



**UNIVERSITI PUTRA MALAYSIA**

***CHARACTERIZATION OF HYBRID BIOCOMPOSITE SYNTHESISED  
WITH MULTI-WALLED CARBON NANOTUBE FOR FINITE STRAIN  
APPLICATION OF AEROSPACE STRUCTURES***

**KHAIRUL IZWAN BIN ISMAIL**

**FK 2018 173**



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APPLICATION OF AEROSPACE STRUCTURES**

By

**KHAIRUL IZWAN BIN ISMAIL**

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

**June 2018**

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

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**Chairman : Assoc. Prof. Mohamed Thariq Bin Hameed Sultan, PhD**  
**Faculty : Engineering**

Generally, synthetic-natural hybrid composites are being introduced to lessen the use of synthetic fibres in composites as well as to deal with the drawbacks of natural fibres in biocomposites. The combination of both properties, from synthetic and natural fibres, results in hybrid biocomposites with surpass properties compared to its elements. Meanwhile, the use of nano-fillers in composites, as reported, can either enhance or diminish the properties of composites, depending on several factors. Therefore, this research was intended to study the effects of multi-walled carbon nanotube (MWCNT) concentration, on the properties of hybrid biocomposites. In this study, high cost synthetic fibres, i.e. glass, carbon and Kevlar, were respectively hybridised with the natural fibre of flax. The epoxy matrix used was modified with 0%, 0.5%, 1.0%, 1.5% and 2.0% of multi-walled carbon nanotubes (MWCNTs) to fabricate five different types of composites for each hybrid combination. In terms of mechanical properties, hybrid flax-carbon biocomposites with 1.0% MWCNT exhibit the highest tensile strength, while these same hybrid combinations possess the highest flexural strength at 0.5 % MWCNT. Analysis of the thermal properties suggested that the inclusion of MWCNT lowered the thermal stability as these nanofillers enhanced the heat diffusion, thus speeding up the degradation. Comparison between the different stacking sequences of hybrid flax-carbon and flax-glass with 1% of MWCNT showed that the flax surface are penetrated by the impactor at 15 J, while the glass and carbon surfaces were both penetrated at 20 J. The higher strength residue of hybrid flax-glass compared to flax-carbon, analysed through the compression after impact testing, was in a good agreement with the less severe damage found on the flax-glass composites.

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

**PENCIRIAN HIBRID BIOKOMPOSIT SINTESIS BERSAMA KARBON  
NANOTIUB UNTUK APLIKASI KEKUATAN TERHADAP STRUKTUR  
AEROANGKASA**

Oleh

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Umumnya, hibrid komposit sintetik-semulajadi telah diperkenalkan untuk mengurangkan penggunaan serat sintetik dalam komposit disamping untuk memperkuatkan kelemahan serat semulajadi dalam biokomposit. Gabungan kedua-dua ciri daripada serat sintetik-semulajadi menjadikan ciri-ciri komposit tersebut lebih baik jika dibandingkan dengan individu elemen tersebut. Manakala, penggunaan pengisi-nano dalam komposit mampu mempertingkatkan atau menyusut ciri-ciri komposit bergantung kepada bebraa factor seperti yang telah dilaporkan. Oleh itu, kajian ini menjurus untuk mengkaji kesan kuantiti karbon nanotiub terhadap ciri-ciri hibrid komposit. Dalam kajian ini, serat sintetik seperti serat kaca, karbon dan Kevlar, telah dihibridkan bersama serat semulajadi iaitu flax. Epoksi yang telah digunakan telah diubahsuai dengan 0%, 0.5%, 1.0%, 1.5% dan 2.0% karbon nanotiub untuk menghasilkan lima jenis komposit yang berbeza bagi setiap gabungan hibrid. Dalam aspek ciri-ciri mekanikal, hibrid biokomposit flax-karbon dengan 1.0% karbon nanotiub menunjukkan peningkatan tertinggi bagi ketegangan, manakala gabungan hibrid komposit yang sama turut menunjukkan keputusan tertinggi dalam kelenturan pada 0.5% karbon nanotiub. Analisa daripada ciri-ciri haba menunjukkan bahawa kehadiran karbon nanotiub mengurangkan kestabilan haba disebabkan karbon nanotiub menyokong pembakaran dengan cepat. Perbandingan antara perbezaan lapisan hibrid biokomposit flax-karbon dan flax-kaca bersama 1.0% karbon nanotiub menunjukkan bahawa permukaan flax telah tembus pada pemberat dengan peringkat tenaga 15 J, manakala permukaan sintetik telah tembus pada peringkat tenaga 20 J. Sisa kekuatan yang lebih tinggi oleh hibrid flax-kaca berbanding flax-karbon, telah dikaji melalui ujian mampatan selepas impak, mempunyai persamaan yang baik dengan tahap kerosakan pada flax-kaca komposit.

