# *CHARACTERIZATION OF MILLET HUSK-FILLED HIGH DENSITY POLYETHYLENE AND POLYLACTIC ACID COMPOSITES*

**HAMMAJAM A. ABBA**

# CHARACTERIZATION OF MILLET HUSK-FILLED HIGH DENSITY POLYETHYLENE AND POLYLACTIC ACID COMPOSITES

By

**HAMMAJAM A. ABBA**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy**

**January 2018**

# DEDICATION

This thesis is dedicated to my family and the entire ummah for their support.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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**January 2018**

**Chairman** : **Associate Professor Nur Ismarrubie Bt Zahari, PhD**
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The millet husk fiber (MHF) is an agricultural byproduct from millet (*pennisetum glaucum*). Currently, the use of thermoplastic composites produced from polymers filled with natural fibers has attracted the attention of many researchers globally. The main objective of this investigation was to study the potential of this agro waste as filler for thermoplastic composites as a substitute to synthetic fibers and other natural fibers. The fibers were treated with sodium hydroxide (NaOH) and pulverized to 250 µm. Fiber loadings of 10 %, 20 % 30 % and 40 % by weight were employed throughout the formulation. Test samples were prepared through melt blend technique followed by compression molding process. The mechanical properties; tensile, flexural and impact test specimens were specified according to ASTM standards and tested using universal testing machine (UTM). The thermal properties were characterized by using thermogravimetric analyzer (TGA).The microstructure observations of fractured surfaces of composites were studied by using scanning electron microscope (SEM).The investigation of water absorption in sea and rain water was determined. The biodegradability was carried out via soil burial technique in municipal and oxisol soils.

Tensile strength of untreated fiber millet husk powder (MHP) filled high density polyethylene (HDPE) and MHP filled poly lactic acid (PLA) composites decreased with increased fiber loading but slightly improved for treated fibers composites. While the tensile modulus of both treated and untreated fibers composites increased by increasing the fiber loadings. Flexural strength of the MHP-HDPE treated fiber composites increased as loading increased, while MHP-PLA composites decreased as loading increased. PLA composites exhibit better flexural properties for treated fiber composites. The flexural modulus for both treated and untreated fiber composites of MHP-PLA and MHP-HDPE increased as fiber loadings increases. The impact properties of treated and untreated fibers MHP-HDPE, MHP-PLA, composites drastically decreased as fiber loading increased. Water absorption increased as fiber loading increased. Composites immersed in sea water uptake at higher rate compare to those immersed in rain water. Thermal degradations of untreated and treated fiber composites show slight difference in terms of weight loss, while decomposition rate varies as the fiber loading increased. Biodegradability in municipal soil showed higher rate of degradation compared to oxisol soil. The degradation level for treated and untreated fiber composites differs slightly in both types of soil. MHP-PLA composites degrade faster in both municipal and oxisol soil compare to MHP-HDPE composites. The fiber treatment has little impact on their biodegradability rates. In view of all the findings, it was appropriate to conclude that millet husk fiber filled thermoplastic is a promising composite.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

# PENCIRIAN SEKAM BIJIRIN YANG DIPENUHI POLIETILENA KETUMPATAN TINGGI DAN KOMPOSIT ASID LAKTIK POLI

Oleh

**HAMMAJAM A. ABBA**

**Januari 2018**

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Serat sekam millet (MHF) adalah hasil sampingan dari millet (pennisetum glaucum). Pada masa ini, penggunaan komposit termoplastik yang dihasilkan daripada polimer bercampur dengan serat semulajadi telah menarik perhatian ramai penyelidik di seluruh dunia. Objektif utama penyelidikan ini adalah untuk mengkaji potensi sisa agro ini sebagai pengisi untuk komposit termoplastik menggantikan serat sintetik dan serat semula jadi lain. Serat ini telah dirawat dengan natrium hidroksida (NaOH) dan dilumatkan kepada 250μm. Pemuatan serat sebanyak 10%, 20% 30% dan 40% berat digunakan sepanjang formulasi. Sampel ujian disediakan melalui teknik campuran cair yang diikuti dengan proses pengacuan mampatan. Sifat mekanik; spesimen uji tegangan, lenturan dan kesan ditentukan mengikut piawaian ASTM dan diuji menggunakan mesin ujian sejagat (UTM). Ciri-ciri termal telah dicirikan dengan menggunakan penganalisis termogravimetrik (TGA). Pemerhatian mikrostruktur permukaan patah komposit telah dikaji dengan menggunakan mikroskop elektron scanning (SEM). Penyiasatan penyerapan air di laut dan air hujan telah ditentukan. Keabadian biodegradasi dilakukan melalui teknik pengebumian tanah di tanah perbandaran dan oxisol.

