



UNIVERSITI PUTRA MALAYSIA

**PREPARATION AND CHARACTERIZATION OF KENAF
CELLULOSEPOLYETHYLENE GLYCOL- POLYETHYLENE
BIOCOMPOSITES**

BEHJAT TAJEDDIN

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**PREPARATION AND CHARACTERIZATION OF KENAF CELLULOSE-
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By

BEHJAT TAJEDDIN

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

November 2009



THIS WORK IS DEDICATED

TO

My HUSBAND (MAHMOOD),
MY LOVELY CHILDREN (MINA & BORNA),

MY FATHER & THE SOUL OF MY MOTHER
WHO SENT ME TO SCHOOL AND ENCOURAGED ME TO FULFILL MY
DREAMS,

AND

MY KIND SISTERS & BROTHERS



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in
Fulfilment of the Requirement for the Degree of Doctor of Philosophy

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Chairman: Professor Russly Abdul Rahman, PhD

Faculty: Engineering

The possibility of using cellulose as natural fiber for the production of bicomposites was investigated in this study that included two stages. The first stage involved the extraction of cellulose from the cell walls of kenaf (*Hibiscus cannabinus* L.), an annual herbaceous crop with many environmental advantages and good mechanical properties. It was done from the bast part of the crop by chemical treatments. Then, mixture of different weights of low density polyethylene (LDPE) and high density polyethylene (HDPE), as a matrix, with the obtained cellulose, and polyethylene glycol (PEG) were blended in order to produce a biocomposite material suitable for food packaging.

For the second stage, the characterization of LDPE- and HDPE-kenaf cellulose bicomposites was performed in order to develop the optimal blends with optimized thermo-mechanical properties and propensity to environmental degradation. Therefore, the mechanical properties including



tensile strength, flexural and unnotched Izod Impact tests were performed using Instron Universal Testing Machine and Izod Impact Tester, respectively. Thermal properties, biodegradability and water absorption of biocomposites were investigated as well. In addition, a scanning electron microscope (SEM) was used to observe the surface morphology of the tensile fracture surface of the samples before and after biodegradation test.

The results showed that the mechanical properties of the LDPE and HDPE-cellulose composites decreased slightly as the cellulose content increased from 0 to 50 wt % in the biocomposite formulation. It is interesting to note that in all treatments, the mechanical behavior of biocomposites retained in an acceptable level of strength except of HDPE composites with 50% cellulose. In general, there is a good homogeneity between samples with PEG that help to find reasonable and acceptable properties. These findings were confirmed by the SEM study.

Thermal analysis of composites is necessary for determining their end use. Therefore, thermal properties of biocomposites were obtained by a thermogravimetry analysis (TGA) and a differential scanning calorimetry (DSC). Addition of cellulose fillers improves the thermal resistance of these biocomposites. The results also showed that PEG has positive role in thermal behavior of composites. This finding gives a good indication that the addition of kenaf cellulose into the body of LDPE and HDPE was capable to increase their thermal degradation properties.

Biodegradability of these biocomposites was performed based on soil burial test to investigate their degradation during 120 days. The findings illustrated

that there is a clear trend of degradation during burial time. The degradability increased as cellulose content was raised in the composite's formulation.

Finally, water absorption was done for biocomposites. The results showed that water absorption value for both composites was higher than those of LDPE and HDPE polymers. Addition of PEG to the formulations reduced the water absorption of the composites.

Generally, it seems that the results of this research may lead to a development of a new type of biocomposites using kenaf cellulose as a natural fiber that can be used to replace plastics for food packaging in the near future.

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

**PENYEDIAAN DAN PENCIRIAN SELULOSA KENAF- POLIETILENA
GLYKOLA- POLIETILENA BIOKOMPOSIT**

Oleh

BEHJAT TAJEDDIN

November 2009

Pengerusi: Professor Russly Abdul Rahman, PhD

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Kajian terhadap selulosa sebagai gentian semula jadi bagi penghasilan biokomposit melibatkan dua peringkat. Peringkat pertama adalah pengekstrakan selulosa daripada dinding sel *bast* pokok kenaf (*Hibiscus cannabinus* L.) melalui kaedah rawatan kimia. Kenaf merupakan sejenis tanaman herba tahunan yang mempunyai sifat mekanikal yang baik disamping memberi kebaikan kepada alam sekitar. Selulosa yang telah diekstrak kemudiannya dicampurkan bersama polimer polietilena berketumpatan tinggi dan rendah iaitu HDPE dan LDPE pada berat yang berbeza-beza dimana ia berfungsi sebagai matrik polimer. Manakala, polietilena glikol (PEG) sebagai bahan pemplastik turut dicampurkan bagi menghasilkan bahan biokomposit yang sesuai digunakan dalam pembungkusan makanan.

