

**DEGRADATION BEHAVIOR OF PINEAPPLE LEAF FIBRE (PALF) REINFORCED  
BIOCOMPOSITES**

**KOSHILA DEVI SIVAKUMAR**

**A report submitted  
in fulfillment of the requirement for the degree of  
Bachelor of Mechanical Engineering (Structural & Material)**

**Faculty of Mechanical Engineering**

**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2016**

## APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Structure & Material).

Signature :



**DR. SITI HAJAR BINTI SHEIKH MD. FADZULLAH**  
PENSYARAH KAWAN  
FAKULTI KEJURUTERAAN MEKANIKAL  
UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Name


:.DR.SITI HAJAR BINTI SHEIKH MD FADZULLAH

Date

:.28 / 06 / 2016

## DECLARATION

I declare that this project entitled “Degradation behavior of Pineapple Leaf Fibre (PALF) Reinforced Biocomposites” is the result of my own work except as cited in the references.

Signature :   
Name : KOSHILA SIVAKUMAR  
Date : 28 / 06 / 2016

## DEDICATION

This report is dedicated to my beloved parents,

Sivakumar Muthiah and Suseela Sinnappan.

## ABSTRACT

This project investigates the degradation behavior of Pineapple leaf fibre (PALF) reinforced PLA composite when subjected to water immersion test. Samples of treated PALF reinforced PLA composite were prepared and subjected to water immersion test to study the water absorption rate by manipulating the duration of immersion period. The specimens were immersed in distilled water solution and incubated at 40 °C for three different immersions period; week 0, 2 and 4. Upon reaching respective immersion period, the specimens were subjected to physical testing, flexural testing, and thermal analysis. The physical and mechanical properties of the specimens reduced as the water absorption rate and the immersion period increased. Moisture induced degradation has significant impact on the mechanical properties such as flexural strength and flexural modulus of the specimen while no drastic changes were observed in terms of the thermal properties.

## **ABSTRAK**

*Projek ini mengkaji kesan kemerosotan bahan komposit matrik poli laktid acid diperkuat serat daun nanas apabila dikenakan ujian rendaman air . Sampel komposit PLA diperkuat serat daun nanas telah disediakan dan dikenakan ujian rendaman air untuk mengkaji kadar penyerapan air dengan memanipulasi jangka masa tempoh rendaman. Spesimen telah direndam dalam larutan air suling pada 40 °C untuk tiga tempoh rendaman yang berbeza; minggu 0 , 2 dan 4. Setelah mecapai tempoh rendaman masing-masing, spesimen dikenakan kepada ujian fizikal , ujian lenturan, dan analisis terma. Didapati bahawa sifat-sifat fizikal dan mekanikal spesimen berkurang dengan peningkatan kadar penyerapan air dan tempoh rendaman. Kelembapan dalam komposit yang menyebabkan degradasi mempunyai kesan besar ke atas sifat-sifat mekanikal seperti kekuatan lenturan dan modulus lenturan manakala tiada perubahan yang ketara ke atas sifat-sifat termal bahan komposit tersebut.*

## ACKNOWLEDGEMENT

I am indebted to each one of the individuals who helped me to complete this report. I would like to express my gratitude to Dr. Siti Hajar Binti Sheikh Md Fadzullah who provided me constant guidance and knowledge throughout the completion of this by sacrificing her precious time and energy to ensure the project turned out to be a successful one.

The contribution and dedication of all the technicians from both Mechanical and Manufacturing Faculty Engineering are worthy of special praises as they were kind enough to accompany and guide me during the handling of equipments and machines. A special thank you to Siti Nur Rabiatur Adawiah, my senior who shared her knowledge and experience to ensure we were equipped with sufficient knowledge before conducting any experiments.

My sincere appreciation goes to my loving family who unconditionally supported and encouraged me by providing necessary support both financially and academically whenever there was a need for it. My dearest friends especially Kesavan Selvan and Ain Nur Haznida who both were my pillar of support and strength when I really needed motivation.

