composites had the highest flexural modulus when compared with plain and twill weave composites. Orientation of the fibers with respect to the loading direction has been found to be a determining factor for composite properties (Mariatti, Nasir, and Ismail, 2000). The fiber orientation in the loading direction was highest in the case of satin weave architecture. Moreover, higher flexural modulus of satin woven sisal fiber/epoxy composites than twill and plain woven sisal fiber/epoxy composites at same fiber content should be a decrease of woven fabrics gaps. It was found that a decrease of crimp angle and the ratio of gap region between adjacent warp fiber per warp yarn width resulted in an increase of flexural moduli (Barbero, 2011).



Figure 4.6 Flexural modulus of pure epoxy and untreated woven sisal fiber/epoxy composites.

The results of flexural strain at maximum flexural strength (%) of woven sisal fiber/epoxy composites are shown in Figure 4.7. The results showed that all composites had lower flexural strain than pure epoxy and flexural strain tended to decrease with increasing fiber content. These may be related to a higher stiffness of fibers. When considering the relationship between flexural strain and weave type, it was found that the composites containing twill weave showed the highest flexural strain.



Figure 4.7 Flexural strain of pure epoxy and untreated woven sisal fiber/epoxy composites.

4.2.3 Impact strength of untreated woven sisal fiber/epoxy composites

The impact strength of untreated woven sisal fiber/epoxy composites with different woven architecture are shown in Figure 4.8. For all fiber loading, composites containing satin weave showed the highest impact strength values and the impact strength increased with increasing fiber contents. The maximum impact strength value of 15.45 kJ/m^2 was found for the composite with 15 % wt. satin weave architecture. This could be because a higher fiber contents in unit volume of

composites compared with plain weave and twill weave. Then a higher load could be transfer from the matrix to the fiber.



Figure 4.8 Notched Izod impact strength of pure epoxy an untreated woven sisal fiber/epoxy composites.

4.2.4 Morphological properties of untreated woven sisal fiber/epoxy composites

SEM micrographs of impact fractured surface of untreated woven sisal fiber/epoxy resin composites and pure epoxy resin are shown in Figure 4.9. It was observed that the pure epoxy resin showed ridged surface. The composites with different weave type are shown in Figure 4.9 (b), (c), and (d). The interstitial regions in the composite that served as crack initiators are clear from the SEM micrograph and more prominent in the case of plain weave and twill weave composite. The lower flexural and impact strength values in the cases of plain and twill weave composite may be correlated with the possible crack initiation and propagation due to higher fiber undulation positions. Similar results were observed by Pothan et al., (2008).



Figure 4.9 SEM micrographs of impact fractured surface of pure epoxy and untreated woven sisal fiber/epoxy composites; (a) pure epoxy, (b) UTPWSF composite, (c) UTTWSF composite, (d) UTSWSF composite. Interstitial regions are indicated by arrows.

4.3 Effect of fiber treatment on physical properties of woven sisal fiber/epoxy composites

4.3.1 The physical properties of untreated and treated woven sisal fiber

From the mechanical properties of untreated woven sisal fiber/epoxy composites, it can be seen that 15 %wt. satin woven sisal fiber/epoxy composites showed the best overall mechanical properties. Therefore, this composite was selected to study the effect of fiber surface treatment. The treatment methods used in this study were chemical treatment which included alkali, silane and alkali-silane treatment. The physical properties of treated satin woven sisal fiber were compared with those of untreated woven fiber in the following section.

4.3.1.1 Tensile properties and diameter of treated woven sisal fiber

The results from tensile tests are tabulated in Table 4.3 and the maximum load of treated and untreated woven sisal fibers are plotted in Figure 4.10. From the results obtained, UTSWSF showed the highest maximum load whereas ATSWSF showed the lowest maximum load. The decrease in maximum load of ATSWSF could be due to the loss of hemicellulose in fiber. Chand, Varma, and Khazanchi (1989) studied tensile strength of alkali treated sisal fiber was reduced.


Figure 4.10 Maximum load of untreated and treated satin woven sisal fiber.

Woven sisal fiber	Range of maximum load (N)	Average maximum load (N)	Extension (mm)		
UTWSF	1029.05-1077.18	1054.60±24.20	3.39±0.10		
ATWSF	689.50-931.03	817.09±42.00	3.25±0.15		
STWSF	967.35-1087.83	1027.00±43.44	3.38±0.23		
ASTWSF	908.67-997.68	945.00±22.70	3.34±0.11		

Table 4.3 Tensile properties of untreated and treated satin woven sisal fibers.

The average diameter of untreated and treated sisal fibers that

cut from woven fiber fabric were also measured using optical microscope. The resulted are shown in Figure 4.11 and summarized in Table 4.4. The results show that the average diameter of all treated fiber was lower than untreated fiber. This might be

because the fiber surface of sisal fiber was etched during treatment process. This result can be confirmed by SEM micrograph of sisal fiber surface (see section 4.3.1.2).



Figure 4.11 Average diameter of untreated and treated sisal fiber.

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Table 4.4 Diameter of untreated and treated sisal fibers.

Sisal fiber	Range of diameter (µm)	Average diameter (µm)
UTSF	480-200	384.00±81.30
ATSF	270-210	240.30±60.09
STSF	340-260	281.50±76.07
ASTSF	240-190	227.40±56.40

4.3.1.2 Morphological properties

The SEM micrographs of untreated and treated sisal fiber are shown in Figure 4.12. It was found that the fiber surface of STSF was rather smooth, whereas ATSF and ASTSF showed a rough surface. This could be because the alkali treatment removed the lignin and hemicellulose from fiber surface and fiber surface was etched during alkali treatment. This result was also confirmed in section 4.3.1.3.



Figure 4.12 The fiber surface of untreated and treated sisal fiber; (a) UTSF, (b) STSF, (c) ATSF, and (d) ASTSF.