Pada peringkat kedua, kajian dijalankan terhadap sifat-sifat biokomposit HDPE dan LDPE-ekstrak selulosa bagi mencapai campuran yang optimum

dengan mengambil kira sifat-sifat mekanikal dan terma serta kecenderungan degradasi terhadap persekitaran. Sifat mekanikal seperti kekuatan tegangan, lenturan dan hentaman dianalisis dengan menggunakan alat Instron Universal Testing Machine dan alat Izod Impact Tester. Selain itu, sifat-sifat terma, biodegradasi dan penyerapan air oleh biokomposit turut dianalisis. Kajian morfologi di atas permukaan tegangan patah sebelum dan selepas degradasi dikaji dengan menggunakan pengimbas elektron mikroskopi (SEM).

Keputusan menunjukkan dengan meningkatnya penambahan ekstrak selulosa dari 0 hingga 50 (% berat) ke atas polimer mengurangkan sifat-sifat mekanikal biokomposit selulosa LDPE dan HDPE. Bagaimanapun sifat-sifat mekanikal biokomposit adalah kekal pada tahap kekuatan yang boleh diterima kecuali biokomposit dengan campuran 50 % ekstrak selulosa. Ini adalah kerana berdasarkan pengimbas elektron mikroskopi campuran yang sekata dan baik diantara selulosa, polimer dan pemplastikan PEG dikenalpasti.

Analisis termal terhadap biokomposit adalah perlu bagi menentukan kegunaan akhir dan sifat-sifat termal biokomposit ini diperolehi dengan menggunakan analisis thermogravimetri (TGA) dan kalorimeter pengimbas separa (DSC). Berdasarkan keputusan yang diperolehi didapati penambahan selulosa meningkatkan rintangan terma biokomposit dan PEG menunjukkan peranan yang positif di dalam sifat terma biokomposit. Penemuan ini memberi petanda yang baik dimana penambahan selulosa di dalam matrik polimer HDPE dan LDPE meningkatkan sifat-sifat degradasi terma.

Proses biodegradasi dijalankan berdasarkan ujian penanaman ke dalam tanah untuk menganalisis degradasi biokomposit dengan mengambil masa selama 120 hari. Penemuan yang diperolehi menggambarkan aliran dengan jelas terhadap degradasi sepanjang masa penanaman dan didapati penambahan selulosa di dalam biokomposit meningkatkan lagi proses degradasi.

Selain itu, berdasarkan ujian penyerapan air oleh biokomposit didapati nilai penyerapan air bagi kedua-dua biokomposit adalah lebih tinggi berbanding polimer HDPE dan LDPE tulen. Penambahan PEG di dalam formulasi biokomposit dikenalpasti dapat mengurangkan penyerapan air.

Secara umumnya, keputusan yang diperolehi daripada penyelidikan ini boleh membawa kepada pembangunan penghasilan biokomposit baru iaitu dengan menggunakan selulosa daripada kenaf sebagai gantian semula jadi yang boleh dijadikan bahan gantian plastik bagi pembungkusan makanan pada masa hadapan.

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I certify that a Thesis Examination Committee has met on 2 November 2009 to conduct the final examination of Behjat Tajeddin on her thesis entitled "Preparation and Characterization of Kenaf Cellulose- Polyethylene Glycol- Polyethylene Biocomposites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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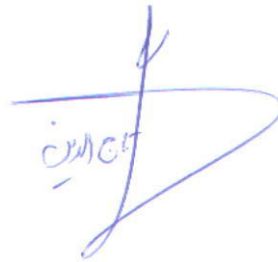
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Date: 14 January 2010



DECLARATION

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

A handwritten signature in blue ink, appearing to be 'Behjat Tajeddin', written over a horizontal line.

