FABRICATION OF PLA/COW DUNG-BASED BIOCOMPOSITE

MOSTAFA YUSEFI
FABRICATION OF PLA/COW DUNG-BASED BIOCOMPOSITE

By

MOSTAFA YUSEFI

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

December 2015
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Dedicated to My Beloved Parents
And
Nature
Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the Degree of Master of Science

FABRICATION OF PLA/COW DUNG-BASED BIOCOMPOSITE

By

MOSTAFA YUSEFI

December 2015

Chairman : Faizah Md. Yasin. PhD

Faculty : Engineering

Various environmental drawbacks such as reduction in land fill space and non-biodegradability lead to systematically investigate the replacing of synthetic composites by using biocomposites. Biocomposites possess suitable characterizations such as light weight, combustible, nontoxic, and biodegradability behavior. However, the final product of the biocomposites carries certain drawbacks in terms of mechanical, physical and thermal properties. In order to determine their characteristic, this research aided to figure out the possibility of applying cow dung (CD) as filler to prepare polylactic acid (PLA) biocomposite. The main objective of this research is to obtain a suitable composition ratio based on the filler and the hosting polymer.

The CD of two different sizes, namely 4.00 mm and 0.5 mm were blended with PLA. PLA/CD biocomposites with different CD ratios (0-60 wt.%) were fabricated using an internal Brabender mixer (W50EHT-3zones) followed by a 40 tones hydraulic compression moulding. The results showed that the addition of CD led to improve flexural properties compared to tensile and impact strength. Biocomposites with 4.00 mm CD (bigger filler) mainly showed higher mechanical properties than those of 0.5 mm CD (smaller filler). Scanning electron microscopy (SEM) of tensile and impact fractured surfaces indicated that the bigger fillers had stronger adhesion and bonding with the matrix. Moreover, the cavities and rough surface of biocomposites increased with the filler content addition. This led to lower mechanical and physical properties of the biocomposites and increased water uptake during water absorption test accordingly. Dynamic mechanical analysis (DMA) technique was also followed to determine both storage and loss modulus of the samples. Neat PLA indicted the lowest storage modulus, while the filler content addition generally improved the storage modulus. Results of thermogravimetric analysis (TGA) indicated that the addition of the filler content prolonged the major degradation temperature. This was due to the higher resistance of the CD filler to the degradation temperature, which induced higher thermal stability of CD compared to the neat PLA.
Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains.

FABRIKASI BIOKOMPOSIT BERAASASKAN PLA/TAHI LEMBU

Oleh

MOSTAFA YUSEFI

Disember 2015

Pengerusi : Faizah Md. Yasin, PhD
Fakulti : Kejuruteraan

Pelbagai kelemahan alam sekitar seperti pengurangan dalam ruang mengisi tanah dan kemerosotan selain daripada segi bio telah menggantikan komposit sintetik dengan menggunakan biokomposit. Biokomposit mempunyai pencirian yang sesuai seperti ringan, mudah terbakar, tanpa toksik, dan kemerosotan dari segi bio. Walau bagaimanapun, produk akhir daripada biokomposit membawa kelemahan tertentu dari segi sifat-sifat mekanikal, fizikal dan terma. Dalam usaha untuk menentukan ciri-cirinya, penyelidikan ini telah dijalankan untuk mengkaji kemungkinan penggunaan tahi lembu (CD) sebagai pengisi untuk menyediakan asid polylactic (PLA) biokomposit. Objektif utama kajian ini adalah untuk mendapatkan nisbah komposisi sesuai berdasarkan pengisi dan polimer hosting.