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## LIST OF ABBREVIATIONS

ASTM	American Society for Testing Materials
CMC	Ceramic matrix composites
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
EP	Neat epoxy
E'	Storage modulus
E''	Loss modulus
FESEM	Field emission scanning electron microscope
FRP	Fibre reinforced polymer
FTIR	Fourier transform infrared spectroscopy
GF	Glass fibre
HDPE	High density polyethylene
HDT	Heat deflection temperature
ISO	International Organization for Standardization
MAPP	Maleic anhydride polypropylene
MDF	Medium density fibreboard
MMC	Metal matrix composites
NDE	Non-destructive evaluation
NFRP	Natural fibre reinforced polymer
PBS	Poly butylene succinate
PLA	Polylactic acid
PMC	Polymer matrix composites
PP	Polypropylene
PS	Polystyrene

PVC	Polyvinyl chloride
SEM	Scanning electron microscopy
SBS	Short Beam Shear
TGA	Thermo-gravimetric analysis
T <sub>g</sub>	Glass transition temperature





## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

The emergence of composites in the materials industries has reduced the dependency on the use of metals, which are heavier, essentially limited in design, susceptible to corrosion, and highly expensive in maintenance. Automotive and aerospace applications are among the critical industries which have demands for lightweight with high strength materials (Gururaja & Rao, 2012). The replacement of metals with fibre reinforced polymers (FRP) in aircraft framework, such as the wing and fuselage, has increased in the development of any new models. For example, the newest model of the Airbus A350 XWB used 52% of composites in its whole structure, which shows the high reliability of composites in this structural application. By 2025, manufacturers involved in car production are reducing the weight of their vehicles which can reduce the petrol usage. It was suggested that carbon and glass fibre composites can offer 50-70% and 25-35% mass reduction respectively over mild steel. An estimated 6-7% fuel savings can be achieved with a 10% mass reduction.

Due to their magnificent performances, synthetic fibres have been extensively used in FRP composites for daily life applications. In spite of that, some issues arise regarding the health and environmental aspects. The inhalation of synthetic fibres during fabrication can cause breathing difficulties, while renewability, degradability and recyclability are almost impossible for all types of synthetic composites (Ramesh, Palanikumar, & Reddy, 2017; Sanjay, Arpitha, & Yogesha, 2015). The high production cost of synthetic fibre as well as its composites drives researchers' attention to introduce lower cost materials with comparable properties to the synthetics (Wambua, Ivens, & Verpoest, 2003).

In an effort to develop new, high performance materials, natural fibres possess outstanding mechanical properties with low production cost, low density, light weight and environmentally friendly benefits, which can overcome the drawbacks of synthetic fibres (Jarukumjorn & Suppakarn, 2009; Malkapuram, Kumar, Negi, & Yuvraj Singh Negi, 2008). Flax fibres, extricated from the bast underneath the surface of the stem of the flax plant, are used commercially in the textile industries. In naturally based composites, studies of flax fibres have been reported with different views as to their potential applications. One of the reported studies suggested an alkaline treatment of flax fibres as being most effective and simplest methods to enhance fibre-matrix bonding, resulting in

better flexural properties of the flax/epoxy composites (Charlet & Béakou, 2011).

In attaining superior properties for flax-reinforced composites, the idea of hybridising flax fibres with the synthetics emerged similarly with other natural-synthetic hybrid composites, such as kenaf/kevlar and bamboo/glass hybrid composites (Nayak, Mohanty, & Samal, 2009; Yahaya, Sapuan, Jawaaid, Leman, & Zainudin, 2014). In the study of any hybrid composites, with the fibres in fabric or mat form, the stacking sequence is one of the factors which varies the performance of the composites. Stacking sequence of natural and synthetic fabric has been seen effects on tensile performance of hybrid composites. By some means, the greater hybrid interface turns out into the improving fracture toughness and inter-laminar shear strength of hybrid composite compared to the non-hybrid, fully glass fibre reinforced composites (Y. Zhang, Li, Ma, & Yu, 2013). This hybridisation results in composites which are comparatively cheaper and equally as strong as the fully synthetic composites (Ramesh, Palanikumar, & Reddy, 2013).