---

## TABLE OF CONTENTS

|                                    | PAGE        |
|------------------------------------|-------------|
| <b>DECLARATION</b>                 |             |
| <b>DEDICATION</b>                  |             |
| <b>ABSTRACT</b>                    | <b>i</b>    |
| <b>ABSTRAK</b>                     | <b>ii</b>   |
| <b>ACKNOWLEDGEMENTS</b>            | <b>iii</b>  |
| <b>TABLE OF CONTENTS</b>           | <b>iv</b>   |
| <b>LIST OF TABLES</b>              | <b>vii</b>  |
| <b>LIST OF FIGURES</b>             | <b>ix</b>   |
| <b>LIST OF APPENDICES</b>          | <b>xiii</b> |
| <b>LIST OF ABBREVIATIONS</b>       | <b>xiv</b>  |
| <b>LIST OF SYMBOLS</b>             | <b>xv</b>   |
| <br>                               |             |
| <b>CHAPTER</b>                     |             |
| <b>1 INTRODUCTION</b>              |             |
| 1.1 Background                     | 1           |
| 1.2 Problem statement              | 3           |
| 1.3 Objective                      | 5           |
| 1.4 Scope of project               | 5           |
| 1.5 Planning and execution         | 6           |
| <br>                               |             |
| <b>2 LITERATURE REVIEW</b>         |             |
| 2.1 Introduction                   | 9           |
| 2.2 Composites                     | 9           |
| 2.2.1 Classification of Composites | 10          |
| 2.3 Biocomposites                  | 11          |
| 2.3.1 Natural Fibres               | 14          |
| 2.3.1.1 Pineapple leaf fibre       | 17          |
| 2.3.2 Polymer matrix material      | 19          |



|          |                                                         |    |
|----------|---------------------------------------------------------|----|
|          | 2.3.2.1 PLA as the biodegradable polymer                | 20 |
| 2.4      | Properties of biocomposites                             | 23 |
|          | 2.4.1 Mechanical properties                             | 23 |
|          | 2.4.1.1 Tensile                                         | 25 |
|          | 2.4.1.2 Impact                                          | 27 |
|          | 2.4.1.3 Flexural                                        | 28 |
|          | 2.4.2 Thermal properties                                | 29 |
|          | 2.4.3 Physical properties                               | 30 |
|          | 2.4.3.1 Density test                                    | 30 |
|          | 2.4.3.2 Water absorption test                           | 31 |
| 2.5      | Alkaline treatment                                      | 35 |
| <br>     |                                                         |    |
| <b>3</b> | <b>METHODOLOGY</b>                                      |    |
| 3.1      | Overview of research                                    | 37 |
| 3.2      | Raw materials                                           | 39 |
|          | 3.2.1 PLA 6100D                                         | 39 |
|          | 3.2.2 Pineapple leaf fibre                              | 41 |
| 3.3      | Fibre surface modification                              | 42 |
| 3.4      | Composite preparation                                   | 44 |
|          | 3.4.1 PLA thin plate fabrication                        | 44 |
|          | 3.4.2 Fabrication of Unidirectional PLA/ PALF Composite | 47 |
|          | 3.4.3 Specimen Preparation                              | 50 |
| 3.5      | Degradation test                                        | 52 |
| 3.6      | Flexural testing                                        | 55 |
| 3.7      | Thermal testing                                         | 58 |
| 3.8      | Physical testing                                        | 60 |
| 3.9      | Surface morphology study                                | 61 |
| <br>     |                                                         |    |
| <b>4</b> | <b>RESULTS AND DISCUSSION</b>                           |    |
| 4.1      | Introduction                                            | 62 |
| 4.2      | Water absorption test                                   | 63 |

|     |                                      |           |
|-----|--------------------------------------|-----------|
| 4.3 | Physical test                        | 73        |
| 4.4 | Flexural test                        | 76        |
| 4.5 | Thermal analysis                     | 82        |
| 4.6 | Surface morphology study             | 83        |
| 5   | <b>CONCLUSION AND RECOMMENDATION</b> |           |
| 5.1 | Conclusion                           | 88        |
| 5.2 | Recommendation                       | 90        |
|     | <b>REFERENCES</b>                    | <b>92</b> |
|     | <b>APPENDICES</b>                    | <b>99</b> |

## LIST OF TABLES

| TABLE | TITLE                                                                                          | PAGE |
|-------|------------------------------------------------------------------------------------------------|------|
| 2.1   | General composition of the constituents in natural fibre                                       | 16   |
| 2.2   | Mechanical properties of natural fibres                                                        | 17   |
| 2.3   | Chemical compositions of different types of fibres                                             | 18   |
| 2.4   | Advantages and disadvantages of PLA                                                            | 22   |
| 2.5   | Mechanical properties of natural and synthetic fibre                                           | 24   |
| 2.6   | Mechanical properties of PLA                                                                   | 24   |
| 2.7   | Comparison of PLA with PS and PET                                                              | 25   |
| 2.8   | Results for monotonic test                                                                     | 34   |
| 2.9   | Mechanical properties of treated and untreated fibre biocomposite                              | 36   |
| 3.1   | Properties of Polylactic Acid (6100D)                                                          | 41   |
| 3.2   | Ratio of constituents                                                                          | 50   |
| 4.1   | pH value of distilled water that has been recorded                                             | 64   |
| 4.2   | Initial mass of the specimens, respective wet weight and corresponding dry mass                | 67   |
| 4.3   | Water absorption percentage (WA) and mass loss of the specimen                                 | 70   |
| 4.4   | Physical test results                                                                          | 74   |
| 4.5   | Comparison of density values of degraded PALF-PLA samples and different types of biocomposites | 74   |

