



**UNIVERSITI PUTRA MALAYSIA**

***DEVELOPMENT AND CHARACTERIZATION OF FLAME RETARDANT  
OIL PALM FILLER/KENAF REINFORCED HYBRID COMPOSITES***

**NAHEED SABA**

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**By**

**NAHEED SABA**

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

**November 2016**

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## DEDICATION

This thesis is exclusively dedicated to:

My beloved parents for their extreme love, sacrifices, encouragements, inspirations,  
compassion and moral support throughout my life

&

My endearing husband and my cute daughter for their support, patience and great  
understanding on me



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

## **DEVELOPMENT AND CHARACTERIZATION OF FLAME RETARDANT OIL PALM FILLER/KENAF REINFORCED HYBRID COMPOSITES**

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**November 2016**

**Chairman: Professor Paridah Md. Tahir, PhD**  
**Institute : Tropical Forestry and Forest Products**

Epoxy is among the most extensively used thermoset polymer in the composite industry, particularly for high performance and advanced applications. However, most cured epoxy systems are extremely brittle, poor resistance to crack initiation, possess lower impact strength and characteristics poor flame retardancy during fire threats, which limits its extensive applications. These weaknesses are more acute when used with natural fibers. To minimize these shortcomings flame retardant (FR) nano filler are incorporated as additive to improve its strength besides its flame retardancy. This study used oil palm empty fruit bunch (OPEFB) fibers obtained from palm oil processing mill for producing FR nano OPEFB. The FR nano sized OPEFB filler was produced through chemical (bromine water and tin chloride) treatment and cryogenic crushing followed by high energy ball milling process. Evaluation under SEM depicts that the surface morphology of the FR nano OPEFB possess amorphous and irregular shape. Thermal analysis revealed that the treated FR nano OPEFB is thermally stable compared to untreated OPEFB fibers. Residual char obtained is 29% for nano OPEFB, 10.85% for untreated (R-OPEFB) and 14.17% for B-OPEFB fibers. Epoxy nanocomposites were fabricated using different nano OPEFB filler loading (1, 3, 5 % by weight) through hand lay-up technique. A marked increase in mechanical properties and flame retardancy were observed for all filler filled epoxy nanocomposites, in particular at 3% loading. Tensile strength of 1% is 21.43MPa, 3% is 29.01 MPa and 5% is 22.61 MPa, while impact strength of 1% is 68.13 J/m, 3% is 98.71 J/m and 5% is 70.62 J/m are observed. LOI value for pure epoxy is 23%, 1% is 25 %, 3% is 29% and for 5% is 28% while, UL-94V ratings for pure epoxy is V-2, 1% is V-1, 3% and 5% is V-0. Mechanical in terms of tensile, impact and elongation at break, morphological, physical, structural, thermal in terms of decomposition temperature and char yield, dynamic mechanical in terms of storage modulus ( $E'$ ) and loss modulus ( $E''$ ),  $T_g$  and damping factor, thermomechanical in terms of coefficient of thermal expansion (CTE) and flame retardancy analysis were conducted by fabricating kenaf/epoxy composites and kenaf/epoxy hybrid nanocomposites each at 40% by weight of kenaf fiber loading. Three types of hybrid nanocomposites namely, nano OPEFB/kenaf/epoxy, montmorillonite

(MMT)/kenaf/epoxy and organically modified montmorillonite (OMMT)/kenaf/epoxy hybrid nanocomposites were fabricated. Considerable improvement in mechanical strength in terms of tensile, impact strength and elongation at break were realized by adding nano OPEFB filler in kenaf/epoxy composites. Tensile strength of kenaf/epoxy composites increases by 24.9% by adding nano OPEFB filler, while 56% increment was recorded by adding OMMT with respect to nano OPEFB/kenaf/epoxy hybrid nanocomposites. Impact strength of kenaf/epoxy increases considerably from 19.13J/m to 24.54 J/m by adding nano OPEFB filler, to 31.32 J/m by adding MMT and to 39.46 J/m by adding OMMT. The profound effects of the nano OPEFB filler addition in reducing void contents and number of fiber pull out from the fractured surface signifies the enhanced adhesion and interfacial bonding between kenaf fibers and matrix. Remarkable improvements in  $E'$ ,  $E''$  and  $T_g$ , while reduction in CTE as function of temperature by adding nano OPEFB filler were also noticed.  $T_g$  value for kenaf/epoxy was increased from 70.1 °C to 80.6 °C by adding nano OPEFB filler. LOI and UL-94V ratings of kenaf/epoxy are 24% and V-2 respectively but the addition of nano OPEFB filler to it increases to 30% and V-0 respectively, for MMT to 28% and V-1 whereas for OMMT to 30% and V-0 rating. Results of the analysis revealed that there are improvements in the properties of the nano OPEFB/kenaf/epoxy nanocomposites which are quite comparable with those of MMT/kenaf/epoxy nanocomposites but lesser than the OMMT/kenaf/epoxy hybrid nanocomposites, except for flame retardancy. In conclusion, the proposed method to develop FR nano OPEFB filler from waste OPEFB fibers represents simple and convenient way in terms of time and energy required for utilizing OPEFB fibers waste efficiently.