4.3.1.3 Thermal properties

The temperature degradation behavior of untreated sisal fiber was studied using TGA. TGA and DTG thermograms of untreated and treated sisal fiber are shown in Figure 4.13 and 4.14 respectively.



Figure 4.13 TGA thermogram of untreated and treated woven sisal fibers.



Figure 4.14 DTG thermogram of untreated and treated woven sisal fibers.

The thermal degradation charcteristics of untreated and treated sisal fiber were summarized in Table 4.5.

Sisal fiber	1 st Transition	2 nd Tra	3 rd Transition	
	Peak (°C)	Initial (°C)	Peak (°C)	Peak (°C)
UTSF	64.37	258.95	287.91	354.24
ATSF	77.92	+ +	-	360.14
STSF	67.81	265.22	296.33	359.46
ASTSF	67.18	HAA.	-	359.10

Table 4.5 Thermal degradation characteristics of untreated and treated sisal fibers.

The TGA curve of sisal fibers shows three transition steps, whilst their decomposition occurs in two main stages. The first transition between 30° C and 100° C is corresponded to the evaporation of moisture from sisal fibers. The second transition step observed between 210° C and 310° C. It is associated to the thermal decomposition of hemicellulose. The third transition step observed in the range of $305-380^{\circ}$ C is corresponded to the degradation of α -cellulose present in the sisal fiber. Yang, Yan, Chen, Dong, and Zhen (2007) reported that cellulose, hemicellulose, and lignin decomposition in a range of $315-400^{\circ}$ C, $220-315^{\circ}$ C, and lower than 200° C - 700° C respectively.

For ATSF and ASTSF, the second transition peak that refers to hemicellulose of sisal fiber disappeared. The third transition of ATSF and ASTSF shifted to a higher temperature compared to UTSF. This indicated that alkali treatment process removed hemicellulose of sisal fiber. In addition, alkali treatment slightly improved thermal stability of the sisal fibers, as revealed by TGA and DTG curves. TGA thermogram of STSF showed second and third transition peak at 296.3°C and 359.5°C, respectively. This indicated that silane treatment did not remove hemicellulose from sisal fiber. The second and third transition peak of STWSF also shifted to a higher temperature compared to UTSF. This might be due to improvement of thermal stability of STSF.

4.3.1.4 Functional groups analysis

ATR-FTIR spectrum of the untreated sisal fiber is shown in Figure 4.15. A broad absorption band in the region 3600-3100 cm⁻¹ corresponded to the O-H stretching vibration of -OH in hemicellulose lignin, and cellulose components. The peaks at 3000-2854 cm⁻¹ were the characteristic band for the C-H stretching vibration of -CH and -CH₂ in hemicellulose lignin, and cellulose components. The peaks at 1765-1712 cm⁻¹ were the characteristic band for the C=O stretching vibration of carbonyl group in hemicellulose and aromatic group in lignin components. The carboxylic groups also presented in the fiber as traces of fatty acids in wax. The absorbance at 1627 cm⁻¹ may be due to the presence of water in fiber. A little peak at 1517 cm⁻¹ is attributed to C=C stretching of aromatic ring of lignin. The absorbance peak at 1430 cm⁻¹ is associated to the CH₂ symmetric bending presented in cellulose. The absorbance peaks at 1384 cm⁻¹ and 1243 cm⁻¹ corresponded to the C-O stretching vibration of the acetyl group in lignin and hemicelluloses component, respectively. The observed peak at 1320 cm⁻¹ in the spectrum indicated the bending vibration of C-H and C-O groups of the aromatic ring in polysaccharides.



Figure 4.15 ATR–FTIR spectrum of untreated sisal fiber.

The absorbance peaks at 1160 cm⁻¹ is due to the anti-symmetrical deformation of C-O-C band. The strong absorption peak at 1035 cm⁻¹ was ascribed to the C-O stretching which belonged to cellulose. The absorbance peaks at 1108 cm⁻¹ corresponded to O-H stretching vibration of C-OH in hemicellulose (Moŕan, Alvarez, Cyras, and Vázquez, 2008). The peak observed at 898 cm⁻¹ was attributed to the presence of β -glycosidic linkages between the monosaccharaides (Rosa, Kenny, Puglia, Santulli, and Sarasini, 2010). The typical functional groups and corresponding bands for each component are summarized in Table 4.6.

Table 4.6 Infrared main transitions of functional groups of sisal fiber (Moŕan,Alvarez, Cyras, and Vázquez, 2008)

Wave number (cm ⁻¹)	Functional group	Fiber component
4000-2995	-ОН	cellulose, hemicellulose, ligin
2,90	н-С-н	cellulose, hemicellulose, ligin
1765-1715	on Bracto Iulago	hemicellulose, ligin
1730-1700	Н-С-Н	ligin
1632	C=C	hemicellulose, ligin
1613, 1450	C=C	ligin
1430	O-CH ₃	ligin
1270-1232	C-O-C	cellulose, ligin
1170-1082	C-O-C	cellulose
1108	-OH	hemicellulose

ATR-FTIR spectra of treated sisal fiber compared with untreated sisal fiber are presented in Figure 4.16. The absorbance at 1743 cm⁻¹ and 1243 cm⁻¹ belonged to C=O stretching vibration of carbonyl group in hemicellulose and lignin respectively. The absorbance at 1743 cm⁻¹ and 1243 cm⁻¹ disappeared when sisal fiber was treated by alkali and alkali-silane treatment. This similar results was reported by Sun, Fang, Mott, and Bolton (2001). Compared to UTSF, a little peak at 852 cm⁻¹ of STSF and ASTSF appeared. This may be due to the presence of Si-O of silane that attached to fiber surface (Khan, Drzal, 2004). Rong et al. (2001) found that the absorption of Si-O and Si-O-Si was also observed at 1285, 1075, and 1018 cm⁻¹. However, the absorption peak of Si-O and Si-O-Si was very close to the strong absorption peak (1035 cm⁻¹) of C-O stretching which belonged to cellulose. This may overlap and may be not clearly observed.





