

**MECHANICAL, THERMAL AND
FLAME RETARDANT PROPERTIES OF
POLY(LACTIC ACID)/
POLY(METHYL METHACRYLATE) BLENDS**

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**MECHANICAL, THERMAL AND FLAME RETARDANT
PROPERTIES OF POLY(LACTIC ACID)/
POLY(METHYL METHACRYLATE) BLENDS**

by

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LIST OF ABBREVIATIONS

APP	Ammonium polyphosphate
ASTM	American Society for Testing and Materials
CD	β -cyclodextrin
CCM	Cone calorimetry test
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
DTG	Derivative of thermogravimetric analysis
EDX	Energy dispersive X-ray
EG	Expanded graphite
ENR	Epoxidized natural rubber
FR	Flame retardant
GF	Glass fiber
HDT	Heat deflection temperature
HNT	Halloysite nanotubes
HRC	Heat release capacity
HRR	Heat release rate
IFR	Intumescent flame retardant
LLDPE	Linear low-density polyethylene
LOI	Limiting oxygen index
MC	Melamine cyanurate
MCAPP	Microencapsulated ammonium polyphosphate
MMT	Montmorillonite
MWNT	Multiwalled carbon nanotube

NR	No rating
OI	Oxygen index
OMMT	Organo-montmorillonite
PER	Pentaerythritol
PHRR	Peak of heat release rate
PLA	Poly(lactic acid)
PLLA	Semicrystalline PLA
PMMA	Poly(methyl methacrylate)
ROP	Ring opening polymerization
RP	Red phosphorus
SEM	Scanning electron microscope
TGA	Thermogravimetric analysis
TG-IR	Thermogravimetric analysis-infrared spectrometry analysis
TEM	Transmission electron microscope
THR	Total heat release
TTI	Time to ignition
UL	Underwriter Laboratory

LIST OF SYMBOLS

E'	Storage modulus
E''	Loss modulus
φ_i	Volume fraction of component i
ΔH_m	Melting enthalpy
ΔH_f	Enthalpy of fusion for 100% crystalline PLA
k	Fitting parameter for Gordon-Taylor equation
$\tan \delta$	Loss factor
T_{cc}	Cold crystallization temperature
T_d	End of decomposition temperature
T_g	Glass transition temperature
T_{gi}	Glass transition temperature of polymer component i
T_{max}	Temperature at maximum decomposition rate
T_m	Melting temperature
T_{o1}	Onset decomposition temperature at first stage
T_{o2}	Onset decomposition temperature at second stage
T_{10}	Temperature at 10% weight loss
χ_c	Degree of crystallinity
χ_{sp}	Interaction parameter between solvent and polymer
χ_{12}	Interaction parameter between polymer 1 and 2
V_i	Molar volume of polymer component i
w_{PLA}	Net weight fraction of PLA
w_i	Weight fraction of polymer component i

**SIFAT-SIFAT MEKANIKAL, TERMA DAN KETAHANAN PENYALAAAN
BAGI ADUNAN POLI(ASID LAKTIK)/POLI(METIL METAKRILAT)**

ABSTRAK

Adunan poli(asidlaktik)/poli(metilmetakrilat) (PLA/PMMA) telah disediakan dengan menggunakan teknik penyebatian leburan. Perencat nyala berasaskan fosforus telah digunakan dengan β -siklodekstrin (CD) untuk meningkatkan sifat-sifat ketahanan penyalaan dan terma bagi adunan PLA/PMMA. Pada peringkat pertama, kajian bertujuan untuk mengenal pasti tahap keterlarutcampuran bagi pelbagai nisbah campuran adunan PLA/PMMA (iaitu 80/20, 60/40, 40/60, dan 20/80). Penilaian dijalankan melalui pengukuran sifat haba [iaitu kalorimeter imbasan pembezaan (DSC) dan penganalisis mekanikal dinamik (DMA)] dan eksperimen pengambilan pelarut. Hanya satu suhu peralihan kaca dikesan pada semua nisbah campuran adunan PLA/PMMA semasa pengukuran DSC dan DMA. Eksperimen pengambilan pelarut menunjukkan bahawa adunan PLA/PMMA20 mempunyai parameter interaksi yang terendah ($\chi_{12} = -0.03$) menurut pengiraan parameter interaksi Flory-Huggins(χ_{12}). Dalam peringkat kedua, perencat nyala fosforus telah ditambah untuk meningkatkan sifat-sifat ketahanan penyalaan dan terma bagi adunan PLA/PMMA dengan nisbah campuran 80/20 dan 60/40. Ujian nyala tegak UL-94 dan indeks oksigen meneghad (LOI) telah dijalankan untuk mengenal pasti sifat ketahanan penyalaan adunan, manakala penganalisis termogravimetrik (TGA) telah digunakan untuk menentukan kestabilan terma adunan tersebut. Kedua-dua adunan PLA/PMMA20/FR dan PLA/PMMA40/FR berjaya mencapai tahap V-0 dalam ujian nyala, tanpa mengira nisbah campuran PLA/PMMA adunan. Keputusan TGA menunjukkan penambahan