Kekuatan tegangan serat sekam tepung dedaunan (MHP) yang tidak diisi polietilena ketumpatan tinggi (HDPE) dan komposisi asid laktik poli (MHA) yang diisi dengan MHP berkurangan dengan peningkatan beban gentian tetapi sedikit bertambah baik untuk komposit serat terawat. Walaubagaimanapun modulus tegangan kedua-dua komposit gentian yang terawat dan tidak dirawat meningkat dengan meningkatkan beban serat. Kekuatan lenturan komposit serat MHP-HDPE yang dirawat meningkat apabila beban meningkat, manakala komposit MHP-PLA menurun apabila beban meningkat. Komposit PLA mempamerkan sifat lentur yang lebih baik untuk komposit serat yang terawat. Modulus lenturan untuk kedua-dua komposit gentian yang terawat dan tidak dirawat oleh MHP-PLA dan MHP-HDPE meningkat apabila kenaikan beban serat meningkat. Ciri-ciri kesan gentian yang dirawat dan tidak dirawat MHP-HDPE, MHP-PLA, komposit secara drastik berkurangan apabila beban serat meningkat. Penyerapan air meningkat apabila beban gentian meningkat. Komposit yang direndam dalam pengambilan air laut pada kadar yang lebih tinggi berbanding dengan yang direndam dalam air hujan. Degradasi termal komposit serat yang tidak dirawat dan dirawat menunjukkan sedikit perbezaan dari segi penurunan berat, manakala kadar penguraian berbeza-beza apabila beban serat meningkat. Biodegradasi di tanah perbandaran menunjukkan kadar degradasi yang lebih tinggi berbanding dengan tanah oxisol. Tahap penguraian untuk komposit serat yang dirawat dan tidak dirawat sedikit berbeza di kedua-dua jenis tanah. Komposit MHP-PLA mengurai lebih cepat di dalam tanah perbandaran dan oxisol berbanding komposit MHP-HDPE.Serat terawat mempunyai sedikit kesan terhadap kadar biodegradasinya. Berdasarkan semua penemuan, adalah wajar untuk membuat kesimpulan bahawa termoplastik yang terisi serat sekam adalah komposit yang berpotensi.

# ACKNOWLEDGEMENTS

I thank Almighty Allah for giving me this precious opportunity and strength to carry out this study. My sincere appreciation to my supervisor Associate Professor Dr Nur Ismarrubie Zahari, co-supervisors Prof. Ir.Dr Mohd Salit Sapuan and Associate Prof. Dr Zulkiffle Leman for spending their ample time and strength in guiding me throughout the period of my study.

I acknowledge my family for their patient during the entire period of study. My appreciations to the entire lecturers and staffs of the Department of Mechanical and Manufacturing Engineering, Universiti Putra Malaysia for their moral assistance during the course of studies.

Let me also extend my appreciation to my senior colleagues, classmates and friends whom we share views and ideas during this research. I also acknowledge the contributions of following persons; Engr. (Dr) Mohammed M Maina, Dr Hafiz, Dr Tarajo Buba Ahmad, Hajiya Ladidi Godowoli, Engr (Dr) M.U Kallamu and Ms Hassana for moral their support.

I certify that a Thesis Examination Committee has met on 26 January 2018 to conduct the final examination of Hammajam A. Abba on his thesis entitled "Characterization of Millet Husk-Filled High Density Polyethylene and Polylactic Acid Composites" 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 Doctor of Philosophy.

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# LIST OF ABBREVIATIONS

| | |
|---|---|
| ADF | Acid Detergent Fiber |
| ADL | Acid Detergent Lignin |
| ATR | Attenuated Total Reflection infrared |
| ASTM | America Standard Testing Materials |
| AS | Standard Association of Australia |
| BC | Before Christ |
| C | Carbon |
| $CH_4$ | Methane |
| CO | Carbon Monoxide |
| $CO_2$ | Carbon Dioxide |
| cm | Centimeter |
| $cm^{-1}$ | Wave number |
| CTAB | Cetyltrimethylammonium Bromide |
| DTG | Derivative Thermogravimetric |
| FTIR | Fourier Transforms Infrared |
| FFT | Fast Fourier Transforms |
| FL | Fiber Loading |
| g | Gram |
| GPa | Giga Pascal |
| H | Hydrogen |
| ha | Hectare |
| HDPE | High Density Polyethylene |
| hr | Hour |

