

PRODUCTION AND CHARACTERIZATION OF
DEGRADABLE POLYETHYLENE
TEREPHTHALETE USING PRO-DEGRADANT
ADDITIVES, POLYLACTIC ACID AND
POLYLACTIC ACID GRAFTED MALEIC
ANHYDRIDE

ASHWINDER A/P CHELLIAH

MASTER OF SCIENCE

UNIVERSITI MALAYSIA PAHANG



SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

(Supervisor's Signature)

Full Name : DR ARUN GUPTA

Position : PROFESSOR

Date : 11th JUNE 2019

(Co-supervisor's Signature)

Full Name : DR NOR HANUNI BINTI RAMLI @ SAID

Position : SENIOR LECTURER

Date : 11th JUNE 2019



STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

(Student's Signature)

Full Name : ASHWINDER A/P CHELLIAH

ID Number : MKC15026

Date : 11th JUNE 2019

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ASHWINDER A/P CHELLIAH

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DEDICATION

I specially dedicate this dissertation to my beloved parents;

Mr. Chelliah and Mrs. Jagender Kaur

Beloved Siblings and Dearest Husband

For their continuous support, guidance and believe in me.

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ABSTRAK

Keprihatinan terhadap alam sekitar yang disebabkan oleh penularan penggunaan bahan plastik sintetik seperti Polietilena tereftalat (PET) telah mewujudkan keperluan untuk pembangunan bahan plastik biodegradasi, terutamanya dalam sektor pembungkusan. Sebagai usaha untuk menggalakkan degradasi, campuran pro-degradasi (CS), asid poli lactic gred botol (PLA) dan agen penserasi (PLA-g-MA) dengan polietilena terephthalate (PET) telah disediakan untuk mengkaji sifat-sifat dan tahap degradasi campuran tersebut. Komposit PET / CS dan campuran PET / PLA dengan dan tanpa agen penserasi disediakan menggunakan teknik penyemperitan dan pengacuan suntikan. Aditif CS dicampurkan dengan PET pada komposisi 0.25, 0.5, 0.75 dan 1 pph masing-masing. Dalam kajian ini, sebanyak 5, 10 dan 15% gred botol PLA dicampurkan dengan PET. PET yang dicampurkan dengan 15% gred botol PLA diserasikan menggunakan komposisi PLA-g-MA sebanyak 2, 4, 6 dan 8 wt %. Campuran polimer yang disediakan dicirikan melalui sifat mekanik (kekuatan tegangan tensil dan fleksural) dan ciri-ciri terma (TGA dan DSC). Kajian tahap degradasi dijalankan menggunakan kaedah pengoksidaan terma dan luluhawa dalam ruang UV. Analisa spektroskopi infra merah (FTIR) dan pengimbasan mikroskop elektron (SEM) telah dijalankan untuk mengkaji perubahan struktur molekul dan morfologi permukaan, sementara tahap degradasi diukur melalui index karbonil. Keputusan menunjukkan bahawa, 0.25CS mencatatkan TS, TM, FS dan FM yang tertinggi berbanding PET tulen. Ini menunjukkan penambahan CS hingga 0.25 pph membantu menguatkan ikatan komposit PET/CS, sekaligus mewujudkan fasa berterusan. Keputusan termal menunjukkan peningkatan kandungan CS dalam PET mengurangkan kestabilan haba PET, manakala entalpi dan penghabuluran sampel komposit berkurangan dengan peningkatan kandungan aditif CS dari 0.25 kepada 1 pph dalam PET. Kajian degradasi menggunakan analisa spektroskopi menunjukkan pertumbuhan intensity yang tidak sekata di sekitar kawasan karbonil sepanjang pendedahan kepada haba kecuali untuk sampel komposit 0.25CS. Walau bagaimanapun, bacaan CI yang rendah (kurang daripada 2.6) menafsirkan tiada kesan degradasi terhadap PET dengan penggabungan CS. Seterusnya, peningkatan penambahan gred botol PLA meningkatkan sifat mekanik PET. PLA15 mencapai TS, TM, FS dan FM tertinggi, masing-masing berbanding PET. Walau bagaimanapun, penambahan PLA-g-MA ke PLA15 telah mengakibatkan pengurangan TS, TM, FS dan FM. Kajian termal menunjukkan campuran PLA15 dengan dan tanpa PLA-MA telah mengalami degradasi dalam satu tahap dan tambahan gred botol PLA dan PLA-g-MA telah mengurangkan kestabilan terma PET. Bagi kajian degradasi, pertumbuhan intensiti pada kawasan karbonil pada campuran PLA15 selepas pendedahan haba bersama-sama dengan peningkatan CI dan penampilan permukaan menunjukkan proses degradasi. Selain itu, tambahan PLA-g-MA dalam campuran PLA15 turut menggalakan proses degradasi berlaku. Secara ringkasnya, pergabungan gred botol PLA dengan PET meningkatkan kualiti PET dari segi ciri-ciri dan tahap degradasi.

