UNIVERSITI PUTRA MALAYSIA

EFFECT OF (3-AMINOPROPYL)TRIMETHOXYSILANE AND (3-AMINOPROPYL)TRIETHOXYSILANE ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF KENAF FIBER REINFORCED POLY(LACTIC ACID)/POLY(BUTYLENE ADIPATE-CO-TREREPHTHALATE) BLENDS

ANITH LIYANA BINTI MOHD SIS

EFFECT OF (3-AMINOPROPYL)TRIMETHOXYSILANE AND (3-AMINOPROPYL)TRIETHOXYSILANE ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF KENAF FIBER REINFORCED POLY(LACTIC ACID)/POLY(BUTYLENE ADIPATE-CO-TREREPHTHALATE) BLENDS

ANITH LIYANA BINTI MOHD SIS

MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA
2012

EFFECT OF (3-AMINOPROPYL)TRIMETHOXYSILANE AND (3-AMINOPROPYL)TRIETHOXYSILANE ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF KENAF FIBER REINFORCED POLY(LACTIC ACID)/POLY(BUTYLENE ADIPATE-CO-TREREPHTHALATE) BLENDS

ANITH LIYANA BINTI MOHD SIS

MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA
2012

EFFECT OF (3-AMINOPROPYL)TRIMETHOXYSILANE AND (3-AMINOPROPYL)TRIETHOXYSILANE ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF KENAF FIBER REINFORCED POLY(LACTIC ACID)/POLY(BUTYLENE ADIPATE-CO-TREREPHTHALATE) BLENDS

By

ANITH LIYANA BINTI MOHD SIS

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, In Fulfilment of the Requirements for the Degree of Master of Science

DECEMBER 2012

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

EFFECT OF (3-AMINOPROPYL)TRIMETHOXYSILANE AND (3-AMINOPROPYL)TRIETHOXYSILANE ON MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF KENAF FIBER REINFORCED POLY(LACTIC ACID)/POLY(BUTYLENE ADIPATE-CO-TREREPHTHALATE) BLENDS

By

ANITH LIYANA BINTI MOHD SIS

DECEMBER 2012

Chairman : Nor Azowa Binti Ibrahim, PhD

Faculty : Science

The effect of (3-aminopropyl)trimethoxysilane (APTMS) and (3-aminopropyl)triethoxysilane (APTES) on composite based poly(lactic acid) (PLA)/poly(butylene adipate-co-terephthalate) (PBAT) blends reinforced kenaf fiber has been prepared by using melt blending technique. Composites were blended for 15 minutes at 170°C until the blends became homogenized. Composites with the ratio of 90:10 PLA/PBAT blend and ratio of 90:10 PLA/PBAT blend with kenaf fiber have been characterized and the results revealed that the mechanical properties have decreased sharply with the addition of kenaf fiber.

However, addition of APTMS and APTES into PLA/PBAT blend reinforced kenaf fiber demonstrated the improvement of its mechanical properties up to 42.46%, 62.71% and 22.00% for tensile strength (52.27 MPa), flexural strength (64.27 MPa) and impact strength (39.79 J/m) respectively.

Morphological observation through scanning electron microscopy (SEM) reveals improved interaction and interfacial adhesion between PLA/PBAT blend and kenaf fiber with addition of APTMS. The fiber was well distributed and pulling into PLA/PBAT blend evenly. Dynamic mechanical analysis (DMA) result shows a decreased in storage modulus (E') for PLA/PBAT blend reinforced fiber, but addition of 2% APTMS, the E' increased. Conversely, the relative damping properties decreased.

Thermogravimetric analysis (TGA) thermogram showed improved thermal properties in the presence of APTMS. Differential scaning calorimetry (DSC) analysis was used to evaluate the crystallization of PLA/PBAT/Kenaf composites. Addition of APTMS, cause the width of crystallization peaks to reduce indicating of an increase in crystallization rate. The composite also shows the inward shifting of melting peaks of polymer constituent indicating improved compatibility between PLA/PBAT and Kenaf.

Beside APTMS, the effect of adding APTES as coupling agent has been investigated. However, it produced contradictory result as addition of 2% APTES does not show any significant changes either in mechanical or thermal properties. The tensile strength increase only by 1.69%, nevertheless, the flexural strength and impact

strength decrease to the tune of 0.05% and 8.39% respectively. These results was supported by SEM micrographs where some of kenaf fiber still pull out after addition of APTES. Moreover, based on DMA graph, composite with APTES added displays lower E' compared to the composite modified by APTMS.

TGA thermogram reveals composite modified by APTES displays 81.32% of degradation, which was 8.48% lower than polymer degradation of composite modified by APTMS. Whereas, differential thermogravimetric (DTG) curve demonstrated there was no significant effect between composite modified by APTMS and APTES since composite/APTMS demonstrated thermal stability at 308.30°C.