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

**PEMBANGUNAN DAN PENCIRIAN KOMPOSIT HIBRID TAHAN  
API DIPERKUKUH DENGAN PENGISI SAWIT/KENAF**

Oleh

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**November 2016**

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Epoksi adalah antara polimer termoset yang paling banyak digunakan dalam industri komposit, terutamanya untuk aplikasi bahan prestasi tinggi dan termaju. Walau bagaimanapun, kebanyakan sistem epoksi adalah amat rapuh, rintangan yang lemah terhadap permulaan retakan, kekuatan impak yang rendah, dan ciri-ciri rencat nyala yang lemah semasa ancaman nyala, yang mana menghadkan aplikasinya yang lebih meluas. Kelemahan-kelemahan ini menjadi lebih berat apabila digunakan bersama gentian semula jadi. Untuk mengurangkan kekurangan ini, pengisi nano kalis nyala diletakkan sebagai bahan tambahan untuk meningkatkan kekuatan selain rencat nyalanya. Kajian ini menggunakan gentian tandan kosong kelapa sawit (OPEFB) yang diperolehi daripada kilang pemrosesan minyak sawit untuk menghasilkan FR nano OPEFB. FR nano OPEFB bersaiz nano telah dihasilkan melalui rawatan kimia (air bromin dan klorida timah) dan penghancuran kriogenik diikuti oleh proses kisar bola tenaga tinggi. Penilaian di bawah SEM menggambarkan bahawa morfologi permukaan OPEFB FR nano yang dihasilkan mempunyai bentuk amorfus yang tidak teratur. Analisis terma mendedahkan bahawa OPEFB FR nano yang dirawat adalah stabil dari segi haba berbanding gentian OPEFB yang tidak dirawat. Sisa menghanguskan diperolehi ialah 29% untuk nano OPEFB, 10.85% untuk tanpa dirawat (R-OPEFB) dan 14.17% untuk serat B-OPEFB. Nanokomposit epoksi telah dihasilkan menggunakan kandungan pengisi OPEFB nano yang berbeza (1, 3, 5% mengikut berat) secara manual. Satu peningkatan ketara dalam sifat mekanik dan rencatan api diperhatikan dalam semua nanokomposit epoksi berisi, khususnya bagi 3% pengisian. Kekuatan tegangan bagi perisian 1% ialah 21.43MPa, 3% ialah 29.01 MPa dan 5% ialah 22.61 MPa. Manakala diperhatikan bahawa kekuatan hentaman perisian 1% ialah 68.13% J/m, 3% ialah 98.71 J/m dan 5% ialah 70.62 J/m. Nilai LOI untuk epoksi tulen ialah 23%, perisian 1% ialah 25 %, 3% ialah 29% dan untuk 5% ialah 28% manakala, pengkadaran UL-94V untuk epoksi tulen ialah V-2, perisian 1% ialah V-1, manakala kedua-dua 3% dan 5% ialah V-0. Peningkatan ketara dalam sifat-sifat mekanikal dan rencat nyala dapat diperhatikan dalam semua nanokomposit epoksi, khususnya pada perisian 3%. Mekanikal dari segi tegangan, kesan