Apart from the superior abilities of the fibres, the good performance of the polymer matrix does contribute to developing the properties of the composites. Previously, a lot of research studies have been conducted to modify the polymer matrix, such as polyamides, polyurethane, epoxy and polypropylene, by adding either nanoparticles or fillers to develop the strength, modulus, thermal stability, and fire retardant of the composites (Chikhi, Fellahi, & Bakar, 2002; S. Kim, Pechar, & Marand, 2006).

Carbon nanotube (CNT), a type of carbon material with one dimension, generally can be segregate into two groups, single-walled and multi-walled CNTs (Yakobson & Avouris, 2001). Even though there are many research on CNTs as filler in polymer nanocomposite, their outstanding performances are still inconsistent due to the dispersion difficulties in the polymers. However, the incorporation of CNTs in most of the polymer composites results in better mechanical and thermal properties (Ma, Siddiqui, Marom, & Kim, 2010a; Mittal, Dhand, Rhee, Park, & Lee, 2015; Y. Zhou, Pervin, Lewis, & Jeelani, 2008).

In this research work, the outcomes of different concentrations of CNTs on the mechanical properties of the flax hybrid with different synthetic fibres were experimentally analysed. The dispersing method of CNT in the epoxy was standardized during the fabrication of all panels.

## **1.2 Research Background**

Carbon nanotubes are used as the additive in a composite product (polymer nano-composite), whilst adding other synthetic fibres such as carbon, glass, Kevlar, and natural fibre such as flax, at the same time making other comparisons between the percentages of multi-walled carbon nanotubes (0wt%, 0.5wt%, 1wt%, 1.5wt% and 2wt%) by weight. Comparisons are made because studying the suitability and comparing the performance of composites that can turn into new materials are important to engineering applications.

This study is also performed to achieve some improvement by studying the effects of CNT distribution and dispersion on the reliability of the cracks in fibre-reinforced polymers (FRPs), and the mechanical loading will also be discussed. The method considered for this study is the wet lay-up process, which is typically used as the composite industry's processing method. This conventional method is used to produce the samples for testing.

Especially in aerospace applications, the implementation of CNT is predicted to have a massive significance on future space vehicles (Khan & Kim, 2011). This is due to their impressive countenance, which include all aspects of thermal, electrical and mechanical properties. Attention is drawn to the future utilization of CNT, which may comprise composite material application, lightning protection for aircraft, aircraft icing mitigation, the reduced weight of airframes/satellites, and the alleviation of the challenges related to future space launch (Volder et al., 2013). This research work also studies the impact of CNT in aerospace applications, as well as any other engineering applications, the current and expected challenges related to their usage in aerospace sciences and large scale manufacturing.

## **1.3 Problem Statement**

Composite plays crucial part in the advancement of the aerospace industry. However, the use of synthetic fibres has created issues such as high cost, low renewability, low biodegradability and low recyclability. These concerns can be overcome by replacing or reducing these synthetic fibres with plant fibres, which would reduce the cost and may allow for such material to be recyclable.

Furthermore, the use of CNT as reinforcement in composite manufacturing has become more commonplace in recent years. Even if it is not to be the main chosen reinforcement, it can still provide new topics for improvement and innovation in composites, although, these are still in the research stage. This research work focuses on creating a new hybrid material with a matrix modified with CNT.

CNT is known to have many virtues as mentioned (Baur & Silverman, 2007; Thostenson, Ren, & Chou, 2001). This work is concerned with developing new potential for a new hybridized material using composite matrix material made from CNT, which can be used as an innovation by replacing the synthetic reinforcement. Furthermore, the research work is designed to enhance the material's properties by combining synthetic fibre and natural fibre, for example glass fibre and flax fibre.

#### **1.4 Objectives**

Based on the literature reviews of previous works, it was found that the area in which research can be conducted is in the field of investigating the influence of embedding CNT in natural composite laminates as well as the impact analysis of the above mentioned laminates. Therefore, the objectives of this research work are as follow:

- I. To study the effect of CNT loading in biocomposites.
- II. To analyse the mechanical properties of hybrid biocomposites modified with MWCNT through a series of quasi-static laboratory tests.
- III. To determine the suitability of CNT in newly developed composite materials for structural applications.
- IV. To access the impact and after-impact properties of hybrid biocomposite modified with MWCNTs by performing LVI test and CAI test.