|     |                                                                                            |    |
|-----|--------------------------------------------------------------------------------------------|----|
| 4.6 | Flexural strength and flexural modulus of specimens from week 0, 2 and 4 immersion period. | 79 |
| 4.7 | Simplified average values of flexural strength and modulus                                 | 80 |
| 4.8 | Average results calculated from DSC analysis                                               | 83 |
| 4.9 | Micrographs from SEM analysis tabulated according to respected immersion period.           | 85 |
| 5.1 | Summary of the prime findings from the relevant test conducted for the degradation study.  | 89 |

## LIST OF FIGURES

| FIGURE | TITLE                                                                                   | PAGE |
|--------|-----------------------------------------------------------------------------------------|------|
| 1.1    | Gantt chart detailing research activities and their respected time frame for PSM I.     | 7    |
| 1.2    | Gantt chart detailing research activities and their respected time frame for PSM II     | 8    |
| 2.1    | Classification of biocomposites                                                         | 13   |
| 2.2    | Classification of natural fibres                                                        | 15   |
| 2.3    | Pineapple plant                                                                         | 17   |
| 2.4    | Classification of biodegradable polymers and their nomenclature                         | 20   |
| 2.5    | The life cycle of biodegradable polymers                                                | 21   |
| 2.6    | Synthesis and chemical structure of PLA                                                 | 22   |
| 2.7    | Tensile properties of PALF/PLA composites                                               | 26   |
| 2.8    | Tensile stress and tensile modulus of PLA/flax composite compared to PP/flax            | 27   |
| 2.9    | Notched Izod impact properties of the neat polymers and the fibre reinforced composites | 28   |
| 2.10   | Flexural properties of the neat polymers and the fibre reinforced composites            | 29   |

|      |                                                                                      |    |
|------|--------------------------------------------------------------------------------------|----|
| 2.11 | Moisture absorption as a function of time for core and<br>bast kenaf fibre           | 32 |
| 2.12 | Percentage of fluid absorption versus degradation time for<br>PLA-PLA-TCP composites | 33 |
| 2.13 | Percentage of mass loss in PLA-PLA-TCP samples versus<br>various degradation time    | 34 |
| 3.1  | Flow chart of the research project                                                   | 38 |
| 3.2  | Steps of PLA process with ring opening polymerization                                | 40 |
| 3.3  | PLA 6100D (pellets form)                                                             | 40 |
| 3.4  | Diluting NaOH in distilled water                                                     | 42 |
| 3.5  | PALF immersed in alkaline solution                                                   | 43 |
| 3.6  | Drying process of treated PALF and Prepared<br>PALF for composite fabrication        | 44 |
| 3.7  | Drying Oven                                                                          | 45 |
| 3.8  | Placement of PLA and Mila sheets in the plate mould                                  | 46 |
| 3.9  | Graph of temperature versus time for PLA plate<br>fabrication process                | 47 |
| 3.10 | Hydraulic Hot Moulding Machine                                                       | 48 |
| 3.11 | Arrangement of PALF in between PLA plates                                            | 48 |
| 3.12 | Graph of Temperature versus time for PALF reinforced<br>PLA composite                | 49 |
| 3.13 | Fabricated PALF reinforced PLA composite                                             | 49 |
| 3.14 | Shearing Machine                                                                     | 50 |
| 3.15 | Specimen prepared for degradation test                                               | 51 |