dan pemanjangan pada takat putus, morfologi, fizikal, struktur, haba dari segi suhu penguraian dan hasil char, dinamik mekanikal dari segi penyimpanan modulus ( $E'$ ) dan kehilangan modulus ( $E''$ ),  $T_g$  dan redaman faktor, termomekanikal dari segi pekali pengembangan haba (CTE) dan analisis nyala retardancy telah dijalankan oleh mereka-reka komposit kenaf/epoksi dan nanokomposit kenaf/epoksi hibrid setiap satu pada 40% berdasarkan berat beban serat kenaf. Tiga jenis nanokomposit hibrid telah dihasilkan iaitu nano OPEFB/kenaf/epoksi, montmorilonit (MMT)/kenaf/epoksi dan montmorilonit organik yang diubahsuai (OMMT)/kenaf/epoksi. Peningkatan yang besar dalam kekuatan mekanikal dari segi kekuatan tegangan, kekuatan impak dan pemanjangan pada takat putus dapat dilihat dengan menambah pengisi OPEFB nano dalam komposit kenaf/epoksi. Kekuatan tegangan bagi kenaf/komposit epoksi meningkat sebanyak 24.9% dengan menambahkan pengisi OPEFB nano, manakala 56% dengan menambahkan OMMT. Kekuatan hentaman komposit kenaf/epoksi meningkatkan daripada 19.13 J/m kepada 24.54J/m dengan menambah pengisi OPEFB nano, kepada 31.32 J/m dengan menambahkan MMT dan kepada 39.46 J/m dengan menambahkan OMMT. Kesan mendalam penambahan pengisi OPEFB nano bagi mengurangkan kandungan liang udara dan bilangan serat yang tertarik keluar dari permukaan patah, melambangkan peningkatan pada rekatan dan ikatan antara muka di antara gentian kenaf dan matriks. Peningkatan yang luar biasa dalam  $E'$ ,  $E''$  dan  $T_g$ , manakala pengurangan CTE sebagai fungsi suhu juga dapat diperhatikan. Nilai  $T_g$  untuk kenaf/epoksi meningkatkan dari 70.1°C ke 80.6°C dengan menambahkan pengisi OPEFB nano. LOI and UL-94V pengkadaran kenaf/epoksi ialah 24% dan V-2 masing-masing tetapi tambahan pengisi OPEFB nano bagi ia meningkatkan kepada 30% dan V-0 masing-masing, untuk MMT kepada 28% dan V-1 manakala untuk OMMT kepada 30% dan V-0 kadar. Keputusan analisis menunjukkan bahawa terdapat penambahbaikan terhadap ciri-ciri nanokomposit hibrid OPEFB nano/kenaf/epoksi yang setanding dengan sifat-sifat MMT/kenaf/epoksi tetapi lebih rendah nanokomposit hibrid OMMT/kenaf/epoksi kecuali bagi rencat nyala. Kesimpulannya, kaedah yang dibangunkan bagi menghasilkan pengisi FR OPEFB nano dengan menggunakan sisa gentian OPEFB merupakan kaedah ringkas dan mudah dari segi masa dan tenaga yang diperlukan bagi penggunaan sisa OPEFB yang efisien.



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**Jazak Allahu Khairun!**

I certify that a Thesis Examination Committee has met on 9 November 2016 to conduct the final examination of Naheed Saba on her thesis entitled "Development and Characterization of Flame Retardant Oil Palm Filler/Kenaf Reinforced Hybrid 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

ASTM	American society for testing and materials
ABS	Acrylonitrile butadiene styrene
AC	Activated carbon
Al <sub>2</sub> O <sub>3</sub>	Aluminium oxide
AZD	Azodicarbonamide
Br	Bromine
B-OPEFB	Brominated OPEFB fibers
BS	Bamboo stem
C	Carbon
CB	Carbon black
CFA	Chemical foaming agent
Cl	Chlorine
CTE	Coefficient of thermal expansion
CNS	Coconut shells
CNSL	Cashew nut shell liquid matrix
CNTs	Carbon nanotubes
DeBDE	Decabromodiphenyl ether
DGEBA	Diglycidyl ether of bisphenol A
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
DTG	Derivative thermogravimetry
<i>E</i> *	Complex modulus
<i>E</i> '	Storage modulus
<i>E</i> ''	Loss modulus
EDX	Elemental dispersive X-ray analysis
EMC	Equivalent moisture content
EP	Ethylene-propylene
ER	Epoxy resin
Fe <sub>2</sub> O <sub>3</sub>	Iron oxide
FRs	Flame retardants
FTIR	Fourier transform infrared spectroscopy
GNI	Gross national income
GO	Graphene oxide
HBr	Hydrogen bromide
HCl	Hydrochloric acid
HDP	Hexadecylpyridinium
HDTMA	Hexadecyltrimethylammonium
HEBM	High energy ball milling
H <sub>2</sub> O	Water
IR	Infrared
LiAlH <sub>4</sub>	Lithium aluminum hydride
LOI	Limiting oxygen index
MFC	Microfibrillated cellulose
MPOB	Malaysian palm oil board
MMT	Montmorillonite