Figure 4.16 ATR–FTIR spectra of untreated and treated sisal fiber.

4.3.2 Flexural properties of treated woven sisal fiber/epoxy composites

The flexural properties of treated woven sisal fiber/epoxy composites are shown in Figure 4.17-4.18. In Figure 4.17, the flexural strength of all treated woven fiber composites was lower than that of untreated woven fiber composites. The decrease in flexural strength of all treated woven fiber/epoxy composites could be due to a decrease of strength of the fiber during treatment process as discussed in section 4.3.1.1. In addition, satin weave woven sisal fibers were treated after weaving process. One reason that may be suggested for the lowering of properties is the change in the nature of the weave architecture. ATWSF composites showed the lowest value of flexural strength. This may be because ATWSF possessed the lowest strength compared to other treated fiber (see section 4.3.1.1). Alkali treatment process has resulted in the shrinkage (Gassan and Bledzki, 1999) and may change the basic nature of the weave pattern of the woven fiber. For ASTWSF composite, the flexural strength of ASTWSF composites was higher than ATWSF. This might be because silane treatment could improve flexural strength of the woven sisal fiber. The flexural modulus of the treated woven sisal fiber/epoxy composites are shown in Figure 4.18. The flexural modulus seemed to be improved for all treated woven sisal fiber composites. The flexural modulus of ATWSF, ASTWSF, STWSF and UTWSF composites was 3.82, 3.62, 3.50, and 3.44 GPa, respectively. The reason for the improvement in modulus can be attributed to the change in the intrinsic structure and thereby the crystallinity of the fibers (Ray, Sarkar, and Rana, 2002). The flexural strain of ATWSF was the lowest. These may be related the highest flexural modulus of ATWSF composites.



Figure 4.17 Flexural strength of treated satin woven sisal fiber/epoxy composites.



Figure 4.18 Flexural modulus of treated satin woven sisal fiber/epoxy composites.



Figure 4.19 Flexural strain of treated satin woven sisal fiber/epoxy composites.

The flexural properties of treated woven sisal fiber are summarized in

Table 4.7.

 Table 4.7 Flexural properties of treated woven sisal fibers/epoxy composites.

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Samples	Flexural strength (MPa)	Flexural modulus (GPa)	Maximum strain (%)		
UTWSF composite	95.73±1.55	3.44±0.03	3.380±0.16		
ATWSF composite	76.93±5.87	3.82±0.07	2.294±0.18		
STWSF composite	83.62±1.70	3.50±0.13	3.480±0.16		
ASTWSF composite	82.83±4.46	3.62±0.37	2.880±0.23		

4.3.3 Impact strength of treated woven sisal fiber/epoxy composites

The impact strength of satin woven sisal fiber/epoxy composites are summarized in Table 4.8 and graphically plotted in Figure 4.20. From the results obtained, the impact strength of the ATWSF and ASTWSF composites was lower than that of UTWSF composite. This might be because the reduction in average fiber diameter led to lower connection between woven plies. Then the stress transferred from the matrix along the fibers in woven plies was decreased. However, the impact strength value of STWSF was not different from the UTWSF composites. This might be because tensile strength of UTWSF and STWSF was not significantly different, although average diameter of STSWSF was lower than UTWSF after fiber treatment process.

Samples	Notched Izod impact strength (kJ/m ²)		
UTSWSF composite	Umafula92 15.45±1.11		
ATSWSF composite	12.01±0.90		
STSWSF composite	15.37±0.83		
ASTSWSF composite	10.90±0.56		

Table 4.8 Impact strength of treated satin woven sisal fibers/epoxy composites.



Figure 4.20 Impact strength of treated satin woven sisal fiber/epoxy composites.

4.3.4 Morphological properties of treated woven sisal fiber/epoxy composites

SEM micrographs of untreated and treated satin woven sisal fiber/epoxy resin composites are shown in Figure 4.21 (a-1)-(d-2). The smooth surface of sisal fiber that pull out from epoxy matrix and large gap were observed in SEM micrograph of UTWSF composites (Figure 4.21 (a-1) and (a-2)). For fractured surface of STWSF composites as shown in Figure 4.21 (b-1) and (b-2), a roughness fiber surface and irregular shape of fiber tips were observed. In addition, it can be seen from Figure 4.21 (c-1), (c-2), (d-1), and (d-2) that the inner gaps of sisal fiber in ATWSF and ASTWSF composites were filled with epoxy resin. However, the impact strength of treated woven sisal fiber/epoxy composites was lower than that of UTWSF

composites. This might be because the reduction of fiber diameter and strength of woven sisal fiber after treatment.



Figure 4.21 The impact fractured surface of untreated and treated satin woven sisal fiber/epoxy composites; (a-1) and (a-2) UTWSF composite, (b-1) and (b-2) STWSF composite, (c-1) and (c-2) ATWSF composite, and (d-1) and (d-2) ASTWSF composite.



Figure 4.21 The impact fractured surface of untreated and treated satin woven sisal fiber/epoxy composites; (a-1) and (a-2) UTWSF composite, (b-1) and (b-2) STWSF composite, (c-1) and (c-2) ATWSF composite, and (d-1) and (d-2) ASTWSF composite (continued).



