

MECHANICAL AND THERMAL PROPERTIES OF MONTMORILLONITE
FILLED NEWSPAPER FIBER REINFORCED RECYCLED POLYETHYLENE
TEREPHTHALATE NANOCOMPOSITES

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UNIVERSITI TEKNOLOGI MALAYSIA

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My dear grandfather, Mr. keramatollah niazi (Babaji), you always encourage me to be a better person. I wish, I could be able to thank you enough.

I want to dedicate this thesis to my supportive husband, who helped me in every single step of this journey.

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ABSTRACT

The feasibility of developing a newspaper fiber (NPF) reinforced poly (ethylene terephthalate) nanocomposites containing organically modified montmorillonite (MMT) was investigated. The nanocomposites were prepared using a counter-rotating twin-screw extruder followed by injection molding. The mechanical properties of the nanocomposites were studied through tensile, flexural and impact testing. The thermal characteristics of the composites were evaluated through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The morphological behavior of the samples was observed via scanning electron microscopy (SEM). The composites were prepared by using 5 to 15 wt% of NPF as reinforcing elements along with 10 phr of SEBS-g-MA as compatibilizer. The results showed that blending PETr with SEBS-g-MA increased the toughness of the recycled resin and significant improvements in the impact strength of the blend were observed. The incorporation of NPF increased both tensile and flexural moduli of the composites but lowered the impact strength. According to DSC data, the inclusion of NPF increased the degree of crystallinity of the composites, which point to the fact that NPF could act as nucleating agent. The TGA curve revealed that NPF addition lowered the thermal stability of the composites. Micro images taken from the surface of the broken specimen showed poor interfacial adhesion between the fibers and the matrix due to the differences in the hydrophobic\hydrophilic nature of the components. Incorporation of MMT resulted in momentous improvements in both Young's and flexural moduli of the nanocomposites at the expense of impact strength of the samples. Significant increase observed in the flexural modulus from 2368 MPa to 3484 MPa for composites with 10 wt% of NPF and nanocomposites incorporating 5 phr of nanoclay respectively. The presence of MMT was shown to have influence on the crystallization behavior of the nanocomposites and decreased the onset crystallization temperature as well as melting temperature (T_m). The thermal stability of the nanocomposites showed improvements upon the incorporation of nanoclay. Overall, the properties point to a product with enhanced mechanical properties as compared to PETr with the potential to be used in wood replacement applications.

ABSTRAK

Nanokomposit daripada gentian surat khabar (NPF) diperkuat poli (etilena terephthalate) PET mengandungi organik montmorilonit (MMT) yang diubahsuai telah dikaji. Nanokomposit disediakan dengan menggunakan ekstruder kaunter berputar skru berkembar diikuti pengacuan suntikan. Sifat-sifat mekanikal seperti kekuatan tensil, kekuatan flexsural dan kekuatan hentaman nanokomposit telah dikaji. Ciri-ciri terma dinilai menerusi analisis thermogravimetric (TGA) dan kalorimetri pengimbasan perbezaan (DSC). Mikroskop imbasan electron (SEM) digunakan bagi mengkaji sifat permukaan sampel. Komposit telah disediakan dengan menggunakan 5 hingga 15% berat NPF sebagai agan penguat bersama-sama dengan 10 phr SEBS-g-MA sebagai pemadan. Keputusan menunjukkan adunan PETr dengan SEBS-g-MA meningkatkan keliatan resin kitar semula dan peningkatan yang ketara dalam kekuatan hentaman. Penambahan NPF didalam adunan meningkatkan modulus tensil dan fleksural komposit namun menurun bagi kekuatan hentaman. Merujuk kepada data DSC, penambahan NPF meningkatkan darjah penghabluran komposit yang membuktikan NPF boleh bertindak sebagai agen penukleusan. Keluk TGA menunjukkan penambahan NPF menurunkan kestabilan terma bagi komposit. Struktur mikro yang diamati dari permukaan patah komposit menunjukkan lekatan antara muka yang lemah diantara gentian dan matrik disebabkan oleh perbezaan sifat hidrophobik/hidrophilik komponen. Penambahan MMT didalam adunan menghasilkan peningkatan dalam modulus young's dan fleksural nanokomposit, namun begitu menurunkan kekuatan hentaman bagi sampel. Peningkatan yang ketara dapat diperhatikan dalam modulus fleksural daripada 2368 Mpa ke 3484 Mpa bagi komposit dengan 10% NPF dan nanokomposit dengan 5 phr nanotanh liat. Kehadiran MMT menunjukkan pengaruh ke atas sifat penghabluran nanokomposit dimana menurunkan suhu permulaan penghabluran serta suhu lebur (T_m). Kestabilan terma bagi nanokomposit menunjukkan peningkatan dengan penambahan nano tanah liat. Secara keseluruhannya, hasil kajian menunjukkan bahawa nanokomposit daripada gentian surat khabar (NPF) diperkuat PET mempunyai sifat-sifat mekanikal yang lebih baik berbanding dengan PETr dimana ia akan menjadikan nanokomposit ini sebagai bahan yang sesuai untuk menggantikan kayu.