NR	Non-rated
OPEFB	Oil palm empty fruit bunch
OPFB	Oil palm fruit bunch
OMMT	Organically modified MMT
OSU	Ohio State University
PBO	Poly p-phenylenebenzobisoxazole
PC	Polycarbonate
PCFC	Pyrolysis combustion flow calorimetry
PEEK	Poly(ether ether ketone)
PHA	Polyhydroxyalkanoates
PI	Phenylethynyl-terminated imide
PLA	Poly lactic acid
PPc	Compatibilized polypropylene
PVC	Poly vinyl chloride
RH	Relative humidity
R-OPEFB	Raw/Untreated OPEFB fiber
Sn	Tin
SnBr <sub>2</sub>	Tin(II) bromide
SnBr <sub>4</sub>	Tin(IV) bromide
SiC	Silicon carbide
SnCl <sub>2</sub>	Tin chloride
SnCl <sub>4</sub>	Tin(IV) chloride
SEM	Scanning electron microscopy
SiO <sub>2</sub>	Silicon dioxide
<i>T<sub>g</sub></i>	Glass transition temperature
Tan $\delta$	Tan delta
TBPA	Tetrabromophthalic anhydride
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
TMA	Thermomechanical analysis
TMWF	Tea mill waste fibers
TiO <sub>2</sub>	Titanium dioxide
TTS	Time-temperature superposition
UHMWPE	Ultra-high molecular weight poly(ethylene)
UL94	Underwriters laboratories 94
UP	Unsaturated polyester
UV	Ultra violet
XRD	X-ray diffraction

## LIST OF SYMBOLS

rpm	Rate per minute
hr	Hour
$\mu\text{m}$	Micrometer
MPa	Megapascal
GPa	Gigapascal
$\text{g}/\text{cm}^3$	Gram per cubic centimeter
nm	Nanometer
$^{\circ}\text{C}$	Degree Celsius
K	Kelvin
J/m	Joule per mole
kJ	Kilojoules
mm	Millimeter
M	Mass
V	Volume
Hz	Hertz
$\text{cm}^{-1}$	Per centimeter
Min	Minute
mg	Milligram
mL/min	Milliliter per minute
Phr	Parts per hundred

## CHAPTER ONE

### INTRODUCTION

#### 1.1. Introduction and Background

##### 1.1.1. Epoxy and polymer composites

Epoxy are one of the most important engineering polymeric materials among thermosets resin, which are commercially available and extensively applied in industrial paintings, coatings, general purpose adhesives, laminated circuit board, electrical and microelectronic materials, electronic insulation, military aircraft industries and in modern industries from past 50 years (Chruściel and Leśniak 2014, Yin et al. 2011). Biocomposites and composites has gained global attention due to increased pressure from environmental activists and growing awareness in society for the development of sustainable and biodegradable polymer composites (Satyanarayana et al. 2009). The term biocomposites and composites are quite interrelated and defined as composite materials in which at least one of the constituents is derived from natural resources. They show potential to replace the conventional traditional materials such as metals and wood for various specific applications, thereby saving weight and reducing energy requirements. Cellulosic fibers are one of most abundant and renewable bio-based materials in nature, grows in crops fields (Majeed et al. 2013) and are used as promising and effective alternate reinforcements or fillers in polymer composites fabrication to the expensive synthetic fibers such as glass, aramid or carbon fiber (Jawaid and Abdul Khalil 2011). Elaborative studies have been reported on variety of cellulosic fibers for the fabrication of polymer composites such as (Srisuwana et al. 2014) Jute (Boopalan et al. 2012) flax (Le Duigou et al. 2010, Modniks and Andersons 2013), hemp (Elkhaoulani et al. 2013) kenaf (Azwa and Yousif 2013) banana (Singh et al. 2012) oil palm empty fruit bunch fiber (OPEFB) (Kasolang et al. 2012) and pineapple fibers (Sapuan et al. 2011) for both thermoset and thermoplastic matrix. The advancement of engineering and nanotechnology based on polymeric materials science coupled with suitable nano filler results new formulation strategies with lighter, thinner, stronger and cheaper nanocomposites having promising applications in construction, buildings-bridges, packaging, aerospace and automotive industries. The term "nanocomposites" appeared in 1994 (Paul and Robeson 2008), and defined as composite materials in which at least one dimensions of the reinforcing filler is in the nano scale (<100 nm) (Biswas and Ray 2001). Currently nanocomposites and hybrid nanocomposites are the highly active area of research with different nano sized fillers for advance structural and non-structural applications (Khare et al. 2014).

##### *Hybrid nanocomposites*

In the composites science, hybrid composites are the systems in which one kind of reinforcing material is incorporated in a mixture of different matrices (poly blends) (Thwe and Liao 2003), or two or more reinforcing and filling materials are present in a

single matrix (Jawaid et al. 2012) or both approaches are combined. Hybrid nanocomposites have shown great promising properties to overwhelm many limitations of traditional composites such as the poor interfacial attraction between the fiber and polymer, hydrophilic properties of cellulosic fibers consequently poor strength which hinder in their industrial applications.