Furthermore, the degradation test on the composite by soil buried in laboratory and at landfill area has been carried out in order to ensure the composite is an environmental friendly materials. Therefore, within nine months, result indicates that presence of kenaf fiber into PLA/PBAT matrix was induced the degradability rate. The weight loss of PLA/PBAT/Kenaf was increase up to 7.1% (soil in laboratory) and 5.48% (soil at landfill area) compared to the PLA/PBAT blend which displays weight loss only by 0.24% and 0.78% respectively. Modification by silane coupling agent, PLA/PBAT/Kenaf/APTMS and PLA/PBAT/Kenaf/APTES shows 5.66% and 6.12% of weight loss respectively for sample that has been soil buried in laboratory. Whereas, demonstrated 6.22% (PLA/PBAT/Kenaf/APTMS) and 6.58% (PLA/PBAT/Kenaf/APTES) of weight loss for sample buried in soil at landfill area.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN (3-AMINOPROPIL)TRIMETOKSISILANE DAN (3-AMINOPROPIL)TRIETOKSISILANE KE ATAS MEKANIKAL, TERMAL DAN MORFOLOGI CAMPURAN POLI(LAKTIK ASID)/POLI(BUTILENA ADIPAT-KO-TEREFTALAT) DAN KENAF FIBER

By

ANITH LIYANA BINTI MOHD SIS

DISEMBER 2012

Pengerusi : Nor Azowa Binti Ibrahim, PhD

Fakulti : Sains

Kesan (3-aminopropil)trimetoksisilane (APTMS) dan (3-aminopropil)trietoksisilane (APTES) ke atas komposit yang berdasarkan pada adunan poli(laktik asid) (PLA) dan poli(butilena adipat-ko-tereftalat) (PBAT) yang decampurkan bersama serat kenaf telah disediakan dengan menggunakan kaedah mencairkan adunan. Komposit ini diadun untuk 15 minit pada 170 darjah selsius sehingga campuran menjadi sebati. Komposit dengan isbah campuran 90:10 PLA /PBAT dan nisbah campuran PLA/PBAT (90:10) dengan serat kenaf telah dikenalpasti dan keputusan menunjukkan bahawa ujian sifat mekanik telah menurun secara mendadak bagi PLA/PBAT yang diadun bersama serat kenaf.

Walaubagaimanapun, pengubahsuaian dengan tambahan APTMS dan APTES ke dalam adunan PLA/PBAT/Kenaf telah dijalankan. Hasil kajian menunjukkan pengubahsuaian oleh 2% APTMS menunjukkan peningkatan sifat mekanikal sehingga 42.46%, 62.71% dan 22.00% untuk ujian kekuatan tegangan (52.27 MPa), kekuatan lenturan (64.27 MPa) dan kekuatan impak (39.79 J/m).

Pemerhatian morfologi melalui mikroskop elektron imbasan (SEM) menunjukkan interaksi mekanisma dan lekatan antara permukaan PLA/PBAT dan serat kenaf bertambah baik dengan penambahan APTMS. Gentian kenaf telah disebarkan secara menyeluruh dan sama rata ke dalam campuran PLA/PBAT. Dinamik mekanikal analisis (DMA) menunjukkan penurunan dalam kekuatan modulus bagi campuran PLA/PBAT/Kenaf namun,menunjukkan peningkatan apabila campuran komposit tersebut ditambah 2% APTMS.

Selain itu, lengkungan termogravimetri analisis (TGA) menunjukkan ciri-ciri terma bertambah baik dalam kehadiran APTMS. Seterusnya, DSC telah digunakan untuk menilai penghabluran komposit PLA/PBAT/Kenaf, penambahan APTMS telah menyebabkan lebar puncak kristal berkurang dimana ini menunjukkan peningkatan dalam proses pengkristalan. Komposit ini juga menunjukkan penurunan nilai dalam proses peleburan dan ini menunjukkan interaksi yang lebih baik antara campuran PLA/PBAT dan serat kenaf.

Satu lagi pengubahsuaian ke atas komposit dengan menggunakan APTES sebagai ejen gandingan juga telah dilakukan. Walau bagaimanapun, ia menghasilkan keputusan yang bercanggah. Tambahan 2% APTES tidak menunjukkan perubahan

ketara sama ada dalam sifat-sifat mekanik atau terma. PLA/PBAT/Kenaf/APTES menunjukkan sedikit peningkatan kekuatan tegangan iaitu hanya 1.69%, namun, kekuatan lenturan dan kekuatan impak menunjukkan pengurangan lebih kurang 0.05% dan 8.39% masing-masing. Keputusan ini telah disokong oleh morfologi dari SEM di mana menunjukkan bahawa serat kenaf serat masih tertarik keluar daripada campuran PLA/PBAT. Berdasarkan lengkungan DMA, komposit yang diubahsuai dengan APTES menunjukksn kekuatan modulus yang lebih rendah berbanding komposit yang diubahsuai oleh APTMS.