Behjat Tajeddin

Date: 13 August 2009

TABLE OF CONTENTS

ABSTRACT	iii
ABSTRAK	vi
ACNOWLEDGMENTS	ix
APPROVAL	x
DECLARATION	xii
TABLE OF CONTENTS	xiii
LIST OF TABLES	xvi
LIST OF FIGURERS	xvii
LIST OF ABBREVIATIONS	xx
CHAPTER	
1 INTRODUCTION	1
1.1 BACKGROUND OF STUDY	1
1.2 RESEARCH OBJECTIVES	5
1.3 THESIS STRUCTURE	6
2 LITERATURE REVIEW	8
2.1 POLYMER MATRIX	8
2.2 BIOCOMPOSITES	12
2.3 BIODEGRADABLE POLYMERS	14
2.4 NATURAL PLANT FIBERS	16
2.4.1 <i>Kenaf</i>	19
2.4.2 <i>Cellulose</i>	21
2.4.3 <i>Justification for the use of cellulose from kenaf</i>	24
2.4.4 <i>Methods of cellulose extraction</i>	25
2.5 PLASTICIZERS	26
2.5.1 <i>Compatibility</i>	28
2.5.2 <i>Processability</i>	28
2.5.3 <i>Polyethylene Glycol (PEG)</i>	28
2.6 CELLULOSE COMPOSITE	30
2.7 MODIFICATION METHODS	31
2.8 COUPLING AGENTS	33
2.9 CHARACTERIZATION AND TESTING OF BIOCOMPOSITES	34
2.9.1 <i>Mechanical Characteristics</i>	34
2.9.2 <i>Scanning Electron Microscopy (SEM)</i>	38
2.9.3 <i>Thermal</i>	38
2.9.4 <i>Biodegradation of Biocomposites</i>	45
2.9.5 <i>Water Absorption</i>	49
2.10 APPLICATION OF BIOCOMPOSITES IN FOOD PACKAGING	51
2.11 SUMMARY	53
3 MATERIALS AND METHODS	54
3.1 MATERIALS	54
3.1.1 <i>Kenaf</i>	54
3.1.2 <i>Chemical materials</i>	54
3.1.3 <i>Polyethylene Glycol (PEG)</i>	55
3.1.4 <i>LDPE and HDPE</i>	55
3.2 SPECIMENS PREPARATION	56
3.2.1 <i>Preparation of Kenaf Fiber</i>	56



3.2.2	<i>Cellulose Processing from Kenaf Fibre</i>	58
3.2.3	<i>Melt Blending</i>	60
3.2.4	<i>Compression- Molding</i>	63
3.3	SPECIMENS FOR TESTING	64
3.4	STATISTICAL SUBJECTS	64
3.5	OVERALL EXPERIMENTAL PROCEDURE	65
4	MECHANICAL PROPERTIES OF PE-KC BIOCOSITES	66
4.1	INTRODUCTION	66
4.2	MATERIALS AND METHODS	68
4.2.1	<i>Materials</i>	68
4.2.2	<i>Methods</i>	68
4.2.3	<i>Surface Morphology Study</i>	73
4.3	RESULTS AND DISCUSSION	73
4.3.1	<i>Tensile Test</i>	73
4.3.2	<i>Izod Impact Test</i>	86
4.3.3	<i>Flexural Test</i>	96
4.4	CONCLUSION	101
5	THERMAL PROPERTIES OF PE-KC BIOCOSITES	104
5.1	INTRODUCTION	104
5.2	MATERIALS AND METHODS	106
5.2.1	<i>Thermogravimetric Analysis (TGA)</i>	106
5.2.2	<i>Differential Scanning Calorimetry (DSC)</i>	106
5.3	RESULTS AND DISCUSSION	106
5.3.1	<i>TGA for LDPE-KC composites</i>	106
5.3.2	<i>TGA for HDPE-KC composites</i>	110
5.3.3	<i>DSC for LDPE-KC Composites</i>	114
5.3.4	<i>DSC for HDPE-KC Composites</i>	116
5.4	CONCLUSION	118
6	BIODEGRADABILITY OF PE-KC BIOCOSITES	120
6.1	INTRODUCTION	120
6.2	MATERIALS AND METHODS	122
6.2.1	<i>Materials</i>	122
6.2.2	<i>Methods</i>	123
6.3	RESULTS AND DISCUSSION	125
6.3.1	<i>Biodegradability of LDPE-KC Composites</i>	125
6.3.2	<i>Biodegradability of HDPE-KC composites</i>	129
6.3.3	<i>Tensile test after biodegradation of PE composites</i>	132
6.3.4	<i>SEM study after biodegradation of PE-KC composites</i>	134
6.4	CONCLUSION	135
7	WATER ABSORPTION BEHAVIOR OF PE-KC BIOCOSITES	137
7.1	INTRODUCTION	137
7.2	MATERIALS AND METHODS	140
7.3	RESULTS AND DISCUSSION	141
7.4	CONCLUSION	145

