

**CHARACTERIZATION AND PROPERTIES OF PALM  
KERNEL SHELL FILLED LOW DENSITY  
POLYETHYLENE BIOCOMPOSITES**

**ROMISUHANI BINTI AHMAD**

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**Characterization and Properties of Palm Kernel Shell  
Filled Low Density Polyethylene Biocomposites**

**By**

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## DECLARATION OF THESIS

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## TABLE OF CONTENTS

	PAGE
THESIS DECLARATION	i
ACKNOWLEDGEMENTS	ii
TABLE OF CONTENTS	iii
LIST OF FIGURES	vii
LIST OF TABLES	xi
LIST OF ABBREVIATIONS, SYMBOLS OR NOMENCLATURES	xii
ABSTRAK	xiv
ABSTRACT	xv
CHAPTER 1. INTRODUCTION	1
1.1 Research Background	1
1.2 Research Objectives	6
CHAPTER 2. LITERATURE REVIEW	7
2.1 Polymer Composites	7
2.2 Matrix	9
2.3 Fillers and Reinforcement	11
2.4 Polyethylene (PE)	12
2.4.1 Application of PE	15
2.4.2 Polyethylene Composites	17
2.4.3 Low Density Polyethylene (LDPE)	18
2.5 Natural Fibres	21
2.5.1 Lignocellulosic	24
2.5.2 Palm Kernel Shell (PKS)	28
2.6 Chemical Modification	29
2.6.1 Compatibilizer	31
2.6.1.1 Polyethylene-co-acrylic acid (PEAA)	32
2.6.2 Coupling Agent	35
2.6.2.1 Acrylic Acid (AA)	36
2.6.2.2 Coconut Oil Coupling Agents (COCA)	38
2.7 Fourier Transform Infrared Spectroscopy (FTIR).	39
2.8 Thermal Properties	40

CHAPTER 3. EXPERIMENTAL	43
3.1 Materials	43
3.1.1 Low Density Polyethylene	43
3.1.2 Filler	43
3.1.3 Filler Treatment	44
3.1.4 Compatibilizer	44
3.1.5. Preparation of Coconut Coupling Agent (COCA)	44
3.1.6 Loss of Ignition Measurement	45
3.2 Compounding of Biocomposites	47
3.2.1 Preparation of LDPE/PKS Biocomposites With Different Filler Loading	47
3.2.2 Preparation of LDPE/PKS Biocomposites With Polyethylene Co-Acrylic Acid (PEAA)	48
3.2.3 Preparation of LDPE/PKS Biocomposites With Acrylic Acid (AA).	48
3.2.4 Preparation of LDPE/PKS Biocomposites With Coconut Coupling Agent (COCA)	49
3.2.5 Compression Molding	50
3.3 Measurement of Mechanical Properties	50
3.3.1 Measurement of Tensile Properties	50
3.3.2 Water Absorption Test	50
3.3.3 Morphology Study	51
3.3.4 Differential Scanning Calorimetry	51
3.3.5 Fourier Transform Infrared Spectroscopy Analysis	52
CHAPTER 4. RESULT AND DISCUSSION	53
4.1 Effect of Palm Kernel Shell Loading on Properties of Low Density Polyethylene/Palm Kernel Shell Biocomposites.	53
4.1.1 Tensile Properties	53

4.1.2	Water Absorption	58
4.1.3	Morphology Study	61
4.1.4	Thermal Properties	63
4.1.4.1	Differential Scanning Calorimetric (DSC)	63
4.2	The Effect of Compatibilizer on Properties of LDPE/PKS Biocomposites	65
4.2.1	Tensile Properties	65
4.2.2	Water Absorption	69
4.2.3	Morphology Study	71
4.2.4	Thermal Properties	72
4.2.4.1	Differential Scanning Calorimetric (DSC)	72
4.2.5	Fourier Transform Infrared Spectroscopy Analysis	74
4.3	The Effect of Acrylic acid on Properties of LDPE/PKS Biocomposites.	76
4.3.1	Tensile Properties	76
4.3.2	Water Absorption	79
4.3.3	Morphology Study	82
4.3.4	Thermal Properties	83
4.3.4.1	Differential Scanning Calorimetric (DSC)	83
4.3.5	Fourier Transform Infrared Spectroscopy Analysis	85
4.4	The Effect of Coconut Coupling Agent (COCA) on Properties of LDPE/PKS Biocomposites.	87
4.4.1	Tensile Properties	87
4.4.2	Water Absorption	90
4.4.3	Morphology Study	92
4.4.4	Thermal Properties	94
4.4.4.1	Differential Scanning calorimetric (DSC)	94
4.4.5	Fourier Transform Infrared Spectroscopy Analysis	96