|      |                                                                                |    |
|------|--------------------------------------------------------------------------------|----|
| 3.16 | Schematic diagram of the specimens prepared for water absorption test          | 52 |
| 3.17 | Samples placed in the bottle containing distilled water to be incubated        | 53 |
| 3.18 | Samples placed in the Incubator                                                | 54 |
| 3.19 | An Instron Universal Testing Machine Model 5585                                | 55 |
| 3.20 | Allowable range of loading nose and support radii in accordance with ASTM D790 | 56 |
| 3.21 | Deflection of the specimen when load was applied                               | 56 |
| 3.22 | Differential Scanning Calorimetry Equipment                                    | 58 |
| 3.23 | Electronic Densimeter                                                          | 60 |
| 4.1  | Change in pH of distilled water versus degradation time                        | 64 |
| 4.2  | Top view of specimen after water uptake tests                                  | 65 |
| 4.3  | Side view of specimen after water uptake test                                  | 66 |
| 4.4  | Water absorption versus degradation time of PALF reinforced PLA composites     | 71 |
| 4.5  | Mass loss versus drying period of PALF-PLA                                     | 71 |
| 4.6  | Mass loss of specimen with their respective immersion period                   | 72 |
| 4.7  | Comparison of fluid uptake rate for PALF-PLA specimen and PLA-PLA-TCP specimen | 73 |
| 4.8  | Comparison of density                                                          | 75 |
| 4.9  | Top view of specimens                                                          | 78 |
| 4.10 | Side view of specimens                                                         | 78 |

|      |                                                                              |    |
|------|------------------------------------------------------------------------------|----|
| 4.11 | Flexural strength versus degradation time for PALF reinforced PLA composites | 80 |
| 4.12 | Flexural modulus versus degradation time for PALF reinforced PLA composites  | 81 |
| 4.13 | Flexural strength of samples before and after immersion test                 | 82 |



## LIST OF APPENDICES

| APPENDIX | TITLE                                                      | PAGE |
|----------|------------------------------------------------------------|------|
| A        | Results of flexural test                                   | 99   |
| B        | Results from DSC analysis                                  | 109  |
| C        | NatureWorks Ingeo Biopolymer 6100D<br>Technical Data Sheet | 114  |

## LIST OF ABBREVIATIONS

|      |                                   |
|------|-----------------------------------|
| CCM  | Carbon-Carbon Composites          |
| CMC  | Ceramic Matrix Composites         |
| DSC  | Differential Scanning Calorimetry |
| MMC  | Metal Matrix Composites           |
| NaOH | Sodium Hydroxide                  |
| PALF | Pineapple leaf fibre              |
| PET  | Polyethylene Terephthalate        |
| PLA  | Polylactic Acid                   |
| PMC  | Polymer Matrix Composites         |
| PP   | Polypropylene                     |
| PS   | Polystyrene                       |
| RNCF | Recycled Newspaper Fiber          |
| ROP  | Ring Opening Polymerization       |
| SEM  | Scanning Electron Microscope      |
| TCP  | Tricalcium Phosphate              |
| TG   | Glass Transition Temperature      |
| WC   | Water Content                     |

## LIST OF SYMBOL

|              |   |                                                                                        |
|--------------|---|----------------------------------------------------------------------------------------|
| $m_w$        | = | Wet weight of specimen after immersion test (g)                                        |
| $m_d$        | = | Dry mass of specimen (g)                                                               |
| $m_o$        | = | Initial mass of specimen (g)                                                           |
| WA           | = | Water absorption percentage (%)                                                        |
| R            | = | Rate of cross head motion, mm/min                                                      |
| Z            | = | Rate of straining of the outer fibre, mm/min = 0.01                                    |
| L            | = | Support span, mm                                                                       |
| d            | = | Depth of beam, mm                                                                      |
| $E_b$        | = | Modulus of elastic in bending, MPa                                                     |
| m            | = | Slope of the tangent to the initial straight line portion of the load deflection curve |
| B            | = | Width of the beam tested, mm                                                           |
| $X_c$        | = | Degree of crystallinity                                                                |
| $T_m$        | = | Melting Temperature                                                                    |
| $\Delta H$   | = | Crystallization enthalpy of composite sample                                           |
| $\Delta H_m$ | = | Crystallization enthalpy of 100% crystalline PLA                                       |
| $\rho_c$     | = | Density of the composite                                                               |
| $M_b$        | = | Buoyancy of the specimen                                                               |
| $M_a$        | = | Weight of the specimen in air                                                          |
| $\rho_w$     | = | Density of the distilled water at room temperature                                     |

## CHAPTER I

### INTRODUCTION

#### 1.1 BACKGROUND

The exploration for the seeking of materials with attractive features such as high performance level, quality assured, environmentally-friendly and competitive price has surged researches to study and develop further on the function and capabilities of composites [1].

Composites are being widely used in the engineering & manufacturing world. Composites can be manipulated to ones' benefit. The researcher/ designer can combine materials to prevail their maximum use of their traits/ virtues by altering the composition and ratio of the materials affecting its mechanical, physical and thermal properties to what they desire for [2]. Through this, the materials deficiency can be minimized.