Cellulosic fibers hybrid nanocomposites are quite less common, although they are potentially useful materials with respect to environmental concerns. Few research works has been reported for cellulosic fibers/nano filler hybrid nanocomposite, such as nanoclay/cellulose/ethylene-propylene copolymer (Singh and Pal 2007), nanoclay/rice husk/high density polyethylene HDPE (Kord 2011), nanoclay/foamed HDPE/wheat straw flour (Babaei et al. 2014), pine cone fiber/nanoclay (Arrakhiz et al. 2013), wood flour/polypropylene/glass fiber (Kord and Kiakojouri 2011), nanoclay/micro crystalline cellulose/ethylene-propylene (EP) (Singh and Pal 2007). However, in contrast, a lot of research works has been conveyed involving nano filler reinforcement in synthetic fibers. Some of the important research works on hybrid nanocomposites are, carbon fiber/glass fiber/nano clay/polyamide (PA-6)(Wu et al. 2001), chopped glass fibers/hectorite-type clays (nano size)/polyamide (PA-6) (Akkapeddi 2000), glass fiber/layered silicate/polyamide (PA-6) (Vlasveld et al. 2005), nanoclay/glass fiber/polyester (Jawahar and Balasubramanian 2006), glass fiber/layered silicate/epoxy (Lin et al. 2006). All these hybrid composite revealed good mechanical and physical properties compared to un-hybridized composites.

## 1.2. Problem statements

Despite of several unique feature and applications of epoxies, the combustibility of epoxies still represents serious constrain in structural, high tech-electronic or automotive applications with high health risks as like other thermosets. As epoxies like other polymers on exposing to high temperatures (300–400°C), decomposes releasing smoke, heat, toxic volatiles, and soot from the cured epoxy laminate which ultimately affecting its mechanical properties (Rakotomalala et al. 2010). In order to overcome this situation epoxies need to be modified. It has been established that nano filler even less than 5% by weight are sufficient to overcome in contrast to 40-60% fibers reinforcements for epoxy modifications. Currently, a lot of approach and research strategy has been counted/considered in order to enhance or modify epoxy properties to overcome its shortcomings through the incorporation of nano sized materials, even they are expensive (Saba et al. 2015a, Shukla et al. 2008), such as carbon nanotubes, carbon nanofibers, polyhedral silsesquioxanes (POSS); nanoclay, montmorillonite (MMT), organically modified MMT (OMMT), silane, boron carbide nano particles or graphene that are quite expensive (Saba et al. 2016a).

To the best of the author knowledge to date, no research work has been reported on the development of flame retardant (FR) nano filler from organic material such as oil palm empty fruit bunch fibers (OPEFB). OPEFB fibers composed of cellulose (~ 65%) hemicellulose (~ 40%) and lignin (~29%), hence bromine water used in this study can easily react with lignin and carbonyl of sugar unit, like coir fibers (Sen and Kumar 2010).



In addition to this, the use of OPEFB fibers wastes as nano FR material will provide an ecofriendly and effective way to minimize the deposition, continuous explosive expansion and their wastes management. About 85 million dry tons of solid wastes biomass per annum including oil palm trunk, frond, fruit mesocarp and OPEFB are generated annually in Malaysia (Shinoj et al. 2011). Mature oil palm tree and OPEFB are displayed in **Figure 1.1**. This research study will fulfil the research gap in the context of potential FR materials from organic source by utilizing abundant huge wastes of OPEFB, for the modification of epoxy resin.



**Figure 1.1: (a) Oil palm tree and (b) Oil palm empty fruit bunch (OPEFB)**  
[Courtesy: Prof. Dr. Ir. Sapuan Mohd Salit]

This research work primarily focused to investigate the simple and convenient preparation method of nano filler FR materials through unique combination of chemical treatment followed by nano making process through cryogenizer and high energy ball milling (HEBM) technique. The developed nano filler are then used to fabricate epoxy nanocomposites and hybrid epoxy nanocomposites with combination of locally and easily available kenaf fibers as reinforcement for different applications.

In the present study 1%, 3%, and 5% by weight FR nano OPEFB filler are dispersed uniformly in epoxy matrix to fabricate epoxy nanocomposites through hand lay-up technique. The developed nanocomposites are then characterized to investigate the optimum nano filler loading in the epoxy. The filler loading which shows best in properties are then continued to fabricate hybrid nanocomposites. All kenaf hybrid nanocomposites are fabricated by keeping 40% kenaf fiber loading by weight. In the literature it has been reviewed that for both hybrid and un-hybrid cellulose fiber reinforced polymer composites, 40% fiber loading of cellulose fiber are optimum to improve the mechanical, thermal and physical properties with considerable better fiber/matrix bonding (Jawaid et al. 2013, El-Shekeil et al. 2014). The physical, mechanical, morphological, thermal, dynamic and flame retardancy analysis of nano OPEFB filler/epoxy composites and nano OPEFB filler/kenaf/epoxy hybrid

nanocomposites are evaluated. The properties improvement in different parameter was compared to analyze the effectiveness of hybrid nano composites.