CHAPTER 5. CONCLUSION AND SUGGESTIONS FOR FURTHER WORK	98
5.1 Conclusion	98
5.2 Suggestions for Further Work	100
<b>REFERENCES</b>	101
<b>APPENDIX A</b>	112
<b>APPENDIX B</b>	113
<b>APPENDIX C</b>	114
<b>APPENDIX D</b>	115
<b>APPENDIX E</b>	116

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## LIST OF FIGURES

NO.		PAGE
2.1	The chemical structure of polyethylene.	13
2.2	Polymerization of polyethylene.	14
2.3	Schematic representation of structures of various Polyethylenes.	15
2.4	Polyethylene Applications.	16
2.5	Molecular structure of cellulose.	26
2.6	Chemical structure of Polyethylene co-acrylic acid (PEAA).	32
2.7	Chemical structure of Acrylic acid.	36
2.8	Schematic representation of the esterification chemical reaction.	37
3.1	The schematic reaction of COCA formation.	45
3.2	Temperature curve with time for loss of ignition of Palm Kernel Shell.	46
4.1	The effect of filler loading on tensile strength of LDPE/PKS Biocomposites.	55
4.2	Scanning electron micrograph of palm kernel shell at magnification 1000X.	55
4.3	The effect of filler loading on elongation of break of LDPE/PKS Biocomposites.	57
4.4	The effect of filler loading on Young's modulus of LDPE/PKS biocomposites.	58
4.5	The percentage of water absorption versus time of LDPE/PKS biocomposites at different filler loading.	60
4.6	The percentage of equilibrium water absorption of LDPE/PKS biocomposites at different filler loading.	60
4.7	SEM micrograph of tensile fracture surface of LDPE at magnification 200X.	61

4.8	SEM micrograph of tensile fracture surface of LDPE/PKS biocomposites (20 php) at magnification 200X.	62
4.9	SEM micrograph of tensile fracture surface of LDPE/PKS biocomposites (40 php) at magnification 200X.	62
4.10	Differential scanning calorimetric (DSC) curve of LDPE/PKS biocomposites at different filler loading.	64
4.11	The effect of filler loading on tensile strength of uncompatibilized and compatibilized LDPE/PKS biocomposites.	66
4.12	The effect of filler loading on elongation at break of uncompatibilized and compatibilized LDPE/PKS biocomposites.	67
4.13	The effect of filler loading on Young's modulus of uncompatibilized and compatibilized LDPE/PKS biocomposites.	68
4.14	The comparison of water absorption of uncompatibilized and compatibilized LDPE/PKS biocomposites at 0, 20, and 40 php.	70
4.15	The percentage of equilibrium water absorption of uncompatibilized and compatibilized LDPE/PKS biocomposites at different filler loading.	70
4.16	SEM micrograph of tensile fracture surface of compatibilized LDPE/PKS biocomposites with PEAA (20 php) at 200X magnification.	71
4.17	SEM micrograph of tensile fracture surface of compatibilized LDPE/PKS biocomposites with PEAA (40 php) at 200X magnification.	72
4.18	Differential scanning calorimetric curve of uncompatibilized and compatibilized LDPE/PKS biocomposites at different filler loading.	73