Terdapat dua saiz CD yang berbeza, iaitu 4.00 mm dan 0.5 mm yang telah dicampur dengan PLA. Biokomposit PLA / CD dengan nisbah CD yang berbeza (0-60 wt.%) telah direka dengan menggunakan pembancuh Brabender dalaman (W50EHT-3zones) diikuti dengan 40 nada pengacuan mampatan hidraulik. Hasil kajian menunjukkan bahawa penambahan CD membawa kepada peningkatan dari segi kelenturan berbanding dengan ketegangan dan kekuatan. Biokomposit bersaiz 4 mm CD (pengisi lebih besar) menunjukkan sifat-sifat mekanikal lebih tinggi daripada 0.5 mm CD (pengisi lebih kecil). Mikroskop imbasan elektron (SEM) tegangan dan kesan permukaan patat menunjukkan bahawa pengisi lebih besar mempunyai lekatan yang kuat dan ikatan dengan matriks. Selain itu, rongga dan permukaan kasar biokomposit juga meningkat dengan kandungan pengisi. Ini telah menyebabkan penurunan sifat-sifat mekanikal, fizikal biokomposit dan juga peningkatan pengambilan air semasa ujian penyerapan air. Analisis mekanikal teknik dinamik (DMA) juga digunakan untuk menentukan simpanan dan kehilangan modulus sampel. PLA yang tulen menunjukkan simpanan modulus yang paling rendah, manakala peningkatan kandungan pengisi secara amnya menambah baik modulus simpanan. Keputusan analisis Termogravimetri (TGA) menunjukkan bahawa penambahan kandungan pengisi telah memanjangkan suhu kemerosotan. Ini disebabkan oleh rintangan pengisi CD yang lebih tinggi kepada suhu degradasi, dan menunjukkan kestabilan haba CD yang lebih tinggi berbanding dengan ketulenan PLA.
ACKNOWLEDGEMENTS

An investigation is an arduous job, which requires obligation to achieve the specified objectives. This work has successfully accomplished by patronage of many identities. I am really grateful to all persons who kindly help to finish this research. First and foremost, I would like to express a great appreciation to my advisor Dr. Faizah Md. Yasin for her advices, honest encouragements and supports. I have been absolutely lucky that the wheel of fortune made a perfect opportunity for me to know Prof. Dr. Luqman Chuah Abdullah, Dr. Mohammad Khalid Siddiqui and Dr. Chantara Thevy Ratnam, my supervisory committee. I am very thankful to them that they always respond my questions throughout the research and without their wholehearted support this research was not feasible. I also appreciate the Head of the Department Dr. Mohamad Amran Mohd Salleh for his cooperation and excellent knowledge.

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Lastly, I really like to express my honest impression and appreciation to my parents who always have heartily behind me to do my best.
I certify that a Thesis Examination Committee has met on 18 December 2015 to conduct the final examination of Mostafa Yusefí on his thesis entitled "Fabrication of PLA/COW Dung-Based Biocomposite" 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 Master of Science