TABLE OF CONTENT

CHAPTER	TITLE	PAGE
	TITLE	i
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENTS	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENTS	vii
	LIST OF TABLES	x
	LIST OF FIGURES	xi
	LIST OF ABBREVIATIONS AND SYMBOLS	xiii
	LIST OF APPENDEX	xvi
1	INTRODUCTION	1
	1.1 Background	1
	1.2 Problem Statement	3
	1.3 Objective of the study	4
	1.4 Scope of Research	5
	1.5 Importance of research	6
2	LITERATURE REVIEW	7
	2.1 Polyethylene terephthalate (PET)	7
	2.2 PET Recycling	8
	2.3 Recycling methods	9

2.4	Natural Fiber	10
2.4.1	Recycled paper reinforced	14
2.4.2	Function of coupling agent	16
2.5	Nanocomposites	18
2.6	Montmorillonite (MMT)	19
2.6.1	MMT distribution	20
2.7	Nanocomposites preparation methods	20
2.8	PET nanocomposites	23
3	MATERIALS AND METHODS	26
3.1	Materials	26
3.1.1	Recycled polyethylene terephthalate (PETr)	26
3.1.2	Newspaper fiber (NPF)	26
3.1.3	Maleic anhydride-grafted poly (styrene-ethylene/butyldiene-Styren)	27
3.1.4	Montmorillonite (MMT)	27
3.1.5	Composition and designation of materials	27
3.2	Sample preparation	29
3.2.1	Premixing	29
3.2.2	Extrusion	29
3.2.3	Injection molding	29
3.3	Materials Properties Characterization	30
3.3.1	Physical and Mechanical Testing	30
3.3.1.1	Density measurement	30
3.3.1.2	Tensile	30
3.3.1.3	Flexural	30
3.3.1.4	Un-notched Izod Impact	31
3.3.2	Thermal Analysis	31
3.3.2.1	Differential Scanning Calorimetry (DSC)	31
3.3.2.2	Thermogravimetry Analysis	32
3.3.3	Morphological Study	32
3.3.3.1	Scanning Electron Microscopy (SEM)	32

4	RESULTS AND DISCUSSION	33
4.1	Effect of different NPF loading on PETr	33
4.1.1	Physical and Mechanical Properties	33
4.1.1.1	Density measurements	33
4.1.1.2	Control materials	34
4.1.1.3	Izod impact properties	36
4.1.1.4	Tensile properties	37
4.1.1.5	Flexural strength	39
4.1.1.6	Flexural modulus	40
4.1.2	Scanning Electron Microscopy (SEM)	42
4.1.3	Thermal properties	45
4.1.3.1	Differential scanning calorimetry (DSC)	45
4.1.3.2	Thermogravimetric analysis (TGA)	47
4.2	Effect of MMT on PETr/NPF/MA-g-SEBS/MMT Nanocomposites	49
4.2.1	Physical and Mechanical properties	49
4.2.1.1	Density measurement	49
4.2.1.2	Izod impact properties	50
4.2.1.3	Tensile properties	51
4.2.1.4	Flexural strength	53
4.2.1.5	Flexural modulus	54
4.2.3	Thermal properties	55
4.2.3.1	Differential scanning calorimetry (DSC)	55
4.2.3.2	Thermogravimetric analysis (TGA)	58
	CONCLUSION AND RECOMMENDATIONS FOR FUTURE WORKS	60
5.1	Conclusion	60
5.2	Recommendation for future works	62
	REFERENCES	63
	Appendices A-B	69