4.19	FTIR spectra of compatibilized and uncompatibilized LDPE/PKS biocomposites.	75
4.20	The schematic reaction mechanism between PKS and PEEA.	75
4.21	The effect of filler loading on tensile strength of untreated and treated of LDPE/PKS biocomposites.	76
4.22	The effect of filler loading on elongation at breaks of untreated and treated of LDPE/PKS biocomposites.	78
4.23	The effect of filler loading on Young's modulus of untreated and treated LDPE/PKS biocomposites.	79
4.24	The percentage of water absorption versus time of untreated and treated LDPE/PKS biocomposites at 0, 20, and 40 php.	81
4.25	The percentage of equilibrium water absorption for nuntreated and treated LDPE/PKS biocomposites at different filler loading.	81
4.26	SEM micrograph of tensile fracture surface of treated LDPE/PKS biocomposites (20 php) at 200X magnification.	82
4.27	SEM micrograph of tensile fracture surface of treated LDPE/PKS biocomposites (40 php) at 200X magnification.	83
4.28	Differential scanning calorimetric (DSC) curve of untreated and treated LDPE/PKS biocomposites at different filler loading.	84
4.29	FTIR spectra of untreated and treated LDPE/PKS biocomposites.	86
4.30	The schematic reaction mechanism between PKS and acrylic acid.	86
4.31	The effect of filler loading on tensile strength of LDPE/PKS biocomposites with and without COCA.	88
4.32	The effect of filler loading on elongation at break of LDPE/PKS biocomposites with and without COCA.	89

4.33	The effect of filler loading on Young's modulus of LDPE/PKS biocomposites with and without COCA.	90
4.34	The comparison of water absorption between LDPE/PKS biocomposites with and without COCA at 0, 20, and 40 php.	91
4.35	The percentage of equilibrium water absorption for LDPE/PKS biocomposites with and without COCA at different filler loading.	92
4.36	SEM micrograph of tensile fracture surface of LDPE/PKS biocomposites with COCA (20 php) at 200X magnification.	93
4.37	SEM micrograph of tensile fracture surface of LDPE/PKS biocomposites with COCA (40 php) at 200X magnification.	93
4.38	Differential scanning calorimetric (DSC) curve of LDPE/PKS biocomposites with and without COCA at different filler loading.	95
4.39	FTIR spectra of LDPE/PKS biocomposites with and without COCA.	96
4.40	The schematic reaction mechanism between PKS and COCA.	97

## LIST OF TABLES

NO.		PAGE
2.1	Physical properties of the Palm kernel shells.	29
2.2	Properties of polyethylene co-acrylic acid (PEAA).	33
2.3	Properties of Acrylic acid	37
2.4	The composition of virgin coconut oil.	39
3.1	Properties of low density polyethylene (LDPE).	43
3.2	The organic and inorganic content in PKS.	47
3.3	Formulation of LDPE/PKS bicomposites with different filler loading.	47
3.4	Formulation of uncompatibilized and compatibilized LDPE/PKS biocomposites with PEAA.	48
3.5	Formulation of untreated and treated LDPE/PKS biocomposites with AA.	49
3.6	Formulation for LDPE/PKS biocomposites with and without COCA.	49
4.1	Parameter DSC analysis of LDPE/PKS biocomposites at different filler loading.	64
4.2	Parameter DSC analysis of uncompatibilized and compatibilized LDPE/PKS biocomposites at different filler loading.	74
4.3	Parameter DSC analysis of untreated and treated LDPE/PKS biocomposites at different filler loading.	85
4.4	Parameter DSC analysis of LDPE/PKS biocomposites with and without COCA at different filler loading.	95

## LIST OF SYMBOLS, ABBREVIATIONS OR NOMENCLATURES

AA	Acrylic acid
ASTM	American Society for Testing and Materials
CAPE	Carboxylated polyethylene
COCA	Coconut Coupling Agent
DSC	Differential scanning calorimetric
DTA	Differential thermal analysis
EAA	Ethylene-acrylic acid
EBAGMA	Ethylene-butyl acrylate-glycidyl metacrylate
EDD	Ethylene diamine dilaurate
EFB	Empty fruit bunch
FTIR	Fourier transform infrared spectroscopy
HDPE	High density polyethylene
HDS	Hexadecyltrimethoxy-silane
HPLC	High performance liquid chromatography
LDPE	Low density polyethylene
LLDPE	Linear low density polyethylene
MAPE	Maleated polyethylene
MAPP	Maleated polypropylene
MPS	Methacryloxypropyltrimethoxy
MRPS	$\gamma$ -mercaptoproxytrimethoxy
Mt	Percentage of water absorption
OHF	Olive husk flour
PALF	Pineapple-leaf fiber
PEAA	Polyethylene co-acrylic acid
PLA	Poly (lactic acid)
PPEAA	Poly (propylene-ethylene acrylic acid)
PKS	Palm kernel shell
POFA	Palm oil fatty acid
PP	Polypropylene
PS	Polystyrene
PVC	Poly (vinyl chloride)