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<tr>
<td>$E^*$</td>
<td>Storage Modulus</td>
</tr>
<tr>
<td>$E'$</td>
<td>Loss Modulus ($E''$)</td>
</tr>
<tr>
<td>$l$</td>
<td>fiber length</td>
</tr>
<tr>
<td>$l_c$</td>
<td>Critical length</td>
</tr>
<tr>
<td>$\tan \delta$</td>
<td>Mechanical Damping</td>
</tr>
<tr>
<td>$\sigma^*_f$</td>
<td>Fiber tensile strength</td>
</tr>
<tr>
<td>$\sigma_{fu}$</td>
<td>Fiber ultimate strength in tension</td>
</tr>
<tr>
<td>$\tau_y$</td>
<td>Interfacial shear stress</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
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<td>CD</td>
<td>Cow Dung</td>
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<td>DMA</td>
<td>Dynamic Mechanical Analysis</td>
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<tr>
<td>INTROP</td>
<td>Institut Perhutanan Tropika &amp; Produk Hutan</td>
</tr>
<tr>
<td>PLA</td>
<td>Polylactic Acid</td>
</tr>
<tr>
<td>PLLA</td>
<td>Poly-l-lactic acid</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>RGP</td>
<td>Refiner Ground Pulp</td>
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<tr>
<td>RH</td>
<td>Relative Humidity</td>
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<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
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<td>TG</td>
<td>Thermogravimetric</td>
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<td>TGA</td>
<td>Thermogravimetric Analysis</td>
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<td>DTG</td>
<td>Derivative Thermal Gravimetric</td>
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<td>FEG</td>
<td>field emission guns</td>
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<tr>
<td>$X_c$</td>
<td>crystallinity</td>
</tr>
<tr>
<td>$T_c$</td>
<td>temperature of crystallization</td>
</tr>
<tr>
<td>$\Delta H_f$</td>
<td>heat of fusion</td>
</tr>
<tr>
<td>CL</td>
<td>cotton linear</td>
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<td>WF</td>
<td>wood fiber</td>
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<td>CFs</td>
<td>chicken feathers</td>
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<td>C</td>
<td>cellulose</td>
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<td>SGC</td>
<td>silane-grafted cellulose</td>
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CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Overusing of synthetic plastic causes several environmental drawbacks such as reduction in landfill space, natural resources depletion and non-biodegradability. This leads to replacing of the synthetic plastic or composites by using biocomposites. Biocomposites possess suitable characterizations such as lightness in weight, combustible, nontoxic, low cost and low density. However, their final production lacks assemblage of mechanical, thermal and physical properties. In addition, biocomposites have some disadvantages such as low thermal stability during processing, high moisture absorption, ultraviolet light resistance and a relatively high price at the current low volume production. Numerous researchers attempt to develop a natural composite for various applications in the automotive, construction, agriculture, building industries and etc (Auras et al., 2004; Joshi et al., 2004). Recently, researchers have applied new materials and modern manufacturing techniques to improve strength of biocomposites.

Since the 1990s, polylactic acid (PLA) has been known as one of the most compostable and renewable polymers. PLA can be derived from renewable resources like corn, scratch, sugarcane, potato and renewable feedstock by bacterial fermentation. It has excellent properties such as stiffness, low energy consumption, low production temperature and biodegradability (Sawyer & Grubb, 1996). It is a linear aliphatic thermoplastic polyester, which is fabricated either by condensation of lactic acid or the ring-opening polymerization of lactic (Garlotta, 2001). However, PLA has its own disadvantages such as inherent brittleness, weak thermal resistance and limited gas barrier. To use PLA in different applications including biomedical, mulching film, packaging and tissue, its undesirable properties such as inherent brittleness, weak thermal resistance and limited gas barrier have been improved by mixing different kinds of fillers and fibers; flax (Bax & Müssig, 2008), kenaf (Han et al., 2012) and nanoparticles (nanaoclays) (Paul et al., 2005).

After chicken, cattle are the second populous livestock in the world with the approximate number of 1.4 billion. As a 450 kg cow produces around 3 kg dung per day, the daily production of dung is virtually 4.2 million tonnes. In addition, it possesses a wide range of protein, lignocellulose, light weight, low cost, a capability as a good filler and it is an animal based fiber with biodegradability behavior (Reddy et al., 2014). It should be noted that to decrease the problems of unusable cow dung (CD) like environmental damages and pollution to ground water, the CD needs improvement of its physical and chemical structure for usage in various applications.

In this study, biocomposite of PLA/CD is systematically described as a composite derived from 100% renewable resources. The additional effects of the two different sizes of CD filler were studied on the mechanical, physical, morphological and thermal properties of PLA/CD biocomposite.
1.2 Problem Statement

Different ratios of fillers and polylactic acid (PLA) are molded to produce biocomposite materials. Biocomposite materials are broadly utilized in many structural applications such as in panels, parts, boards and sheets. For this aim, the sample’s properties play a key role with respect to their applications including compost bags, tea bags, food packaging and also mulch film. Therefore, it is necessary to study the mechanical, thermal and physical properties of the biocomposite materials. PLA has a brittle microstructure and it also has a high cost. Besides that, high amount of un-usable cow dung (CD) results in environmental pollution to ground water. Therefore, to decrease the drawbacks of both PLA and CD, the effect of CD to PLA biocomposite properties is systematically studied in this first hand study.