| | |
|---|---|
| $H_2SO_4$ | Sulpheric  Acid |
| $H_2O$ | Water |
| IR | Infrared |
| ISO | International Standard Organization |
| KBr | Potassium Bromide |
| kg | Kilogram |
| kJ | Kilo Joule |
| kN | Kilo Newton |
| kV | Kilovolt |
| MAPE | Maleic Anhydride Grafted Polyethylene |
| Mc | Moisture content |
| $M_{cw}$ | Mixer capacity |
| MHF | Millet Husk Fiber |
| MHP | Millet Husk Powder |
| min. | Minute |
| mL | Milliliter |
| Mol | Mole |
| MPa | Mega Pascal |
| N | Nitrogen |
| N | Newton |
| NaOH | Sodium Hydroxide |
| NDF | Neutral Detergent Fiber |
| O | Oxygen |
| OH | Hydroxyl |

| | |
|---|---|
| PC | Personal computer |
| PE | Polyethylene |
| PLA | Poly Lactic Acid |
| pH | Power of Hydrogen |
| ROM | Rule of Mixer |
| rpm | Revolution Per Minute |
| SEM | Scanning Electron Microscope |
| STD | Standard Deviation |
| UTM | Universal testing machine |
| TGA | Thermogravimetric Analysis |

# LIST OF NOTATIONS

| **NOTATION** | **UNIT** |
|---|---|
| $b$ Width | m |
| $h$ Height | m |
| $l$ Length | m |
| $D$ Diameter | mm |
| $d$ Thickness | m |
| $V$ Volume | m$^3$ |
| $^oC$ Temperature | $^oC$ |
| $S$ Displacement | mm |
| $W$ Load at yield (maximum load) | N/m |
| $I$ Moment of area | mm |
| $F$ Force applied on the test specimen | kN |
| □Strain of the test sample | m/m |
| $A$ Crossectional area of the test specimen | m$^2$ |
| $\delta$ Applied stress on the test specimen | N/m$^2$ |
| $E$ Flexural modulus | GPa |
| □Flexural strength | MPa |
| $\mu m$ Micrometer | |
| % Percentage | |
| $Wt$ Weight percentage | |
| $W_{hdpe}$ Weight of high density polyethylene | |
| $W_{mf}$ Weight of millet fiber | |
| $W_m$ Weight of matrix | |

$W_f$ Weight of fiber

$Ts$ Glass temperature

$Tr$ Transient temperature

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background

The use natural fibers for composite material manufacturing have received great attention because of their sustainability and renewability. Research and advancement on natural fiber composites are continually rising and their function is growing into novel fields of usage (Jumaidin et al., 2017; Kiruthika, 2017; Merkel et al., 2014; Sapuan and Mansor, 2014). The attributes of natural fibers such as availability, low density, cost-effective, renewability, and biodegradability act as agents in the replacement of usual fillers/reinforcements of composites, like carbon and glass. However, problems associated with the use of synthetics materials have forced legislation to enact laws that protect the environment in many countries around the globe (Sain and Panthapulakkal, 2006; Samper et al., 2015; Sapuan and Mansor, 2014). In addition, the search for low cost and superior materials in many advanced technology fields such as automotive, nautical, building as the alternative to synthetic materials has received prompt attention (Mahjoub et al., 2014).

Therefore, the utilization of these fibers can save the environment and humanity from becoming the house for microorganisms that harbor diseases. The use of thermoplastic polyethylene (PE) in demanding technology applications have increased in recent time (Boopalan et al., 2013). Due to the attractive properties of high melting point, high density and good chemical inertness, it becomes an important choice for husk, fiber filler, compounding and blending work (Ridzuan et al., 2016). Because of its nature, it can be remelted and recycled for various purposes, thus PE composites are profitable and environmentally friendly. Although good work has been done in a variety of areas on agro waste composites, the current literature on millet husk-filled poly lactic acid has not been reported with respect to their composite formations and thus this topic forms the basis for this study. Therefore, the usage of millet husk in natural fiber thermoplastic or polymer composite manufacturing in Sahel sub-Saharan African countries is limited. In the research on the millet husk here, the chemical compositions, fiber size chemistry and thermal degradation characteristics were determined with the aim to establish the viability in the processing and production of composites. Consequently, it is suggested as a possible filler for plastic composites, since research has shown that synthetic fibers have a direct influence on the environment and this substitute material does not endanger the natural forest product which is waning in resource and becoming costly.