ABSTRACT

Overwhelming environmental concern due to the usage of synthetic plastics such as polyethylene terephthalate (PET) has continuously evoke to the development of biodegradable plastic in the packaging segment. As an effort to promote degradation, pro-degradant (CS), bottle grade polylactic acid (PLA) and functionalised maleated polylactic acid (PLA-g-MA) were blended with polyethylene terephthalate (PET) to study on its properties and degradation behaviour. PET/CS composites, PET/PLA blends with and without compatibilizers were prepared using extrusion and injection molding technique. CS additives were incorporated into PET at a composition of 0.25, 0.5, 0.75 and 1 pph, respectively. In this study the polymer blend was prepared by adding bottle grade PLA composing of 5, 10 and 15 wt % with PET. PET blended with 15 wt % of bottle grade PLA (PLA15) were compatibilized using PLA-g-MA at 2, 4, 6 and 8 wt %, respectively. The formulated samples were then characterized by mechanical properties (tensile and flexural strengths) and thermal characteristics (TGA and DSC). Degradation studies were done using thermo-oxidative and accelerated weathering testing. Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectroscopy and scanning electron microscope (SEM) analysis were performed to study on the molecular structural changes and surface morphologies, while degree of degradation of the polymeric blends were measured using Carbonyl Index. The results indicated that, 0.25CS recorded highest TS, TM, FS and FM compared to pure PET. This showed that addition of CS additives up to 0.25 pph promoted an improved strength and interfacial adhesion in the composites. Thermal studies showed that increasing CS loading from 0.25 to 1 pph reduced the thermal stability of PET, while enthalpy and crystallinity of the composites reduced with increasing CS loading amount in PET. Degradation studies revealed an uneven growth around the carbonyl region of all the samples in the FTIR spectrum upon thermal exposure except for 0.25CS composite sample. However, reduced CI reading (less than 2.6) on all the composite samples interprets absence of degradation in the samples. The increasing addition of bottle grade PLA improved mechanical properties of PET. PLA15 achieved the maximum TS, TM, FS and FM, respectively compared to PET. Nevertheless, addition of PLA-g-MA into PLA15 has resulted in reduced TS, TM, FS and FM. Thermal studies showed that PET/PLA blends with and without PLA-g-MA have undergone a single step decomposition and increasing loading effect of PLA and PLA-g-MA have reduced the thermal stability of PET favoring for degradation to take place. As for the degradation studies, growing carbonyl region of the PLA15 blend after thermal exposure along with increasing CI reading and surface appearance stipulates the degradation process. Likewise, increasing addition of PLA-g-MA in PLA15 blends was observed to promote degradation compared to a compatibilized effect in the PET/PLA blends. Conclusively, blending of bottle grade PLA with PET increases the added value of the polymeric blend with regards to its properties and susceptibility towards degradation.

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LIST OF SYMBOLS

%	Percentage
\geq	More than or equal to
=	Equal
\times	Multiply
\pm	Plus minus
$^{\circ}\text{C}$	Degree Celsius
s	Second
g	Gram
m	Mass
kg	Kilogram
hr	Hour
wt	Weight
N_2	Nitrogen gas
min	Minute
rpm	Revolutions per minute
phr	Parts per hundred
mL	Millilitre
kN	Kilo newton
MPa	Mega pascal
GPa	Giga Pascal
kv	Kilovolts
cm^{-1}	Reciprocal centimetre
g/cm^3	Gram per cubic centimetre
ml/min	Millilitre per minute
$^{\circ}\text{C/min}$	Degree Celsius per minute
J/g	Joule per gram
W/m^2	Watt per square metre
dl/g	Decilitre per gram
V	Volume
N	Normality
A.N	Acid number

G_r	Grafting of Maleic Anhydride
T_g	Transition glass temperature
T_m	Melting temperature
X_c	Degree of crystallinity
ΔH_f°	Heat of fusion

LIST OF ABBREVIATIONS

ABS	Acrylonitrile butadiene styrene
ATR-FTIR	Attenuated Total Reflectance Fourier Transmission Infra-Red
CI	Carbonyl Index
CS	Cobalt stearate
DCP	Dicumyl peroxide
DSC	Differential Scanning Calorimetry
EB	Percentage of elongation of break
EG	Ethylene Glycol
FM	Flexural modulus
FS	Flexural strength
HDPE	High density polyethylene
IV	Intrinsic Viscosity
KOH	Potassium hydroxide
LDPE	Low density polyethylene
MA	Maleic anhydride
UV	Ultraviolet
PBAT	Poly(butylene adipate-co-terephthalate)
PBS	Polybutylene Succinate
PBSL	Poly (butylene succinate-co-L-lactate)
PF	Phenol formaldehyde
PCL	Polycaprolactone
PDLA	Poly (D-Lactide)
PDLLA	Poly(DL-Lactide)
PE	Polyethylene
PET	Poly(Ethylene terephthalate)
PET/PLA	Polyethylene terephthalate polylactic acid blends
PHA	Polyhydroxy acid
PHB	Polyhydroxyl butyrate
PLA	Poly (Lactic acid)
PLA-g-MA	Maleated Polylactic acid

PLLA	Poly (L-Lactide)
POE-g-MA	Maleated polyethylene octane
POP	Persistent organic pollutants
PP	Polypropylene
PP-g-MA	Maleated polypropylene
PS	Polystyrene
PVC	Polyvinyl chloride
ROP	Ring of polymerization
SEM	Scanning Emission Morphology
sPS-g-MA	Maleated syndiotactic polystyrene
TGA	Thermogravimetric Analysis
TM	Tensile modulus
TPS-g-MA	Maleated thermoplastic
TS	Tensile strength

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