RHDPE	Recycled high density polyethylene
SEM	Scanning electron microscopy
$T_c$	Crystallization
TDM	Titanium-derived mixture
$T_g$	Glass transition temperature
TGA	Thermogravimetric analysis
$T_m$	Melting point
TS	Tensile strength
Wd	Original dry weight
Wn	Weight after immersed
WOWP	Waste office white paper
WP	Waste paper
$X_{biocom}$	Crystallinity of biocomposites

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**PENCIRIAN DAN SIFAT-SIFAT BIOKOMPOSIT TEMPURUNG KELAPA  
SAWIT (PKS) TERISI POLIETILENA BERKETUMPATAN RENDAH (LDPE).**

**ABSTRAK**

Biokomposit berdasarkan tempurung kelapa sawit (PKS) dan polietilena berketumpatan rendah (LDPE) telah dikaji. Biokomposit disediakan dengan menggunakan pencampur bilah-Z pada 180°C dengan kelajuan rotor 50rpm. Kesan pembebanan pengisi tempurung kelapa sawit (PKS) terisi di dalam LDPE keatas sifat-sifat mekanikal, penyerapan air, morfologi, sifat terma dan spektroskopi inframerah transformasi fourier (FTIR) telah dikaji. Keputusan menunjukkan bahawa peningkatan pembebanan PKS menyebabkan kekuatan tensil dan pemanjangan pada takat putus berkurang tetapi modulus Young dan penyerapan air didapati meningkat. Kajian morfologi menggunakan mikroskop elektron pengesanan (SEM) menunjukkan bahawa pembebanan pengisi yang tinggi menyebabkan interaksi antara muka adalah lemah antara PKS dan LDPE. Penghabluran biokomposit meningkat dengan semakin meningkatnya pembebanan PKS. Untuk meningkatkan sifat-sifat mekanikal biokomposit, polietilena ko-akrilik asid (PEAA) digunakan sebagai bahan pengserasi. Kesan modifikasi kimia ke atas biokomposit dengan polietilena ko-akrilik asid (PEAA), asid akrilik (AA) dan agen pengkupel kelapa (COCA) telah meningkatkan kekuatan tensil, modulus Young dan penghabluran tetapi mengurangkan pemanjangan takat putus dan penyerapan air. Kajian mikroskop elektron pengesanan (SEM) juga menunjukkan bahawa interaksi antara pengisi-matrik didapati meningkat dengan kehadiran PEAA, AA dan COCA. Keputusan FTIR menunjukkan nombor gelombang kumpulan hidroksil untuk kesemua biokomposit semakin berkurang yang mana menunjukkan bahawa interaksi diantara kumpulan hidroksil daripada PKS dengan bahan pengserasi dan agen pengkupel.



**CHARACTERIZATION AND PROPERTIES OF PALM KERNEL SHELL  
(PKS) FILLED LOW DENSITY POLYETHYLENE (LDPE) BIOCOMPOSITES.**

**ABSTRACT**

Biocomposites based on palm kernel shell (PKS) and low density polyethylene (LDPE) was investigated. The biocomposites were prepared by using Z-Blade mixer at processing temperature 180 °C and rotor speed 50 rpm. The effect of filler loading of PKS as filler in LDPE on mechanical properties, water absorption, morphology, thermal properties and fourier tranformation infrared spectroscopy (FTIR) were studied. The results show that the increasing of filler loading have decreased the tensile strength and elongation at break but increased the Young's modulus and water absorption. The morphology study using scanning electron microscopy (SEM) shows poor interfacial interaction between PKS and LDPE with increasing of the filler loading. The crystallinity of the biocomposites increased with increasing of PKS loading. To improve the mechanical properties, compatibilizer, Polyethylene co-acrylic acid (PEAA) was used. The effect of chemical modification of biocomposites with polyethylene co-acrylic acid (PEAA), acrylic acid (AA) and coconut coupling agent (COCA) enhanced the tensile strength, Young's modulus and crystallinity of the biocomposites but reduced the elongation at break and water absorption. The morphology study (SEM) shows that the filler-matrix interaction was improved with incorporation of PEAA, AA and COCA. The FTIR results show that the wave number of hydroxyl group for all biocomposites shifted to lower wave number which indicates that interaction between the hydroxyl groups from PKS with compatibilizer and coupling agent.