CHAPTER V

CONCLUSIONS

5.1 Conclusions

In this study, woven sisal fiber with three different weave type including plain, twill, and satin weave were used as reinforcement for epoxy resin. Woven sisal fiber contents used in composite preparation were 5, 10, and 15 % wt. The flexural, impact, and morphological properties of untreated woven sisal fiber/epoxy composites were studied. Flexural modulus and flexural strength of untreated woven sisal fiber/epoxy composite were increased with increasing fiber content. 15 %wt. satin woven sisal fiber/epoxy composite showed the highest flexural strength and flexural modulus. Flexural strain of pure epoxy resin was higher than that of untreated woven sisal fiber/epoxy composites. An increase of fiber content in untreated woven sisal fiber/epoxy composite resulted in a decrease of flexural strain for all type of untreated woven fiber composite. The notched Izod impact strength of all untreated woven sisal fiber/epoxy composite was significantly higher than that of pure epoxy resin. The maximum impact strength value of 15.45 kJ/m^2 was found for the composite with 15 %wt. satin weave architecture. The SEM micrograph of untreated woven sisal fiber revealed that the fibers pull out and large gaps appeared in all of untreated woven sisal fiber composites. From flexural and impact properties of untreated woven sisal fiber/epoxy composite, 15 % wt. satin woven sisal fiber/epoxy composite had the best overall mechanical properties. Therefore, this composite was selected to study the effect of fiber surface treatment.

Alkali, silane, and alkali-silane treatment process were applied to satin woven sisal fiber. The maximum load and average diameters of fiber cut from treated satin woven sisal fiber were decreased. The maximum load of treated satin woven sisal fiber was in the order of STWSF>ASTWSF>ATWSF. ASTWSF showed the lowest average diameter, whereas STWSF showed the highest average diameter. The result from FTIR and TGA illustrated that hemicelluloses was removed from the fiber surface. The SEM micrographs revealed the rough surface of ATWSF and ASTWSF. FTIR results also confirmed the attachment of Si-O group of silane on the fiber surface.

The flexural and impact properties of treated satin woven sisal fiber/epoxy composites were studied. ATWSF composites possessed the highest flexural modulus. The flexural strength of treated satin woven sisal fiber/epoxy composites was lower than untreated satin woven sisal fiber composites. The impact strength of ASTWSF composites was the lowest whereas the impact strength of STWSF composite was not significantly different from UTWSF composite. The SEM micrographs of impact fractured surface of ATWSF and ASTWSF composites showed that the inner gaps of sisal fiber were filled with epoxy resin.

5.2 Suggestions for further work

The main interesting topics for the further study related to this research should be as follows:

 (i) an investigation of the effect of other types of fiber yarn on mechanical properties of woven sisal fiber/epoxy composite

- (ii) a study of the effect of woven fiber preparation techniques on mechanical properties of woven sisal fiber epoxy composite and
- (iii) a study of possibility of applying woven sisal fiber/epoxy composites in engineering application.



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APPENDIX A PUBLICATIONS

List of Publications

- Srisuwan, S. and Chumsamrong, P. (2010). Effect of fiber orientation and fiber content on physical properties of sisal fiber/epoxy composites. Proceeding of Pure and Applied Chemistry International Conference 2010. (pp 464-466).
- Srisuwan, S. and Chumsamrong, P. (2010). Effects of weave type and fiber content on physical properties of sisal fiber/epoxy composites. Advance Materials Research. 123-125: 1139-1142.
- Srisuwan, S. and Chumsamrong, P. (2010). Influence of fiber orientations and fiber content on mechanical properties of sisal fiber/epoxy composites. The 1st
 Research Symposium on Petroleum, Petrochemicals, and Advanced Materials and 16th PPC Symposium on Petroleum, Petrochemicals, and Polymers. (pp 82). Bangkok, Thailand.
- Srisuwan, S. and Chumsamrong, P. (2011). The effect of sisal fiber treatment on mechanical properties of woven sisal fiber/epoxy composites. The 2nd
 Research Symposium on Petroleum, Petrochemicals, and Advanced Materials and 17th PPC Symposium on Petroleum, Petrocchemicals, and Polymers. (pp 82). Bangkok, Thailand.
- Srisuwan, S. and Chumsamrong, P. (2012). Effects of fiber architecture and fiber surface treatment on physical properties of sisal fiber/epoxy composites. Advance Materials Research. 410: 39-42.

Effects of fiber orientation and fiber content on physical properties of sisal fiber/epoxy composites

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Abstract: Traditionally, synthetic fibers are used as reinforcing fiber in epoxy composites. However, increasing of environmental concern has resulted in a development of sustainable materials that can replace synthetic fibers. The natural fibers seem to be good alternative because they are high specific strength, low density and cost effectiveness [1]. Sisal fiber is one of the most interesting natural fibers that can be found in Thailand. The aim of this work was to investigate the effects of fiber orientation in woven form with different woven space and fiber content which varied from 1 wt.%, 3 wt.% and 5 wt.% of untreated sisal fiber on flexural and impact properties of sisal/fiber epoxy composites. The samples were produced by hand lay up process and they were cured at room temperatures for 24 hours in two plate moulds. After that the samples were subjected to flexural (three-point bending) and impact testing according to ASTM D790-02 and ASTM D256-02 respectively.

Introduction

The use of natural fibers as reinforcements in polymer composites is currently significance. This is due to the potential environmental benefits. One of the most interesting fibers is sisal fiber. Sisal plants can be found in Thailand especially in Nakhon Ratchasima. Sisal fiber is widely used in making ropes, mats, carpets because it has good mechanical properties such as low density, high specific strength [1-3]. In addition, it is renewable and low cost [4].

In this work, the investigation of mechanical properties of woven sisal fiber reinforced epoxy composites was focused. The influences of woven space and fiber content of sisal fiber on the impact and fractural properties of sisal fiber/epoxy composite were evaluated.

Materials and Methods

Sample preparation

Sisal fibers used in this study were obtained from Nakhon Ratchasima, Thailand. Woven sisal fibers were manufactured by hand weaving process. Two different woven patterns as shown in Figure 1 were used.