LIST OF TABLES

TABLE NO.	TITLE	PAGE
2.1	Natural fibers and their origins	10
2.2	Production of plant fibers annually	11
2.3	Structure and chemical composition of natural fiber	12
2.4	Comparison of natural fiber and other fibers in terms of mechanical properties	13
3.1	Sample composition of PETr/SEBS-g-MA/NPF composites	28
3.2	Sample composition of rP/S10/N10 with different MMT contents	28
4.1	Density variations of rP, rP/S10 blend and rP/S10/NPF composites	34
4.2	Mechanical properties of rP and rP/S10 blend	35
4.3	Flexural modulus/Density of rP, rP/S10 blend and rP/S10/NPF composites	41
4.4	Thermal characteristic of rP, rP/S10 and rP/S10/NPF composites (first heating)	46
4.5	Thermal characteristic of rP, rP/S10 and rP/S10/NPF composites (cooling)	47
4.6	Thermal characteristics of nanocomposites (first heating)	56
4.7	Thermal characteristics of rP, rP/S10 and rP/S10/NPF composites (cooling)	57
4.8	TGA results of rP/S10/N10 composite and rP/S10/N10/MMT nanocomposites	59

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
2.1	Structure of 2:1 phyllosilicates	19
2.2	Three possible structures of polymer-layered silicate composites	20
2.3	Schematic representation of polymer nanocomposite Obtained by in-situ polymerization	21
2.4	Schematic representation of polymer nanocomposite Obtained By intercalation of polymer from solution	22
2.5	Schematic representation of polymer nanocomposite Obtained by direct melt intercalation	23
4.1	Effect of fiber loadings on Impact strength of rP/S10 blend	36
4.2	Effect of fiber loadings on Tensile strength of rP/S10 blend	37
4.3	Effect of fiber loadings on Young's modulus of rP/S10 blend	38
4.4	Effect of fiber loadings on Elongation at break of rP/S10 blend	39
4.5	Effect of fiber loadings on Flexural strength of rP/S10 blend	40
4.6	Effect of fiber loadings on Flexural modulus of rP/S10 blend	41
4.7	SEM micrographs of impact fractured surfaces :(a) NPF , (b) rP/s10, (c) rP/S10/N5, (d) rP/S10/N10, (e) rP/S10/N15	44
4.8	PETr-co-SEBS-g-MA copolymer	45
4.9	Thermogravimetric analysis of PETr and PETr/SEBS-g-MA blend	48
4.10	Thermogravimetric analysis of PETr/SEBS-g-MA/NPF composites	48
4.11	Density changes with different MMT loadings	49
4.12	Effect of MMT content on Impact strength of	

	rP/S10/N10 composite	50
4.13	Effect of MMT content on tensile strength of rP/S10/N10 composite	51
4.14	Effect of MMT content on young's modulus of rP/S10/N10 composite	52
4.15	Effect of MMT content on Elongation at break of rP/S10/N10 composite	53
4.16	Effect of MMT content on flexural strength of rP/S10/N10 composite	54
4.17	Effect of MMT content on flexural modulus of rP/S10/N10 composite	55
4.18	TGA curves for rP/S10/N10 composite and rP/S10/N10/MMT nanocomposites	58

LIST OF ABBREVIATIONS AND SYMBOLS

ASTM	American Society for Testing and Material
CMC	Cetyltrimethylammonium chloride
DMA	Dynamic mechanical analysis
DMT	Dimethyl terephthalate
DSC	Differential scanning calorimeter
d	Average distance between MMT layers
EG	Ethylene glycol
EPDM	Ethylene–propylene–diene monomer
EBGMA	Ethylene/n-butyl acrylate/glycidyl methacrylate
EMA	Ethylene and methyl acrylate
FDA	Food and Drug Administration
f	weight fraction of the component
HAR	High aspect ratio
HIPP	High impact polypropylene
MA	Maleic anhydride
MAPP	Maleic anhydride grafted polypropylene
MA-g-EPDM	Maleic anhydride grafted ethylene propylene dien monomer
MMT	Montmorillonite
NPF	Newspaper fiber

n	Diffraction number
OMLS	Organically modified layered silicates
PET	Polyethylene terephthalate
PETG	Polyethylene terephthalate glycol
PETr	Recycled polyethylene terephthalate
PP	Polypropylene
Ps	Paper sludge
SEBS-g-MA	Styrene-ethylene-butylene-styrene grafted maleic anhydride
SEM	Scanning electron microscopy
TEM	Transmission electron
TGA	Thermo gravimetric analysis
TPA	Terephthalic acid
T _g	Glass transition temperature
T _m	Melting temperatures
T _c	Crystallization
U.S	United States
UV	Ultra violet
WF	Wood flour
XRD	X-ray diffraction
X _c	Degree of crystallinity
θ	Diffraction angle
λ	Inlet X-ray wavelength
ΔH_m	Measured heat fusion