8	SUMMARY, GENERAL CONCLUSION AND RECOMMENDATIONS FOR FUTURE RESEARCH	147
	REFERENCES	153
	APPENDICES	164
	BIODATA OF THE STUDENT	172
	LIST OF PUBLICATIONS	173



LIST OF TABLES

Table	Page
2-1. SOME COMMERCIALY IMPORTANT PROPERTIES OF PE	12
2-2. CHEMICAL COMPOSITION OF SOME COMMON NATURAL VEGETABLE FIBERS	18
2-3. CHEMICAL CONTENT OF KENAF STEM (% OF TOTAL)	21
2-4. CRYSTALLINITY PERCENT OF COMMON POLYMERS	39
3-1. DATA SHEET OF LDPE AND HDPE POLYMERS	55
3-2. THERMO HAAKE POLYDRIVE CONDITION	60
3-3. FORMULATION OF LDPE- KC- PEG COMPOSITES	61
3-4. FORMULATION OF HDPE- KC- PEG COMPOSITES	62
3-5. CONDITION OF COMPRESSION MOLDING FOR LDPE AND HDPE SAMPLES	64
5-1. DSC DATA FOR LDPE-KC COMPOSITES	115
5-2. DSC DATA FOR HDPE-KC COMPOSITES	117
6-1. TS FOR ALL BIOCOMPOSITES BEFORE AND AFTER BIODEGRADATION TEST	133

LIST OF FIGURES

Figure	Page
2-1. A CLASSIFICATION OF POLYMERS (COWIE, 2008)	9
2-2. MOLECULAR STRUCTURE OF LDPE (A), HDPE (B) AND CROSS-LINKED (C); (HANLON ET AL., 1998)	11
2-3. CLASSIFICATION OF BIOBASED COMPOSITES (MOHANTY ET AL., 2005)	13
2-4. CLASSIFICATION OF NATURAL FIBERS (NISHINO, 2004; SUDDLELL & EVANS, 2005)	17
2-5. EXAMPLES OF NATURAL VEGETABLE FIBERS (NISHINO, 2004; SUDDLELL & EVANS, 2005)	17
2-6. KENAF PLANT (SOURCE OF PAINTED PHOTO: VISIONPAPER, 2005)	21
2-7. STRUCTURE OF CELLULOSE (HERRERA ET AL., 2005; NISHINO, 2004)	22
2-8. MECHANISM OF COMPATIBILIZATION OF MAPP OR MAPE (ROWELL, 2006)	33
2-9. MODULUS-TEMPERATURE CURVE OF AN AMORPHOUS POLYMER, POLYCARBONATE (BARRY & ORROTH, 2000)	40
2-10. MODULUS-TEMPERATURE CURVE OF A SEMI CRYSTALLINE POLYMER, POLYPROPYLENE (BARRY & ORROTH, 2000)	40
2-11. AN EXAMPLE OF TG CURVE (EHRENSTEIN ET AL., 2004); T_A = ONSET TEMPERATURE/TIME, T_B = END TEMPERATURE/TIME, T_C = MIDPOINT TEMPERATURE/TIME, M_S = STARTING MASS, M_F = FINAL MASS, M_I = MIDPOINT BETWEEN M_{B1} AND M_{A2} .	41
2-12. AN EXAMPLE OF A TWO-STEP CHANGE OF MASS (EHRENSTEIN ET AL., 2004); $M_{L1}=M_{L2}$ =LOSS OF MASS AND $T_{P1}=T_{P2}$ =PEAK TEMPERATURE ON THE DTG CURVE.	42
2-13. SCHEMATIC DIAGRAM OF A DSC CURVE (EHRENSTEIN ET AL., 2004)	44
2-14. BIODEGRADABILITY TESTING OF SOLID POLYMERS (ITAVAARA & VIKMAN, AS CITED IN SRIDACH ET AL., 2006)	48
2-15. SCHEMATIC PRESENTATION OF BIOBASED POLYMERS BASED ON THEIR ORIGIN AND METHOD OF PRODUCTION FOR FOOD PACKAGING (WEBER, 2000)	52
3-1. SEPARATING THE BAST FROM CORE IN KENAF STALK	56
3-2. CUTTING THE KENAF BASTS INTO SMALL PIECES AFTER DRYING	56
3-3. LABORATORY MILL FOR GRINDING OF KENAF BAST	57
3-4. SIEVING THE SAMPLES BY SHAKER AND PASS 40 MESH (0.40 MM)	57
3-5. PROCEDURE OF CELLULOSE PROCESSING (HAN & ROWELL, 1996)	58
3-6. KC UNDER VACCUM PACKAGING	59
3-7. THERMO HAAKE POLYDRIVE R600/610 BLENDING MACHINE WITH RHEOMIX	60
3-8. HOT PRESS MACHINE	63
3-9. THE STUDY PLAN OF THE THESIS	65
3-10. OVERAL EXPERIMENTAL DESIGN FOR THE RESEARCH	65
4-1. DUMBBELL-SHAPED CUTTER	69
4-2. MEASURING DIMENSIONS OF SEVEN SPECIMENS FOR EACH COMPOSITION	69
4-3. INSTRON UNIVERSAL TESTING MACHINE FOR TENSILE TEST	70
4-4. BAND SAW MACHINE	71
4-5. IZOD/CHARPY IMPACT TESTER	72
4-6. GOLD COATED SAMPLES READY FOR SCANNING BY PHILIPS XL 30 SEM	73



