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-TEREPTHALATE) BLENDS

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FS 2012 74
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By

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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
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 scanning 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.
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

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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. Tambah 2% APTES tidak menunjukkan perubahan
ketara sama ada dalam sifat-sifat mekanik atau terma. PLA/PBAT/Kenaf/APTES menunjukkan sedikit peningkatan kekuatan kegangan 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 menunjukkan 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.
ACKNOWLEDGEMENT

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 Universiti Putra Malaysia [P.U(A) 106] 15 March 1998. The Committee recommends that the student be awarded the degree of Master of Science.

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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

<table>
<thead>
<tr>
<th>ABSTRACT</th>
<th>ii</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRAK</td>
<td>v</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENT</td>
<td>ix</td>
</tr>
<tr>
<td>APPROVAL</td>
<td>x</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>xii</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>xvii</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xx</td>
</tr>
<tr>
<td>LIST OF ABBREVIATION</td>
<td>xxi</td>
</tr>
</tbody>
</table>

## CHAPTER

### 1 INTRODUCTION

1.1 General Introduction 1
1.2 Problem Statement 3
1.3 Scope of Research 5
1.4 Research Objective 6

### 2 LITERATURE REVIEW

2.1 Preliminary Study 7
2.1.1 Non Degradable Plastic 7
2.1.2 Degradable Plastic 9

2.2 Composite 11
2.2.1 Advantages of Composite 12
2.2.2 Application in Industry 13

2.3 Thermoplastic 13

2.4 BiodegradablePolymer 14
2.4.1 Poly(lactic acid) 15
2.4.2 Poly(butylene adiphate-co-terephthalate) 17
2.4.3 Polymer Blend (PLA/PBAT Blend) 18
2.5 Fiber as Reinforcement
  2.5.1 Advantages of Fiber
  2.5.2 Introduction of Kenaf

2.6 Problems on Polymer/Fiber Composite
  2.6.1 Fiber Loading
  2.6.2 Thermal Properties

2.7 Modification of Polymer/Fiber Composite Interaction
  2.7.1 Compatibilizer
  2.7.2 Coupling Agent

2.8 Modification by Silane Coupling Agent
  2.8.1 Chemical Structure of Silane Coupling Agent

2.9 Blending
  2.9.1 Mixing Duration

3 MATERIALS AND METHODS

3.1 Materials

3.2 Experimental Stages

3.3 Preparation of Composite
  3.3.1 Compounding of composite Materials
  3.3.2 Effect of PBAT Loading
  3.3.3 Effect of Kenaf Loading
  3.3.4 Effect of APTMS/APTES Loading
  3.3.5 Sample Preparation

3.4 Mechanical Testing
  3.4.1 Tensile Test
  3.4.2 Flexural Test
  3.4.3 Impact Izod Test
  3.4.4 Water Absorption Test
  3.4.5 Degradability Test
3.5 Characterization of The Composites 47
3.5.1 Scanning Eelectron Microscopy (SEM) 47
3.5.2 Thermogravimetric Analysis (TGA) 47
3.5.3 Differential Scanning Calorimetry (DSC) 48
3.5.4 Dynamic Mechanical Analysis (DMA) 48

4 RESULTS AND DISCUSSION

4.1 Tensile Propertise of Composite 50
4.1.1 Effect of PBAT Loading on Tensile Strength and Tensile Modulus of PLA/PBAT Blend 50
4.1.2 Effect of Kenaf Fiber Loading on Tensile Strength and Tensile Modulus of PLA/PBAT/Kenaf 52
4.1.3 Effect of APTMS and APTES Loading on Tensile Strength and Tensile Modulus of PLA/PBAT/Kenaf/APTMS and PLA/PBAT/Kenaf/APTES 54

4.2 Flexural Propertise of Composite 58
4.2.1 Effect of PBAT Loading on Flexural Strength and Flexural Modulus of PLA/PBAT Blend 58
4.2.2 Effect of Kenaf Fiber Loading on Flexural Strength and Flexural Modulus of PLA/PBAT/Kenaf 59
4.2.3 Effect of APTMS and APTES Loading on Flexural Strength and Flexural Modulus of PLA/PBAT/Kenaf/APTMS and PLA/PBAT/Kenaf/APTES 60

4.3 Impact Propertise of Composite 63
4.3.1 Effect of PBAT Loading on Impact Strength of PLA/PBAT Blend 63
4.3.2 Effect of Kenaf Fiber Loading on Impact Strength of PLA/PBAT/Kenaf 64
4.3.3 Effect of APTMS and APTES on Impact Strength of PLA/PBAT/Kenaf/APTMS and PLA/PBAT/Kenaf/APTES 65
4.4 Scanning Electron Microscopy (SEM) 71

4.5 Thermal Properties 72
   4.5.1 Thermogravimetric Analysis (TGA) 72
   4.5.2 Differential Scanning Calorimetry (DSC) 76

4.6 Dynamic Mechanical Analysis (DMA) 78
   4.6.1 Storage Modulus of Composite 78
   4.6.2 Tan Delta of Composite 80

4.7 Water Absorption Test 82
   4.7.1 Percentage of Water Uptake 82

4.8 Biodegradability Test 84
   4.8.1 Percentage of Weight Loss 84

5 CONCLUSIONS

5.1 Conclusion 87

5.2 Recommendations 88

REFERENCES 90
BIODATA OF STUDENT 102