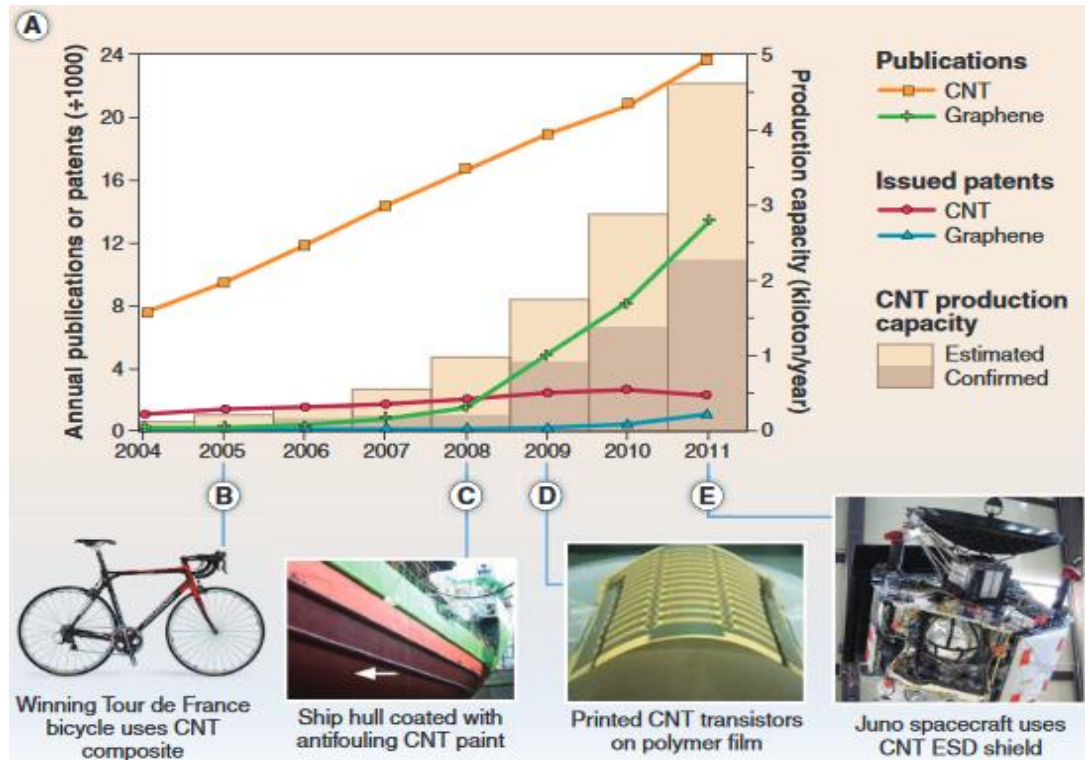
#### **1.5 Research Scope**

A hybrid biocomposite of reinforced epoxy modified with CNT is a combination of synthetic and natural fibres, in which the epoxy resin used for the manufacturing process of the hybrid biocomposite has been modified with MWCNT. In this research work, certain work was necessary to achieve the objectives. The work scope of this research is given below:

- i. Modification of the resin with MWCNT.
- ii. Fabrication of the hybrid biocomposite by the wet lay-up method.
- iii. Checking of CNT dispersion by FESEM.
- iv. Identifying the mechanical performance through a series of quasi-static tests (Tensile test, Flexural test, Impact test, Compression After Impact test and Thermo-gravimetric Analysis (TGA)).

CNT was discovered in 1991 by the Japanese Physicist, Sumio Iijima, and has become the most interesting development for the nanoscience community. CNT is known to have outstanding performances. It is very small but gives

good advantages. Figure 1.1 shows some developments of CNT and graphene in industry; A) Publications, issued patents and annual production. B) to E) Selected CNT products: Composite bicycle frame, antifouling coatings, printed electronics and electrostatic discharge shielding.



**Figure 1.1 : Development of CNT and graphene in industry**  
(Source: Volder, Tawfick, Baughman, & Hart, 2013)

At present, carbon nanotubes are widely utilised in nano-engineering fields, with various physical, chemical and mechanical applications, so that many research studies on the dynamic behaviours of CNT have been published and presented. Since CNT was discovered, the studies on physical and mechanical behaviour of CNT has greatly increased. Although the applications of CNT in engineering fields have been potentially impacted, there is still lack on detailed part of the characteristics of CNT, thus the best design for devices and mechanical application has still not been greatly explored.

## 1.6 Thesis organisation

This book is organised into five different chapters that describe the whole study of a hybrid biocomposite modified with MWCNT. The descriptions of each chapter are given as follows:



- i. Chapter 1; The introduction to the whole research work suggesting natural fibres as reinstatement for synthetic fibres in aerospace industrial applications. This chapter also explained the objectives, problem statements and scope in this research work.
- ii. Chapter 2; Gives literature studies of the common synthetic fibres used in the aerospace industry and previous research on polymer nano-composites. Besides this, the benefits of natural fibres and nano-materials in polymer composites have also been identified.
- iii. Chapter 3; The whole process involved in fabricating a hybrid bio-composite and a polymer nano-composite has been described in this chapter. It also includes all the quasi-static, impact and thermal loading laboratory work for this research work.
- iv. Chapter 4; This depicts the results and discussion of the findings from the experimental analysis. Figures and tables are presented in this chapter to highlight the findings clearly.
- v. Chapter 5; Draws the conclusion from the results and summarizes the findings from the whole project. In this last chapter, potential improvements and future work has also been suggested.