Figure 1. Schematic representation of woven sisal fibers used as reinforcement in epoxy matrix

The fiber content in epoxy composites was fixed at 1 wt.%, 2 wt.% and 3 wt.%. This was achieved by varying the number of woven plies. Weight per area and the number of woven plies of woven sisal fibers in composites are tabulated in Table 1.

Table 1: Weight per area of woven sisal fibers and the number of woven plies in composites

Woven pattern	Weight/area (g/cm ²)	wt.% of fiber in composite	The number of woven plies
		1%	1
А	5.649x10 ⁻³	3%	4
		5%	7
		1%	2
В	3.286x10 ⁻³	3%	7
		5%	12

To produce test samples, Epiclon 850 epoxy resin and Luckamide hardener supplied by Siam chemical industry co., ltd. were used. Stoichiometric ratio of epoxy and hardener was 100:40 by weight. The mixture was then carefully and slowly poured into the mould cavities about a half of mould depth after that the woven sisal fibers were slowly embedded to avoid the formation of voids. After all the remaining resin

464 PACCON2010 (Pure and Applied Chemistry International Conference) was carefully poured into the mould cavities, the mould covers were tightened. The samples were allowed to cure at room temperatures for 24 hours in the mould and the samples were post cured at 120° C for 2 hour.

Sample testing

Determination of flexural strength and flexural modulus of samples was done at room temperature using an Instron testing machine following ASTM D790-02. Each sample was loaded over the support span length of 96 mm and crosshead speed of 2.56 mm/min until the sample fractures. The testing for impact strength was done using impact testing machine following ASTM D256-02. At least five samples were used for both impact and flexural testing.

Results and Discussion

The flexural and impact properties of pure epoxy resin and composites are summarized in Table 2.

Table 2: Flexural and impact properties for pure epoxy resin and epoxy/woven sisal fiber composites

	Flex	Impact	
Epoxy composites	σ (MPa)	E (GPa)	J/mm ²
Pure epoxy	100.95	2.323	0.053
Epoxy /1% pattern A	99.61	2.450	0.007
Epoxy /3% pattern A	82.73	2.666	0.009
Epoxy /5% pattern A	74.01	2.484	0.023
Epoxy /1% pattern B	97.95	2.526	0.012
Epoxy /3% pattern B	67.67	2.625	0.023
Epoxy /5% pattern B	63.00	2.690	0.007

For the flexural test, the results are graphically compared in Figures 2-3. It was found that the flexural strength of composites containing fiber with different woven pattern decreased when sisal fiber content increased. This could be because the natural fibers have the poor wettability and absorbability toward polymers resulting from the hydophilicity of plant fibers [5-7]. Nevertheless, the composites prepared from smaller woven space (pattern A) seemed to show slightly higher flexural strength than those prepared from larger woven space (pattern B). This might be because the number of plies of small woven space (pattern A) was lower than those of large woven space resulted in less interfacial weak points. From Figure 3, it was found that the flexural modulus of all composites were higher than that of pure epoxy resin. This is as expected because the modulus of sisal fiber is high. The composites containing woven fibers with woven space of 1x1cm (pattern B) increased with an increase of sisal fiber. The maximum flexural modulus was 2.69 GPa. In case of the composites prepared from

woven fiber with woven space of 0.5x0.5cm (pattern A), the maximum modulus was about 2.67 GPa at 3 wt.% sisal fibers. However, when sisal fibers content was increased to 5 wt.% the flexural modulus of the composites prepared from woven fiber with woven space of 0.5x0.5cm slightly dropped to 2.48 GPa. This might be because more voids were trapped in sisal fiber and epoxy matrix when higher fiber content of small woven space was used.



Figure 2. Variation of flexural strength of sisal fiber reinforced epoxy resin as a function of fiber content and woven pattern.



Figure 3. Variation of flexural modulus of sisal fiber reinforced epoxy resin as a function of fiber content and woven pattern

The results of impact strength are given in Figure 4. It was found that the impact strength of sisal fiber/epoxy composites made from both type of woven pattern was lower than that of pure epoxy samples. This could be due to poor interfacial adhesion between epoxy matrix and sisal fiber [8]. The gap between sisal fiber and epoxy matrix acted as stress concentrator leading to low impact strength. At 1 and 3 wt.% fiber content, the impact strength values of composites containing fibers with woven space of 0.5x0.5mm (pattern A) were lower than those of the composites containing fibers with woven space of 1x1mm (pattern B). This could be because small woven space was tighter and difficult to relax leading to more brittle characteristic. However, when fiber content was

increased to 5 wt.%, the present of more voids in composites containing fibers with woven space of 0.5x0.5mm (pattern A) led to more energy dissipation. The composite containing woven fiber with woven space of 1x1mm (pattern B) showed the lowest impact strength at 5 wt.% fiber. This might be because higher plies (12 plies) in composites containing 5 wt.% fibers with woven space of 1x1mm (pattern B) led to more weak point of interfacial adhesion between fiber and matrix. This is in agreement with the explanation given earlier.



Figure 4. Variation of impact strength of sisal fiber reinforced epoxy resin as a function of fiber content and woven pattern

Conclusions

The flexural strength of all composites was lower than that of pure epoxy resin. However, the composites prepared from smaller woven space (pattern A) seemed to show slightly higher flexural strength than those prepared from larger woven space (pattern B). Flexural modulus of all composites was higher than that of pure epoxy resin. In addition, with an exception of the composites containing 5 wt.% woven fiber with woven space of 0.5x0.5cm (pattern A), the flexural modulus tended to increase with increasing fiber content. The impact strength of all composites was lower than that of pure epoxy resin. The highest impact strength of composite prepared from woven sisal fiber with woven space of 0.5x0.5cm (pattern A) appeared at 5 wt.% fiber but the highest impact strength of composite prepared from woven sisal fiber with woven space of 1x1 cm (pattern B) appeared at 3 wt.% fiber. The results obtained implied that both fiber content and woven pattern have affected the flexural and impact properties of the composites. Moreover, the interfacial adhesion between fiber and matrix also affected the properties of composites. Therefore, the modification of sisal fiber surface will be investigated in furthur study.