4-7. EFFECT OF KC ON TENSILE STRENGTH OF LDPE-KC COMPOSITES. THE DATA ARE MEAN OF 7 REPLICATIONS OF EACH TREATMENT.	74
4-8. EFFECT OF PEG ON TENSILE STRENGTH OF LDPE- KC COMPOSITES	76
4-9. SEM (MAG. × 200) FOR TENSILE FRACTURE SURFACE OF LDPE-KC COMPOSITES AT DIFFERENT CONCENTRATIONS: A) 70:30, B) 60:40, AND C) 50:50	78
4-10. SEM FOR TENSILE FRACTURE SURFACE OF LDPE-KC-PEG COMPOSITES; A) 30%KC+7%PEG (MAG. × 101) AND B) 50%KC+7%PEG (MAG. × 200).	79
4-11. EFFECT OF KC LOADING ON TENSILE STRENGTH OF HDPE- KC BIOCOMPOSITES	81
4-12. EFFECT OF PEG ON TENSILE STRENGTH OF HDPE- KC COMPOSITES	82
4-13. SEM (MAG. × 300) OF THE TENSILE FRACTURE SURFACE OF HDPE-KC COMPOSITES AT DIFFERENT CONCENTRATIONS: A) 70:30, B) 60:40, AND C) 50:50.	84
4-14. SEM FOR TENSILE FRACTURE SURFACE OF HDPE-KC-PEG COMPOSITES; A) 30%KC+5%PEG (MAG. × 300) AND B) 40%KC+5%PEG (MAG. × 200).	85
4-15. EFFECT OF KC ON UNNOTCHED IMPACT STRENGTH OF LDPE-KC COMPOSITES	87
4-16. EFFECT OF PEG ON UNNOTCHED IMPACT STRENGTH OF LDPE- KC COMPOSITES	88
4-17. SEM FOR IMPACT FRACTURE SURFACE OF LDPE-KC COMPOSITES AT DIFFERENT CONCENTRATIONS: A) 70:30, B) 60:40, AND C) 50:50	90
4-18. SEM FOR IMPACT FRACTURE SURFACE OF LDPE-KC-PEG COMPOSITES; A) 40%KC+7%PEG (MAG. × 100) AND B) 30%KC+7%PEG (MAG. × 101).	91
4-19. EFFECT OF KC ON UNNOTCHED IMPACT STRENGTH OF HDPE-KC COMPOSITES	92
4-20. THE EFFECT OF PEG ON IMPACT STRENGTH OF HDPE- KC COMPOSITES	93
4-21. SEM (MAG. × 300) OF THE IMPACT FRACTURE SURFACE OF HDPE-KC COMPOSITES AT DIFFERENT CONCENTRATIONS: A) 70:30, B) 60:40, AND C) 50:50.	95
4-22. SEM (MAG. × 300) FOR IMPACT FRACTURE SURFACE OF HDPE-KC-PEG COMPOSITES; A) 30%KC+5%PEG AND B) 40%KC+5%PEG.	96
4-23. EFFECT OF KC CONTENT ON FLEXURAL STRENGTH OF LDPE BIOCOMPOSITES. THE DATA ARE MEAN OF 7 REPLICATIONS OF EACH TREATMENT.	97
4-24. EFFECT OF PEG ON FLEXURAL STRENGTH OF LDPE- KC COMPOSITES	98
4-25. EFFECT OF KC CONTENT ON FLEXURAL STRENGTH OF HDPE BIOCOMPOSITES	99
4-26. EFFECT OF PEG ON FLEXURAL STRENGTH OF HDPE-KC COMPOSITES	100
5-1. TG CURVES FOR LDPE-KC COMPOSITES WITH 0, 30, 40, AND 50% KC	107
5-2. DTG CURVES FOR LDPE-KC COMPOSITES WITH 0, 30, 40, AND 50% KC	107
5-3. TG CURVES FOR EFFECT OF PEG8000 ON LDPE-KC COMPOSITES	110
5-4. TG CURVES FOR HDPE-KC COMPOSITES WITH 0, 30, 40, AND 50% KC	111
5-5. DTG CURVES FOR HDPE-KC COMPOSITES WITH 0, 30, 40, AND 50% KC	111
5-6. TG CURVES FOR EFFECT OF PEG8000 ON HDPE-KC COMPOSITES	113
5-7. DSC CURVE OF PURE LDPE SAMPLE	114
5-8. DSC CURVE OF PURE HDPE SAMPLE	116
6-1. DISTRIBUTION OF SAMPLES IN THE SOIL INSIDE THE POT	123
6-2. SPECIMENS IN THE SOIL BURIAL TEST	124
6-3. PLASTIC FILM COVERING THE POTS	124