# CHAPTER 1

## INTRODUCTION

### 1.1 Research Background

A biocomposite are composite materials comprising one or more phases derived from abiological origin. In terms of the reinforcement, this could include plant fibres such as cotton, flax, hemp, and the likes or fibres from recycled wood or waste paper, or even by-products from food crops. Regenerated cellulose fibres (viscose/rayon) are also included in this definition, since ultimately, they too come from a renewable resources (Paul et al., 2006). Biocomposites from plant and wood based fibers are used in a wide range of products, including aerospace, automotive, and building materials. Using biomass fibers to reinforce plastics has several advantages over synthetic ones. They offer environmental benefits because of their renewable nature and low energy consumption in production (Maya & Sabu, 2008).

© Biocomposites are obtained by the combination of biodegradable polymer as the matrix material and biodegradable fillers (e.g., lignocellulosic fillers). Since both components are biodegradable, the composite as the integral part is also expected to be biodegradable (Ave´rous et al., 2006). For short-term applications, biocomposites present strong advantages, and many number of papers have been published on this topic (Barreto et al., 2010; Fransisco et al., 2010; Ave´rous et al., 2006).

Currently, the main market for biocomposites are in the automotive and construction sectors which further development and improvement are still taking places. However, new opportunities and application will likely arise. Significant opportunities are likely to occur in the built environment at this sector which responsible for producing huge volumes of waste at a time. This environmental impact of industries is coming under close scrutiny.

The specific mechanical properties of natural fibres are comparable to those of traditional reinforcement. Thus, the intrinsic properties of natural fibres can satisfy the request of the global market especially for those industries concerned in weight reduction (i.e., automotive). That is why they can be a potential substitute for non-renewable synthetic fibres.

Natural fibres are subdivided based on their origins, coming from plants, animals or minerals. All plant fibres are composed of cellulose while animal fibres consist of proteins (hair, silk, and wool). Plant fibres include bast (or stem or soft sclerenchyma) fibres, leaf or hard fibres, seed, fruit, wood, cereal straw, and other grass fibre. Over the last few years, a number of researchers have been involved in investigating the exploitation of natural fibres as load bearing constituents in composite materials. Natural organic fibres from renewable natural resources often the potential to act as a biodegradable reinforcing materials alternative for the use of glass or carbon fibre and inorganic fillers. These fibres often several advantage including high specific strength and modulus, low cost, low density, renewable nature, biodegradability, absence of associate health hazards, easy fibre surface modification, wide availability, and relative non – abrasiveness (Al-Kaabi et al., 2005; Bledzki & Gassan, 1999; Thwe & Liao, 2002; Kim et al., 2004).

Many researches have been done on virgin thermoplastic and natural fibre composites, which have successfully proven their applicability to various fields of technical application, especially for load-bearing application. Thermoplastics have been compounded with natural fibres such as polyethylene (PE) with flax fibre (Foulk et al., 2004), PE with henequen (Herrera-Franco et al., 2005), PE with wood flour (Bengtsson & Oksman, 2006), PE with curaua (Araujo et al., 2008), polypropylene (PP) with kenaf (Tajvidi, 2005), PP with jute fibre (Cabral et al., 2005; Li & Sain, 2005), poly (vinyl chloride) (PVC) with wood/flour (Guffey & Sabbagh, 2002), PVC with baggase fibre (Zheng et al., 2007), polystyrene (PS) with sisal fibre (Antich et al., 2006), and poly (lactic acid) with flax fibre (Oksmana et al., 2003), PLA with abaca fibre (Bledzki et al., 2009).

The interest in cellulose fibers as reinforcement agents in composite materials with polymer matrices has increased dramatically in the last decade, as detailed in recent reviews (Aparecido et al., 2009). Lignocellulosic materials, which predominantly consist of cellulose, lignin and hemicelluloses, in the production of the plastic composites has gained momentum in recent years which have stimulated much interest in the manufacture of composite during the past decade, i.e. used as filler material instead of conventional filler such as mica, clay and glass fiber.