FR mempercepat penguraian terma adunan PLA/PMMA20/FR dan PLA/PMMA40/FR pada suhu rendah, tetapi meningkatkan kestabilan terma adunan pada suhu tinggi. LOI yang lebih tinggi diperolehi oleh PLA/PMMA20/FR (iaitu 31.3%) daripada PLA/PMMA40/FR (iaitu 28.0%), menunjukkan bahawa PLA/PMMA20/FR mempunyai sifat ketahanan penyalaan yang lebih tinggi daripada PLA/PMMA40/FR. Dalam peringkat ketiga, separuh FR telah diganti dengan CD dan kecekapan kombinasi dalam meningkatkan sifat-sifat ketahanan penyalaan dan terma adunan PLA/PMMA telah dikaji. Semasa ujian kemudahbakaran, sifat menitis PLA/PMMA20/FR/CD telah meningkat dan ketahanan penyalaan masih dikekalkan (iaitu UL-94 V-0; LOI = 29.3%). Suhu penguraian terma maximum (T_{max}) yang lebih tinggi diperolehi oleh PLA/PMMA20/FR/CD semasa pengukuran TGA, disebabkan penguraian terma telah dilambatkan dalam kehadiran CD. Mikroskop elektron imbasan (SEM) menunjukkan arang yang banyak dan padat terbentuk pada permukaan pembakaran PLA/PMMA20/FR/CD, membuktikan bahawa CD adalah berkesan dalam membantu FR demi meningkatkan ketahanan penyalaan PLA/PMMA20/FR/CD.

MECHANICAL, THERMAL AND FLAME RETARDANT PROPERTIES OF POLY(LACTIC ACID)/POLY(METHYL METHACRYLATE) BLENDS

ABSTRACT

Poly(lactic acid)/poly(methyl methacrylate) (PLA/PMMA) blends were prepared by melt compounding technique. Phosphorus-based flame retardant (FR) was used together with β -cyclodextrin (CD) to improve the flame resistant and thermal properties of PLA/PMMA blends. In the first stage, study was focus on the evaluation on the miscibility of PLA/PMMA blends at various blending ratio (i.e., 80/20, 60/40, 40/60, and 20/80). The evaluation was performed through thermal properties measurement [i.e., differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA)] and solvent uptake experiment. Single glass transition temperature (T_g) was detected on PLA/PMMA blends over entire composition range during DMA measurement. The solvent uptake experiment showed that PLA/PMMA20 blend exhibited smallest interaction parameter ($\chi_{12} = -0.03$) according to the calculation of Flory-Huggins interaction parameter (χ_{12}). In the second stage, phosphorus-based flame retardant (FR; 20 part per hundred resin) was added to improve the flame resistant and thermal properties of PLA/PMMA blends at blending ratio of 80/20 and 60/40. UL-94 vertical burning test and limiting oxygen index (LOI) were conducted to characterize the flame resistant properties of the blends, while thermogravimetric analyzer (TGA) was used to determine the thermal stability of the blends. Both of the PLA/PMMA20/FR and PLA/PMMA40/FR successfully achieved V-0 rating in the burning test, regardless of the PLA/PMMA blending ratio. TGA results showed that addition of FR had accelerated the thermal decomposition of PLA/PMMA20/FR and