Tapping into the diversity of composites, studies and innovation has motivated the development of biocomposites. Biocomposites are composites that are products from polymers and natural fibres [3]. Polymers can be classified into synthetic polymers and also natural polymers. Polylactic acid (PLA) is a natural polymer that is being explored vastly due to its environmentally friendly properties. Due to the superseding of advancement in industrial technologies, PLA has been generated to possess high molecular weight that contributes the

sustainability as for structural materials with sufficient lifespan and good weatherability to maintain its mechanical properties [4].

Natural fibres on the contrary are opted as they are renewable and a much cheaper substitute for synthetic fibres. Possessing a number of advantages such as low density, high toughness, acceptable specific strength properties not neglecting its ease of separation and biodegradability has prompted researches to cultivate the growth of natural fibre-reinforced composites [4].

All the advantages that the combination of polymers and natural fibres which results in biocomposites is very generous, however they still do have a few major drawbacks. Amongst them would be its poor resistance towards water absorption and how it affects the biocomposites degradation behaviour [6]. Water absorption of natural fibre reinforced composites are mostly because of the hydrophilic nature of the natural fibre itself and it is proportionally dependent with the volume fractions of fibres [13]. A change by the improvement of water resistance of natural fibre reinforced composite would altogether improve their qualities and mechanical properties. Other than that, water content influences the dielectric properties of composites to be utilized for insulation purpose.

## 1.2 PROBLEM STATEMENT

The consumption of petroleum assets to the extreme causing depletion of petroleum resources, plastic disposal issues and emission amid incineration along increasing environment regulations has prompted development of materials that are compatible with the environment and independent of fossil fills [5].

New materials with high efficiency at accessible costs are progressively required with the raising of environment conscience. Exploration has been focused specifically on materials respectful of environment [10]. The environment friendly materials include the fields of green chemistry, biodegradable and bio-based materials at the structural, chemical and physical level, as well as the utilization of principles to and the usage of standards to diminish or dispose of hazardous substances in the outline, assembling and application of chemical products.

Numerous successful attempts have prompted opening up an extensive scope of materials leading to the advancement of biodegradable materials, which brought great impetus for the improvement of industrial viable biodegradable composites with expanded focus in eco-friendliness. Natural polymers such as polylactic acid (PLA) which are typically biodegradable is a vital outline parameter for opening up new materials opportunities for the advancement of value added products from natural polymer feed stocks [5, 11].

However, biocomposites undergo degradation that is the change in the properties (tensile, strength, odour, shape and etc) under the impact of one or more ecological elements for



example light, water, heat or chemicals. These progressions are normally undesirable for instance changes during use, cracking and depolymerisation of products and it is desirable as in biodegradation or deliberately bringing down the molecular weight of the composite for reusing or decomposing purpose. The changes in properties are frequently termed as 'aging'. In a finished product, such as a change is to be prevented or delayed.

Degradation can be induced to assist structure determination. Polymeric molecules are huge (on the atomic scale) and their unique and useful properties is a result of their size thus a loss in chain length brings down their tensile strength and it is the essential driver of premature cracking. Composites poor resistance towards water absorption that disrupts their mechanical properties and ways to improve the resistance is yet and still being studied. Water absorption is caused by hydrogen bonding between free hydroxyl groups in cellulose molecules and water molecules as well as the adhesion between fibre and the matrix. Considering these aspects gives rise to the water content in natural fibre reinforced biocomposite as a critical parameter to be concentrated on [44].

The percentage/ rate of water resided in a biocomposite can be examined by distinctive technique, such as water absorption test and thermogravimetric analysis. Results on water absorption behaviour should be studied heavily about when designing a part on the grounds that moisture prompts an increment in dimensions as previously seen to a reduction in tensile and flexural properties of the biocomposite. Before searching for solutions to lessen the water absorption of the biocomposite their behaviour needs to be completely understood hence the suitable solution can be recommended.

Thus, this study aims to study the degradation behaviour of PALF reinforced biocomposite via water absorption test and followed by mechanical tests conducted on the biocomposite specimens after is has been immersion in distilled water solution for a specific period of time (0, 2, 4 weeks).

### **1.3 OBJECTIVE**

The objectives for pursuing the current research topic are:

1. To fabricate of biocomposites where PLA is the matrix and long fibre from pineapple leaves (PALF) acts as the reinforcement.
2. To study the degradation behaviour of the biocomposite due to the effects of water absorption.
3. To study the effect of degradation on the mechanical properties (flexural strength) of the biocomposites.

### **1.4 SCOPE OF PROJECT**

The scope of this project is listed as below:

- i. PALF pretreatment.
- ii. Fabrication of PLA thin films.
- iii. Producing the biocomposite plate.