Lengkung TGA mendedahkan komposit yang diubahsuai oleh APTES menunjukkan 81.32% degradasi, yang mana ia adalah 8.48% lebih rendah daripada degradasi polimer komposit yang telah diubahsuai oleh APTMS. Manakala, berdasarkan lengkungan DTG pula, ia menunjukkan tiada kesan yang ketara antara komposit diubahsuai oleh APTMS atau APTES.

Tambahan pula, ujian degradasi apabila sampel ditanam di dalam tanah yang dilakukan di dalam makmal dan di kawasan tapak pelupusan telah dijalankan untuk memastikan komposit ini adalah bahan mesra alam. Oleh itu, dalam tempoh sembilan bulan, hasilnya menunjukkan bahawa kehadiran serat kenaf ke dalam campuran PLA/PBAT telah meningkatkan kadar degradasi. Kehilangan berat PLA/PBAT/Kenaf telah meningkat sehingga 7.1% (tanah di makmal) dan 5.48% (tanah di kawasan tapak pelupusan) berbanding dengan campuran PLA/PBAT yang memaparkan penurunan berat badan hanya dengan masing-masing 0.24% dan 078%. Pengubahsuaian oleh ejen gandingan silane, PLA/PBAT/Kenaf/APTMS dan PLA/PBAT/Kenaf/APTES menunjukkan 5.66% dan 6.12% untuk kehilangan berat

masing-masing bagi sampel yang telah tanah ditanam di dalam makmal. Sementara itu, menunjukkan 6.22% (PLA/PBAT/Kenaf/APTMS) dan 6.58% (PLA/PBAT/Kenaf/APTES) menunjukkan kehilangan berat bagi sampel ditanam di tanah di kawasan tapak pelupusan.

ACKOWLEDGEMENT

I would like to express my sincere appreciation to my project supervisor, Dr. Nor Azowa Ibrahim for her encouragement, guidance and support throughout this research project. Beside, I would also like to extend my gratitude to my co-supervisors, Prof. Dato' Dr. Wan Md Zin Wan Yunus and Dr. Yusran Sulaiman. Thanks for their supervision throughout the research.

Special thanks to all staffs at Faculty of Science, Universiti Putra Malaysia for their helps and advice. Furthermore, my deepest gratitude to Cheing Buong Woei and Then Yoon Yee, my seniors who helped me a lot during experiments. My sincere appreciation also extends to all my colleagues in polymer group and others who have provided help at various occasions. Their views and tips are useful indeed.

In addition, I would like to gratefully acknowledge the sponsorship from National Science Fellowship (NSF), Ministry of Science, Technology and Innovation (MOSTI).

Last but not least, I am internally indebted to my parents for always being believe in me. A lot of appreciate for their guidance, care and support during the hardest time in my dissertation. Thanks a lot.

I certify that a Thesis Examination Committee has met on 7th December 2012 to conduct the final examination of Anith Liyana Binti Mohd Sis on her thesis entitled "Effect Of (3-Aminopropyl)trimethoxysilane and (3-Aminopropyl)triethoxysilane on Mechanical, Thermal and Morphological Properties of Kenaf Fiber Reinforced Poly(Lactic Acid)/Poly(Butylene Adipate-Co-Trerephthalate) Blends" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiyi Putra Malaysia [P.U(A) 106] 15 March 1998. The Committee recommends that the student be awarded the degree of Master of Science.

Members of the Thesis Examination Committee were as follows:

Mohamed Ibrahim Mohamed Tahir, PhD
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Mansor Hj Ahmad, PhD
Assoc. Prof.
Faculty of Science
Universiti Pertahanan Nasional Malaysia
(Member)

Mohamad Zaki Abd Rahman, PhD
Assoc. Prof.
Faculty of Science
Universiti Putra Malaysia
(Member)

BUJANG KIM HUAT, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science.The members of Supervisory Committe were as follows:

Nor Azowa Ibrahim, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Wan Md Zin Wan Yunus, PhD
Professor
Faculty of Science
Universiti Pertahanan Nasional Malaysia
(Member)

Yusran Sulaiman, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

_____

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declare that the 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 Putra Malaysia or other institutions.

_____

ANITH LIYANA BINTI MOHD SIS

Date: 7 DECEMBER 2012

TABLE OF CONTENT