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## BIODATA OF STUDENT

Khairul Izwan Ismail was born in Hospital KL, W. Persekutuan in February 1993 - successfully finishing primary school at the Methodist Boys Primary School (MBPS) and Sek. Keb. Bandar Baru Sentul (SKBBS). The eldest child of En. Ismail Harun and Pn. Rafeah Abd Rahman continued their successful journey in one of the boarding schools in Gombak. After five years in Gombak Integrated Boarding School (INTEGOMB), he pursued his study at Perak Matriculation College in Gopeng for one year. Then, he continued his Bachelor Degree in Aerospace Engineering and Masters of Science at Universiti Putra Malaysia (UPM). Progressing well in his research studies on polymer nano-composites and hybrid composites, under the supervision of dedicated supervisors, Khairul is now completing his Master of Science in his fourth semester.



## LIST OF PUBLICATIONS

### Journals

**Ismail, K. I.**, Sultan, M. T. H., Shah, A. U. M., Mazlan, Norkhairunnisa., Hamdan, A. (2018). Tensile Properties of Hybrid Biocomposite Reinforced Epoxy Modified with Carbon Nanotube (CNT). *Bioresources*, 13(1), 1787-1800. DOI: 10.15376/biores.13.1.1787-1800. ISMAIL – Published

**Ismail, K. I.**, Sultan, M. T. H., Shah, A. U. M. (2018), Impact and After-impact Properties of Hybrid Biocomposite Modified with Multi-walled Carbon Nanotube. *Journal of Composite Part B* – Submitted

**Ismail, K. I.**, Sultan, M. T. H., Shah, A. U. M. (2018), Thermogravimetric Analysis of Hybrid Biocomposite Modified with Multi-walled Carbon Nanotube. *Journal of Material Research and Technology* - Submitted

### Book chapters

Ain U, M. S., Azmi, A. M. R., Safri, S. N. A., **Ismail, K. I.**, A. F. M., Razali, N., Sultan, M. T. H., (2017). Numerical and Experimental Analysis of Delamination in Fibre Reinforced Polymer Composites Failure Analysis in Biocomposites, Fibre Reinforced Composites and Hybrid Composites - Accepted

### Conference

**Ismail, K. I.**, Sultan, M. T. H., Shah, A. U. M., Nor, A. F. M., Azmi, A. M. R., Hamdan, A., (2018). Effect of Carbon Nanotube (CNT) Concentration on Flexural Properties of Flax Hybrid Biocomposite. 6<sup>th</sup> International Conference on Advanced Material Engineering and Technology – Published

Nor, A. F. M., Sultan, M. T. H., Shah, A. U. M., Azmi, A. M. R., & **Ismail, K. I.** (2018). Effects of Carbon Nanotube (CNT) as the Nanofillers into Bamboo/Glass Hybrid Composites on Tensile, Flexural and Impact Properties. 6<sup>th</sup> International Conference on Advanced Material Engineering and Technology – Published

Azmi, A. M. R., Sultan, M. T. H., Shah, A. U. M., Nor, A. F. M., & **Ismail, K. I.** (2018). Tensile Properties of a Kenaf/X-Ray Film Hybrid Composites. 6<sup>th</sup> International Conference on Advanced Material Engineering and Technology – Published

## **Workshops and Seminars**

1. 3<sup>rd</sup> Malaysia Super Satellite Campus (MSSC) International Workshop on Engineering and Health Science 2017: Collaboration for Bioengineering, Mechanics and Nano-materials, Aerospace Manufacturing Research Centre, UPM, 31 January 2017 – Participant
2. Global Aerospace Industry Outlook and Insight into Malaysia's Aerospace Initiatives Talk, Aerospace Manufacturing Centre, UPM, 2 March 2017 – Participant
3. Composite Technology - Current and Future Trends Talk, Aerospace Manufacturing Research Centre, UPM, 16 March 2017 – Participant
4. International Workshop on Advanced Composites and its Manufacturing, Aerospace Manufacturing Research Centre, UPM, 10 April 2017 – Participant
5. Sharing Session on Student – Supervisor Relationship, Aerospace Manufacturing Research Centre, UPM, 2 May 2017 - Participant



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