There is a growing demand for an innovative research trend to utilize the waste bio-resources as a cheaper and easily available FR material to convey opportunities for effective waste management issues for extremely lively, safe-secured and exciting upcoming future. However, challenges still involve in achieving multifunctional composites with high-performance mechanical properties and effective flame retardancy at the same time to broaden the range of engineering and domestic goods applications (Saba et al. 2016a).

### **1.3. Research objectives**

The primary aim of this current study is to develop FR nano OPEFB filler by treatment followed by cryogenizer and high energy ball milling techniques, to fabricate and characterize epoxy composites, epoxy nanocomposites and kenaf/epoxy hybrid nanocomposites by incorporating FR nano OPEFB filler. The specific research objectives are as follows:

1. To characterize FR nano OPEFB filler prepared from wastes OPEFB fibers.
2. To evaluate the mechanical, morphological and flame retardancy properties of epoxy nanocomposites.
3. To determine physical, mechanical and morphological properties of nano OPEFB filler/kenaf/epoxy hybrid nanocomposites.
4. To analyse the thermal and flame retardancy properties of nano OPEFB filler/kenaf/epoxy hybrid nanocomposites.
5. To evaluate the effectiveness of nano OPEFB filler/kenaf/epoxy hybrid nanocomposites with MMT/kenaf/epoxy hybrid nanocomposites and OMMT/kenaf/epoxy hybrid nanocomposites in terms of physical, mechanical, morphological, structural, thermal and flame retardancy properties.

### **1.4. Significance of study**

- a. The findings from the current study are expected to enhance the knowledge in developing flame retardant nano material derived from wastes oil palm empty fruit bunch fibers through simple chemical treatment.
- b. It is also expected that the development of nano materials from oil palm empty fruit bunch fibers may help to address environmental problems of disposing the huge amounts of wastes derived from oil palm mill. Hence, they could serve as good alternative to commercially available expensive nano sized fillers.
- c. Time problems associated with the process of developing nano filler only through high energy ball milling can be overcome through unique combination of cryogenizer and high energy ball milling.
- d. In term of waste disposal and its management issue, this research finding delivers a new platform for proper utilizing the huge underutilized

- lignocellulosic wastes derived from agricultural products into valuable flame retardant nano filler and its polymer composites.
- e. The fruitful development of such nano filler from waste of oil palm trees would provide opportunities in fulfilling the growing global need for green and nano products and together contribute towards Malaysia's gross national income (GNI) and will improve the living standard by generating high value jobs for the benefit of Malaysians.
  - f. This study may also add to the effort to unveil the potential application of flame retardant nano filler in modifying the properties of conventional polymer such as epoxy, by fabricating epoxy nanocomposites.
  - g. This study also directs the successful development and fabrication of the epoxy based hybrid nanocomposites with locally, cheaper and abundantly available kenaf fibers, in order to improved physical, mechanical, thermal, thermomechanical along with flame retardancy.

### **1.5. Scope of the study**

The aim of present research work is to fabricate and characterize the feasibility of using FR nano OPEFB filler and kenaf fibers as reinforcement in epoxy matrix. Present study deals with the development of epoxy nanocomposites as well as their hybrid epoxy nanocomposites. The literature survey on hybrid composites and nanocomposites based on epoxy resins indicates that until now, no work has been reported on FR nano OPEFB filler/kenaf/epoxy hybrid nanocomposites. Presently, OPEFB fibers and kenaf bast fibers which is easily available in Malaysia, can be utilized as a reinforcing material along with epoxy resin, as they are still underutilized, thus the concerns about the raw material application problems of this by-product can be solved. Moreover the advantages that shows eminent significance in composite fabrication and applications are offered by both OPEFB fibers and kenaf fibers, comprises cheaper, renewable, biodegradable, easier processing and disposal properties. The novel preparation method of nano filler from OPEFB fibers through distinctive combination of cryogenizer and HEBM method will reduced the cost and save time. As the nano filler until now developed from only high energy ball milling technique requires 32- 60hr at 400-600 rate per minute (rpm). The new combination of nano filler derived from agricultural wastes and cellulosic fiber composite materials frequently exhibit remarkable improvements of physical, mechanical, thermal and flame retardancy compared with conventional composites OPEFB and kenaf fibers reinforced polymer composites.