$\Delta H_{\text{m ideal}}$

Enthalpy of fusion for 100% crystalline

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A.	Conference proceeding (MAMIP 2012), Malaysia.	69
B.	Conference Proceeding (ACCM-8), Malaysia.	70

CHAPTER 1

INTRODUCTION

1.1 Background

Polyethylene terephthalate (PET) is one of the most widely used polymers in today's world. It has the second highest scrap value and has been extensively used for the fabrication of soft drink bottles, polyester fiber, photographic films and video/audio tapes. The widespread use of PET has serious economic and environmental concerns, and its recycling is a promising choice for the resource conservation. Due to its availability and low cost, PET has received considerable attention in terms of recycling of post-consumer bottle (Bartolome *et al.*, 2012).

PET has been used in various applications such as packaging, textile, automotive and electro-technical but the largest market for PET is carbonated soft drink bottles and water bottle, which is the second largest PET consuming market globally. Beside Asia, The Middle East and African demand for PET is the second fastest growing in the world. Approximately, the Middle East and African Consumers used 1.2 million tons of PET where this number for Asian demand was almost 4.7 million tons in 2009. Because of the increasing usage of PET in plastics industry, finding suitable methods of recycling is an evolving challenge from economic and ecological points of view (Sirsat *et al.*, 2010).

Two methods have been used for recovering of PET: chemical and physical recycling. Physical recycling is an appropriate way and becoming a priority because of economic and environmental concerns (Bartolome *et al.*, 2012). Other method in reusing is to add synthetic or natural fibers (date palm tree, waste papers, palm oil tree, kenaf, sugarcane bagasse, coconut) as the reinforcements in polymers. The natural fibers offered significant advantages (in terms of reduced composite weight and biodegradability) over mineral fillers such as glass fiber, calcium carbonate, and talc (Corradini *et al.*, 2009). Due to the biodegradability of fibers, their composites with polymers propose a new group of materials, which can offer environmental protection.

There are several important features of natural fibers such as; low density, low cost, nonabrasive nature, low energy consumption, possibilities of powerful loading level, biodegradability, high specific properties, availability of a wide variety of fibers throughout the world, and generation of agriculture-based economy (Joseph *et al.*, 1996; Kalia *et al.*, 2009; Nair *et al.*, 1996; Sinha Ray and Okamoto, 2003; Sreekala *et al.*, 2000; Taj *et al.*, 2007b; Zhang *et al.*, 2007). Natural fiber-reinforced composites have been studied extensively due to low cost, environmental friendly, degradability and low energy requirement (Nair *et al.*, 1996; Taj *et al.*, 2007a).

Waste paper is a type of natural fiber, which is mainly made up of cellulose, hemicelluloses and lignin (Overend *et al.*, 1987). The main component in municipal solid waste streams is paper and the sub products that come from it. Paper can be divided into three categories, old newspaper, office paper and old corrugated. Some types of papers have contaminated with ink, dyes, metal fid and plastic. Using recycled newspaper in paper industry requires specific refinement but according to English (1994) for composite industry this modification requirement is lower. Since last decade, natural fiber-reinforced composites have received substantial attention due to the technology development and economic factors.

Newspaper fiber feed is a critical requirement in plastic composite formulation. More than 40 wt% of paper affects the process ability of the blend.

Reused papers have been used in composite industry via different methods. Today, recycled paper is used in a variety of applications such as producing new papers or using it as reinforcement in composite industry (English, 1994).

Annually, around 60 million tons of polyethylene terephthalate (PET) are produced in beverage industry for packaging sodas, mineral water, juice and, etc. (Lepoittevin and Roger, 2011). The recycling of newspaper and PET bottles can play a major role in addressing the economic and environmental issues and offers the advantages of clean air and the reduction of using virgin timber. Each people in the United States use 290 kg of paper and paperboard which is as high as 54 million metric tons each year (World Book, 1991).

Old newspaper and recycled polyethylene terephthalate (PETr) can offer benefits over using synthetic fibers such as low cost, biodegradability and lightweight in the composite industry. A major problem in using old newspaper in the composite structure is the compatibility between the fiber and the matrix (Al - Khanbashi *et al.*, 2005; Herrera-Franco and Valadez-Gonzalez, 2005; Soleimani *et al.*, 2008). Compatibility between NPF and PETr is the main problem of this combination. It is expected the addition of compatibilizer such as, styrene-ethylene-butylene-styrene grafted maleic anhydride (SEBS-g-MA) can resolve this issue.