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Effects of Weave Type and Fiber Content on Physical Properties of Sisal Fiber/Epoxy Composites

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Keywords: sisal fibers, epoxy composite, alkali treated fibers

Abstract. In this study, the effects of weave type and fiber content on the physical properties of woven sisal fiber/epoxy composites were investigated. Sisal fibers used in this work were obtained from Nakhon Ratchasima, Thailand. Both untreated and alkali-treated fibers were employed. The woven sisal fibers were manufactured by hand weaving process. The fiber content in sisal fiber/epoxy composites were 3 wt.%, 5 wt.% and 10 wt.%. The composites were cured at room temperatures. In order to determine mechanical properties of the composites, flexural and impact tests were applied. Flexural strength and flexural modulus of all composites were higher than those of pure epoxy resin and tended to increase with increasing fiber content. The impact strength of all composites was lower than that of pure epoxy resin. The composites containing 10 wt.% sisal fibers showed the highest impact strength. There was no definite influence of weave type on flexural properties of the composites. At 3 and 5 wt.% fiber, the composites containing plain weave fibers seemed to show a higher impact strength than the composites containing other weave types.

Introduction

The use of sisal fibers as reinforcements in polymer composites to replace synthetic fibers like glass fibers are presently increasing attention because sisal fibers has interesting mechanical properties including low density, high specific strength. In addition, it comes from renewable resources [1]. Generally, natural fiber reinforcement can be used as short randomly distributed fibers, long oriented fibers, or as a fiber fabric. Some of mechanical properties such as tensile and flexural properties of composites were found to increase when long fiber [1] and woven fabric[2] were used. Two-dimensional woven fabrics are the most widely used forms for composite reinforcement. The different fabric weave types have different characteristics. For example, plain weave provides the maximum fabric stability and firmness with minimum yarn slippage. Basket weave is less crimped and the fabric is more conformable than in plain weave [3].

In this work, the investigation of physical properties of woven sisal fiber reinforced epoxy composites was focused. The influences of woven weave type and fiber content of sisal fiber on the impact and fractural properties of sisal fiber/epoxy composites were evaluated.

Materials and Methods

Sisal fibers employed in this work were obtained from Nakhon Ratchasima, Thailand. Woven sisal fibers were manufactured by hand weaving process. Three different woven weave types as shown in Fig. 1 were used. The epoxy resin (Epiclon 850) and hardener (lackamide) were supplied by Siam chemical industry co., ltd.

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Alkali-treated sisal fibers were prepared by soaking in a 2 wt.% NaOH solution for 2 h at room temperature. After that the woven sisal fiber was washed thoroughly with water to remove the excess NaOH and air dried at 70°C overnight.

Sisal fiber/epoxy composite laminates with fiber content of 3 wt.%, 5 wt.% and 10 wt.% were prepared as follows. Treated and untreated fibers were dried at 110 °C for 2 h before use. Stoichiometric ratio of epoxy and hardener was 100:40 by weight. The mixture of epoxy and hardener was then carefully poured into the mould cavities about a half of mould depth after that the woven sisal fibers were embedded. After all the remaining resin was carefully poured into the mould cavities, the mould covers were tightened. The samples were allowed to cure at room temperatures for 24 h in the mould and the samples were post cured at 120 °C for 2 h. Weight per area and the number of woven plies of woven sisal fibers in the composites are tabulated in Table 1.

Designation	Woven weave type	Weight/area (g/cm ²)	wt.% of fiber in composite	The number of woven plies
UTSFP	Untreated sisal plain weave	2.543 x 10 ⁻³	3	1
UTSFB	Untreated sisal basket weave	2.510 x 10 ⁻³	5	2
UTSFR	Untreated sisal right hand twill weave	2.492 x 10 ⁻³	10	4
ATSFP	Alkali-treated sisal plain weave	2.524 x 10 ⁻³	3	1
ATSFB	Alkali-treated sisal basket weave	2.498 x 10 ⁻³	5	2
ATSFR	Alkali-treated sisal right hand twill weave	2.485 x 10 ⁻³	10	4

Table 1 Weight per area of woven sisal fibers and the number of woven plies in the composites.

Determination of flexural strength and flexural modulus of samples was done at room temperature using an Instron testing machine. Each sample was loaded over the support span length of 96 mm and crosshead speed of 2.56 mm/min until the sample was fractured. The testing for impact strength was done using impact testing machine. At least five samples were used for both impact and flexural testing.

Results and Discussion

Flexural properties of woven sisal fiber/epoxy composites

The results from flexural tests are shown in Fig. 2-3. In general, flexural strength of the composites seemed to higher than that of pure epoxy resin as seen in Fig. 2. This might be because the fibers act as carrier of load and stress is transferred from the matrix along the fibers leading to stress distribution. In addition, flexural strength tended to increase with an increase of fiber content. At low fiber content, the fibers are incapable of transferring load to one another and stress gets accumulated at certain points of composite leading to low flexural strength. At higher fiber content, the fibers actively participate in stress transfer [4].

In Fig. 3, flexural modulus of all composites were higher than that of pure epoxy resin. Moreover, flexural modulus also increased with increasing fiber content. This is as expected because the modulus of sisal fiber is high [5].

Woven weave type and fiber treatment only slightly affected the flexural properties of the composites. However, there was no definite influence of weave type and fiber treatment on flexural properties of the composites.