### **1.6. Thesis organization**

The layout of this thesis is in accordance with Universiti Putra Malaysia alternative thesis format based on publications, in which each research chapter (3 – 10) represent a separate study that has its own: 'Introduction', 'Materials and methods', 'Results and discussion' and 'Conclusions'. Thus, it is important to note that there is no separate chapter dedicated to the materials and methodology. The details of the thesis structure are presented beneath.

## *Chapter 1*

The problems that necessitate this research as well as the research objectives were clearly highlighted in this chapter. In addition, the significant contribution, major challenges/gaps and scope of this study were also explicated within the chapter.

## *Chapter 2*

A comprehensive literature review or scientific information on essential areas connected to the topic (such as, biomass, cellulosic fibers, polymer matrix, nano filler, composites, manufacturing techniques, nanocomposites, natural fiber reinforced polymer nanocomposites, hybrid composites, natural fiber/nano filler hybrid nanocomposites, nanotechnologies application and challenges) of this thesis was presented in this chapter.

## *Chapter 3*

This chapter presents the first article entitled “**Preparation and characterization of fire retardant nano filler from oil palm empty fruit bunch fibers**”. In this article, the preparation of oil palm nano filler from oil palm empty fruit bunch fibers through cryogenizer and high energy ball milling techniques and the different characterization of developed oil palm nano filler was investigated.

## *Chapter 4*

This chapter entails the second article entitled “**Fabrication of epoxy nanocomposites from oil palm nano filler: mechanical and morphological properties**”. The effect of incorporating different loading percentage by weight (1%, 3%, 5%) of oil palm nano filler on tensile, impact properties and on the morphologies of epoxy composites were evaluated in this chapter.

## *Chapter 5*

This chapter presents the third article entitled “**Flame retardancy behavior of oil palm nano filler based epoxy nanocomposites**”. In this article the improvement in fire retardant properties of epoxy composites offered by the incorporation of different loading percentage by weight (1%, 3%, 5%) of oil palm nano filler are analysed. The best loading percentage of oil palm nano filler are intended to be utilized for improving the properties of kenaf fibers reinforced epoxy composites.

## *Chapter 6*

This chapter presents the fourth article entitled “**Effect of oil palm nano filler on mechanical and morphological properties of kenaf reinforced epoxy composites**”. This article deals with the fabrication of oil palm nano filler filled kenaf/epoxy hybrid nanocomposites and its characterization to analyze the effect of nano filler on tensile, impact and morphological properties of kenaf fibers reinforced epoxy composites. Moreover comparative study of nano OPEFB filler/kenaf/epoxy hybrid composites with the MMT/kenaf/epoxy and OMMT/kenaf/epoxy were also investigated in detail.

## *Chapter 7*

This chapter shows the fifth research article entitled “**Thermal properties of oil palm nano filler/kenaf reinforced epoxy hybrid nanocomposites**”. This article deals the fabrication of hybrid nanocomposites and its characterization to analyze the effect of nano OPEFB filler on thermal properties of kenaf fibers reinforced epoxy composites. It also elaborates the comparative study of nano OPEFB filler/kenaf/epoxy hybrid composites with MMT/kenaf/epoxy and OMMT/kenaf/epoxy hybrid nanocomposites.

## *Chapter 8*

This chapter shows the sixth research article entitled “**Dynamic mechanical properties of oil palm nano filler/kenaf/epoxy hybrid nanocomposites**”. In this study the dynamic mechanical properties of nano OPEFB filler/kenaf/epoxy hybrid composites were analyzed and compared with kenaf fibers reinforced epoxy composites. This article also includes the comparative study of nano OPEFB filler/kenaf/epoxy hybrid composites with the MMT/kenaf/epoxy and OMMT/kenaf/epoxy hybrid nanocomposites.

## *Chapter 9*

This chapter shows the seventh research article entitled “**Physical, structural and thermomechanical properties of oil palm nano filler/kenaf/epoxy hybrid nanocomposites**”. In this study the physical, thermomechanical and structural properties of nano OPEFB filler/kenaf/epoxy hybrid composites were analyzed and compared with kenaf fibers reinforced epoxy composites. Moreover, this article also holds the comparative study in physical, thermomechanical and structural properties improvement with the MMT/kenaf/epoxy and OMMT/kenaf/epoxy hybrid nanocomposites.