1.2 Problem Statement

According to the literature, several researchers have done work on natural fiber/poly propylene composite (Ismail and Bakar, 2005; Jang *et al.*, 2000; Jang and Lee, 2000; Qiao and Zhang, 2004; Qiao *et al.*, 2003; Son *et al.*, 2001) , but natural fiber/PETr composite received less attention. The present study focuses on the effect of adding newspaper fiber (NPF) into PETr. The effect of adding NPF with different

fiber loadings into the recycle PET has not yet been reported. It is expected that mechanical and thermal properties will be improved.

1.3 Objective of the study

The aim of the research was to develop plastic composites based on NPF reinforced PETr. The composites have the potential application in indoor articles such as doorframes, trim, furniture and any other wood replacement. The main objectives can further be divided into:

1. To study the effect of NPF loading on thermal (differential scanning calorimeter (DSC), thermo gravimetric analysis (TGA)) and mechanical (impact, tensile and flexural) properties of the PETr composites.
2. To study the feasibility of producing hybrid NPF reinforced PETr composite by the incorporation of nano sized filler Montmorillonite (MMT) through mechanical and thermal analysis.

1.4 Scope of research

In order to achieve the goals of this research, the following activities were carried out:

1) Sample preparation

In this research project, sample preparation and blending were performed via melt extrusion. This includes:

- i) Papering the newspaper fiber.
 - ii) Blending of PETr with NPF fibers by using a twin-screw extruder in a single extrusion step.
 - iii) Blend fabrication into test specimens via injection molding according to the ASTM standard.
- ### **2) Physical and mechanical analysis**
- i) Density measurement
 - ii) Tensile
 - iii) Flexural
 - iv) Unnotched Izod impact

3) Thermal properties analysis

i) Differential scanning calorimeter (DSC)

ii) Thermo gravimetric analysis (TGA)

4) Morphological study

Scanning electron microscopy (SEM)

1.5 Importance of Research

The current study developed acceptable properties in terms of physical (low density), mechanical (Impact, tensile and flexural) and thermal properties for PETr/NPF composite. The success of this research will widen the applications of PETr/NPF composite such as floor coverings, apparel, roofing sheets, window and door frames, furniture, objects of art (decorations, plant pots), seats at a stadium, stationary (stands for pencils and documents), post office box, etc.

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A. Conference proceeding

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Study on the mechanical properties of recycled poly (ethylene terephthalate) reinforced newspaper fiber composite

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Abstract: Newspaper fiber/recycled polyethylene terephthalate (PETr) composites which have many advantages such as low cost, light weight and good mechanical properties were produced. In this study, the mechanical properties of PETr/newspaper fiber (NPF) were investigated. Maleic anhydride grafted styrene-ethylene-butylene-styrene (MA-g-SEBS) was used as compatibilizer. It was found that impact strength and elongation at break improved by the addition of MA-g-SEBS. At different fiber loadings (5, 10 and 15 wt %), the tensile modulus increase, while the tensile strength decreased.

B. Conference proceeding

THE 8TH ASIAN-AUSTRALASIAN CONFERENCE ON COMPOSITE MATERIALS(ACCM-8)

Study on mechanical and crystallization behavior of recycled poly (ethylene terephthalate)/ newspaper fiber/ Montmorillonite nanocomposite

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ABSTRACT

In this study, recycled poly (ethylene terephthalate) (PETr) / 10 wt% newspaper fiber (NPF) / 10 Phr styrene-ethylene-butylene-styrene grafted maleic anhydride) (SEBS-g-MA) and (1, 3 and 5 phr) montmorillonite (MMT) nanocomposites were prepared using a co-rotating Brabender twin-screw extruder followed by injection molding. Improvements in tensile modulus by 15% in the case of 3 phr MMT content was observed. However, the tensile strength of PETr/NPF/MA-g-SEBS with 1 phr MMT content remained constant and decreased with higher MMT loadings. Flexural modulus showed significant improvements up to 47% by the addition of 5 phr of MMT. The flexural strength of all hybrid nanocomposites showed enhancements up to 8%. Elongations at break in all samples were less than that of PETr/SEBS-g-MA/NPF composite.