## 1.2 Problem statements

This study tries to find solutions to the environmental and health-related problems caused by agricultural wastes such as respiratory infections, diarrhea, malaria, intestine nematode, trachoma, sleeping sickness etc. as reported by Smith et al., (2004); Smith et al., (2000); and WHO (2002) through converting the waste into polymer-filled composites. Millet husks fiber is an agro waste left on the farm site after harvest. Due to the underutilization of this waste, it became home for microorganisms that harbor diseases and consequently resulted in environmental pollution in Sub-Saharan Africa were large amount of millet husks are deposited on farm lands. According to the world health report, an estimated 24% of the world disease burden and 23% of all deaths can be associated with environmental factors (WHO, 2002). However, the greater percentages are from developing nations. This study seeks a way of converting this waste into fillers for plastic composites applications.

## 1.3 Work scope

The investigation of agro waste millet husk powder (MHP)-filled high-density polyethylene (HDPE) and poly lactic acid (PLA) composites was conducted in this work. The study started with the preparation of millet husk, poly lactic acid, sodium hydroxide and high-density polyethylene and is followed by the treatment of fibers, testing and characterization of the composites. Municipal soil (city soil, black with loose sandy appearance) was obtained from Damaturu city area, Nigeria with a pH value of 6.8 and soil temperature of 34 $^{o}$C in June 2015. Oxisol soil (tropical rain forest soil), sometimes called red earth, was obtained from Lekki area of Lagos. Seawater was collected from the Atlantic Ocean off the shore of Lagos, Nigeria in May 2015. Millet husk powder was used as the composite filler while high-density polyethylene and poly lactic acid were used as polymer matrices. The effects of fiber loading on the mechanical, physical, and thermal properties were investigated. In addition, the biodegradability, microstructure and water absorption of the composites were also investigated.

## 1.4 Significance of the study

This investigation presents the findings on the applicability of millet husk fiber as filler in high-density polyethylene (HDPE) and poly lactic acid (PLA) composites. Several other vital investigations were carried out on some cereal fibers to develop industrial products. There is a large quantity of millet husks across sub-Saharan Africa that was left as agro wastes; however, the development of these husks into poly lactic acid-filled composite was not reported. Millet husk is selected for this investigation as the filler because of its availability, environmental and health impact. Furthermore, the forecast cost benefits include domestic utilities, environmental safety through end product recycling, preventing damages to ecosystems and occupation of utilizable land, as well as cost reduction in manufacturing. It is presumed that these composites could be used in domestic products (lampshade, partition board) and office utilities especially where load bearing is not a critical parameter.

## 1.5 Objectives

The aim of the investigation is to see the possibility of using millet husk powder (MHP) fiber as the filler in high-density polyethylene (HDPE) and poly lactic acid (PLA) polymer matrix to form composites. The specific objectives are;

- To evaluate the effect of fiber treatment on the mechanical properties of MHP-filled HDPE and PLA composites.
- To determine the effect of water absorption on the mechanical properties of MHP-filled HDPE and PLA composites.
- To evaluate the biodegradability of MHP-filled HDPE and PLA composites.
- To determine the effect of fiber loading on the thermal properties of MHP-filled HDPE and PLA composites.

## 1.6 Thesis structure

This thesis is structured into five chapters as follows; Chapter 1 presents the background of studies, the work scope, the significance, and the objectives of the study as well as the thesis structure.

Chapter 2 contains the literature assessment of research work in different areas relevant to this study. It began with fiber treatment, fabrications and properties characterizations of the composite products made from agro waste husk, fiber and plastic. Mechanical, microstructure, physical, thermogravimetric, degradability and water absorption properties of millet husk were reviewed. Studies of composite preparation and properties are also included in this section.

Chapter 3 describes the methodology of the study. It also presents the techniques to determine the physical, microstructure, thermogravimetric, degradability, water absorption and mechanical properties of the composites. The preparation steps of the composites were also presented.

Chapter 4 reports the results and discussion of the study. The discussion on the mechanical properties of the composites such as tensile, flexural and impact tests was presented. The morphology of the fractured surface of the composites using the scanning electron microscope (SEM) was also presented. In addition, the physical, water absorption, biodegradability, thermogravimetric analysis of the composites were investigated and the findings presented.

Finally, Chapter 5 presents the conclusion and recommendations for further investigations on the study.

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