6-4. BIODEGRADABILITY OF LDPE-KC COMPOSITES DURING BURIAL TIME	125
6-5. EFFECT OF PEG8000 ON BIODEGRADABILITY OF LDPE-KC COMPOSITES CONTAINING: A) 30%, B) 40%, AND C) 50% KC DURING BURIAL TIME	127
6-6. BIODEGRADABILITY OF LDPE-PEG COMPOSITES DURING BURIAL TIME	128
6-7. BIODEGRADABILITY OF HDPE/KC COMPOSITES DURING THE TIME	129
6-8. BIODEGRADABILITY OF HDPE-KC COMPOSITES CONTAINING: A) 30%, B) 40% AND C) 50% KC WITH 5 AND 7% PEG8000 DURING BURIAL TIME	130
6-9. BIODEGRADABILITY OF HDPE/PEG COMPOSITES DURING BURIAL TIME	131
6-10. DISPERSION OF INTEGRITY AND BREAKUP OF PE-KC SAMPLES DURING BURIAL TIME	134
6-11. SEM (MAG. × 500) OF THE FRACTURE SURFACE OF 50% KC + 5% PEG8000 FOR LDPE (LEFT) AND 50% KC + 7% PEG8000 FOR HDPE (RIGHT) COMPOSITES AFTER 4 MONTHS IN THE SOIL.	135
7-1. WATER ABSORPTION TEST FOR PE-KC COMPOSITES	140
7-2. EFFECT OF KC AND PEG LOADING ON WA OF LDPE-KC COMPOSITES DURING THE FIRST 24 HOURS	141
7-3. EFFECT OF KC AND PEG LOADING ON WA OF HDPE-KC COMPOSITES DURING THE FIRST 24 HOURS	142
7-4. EFFECT OF BIOCOMPOSITE FORMULATIONS ON WA OF LDPE-KC COMPOSITES DURING 2 MONTHS	143
7-5. EFFECT OF BIOCOMPOSITE FORMULATIONS ON WA OF HDPE-KC COMPOSITE DURING 2 MONTHS	144



LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
Da	Daltons
DP	Degree of polymerization
DSC	Differential Scanning Calorimetry
DTG	Degradation Temperature (Derivative Thermogravimetric)
EU	European Union
FS	Flexural Strength
HDPE	High Density Polyethylene
INTROP	Institute of Tropical Forestry and Forest Products
IS	Impact Strength
KC	Kenaf cellulose
LDPE	Low Density Polyethylene
LLDPE	Linear Low Density Polyethylene
MA	Maleic Anhydride
MAPP	Maleic Anhydride polypropylene
MARDI	Malaysian Agricultural Research and Development Institute
M_n	The number average molecular weight of the polymer
M_w	Molecular weight (The sum of the atomic weights of all the atoms in a molecule)
OECD	Organization for Economic Cooperation and Development
PCL	Polycaprolactone
PE	Polyethylene
PEA	Polyesteramide
PEG	Polyethylene glycol