Figure 2. Variation of flexural strength of sisal fiber/epoxy resin composites as a function of fiber content and sisal fiber weave type.

Figure 3. Variation of flexural modulus of sisal fiber/epoxy resin composites as a function of fiber content and sisal weave type.

Impact properties of woven sisal fiber/epoxy composites

The results of impact tests are presented in Fig. 4. It can be seen that the impact strength of sisal fiber/epoxy composites made from different weave type was lower than that of pure epoxy samples. This could be due to poor interfacial adhesion between epoxy matrix and sisal fiber [6]. The gap between sisal fiber and epoxy matrix acted as stress concentrator leading to large different value of impact strength between pure epoxy and the composites. The impact strength seemed to increase with an increase of fiber content. As explained above, the higher fiber content may lead to better stress transfer. At 3 and 5 wt.% fiber, the impact strength of composites containing plain weave fibers were slightly higher than those containing other types of fibers. It might be because the plain weave procures the maximum fabric stability then it has good strength in the two yarn direction. However, when the fiber content was increased to 10 wt. %, the weave type seemed to show no definite influence on impact strength. Generally, alkali-treated sisal fiber did not clearly lead to an increment of impact strength. This is probably because fiber treatment in this work does not significantly improve interfacial adhesion between woven fiber and epoxy matrix.

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Figure 4. Variation of impact strength of epoxy resin and sisal fiber/ epoxy composites as a function of fiber content and sisal fiber weave type.

Conclusions

Flexural strength and flexural modulus of all composites were higher than those of pure epoxy resin and tended to increase with increasing fiber content. The impact strength of all composites was lower than that of pure epoxy resin. The composites containing 10 wt.% sisal fibers showed the highest impact strength. The results obtained disclosed that the flexural and impact properties of composites containing treated and untreated sisal fibers were insignificantly different. At 3 and 5 wt.% fiber, the composites containing plain weave fibers seemed to show a higher impact strength than the composites containing other weave types.

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Poster Sessions

SUT-PE

PA-P(9)-15 Influence of Fiber Orientations and Fiber content on Mechanical Properties of Sisal Fiber/Epoxy Composites

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The aim of this work was to investigate the influence of fiber orientation in woven form with different woven space and fiber content which varied from 1 wt.%, 3 wt.% and 5 wt.% on flexural and impact properties of sisal/fiber epoxy composites. The results obtained implied that both fiber content and woven pattern have affected the flexural and impact properties of the composites. As for the case of flexural properties, the composites prepared from woven space of 0.5cm x 0.5cm seemed to show slightly higher flexural strength than those prepared from woven space of 1.0cm x 1.0cm but the flexural strength of all composites was lower than that of pure epoxy resin. In the case of impact properties, the impact strength of all composites was lower than that of pure epoxy resin. In the case of impact strength of composite prepared from woven space of 0.5cm \times 0.5cm seemed to show slightly higher flexural strength than those prepared from woven space of 1.0cm x 1.0cm but the flexural strength of all composites was lower than that of pure epoxy resin. In the case of impact strength of all composites was lower than that of pure epoxy resin. The highest impact strength of composite prepared from woven space of 0.5cm.5cm appeared at 5 wt.% fiber but the highest impact strength of composite prepared from woven sisal fiber with woven space of 1.0x1.0 cm appeared at 3 wt.% fiber.



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Poster Sessions

PA-P(9)-14 The Effect of Sisal Fiber Surface Treatment on Mechanical Properties of Woven Sisal Fiber/Epoxy Composites

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The aim of this work was to investigate the effect of fiber surface treatment on flexural and impact properties of woven sisal fiber/epoxy composites. A 2 wt% sodium hydroxide aqueous solution and a 2 wt% γ -glycidoxypropyltrimethoxysilane aqueous solution were used to treat the fiber. The untreated sisal fiber (UTSF), alkali treated sisal fibers (ATSF) and silane treated sisal fiber (STSF) were characterized using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Woven sisal fiber/epoxy composites with different fiber surface treatment were prepared by hand lay up process. Flexural and impact properties of the composites were examined according to ASTM D790-02 and ASTM D256-02 respectively

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The Effects of Fiber Architecture and Fiber Surface treatment on Physical properties of Woven Sisal Fiber/Epoxy Composites

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Abstract. The aim of this work was to investigate the effects of fiber architecture and fiber surface treatment on flexural and impact properties of woven sisal fiber/epoxy composites. The woven sisal fibers with three different weave types including plain weave (P-weave), harness satin weave (S-weave) and right hand twill weave (R-weave) were used. For untreated fiber/epoxy composites, the fiber contents in the composite were 0, 5, 10 and 15% by weight (%wt). The untreated S-weave sisal fiber/epoxy composites showed the best overall properties and the composites with 15% fiber loading showed the highest properties. When compared to pure epoxy, flexural strength, flexural modulus and impact strength of the composite with 15% wt fiber increased by 4.5%, 60.6% and 150% respectively. Therefore, the composite of 15% wt silane treated S-weave and epoxy was prepared in order to study the effect of fiber surface treatment. The results showed that the composite containing 15% wt silane treated fiber possessed nearly the same properties with the untreated fiber/epoxy composite.

Introduction

Epoxy composites for engineering applications such as aircraft, sporting goods and structural applications require high strength, high stiffness, impact resistance and lightweight. In the early stage, carbon and glass fiber were commonly used as reinforcement materials. However, these materials are expensive and cause of environmental problems. The trend in environmental awareness has contributed to the development, improvement and use of natural fibers as reinforcing materials in polymeric composites. The advantages of natural fiber are high specific stiffness, high specific strength, low density, low cost, recyclability and biodegradability [1-3]. In the past decade, natural fibers such as hemp, flax, jute, bamboo and sisal fiber were applied by some researchers as fiber reinforcement for thermoset composites [4]. These natural fibers can be used in the forms of chopped, staple or short fibers, mats and woven fabrics for thermoset composites. The woven fabrics composites showed more balanced mechanical properties, easier applied to processing method such as hand lay up process and good for structural design [5]. Generally, the common woven fiber architecture such as plain weave, basket weave and twill weave were used in lamination polymer composites.