## *Chapter 10*

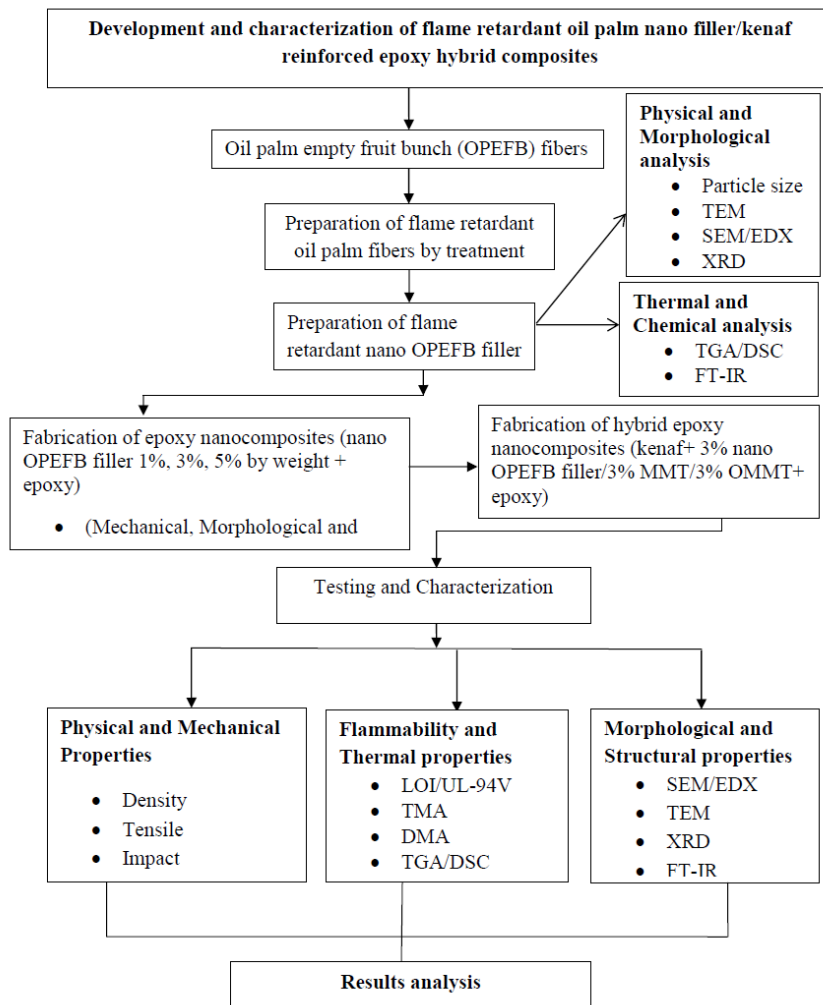
The present chapter displays the eighth research article entitled “**Flame retardancy behavior of oil palm nano filler/kenaf/epoxy hybrid nanocomposites**”. In this article the fire retarding properties of nano OPEFB filler/kenaf/epoxy hybrid composites were analyzed and compared with kenaf fibers reinforced epoxy composites. Furthermore, this article also embraces the comparative study in fire retarding properties improvement in nano OPEFB filler/kenaf/epoxy hybrid composites with the MMT/kenaf/epoxy and OMMT/kenaf/epoxy hybrid nanocomposites.

## *Chapter 11*

Finally, the overall conclusions from the various research articles as well as relevant suggestions for future research were presented in this chapter.

### **1.7. Research methodology flow**

**Figure 1.2** shows the research methodology flow for this thesis. The methodology flow covers every single experiment conducted in this work; ranging from the preparation of flame retardant nano filler from OPEFB fibers to the fabrication of epoxy nanocomposites and kenaf hybrid nanocomposites and their characterization. The photographic images of all equipment used for characterizing the physical, mechanical, morphological and thermal properties of nano OPEFB filler, nanocomposites and hybrid nanocomposites are presented in **Appendix A**.



**Figure 1.2: Research methodology flow chart**

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## LIST OF PUBLICATIONS

### Journal Articles

- Saba N**, Paridah MT, Jawaid M: A Review on Potentiality of Nano Filler/Natural Fiber Filled Polymer Hybrid Composites. *Polymers* 08/2014; 6(8). [Q2, Impact factor: 2.944]
- Saba N**, Paridah MT, Abdan K, Ibrahim NA: Preparation and Characterization of Fire Retardant Nano Filler from Oil Palm Empty Fruit Bunch Fibers. *BioResources* 05/2015; 10(3). [Q1, Impact factor: 1.334]
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- Saba N**, Paridah MT, Abdan K, Ibrahim NA: Physical, Structural and Thermomechanical Properties of Oil Palm Nano Filler/Kenaf/Epoxy Hybrid Nanocomposites. *Materials Chemistry and Physics* 09/2016; 184. [Q2, Impact factor: 2.101]

### Conference paper

- Saba N**, Paridah MT, Abdan K, Ibrahim NA: Thermal Properties of Oil Palm Nano Filler/Kenaf Reinforced Epoxy Hybrid Nanocomposites. *International Congress on Fundamental and Applied Sciences (ICFAS 2016)*. 15th to 17th August 2016. Kuala Lumpur, Malaysia.





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