1.3 Objective

The aim of this study is attempted to prepare suitable biocomposite samples and investigate their mechanical, thermal, and physical properties. The influence of this study will aid to figure out the possibility of applying cow dung (CD) as filler to produce polylactic acid (PLA) biocomposite. The ability of manufacturing development of the biocomposite will be understood from this study as using of CD filler can decrease final price of the PLA biocomposite.

To completely obtain the final objective of this study, the following contributions will be investigated.

1) The fabrication of the biocomposite films at different CD ratios and sizes.

2) The assessment of the mechanical, physical, thermal, and dynamic properties of the fabricated biocomposites.

3) The study of the interfacial interaction using morphological analysis.

This research will evaluate suitable methods to obtain main contributions of the relation between the samples preparation and their characterizations.

1.4 Scope

Using biodegradable substances has become more popular in the modern societies due to increasing of plastic waste and environmental problems. However, a major disadvantage of the biocomposite is poor adhesion between matrix and filler that it leads to weak physical, thermal and mechanical properties. Sorts of natural polymers with different natural fillers have been examined to fabricate strong biocomposite samples. Advantages of natural filler (low cost, light weight and low density) may improve the mechanical, thermal, morphological and dynamic properties of polymer composites with lower cost. The high temperature of biocomposite fabrication influences the mechanical stress that it leads to their thermal and mechanical degradation (Klemm, 1998). This effects the strength of the final biocomposite samples (Gassan & Bledzki, 2001). Polylactic acid (PLA) is comprised of different natural fillers to improve its strength. Cow dung with its sufficient cellulose content can be used as a new natural filler to improve mechanical, thermal, dynamic and morphological properties of PLA composites. In addition, CD with its low cost and biodegradability behavior can decrease the final cost of PLA production.
1.5 **Significance of the Study**

This research has taken the full advantages of polylactic acid (PLA) matrix and cow dung (CD) filler for PLA/CD biocomposite samples preparation. Low cost of CD and suitable properties of the PLA lead to the obtainment of the low final cost and improvement of the mechanical and dynamic properties of the biocomposite, respectively. Besides, PLA samples comprise of various sizes of CD filler in different content ratios which leads to the improved formulation of PLA/CD biosomposite. Therefore, different biocomposite formulations can be used for all sorts of applications such as mulch film, disposable tableware as well as food container. The consequence of this study will lead to understanding the ability of replacing synthetic composite using PLA/CD biocomposite which is 100 % biodegradable to decline the environmental problems. Therefore, the mechanical, physical, thermal and morphological properties of PLA/CD biocomposite are evaluated, systematically.

1.6 **Chapter Outline**

Chapter 1 briefly describes introduction, problem statement and objectives of this research.

Chapter 2 is “Literature Review”, which explains a review of the previous works on types of composites, natural fiber or filler and polymers. It presents the challenges and suitable properties of different composites and ends with the summary.

Chapter 3 is “Research Methodology”, which presents the methodology of this research. It outlines the materials, method of PLA/CD biocomposites preparation and the apparatuses employed for all sorts of mechanical, thermal, physical and morphological analysis and tests.

Chapter 4 is “Results and Discussion”, which explains and discusses the results and findings of mechanical, physical, thermal and morphological analysis and tests of PLA/CD biocomposites.

Chapter 5 is “Conclusion”, which gives an outline of all the conclusions that have been obtained from this study and also presents the direction for future research.
REFERENCES


Molins, Gemma, *et al.* CHICKEN FEATHERS BASED BIOCOMPOSITES: A NEW SUSTAINABLE PRODUCT?