Although natural fibers are interested for using as reinforcement in polymer composites, one main drawback of natural fibers is poor adhesion with polymer matrix due to its hydrophilicity. The adhesion between reinforcement and matrix can be enhanced by chemical treatment such as alkalization, silanization and acetylation.

In this work, the mechanical properties of untreated woven sisal fiber/ epoxy composites with difference fiber architecture were firstly investigated. The fiber with woven architecture that gave the best overall mechanical properties was subjected to silanization and used in the composite preparation.

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Experimental

Materials. Sisal fibers used in this study were obtained from Nakhon Ratchasima, Thailand. γ -Glycidoxypropyltrimethoxy silane (A-187) was purchased from Shin-Etsu silicones Co., Ltd. Bisphenol-A based epoxy resin (Epiclon 850) and polyamide hardener (Luckamiode GL120) were supplied by Siam Chemical Industry Co., Ltd.

Preparation of woven sisal fiber. The woven sisal fibers were manufactured by hand weaving process. Three different woven architecture as shown in Fig. 1 were used.



Fig. 1 Schematic representation of woven sisal fiber architecture.

Silanization of woven sisal fiber. The solution of γ -glycidoxypropyltrimethoxy silane was prepared by dissolving 2 g of γ -glycidoxypropyltrimethoxy silane in 98 g of water. The 2% solution of γ -glycidoxypropyltrimethoxy silane were adjusted to pH value of 3.5 by acetic acid. The untreated harness satin woven sisal fiber were soaked in a solution of 2% γ -glycidoxypropyltrimethoxy silane for 2 h at room temperature. After that, the woven sisal fibers were washed thoroughly with water and air dry at 70 °C overnight.

Preparation of woven sisal fiber/epoxy composites. Woven sisal fibers were dried at 110 $^{\circ}$ C for 3 h in a mechanical convection oven before use. Stoichiometric ratio of epoxy and hardener was 100:40 by weight. The mixture was then carefully and slowly poured into the mold cavities about a half of mold depth after that the woven sisal fibers were slowly embedded to eliminate the formation of air bubbles. After all the remaining resin was carefully poured into the mold cavities, the mold covers were tightened. The samples were allowed to cure at room temperatures for 24 h in the mold and the samples were post cured at 120 $^{\circ}$ C for 2 h. The test samples were cut to shape for impact and flexural tests.

Mechanical property measurements. Flexural properties were determined using Instron univeral testing mechine model 5565 according to ASTM D790-02 standard. The testing for impact strength was done using impact testing machine following ASTM D256-02.

Results and discussion

The flexural properties of untreated woven sisal fiber/epoxy composites are shown in Fig. 2. It was found that the flexural strength of composites tended to be affected by woven type. The composites prepared from untreated harness satin woven sisal fiber showed the best flexural strength for all fiber loading. This could be because harness satin weave has long float length, loose pattern, small amount of fiber bundle crimp and fewer interlace point than the other woven architecture [6]. The fiber bundles in loose pattern can closely pack together leading to a higher fiber per unit volume. In addition, the fewer interlace point of harness satin woven sisal fiber resulted in a reduction of warp and weft crimp angle. This can reduce voids during the preparation of composites. Flexural moduli of all untreated fiber/epoxy composites were higher than that of pure epoxy resin and increased with an increase of fiber content. The harness satin woven fiber/epoxy composite with 15% fiber loading possesed the highest modulus which is 3.42 GPa.

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The impact strength of untreated woven sisal fiber/epoxy composites with different woven architecture was shown in Fig. 3. For all fiber loading, composites containing harness satin weave showed the highest impact strength values and the impact strength increased with increasing fiber loading. The maximum impact strength value of 15.45 kJ/m² was found for the composite with 15% wt harness satin woven fiber. This could be because a higher fiber contents, a higher load was transfer from the matrix.





Fig. 2 Variation of flexural strength of woven sisal fiber/epoxy composites as a function of fiber content and woven pattern.

Fig. 3 Variation of flexural modulus of woven sisal fiber/epoxy composites as a function of fiber content and woven pattern.



Fig. 4 The impact strength of untreated woven sisal fiber/epoxy composites as a function of fiber content and woven pattern.

From the flexural and impact properties, it can be seen that, for untreated fiber/epoxy composites, the composite of 15% wt untreated harness satin woven sisal fiber and epoxy resin possessed the best properties. Therefore, the composite of 15% wt silane treated harness satin woven fiber and epoxy resin was prepared and further studied.

The flexural properties and impact strength of the composites prepared from 15% wt silane treated harness satin woven sisal fiber are compared with those of untreated fiber/epoxy composite in Fig. 4 and 5 respectively. It was found that the composite containing silane treated fiber did not

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show the difference in flexural strength, flexural modulus and impact strength from the untreated one. This may be because the silane treated sisal fiber did not sufficient to improve fiber/matrix adhesion.





Fig. 5. Variation of (A) flexural strength and (B) flexural modulus of 15% wt. silane treated harness satin woven sisal fiber/epoxy composites

Fig. 6. Variation of impact strength of 15% wt. silane treated harness satin woven sisal fiber/epoxy composites.

Conclusions

The flexural and impact properties of woven sisal fiber/epoxy composites were found to change with change of woven architecture. The composites with harness satin weave sisal fiber showed the best overall properties. The flexural strength, flexural modulus and impact strength of harness satin woven sisal fiber/epoxy composites were not improved after silane treatment.

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BIOGRAPHY

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