© Palm Kernel Shell is one of the lignocellulosic biofibres derived from waste palm kernel oil. Palm Kernel Shell is an agricultural by-product from palm oil mills, and it is one of the main agricultural wastes in Malaysia. Palm Kernel Shell can be utilized to a great extend as construction material. At present, it uses is limited mostly to fuel for burning and as finishes traditional mud houses. There is a great interest to find some value-added applications for Palm Kernel Shell.

Low-density polyethylene (LDPE) is important thermoplastic due to their having a good combination of properties, such as fluidity, flexibility, transparency and a glossy surface. LDPE is used mainly as a food packing material, sheet and film.

However, the most important issue associated with these composites is the interfacial adhesions between the natural reinforcing fillers and matrix polymers. The compatibility problem may be due to the fact that the polyolefin is non-polar and hydrophobic, whereas the natural polymer, which is a lignocellulosic material, is polar due to the –OH groups in the cellulose. This results in poor adhesion and prevents the reinforcing filler from acting effectively in the composite. The good properties of these composites can be obtained by improving the compatibility between these two materials. In order to solve these problems, studies have been performed on surface modification or treatment of filler using a compatibilizing agent or coupling agent in order to reduce the hydrophilicity of the filler.

Cellulosic fillers can be modified by physical and chemical methods. Extensive studies are currently being performed on polyolefins (polypropylene and polyethylene) and various natural reinforcing fillers, in conjunction with various chemicals that could affect the interface. Among various ways of chemical modification for polyolefin/natural fibre composites were used such as effect of crosslinking on polyethylene (Cousin et al., 1989; Benedetto et al., 1989), effect of silane coupling agent (Raj et al., 1989a; Raj et al., 1990; Raj et al., 1989b), and the effect of different coupling agent using poly[methylene (polyphenyl isocyanate)] and silanes (A-172, A-174, A-1100) (Maldas et al., 1989). The functionalized polyolefins are most often tested because of their efficiency and commercial availability.

The improvement in the mechanical properties of the composites can be achieved by increasing the filler volume fraction up to the certain degree of filler loading depending on the packing arrangement of the filler. The filler matrix interaction may be improved by making chemical or physical modification to the filler. Chemical modification of natural filler such as acrylation, silylation and other treatments reduce and other treatment reduces their moisture sensitivity. Much remain to be done to change or modify and improve bulk and surface characteristics in order to improve composites properties. The strong interfacial bonding strength obtained by improving the compatibility between the hydrophilic filler and hydrophobic matrix polymer can improve the physical, mechanical and thermal properties of the composite system (Kim, 2008).

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## 1.2 Research Objectives

The prime objective of this study is to determine the properties and characterizations of palm kernel shells (PKS) filled low density polyethylene (LDPE) on a different composition. Several different compositions of LDPE/PKS biocomposites were prepared in order to study:

1. To study the effect of palm kernel shell loading on mechanical properties, water absorption, morphology, and thermal properties of LDPE/PKS biocomposites
2. To study the effect of compatibilizer (PEAA) on properties of LDPE/PKS biocomposites
3. To study the effect of chemical modification (acrylic acid) on properties of LDPE/PKS biocomposites
4. To study the effect of coconut coupling agent on properties of LDPE/PKS biocomposites

The testing of LDPE/PKS biocomposites has been done by tensile test, water absorption, morphology, thermal analysis using differential scanning calorimetry (DSC), and fourier transform infrared spectroscopy (FTIR) Analysis.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Polymer Composites

Composites materials have been utilized to solve technological problems for a long time and these materials start capturing the attention in industries with the introduction of polymeric-based composites. Since then, composites materials have become common engineering materials and are designed and manufacturing for various application including automotive components, sporting goods, aerospace parts, consumer goods, and in the marine and oil industries. As early as 1908, the first composite materials were applied for the fabrication of large quantities of sheets, tubes and pipes for electronic purposes (paper or cotton to reinforce sheets, made of phenol- or melamine-formaldehyde resins). In 1896 for example, aeroplane seats and fuel-tanks were made of natural fibers with a small content of polymeric binders (Bledzki & Gassan, 1999).

The growth in composite usage also came about because of increase awareness regarding product performance and increase competition in the global market for lightweight components. Today, it appears that composites are materials of choice for many engineering applications. Daniel et al., (2003) has defined composites as an engineering material that made from a mixture of two or more constituent material. There are two categories of constituent materials which are matrix and reinforcement. At least one portion of each type is required. The matrix material surrounds and