PHA	Polyhydroxyalkanoates
PHB	Polyhydroxybutyrate
PLA	Polylactic acid
PP	Polypropylene
RPE	Recycled high density Polyethylene
rpm	Revolution per minute
SEM	Scanning Electron Microscope
T_d	Decomposition Temperature
T_g	Glass transition temperature
TG	Thermogravimetric
TGA	Thermogravimetric Analysis
T_m	Melting Temperature
TS	Tensile Strength
UPM	Universiti Putra Malaysia
WA	Water absorption
WF	Wood flour

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Packaging technology is a mixture of mechanical engineering, mathematics, chemical engineering, physics, packaging science, etc. They make a body of knowledge related to packaging that might be considered the foundation for a technology. Food packaging, for example, is intended to help maintain the quality and shelf life of food products by controlling the transfer of moisture, oxygen, carbon dioxide, lipids, aromas, flavors, and food additives. Han, (2005) said that a good package protects the food quality and also considerably contributes to a business profit. Quality of package depends on its materials. Therefore, the most important function of a packaging material is the preservation of the packed goods quality for storage, transportation and end-use.

Plastic materials are one of the most important materials that are used in food packaging. Plastics are defined as processable materials based on polymers. Because of plastics' advantages in compared with other materials, they have been extensively adopted in food packaging. These advantages are reflected in the physical, mechanical and chemical properties of plastics. They are stable in ambient and many hostile environments and not subject to degradation in normal use. It means that they will not change in properties or performance during the package life (Brown, 1992). Plastics can be formed into end products, such as bottles, containers, films, coatings, etc. The



development of self-service stores with their large variety of products is unbelievable without plastics.

Polyethylene (PE) is used as a common and cheap polymer in food packaging. It is one of the usual synthetic polymers with high hydrophobic level and high molecular weight. In natural form, it is not biodegradable. Thus, their use in the production of disposal or packing materials causes many problems (Abd El-Rehim et al., 2004). The main problem is environmental pollution troubles by synthetic polymers and in conjunction with the land shortage problems for solid waste management. Latest and harder ecological policies enforce industries like the automotive, packaging and construction industry to look for new materials (Espert et al., 2004). Thus, attempts have been made to solve these problems and so, the need for environmentally degradable and “environmental friendly” polymers has arisen.

There is a considerable interest in replacing some or all of the synthetic plastics by natural or biodegradable materials in many applications. Since the food industry uses a lot of plastics, even a small reduction in the amount of materials used for each package would result in a significant polymer reduction, and may improve solid waste problems (Chandra & Rustgi, 1998; Han, 2005). It is clear that the use of biodegradable polymers for packaging offers an alternative and partial solution to the problem of gathering of solid waste composed of synthetic inert polymers (Jayasekara et al., 2003).

Plant fibers have attracted more and more research interests owing to their advantages like renewable, environmental friendly, low cost, light weight (low



density), high specific mechanical performance (acceptable specific strength properties), easily recyclable, ease of separation, carbon dioxide sequestration, and biodegradability (Zhang et al., 2005).

There are some biocomposites consisting of biodegradable polymers as the matrix material and biodegradable fillers, usually biofibres (e.g. lignocellulose fibers). Since both components are biodegradable, the composite as the integral part is also expected to be biodegradable (Le Digabel & Avérous, 2006). However, these materials may have problems in the mechanical and physical properties.

Nowadays, the blending of biodegradable polymers with inert polymers has been accepted as a possible application in the waste disposal of plastics. In principal, the way of thinking behind this method is that if the biodegradable section is present in enough quantities and if microorganisms in the waste disposal environment degrade it, the plastic or film containing the residual inert component should lose its integrity, fall to pieces and fade away. The best-known renewable resources able to create biopolymer and biodegradable plastics are starch and cellulose (Chandra & Rustgi, 1998; Selke, 2000).

Weber et al. (2002) believed that the only biobased food-packaging materials in use commercially on a major scale are based on cellulose; however, many studies were done on starch-based products including thermoplastic starch, starch and synthetic aliphatic polyester blends, and starch and other polymer blends. Since, starch is a source of energy and it has an important role in human food, research should be focused on new subject that is not human