PLA/PMMA40/FR blends at low temperature, but improved the thermal stability of blends at high temperature. Higher LOI value was attained by PLA/PMMA20/FR (i.e., 31.3%) than PLA/PMMA40/FR (i.e., 28.0%), indicating PLA/PMMA20/FR having higher flame resistant properties than PLA/PMMA40/FR. In the third stage, half of the FR was replaced by CD and the efficiency of the combination in improving flame resistant and thermal properties of PLA/PMMA blend was investigated. During flammability tests, dripping behavior of the PLA/PMMA20/FR/CD was improved while maintaining its flame retardancy (i.e., UL-94 V-0; LOI = 29.3%). Higher maximum degradation temperature (T_{max}) was obtained for PLA/PMMA20/FR/CD during TGA measurement, suggested the thermal degradation was delayed in the presence of CD. Scanning electron microscope (SEM) showed an extensive and compact char layer was formed on the burning surface of PLA/PMMA20/FR/CD, proving that CD could be an effective adjuvant for FR in improving flame retardancy of PLA/PMMA20/FR/CD.

CHAPTER ONE

INTRODUCTION

1.1 Background of research

In recent years, sharp rise in environmental problems including natural resources depletion, white pollution, and waste disposal have been of great concern to all. The consumption and production of petroleum resource based plastics will become a problem if the persistent plastic wastes have not properly managed. Due to the rapid rise in the petroleum's price and limited petroleum resource, extensive researches have been done on the biodegradable polymer in order to develop eco-friendly and renewable plastics. One of the naturally renewable biodegradable polymer that have drawn attraction of academic and industrial interest is poly(lactic acid) (PLA).

Poly(lactic acid) (PLA) has emerging as a green alternative to petrochemical based polymers and successfully reaches commercial scale due to its advantageous properties, such as high modulus and strength, good processability, biocompatibility, good transparency, and nontoxicity (Vilay et al., 2010; Khankruea et al., 2014; Liang et al., 2014). It is derived from lactic acid made from renewable resources such as corn, rice, beets, sugarcane, wheat and potatoes (Pang et al., 2010). Besides, PLA possess several environmental advantageous over the conventional petrochemical-based polymers, such as biodegradability, less fuel energy consumption, and low greenhouse gas emission (Vink et al., 2004; Shukor et al., 2014). However, it still faces some drawbacks such as inherent brittleness (Krishnan et al., 2016), low thermal stability (Liu et al., 2012), and ease of ignition (Tao et al., 2011), which limit its application in electronic housing and automotive industries.

Modification of PLA through blending is a promising and economic approach to overcome its limitations. Polymer blending is a convenient industry process that able to improve the properties of existing materials by combining the unique properties of available materials. Several biodegradable and conventional synthetic polymers have been blended with PLA, such as polyamide 11 (Stoclet et al., 2011), natural rubber (Bitinis et al., 2011), ethylene-*co*-vinyl acetate (Ma et al., 2011), poly(hydroxybutyrate-*co*-hydroxyvalerate) (Gerard & Budtova, 2012), poly(3-hydroxybutyrate-*co*-3-hydroxyvalerate) (Zembouai et al., 2013), poly([R,S]-3-hydroxy butyrate) (Bartczak et al., 2013), poly(urethane) elastomer (Imre et al., 2013), and polycarbonate (Phuong et al., 2014). These PLA blend systems have demonstrated significant improvement in ductility and toughness compare to the neat PLA.

Poly(methyl methacrylate) (PMMA) is a transparent thermoplastic that being used in various applications, including automotives, outdoors, and electrical appliances. It is valued for several properties, such as good mechanical properties, high scratch resistance, outstanding weathering resistance, good surface hardness, and low water absorption capacity. Despite of its numerous advantageous, it still poses some limitations in toughness, thermal stability, flame resistant, and barrier properties. PMMA degrades and generates large amount of monomers during thermal decomposition. It is highly combustible material and releases heat, smoke, and toxic gas during combustion (Zeng et al., 2002b).

Recently reports showed that blending PLA with PMMA had drawn significant attention due to the miscibility of these blends. Zhang et al. (2003) had successfully prepared miscible blends comprises of amorphous poly(D,L-lactide)/PMMA and crystalline poly(L-lactide)/PMMA blends by solution and precipitation method. Woo and Wang (2012) had demonstrated the formation of a new