

# DEVELOPMENT AND CHARACTERIZATION OF RECYCLED CARBON-KENAF FILLED CARDANOL HYBRID COMPOSITES

ZAHRA DASHTIZADEH

**IPTPH 2018 11** 



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By

ZAHRA DASHTIZADEH

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

May 2018

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## **DEDICATION**

This thesis is dedicated to:

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

My respected parents-in-law for their kind support and encouragement,

My beloved husband for his non-stop love, caring and support,

My brothers for their reliable love

&

My lovely son for being such an understanding kid. He is my motivation for being the better version of myself.

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

#### DEVELOPMENT AND CHARACTERIZATION OF RECYCLED CARBON-KENAF FILLED CARDANOL HYBRID COMPOSITES

By

#### ZAHRA DASHTIZADEH

May 2018

#### Chairman: Assoc. Prof. Khalina Abdan, PhD Faculty: Institute of Tropical Forestry and Forest Products

Kenaf fibers have shown a great potential to be a replacement for synthetic fibers due to their high specific strength, light weight, low cost and renewable source. However, like all other natural fibers, the mechanical properties of kenaf fiber are highly affected by its hydrophilic nature. Researchers suggest chemical treatment for fibers to improve its strength and compatibility with a variety of resins. In this research, the results indicate that alkaline and silane treatment with specific concentration increase single fiber tensile strength. The increment is higher for alkaline treatment compared to silane treatment. However, tensile strength of the composites made of treated kenaf fiber and cardanol (bio-phenolic derived from cashew nut shell liquid, CNSL) composite showed small improvement as compared to untreated kenaf fiber and cardanol composites.

Different fiber volume fractions were fabricated, 0%w Kenaf, 30%w Kenaf, 40%w Kenaf, 50%w Kenaf and 60%w Kenaf. Then the mechanical, thermal, physical, chemical and flammability properties of the specimens were studied. The Results showed that composite with 50%w Kenaf shows the highest tensile and flexural strength, while impact strength increased up to 60%w kenaf.

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For further improvement of the mechanical properties, recycled carbon was used to fabricate the hybrid composites. Hybridization with recycled carbon enhanced the mechanical properties of cardanol/ kenaf composites. Also, hybridization of treated kenaf fibers with carbon fibers demonstrated higher mechanical properties.

In addition, thermal characterizations of the specimens indicate that cardanol improved the thermal stability of kenaf also hybridization with recycled carbon further improves the thermal stability of the specimens. Flammability UL 90HB test determines the flame retardancy property of all specimens.

It was observed that the peak of tan  $\delta$  for cardanol was higher than all other specimens which also agreed with the previous results that showed cardanol had a lower damping property and was more brittle than the kenaf/cardanol composites, the shift of tan  $\delta$  and reduction of the absolute value indicate the interaction between kenaf and cardanol which enhanced the mechanical properties of the kenaf/cardanol composites. It was also observed that the specimen with higher fiber volume fraction showed the lower tan  $\delta$ and higher damping property.



Abstrak tesis yang dibentangkan kepada Senat Universiti Putra Malaysia sebegai memenuhi keperluan untuk ijazah Doktor Falsafah

#### PEMBANGUNAN DAN PENCIRIAN KARBON-KENAF DIKENALI KANOAN KOMPOSIT HYBRID CARDANOL

Oleh

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#### Pengerusi : Prof.Madya Khalina Abdan, PhD Fakulti: Institut Perhuntanan Tropika dan Produk Hutan

Gentian Kenaf telah menunjukkan potensi yang besar untuk menjadi pengganti gentian sintetik kerana kekuatan khusus mereka yang tinggi, ringan, kos rendah dan sumber yang boleh diperbaharui. Walau bagaimanapun, seperti semua gentian semulajadi lain, sifat mekanik gentian kenaf sangat terjejas oleh sifat hidrofiliknya. Penyelidik mencadangkan rawatan kimia kepada gentian untuk meningkatkan kekuatan dan keserasiannya dengan pelbagai resin. Dalam kajian ini, hasil menunjukkan bahawa rawatan alkali dan silane dengan kepekatan tertentu meningkatkan kekuatan tegangan gentian tunggal. Peningkatan ini adalah lebih tinggi untuk rawatan alkali berbanding dengan rawatan silane. Walaubagaimanapun, kekuatan tegangan komposit yang diperbuat daripada gentian kenaf dan kardanol yang dirawat (bio-fenolik yang diperolehi daripada cecair cashew kacang cecair, CNSL) menunjukkan peningkatan kecil berbanding dengan gentian kenaf yang tidak dirawat dan komposit kardanol.

Pecahan jumlah gentian yang berbeza dibuat, 0% w Kenaf, 30% w Kenaf, 40% w Kenaf, 50% w Kenaf dan 60% w Kenaf. Kemudian sifat-sifat mekanik, haba, fizikal, kimia dan mudah terbakar dari spesimen telah dipelajari. Keputusan menunjukkan bahawa komposit dengan 50% w Kenaf menunjukkan kekuatan tegangan dan lenturan tertinggi, manakala kekuatan impak meningkat sehingga 60% w kenaf.

Untuk penambahbaikan lagi sifat-sifat mekanik, karbon yang dikitar semula digunakan untuk mengarang komposit hibrid. Hibridisasi dengan karbon kitar semula meningkatkan sifat mekanik kardanol / kenaf komposit. Selain itu, hibridisasi gentian kenaf yang dirawat dengan gentian karbon menunjukkan sifat mekanikal yang lebih tinggi.

Di samping itu, ciri-ciri haba spesimen menunjukkan bahawa kardanol meningkatkan kestabilan haba kenaf begitu juga hibridisasi dengan karbon kitar semula seterusnya meningkatkan kestabilan terma spesimen. Ujian UL 90HB mudah terbakar menentukan sifat kelambatan api semua specimen.

Telah diperhatikan bahawa puncak tan  $\delta$  untuk kardanol adalah lebih tinggi daripada semua spesimen lain yang juga bersetuju dengan keputusan sebelumnya yang menunjukkan bahawa kardanol mempunyai sifat redaman yang lebih rendah dan lebih rapuh daripada kenaf/kardanol komposit, perubahan tan  $\delta$  dan pengurangan nilai mutlak menunjukkan interaksi antara kenaf dan kardanol yang meningkatkan sifat mekanik komposit kenaf / kardanol. Ia juga diperhatikan bahawa spesimen dengan pecahan isipadu gentian yang lebih tinggi menunjukkan tan  $\delta$  yang lebih rendah dan sifat redaman yang lebih tinggi.



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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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# LIST OF ABBREVIATIONS

AFM	Atomic force microscopy
ASTM	American society for testing and materials
С	Carbon
CL	Cardanol
CNSL	Cashew nut shell liquid
CNSO	Cashew nut shell oil
СМ	Compression molding
cm	centimeter
CTBN	Carboxyl-terminated butadiene acrylonitrile
DGEBA	Diglycidyl ether bisphenol A
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
ENR	Epoxidized natural rubber
g	Gram
GFRP	Glass fiber reinforced polymer
HEXA, HMTA	Hexamethylenetetramine (Hexamine)
INTROP	Institute of tropical forestry and forest products
KSBF	Kenaf short bast fiber
kg	kilogram
KŠ	Kenaf stem
MALDI	Matrix assisted laser desorption ionization
MJ	Mega Joule
mm	Millimeter
MPa	Mega Pascal
NaOH	Sodium hydroxide
PLA	Poly lactic acid
PMMA	Polymethyl methacrylate
PP	Polypropylene
PVC	Poly (vinyl chloride)
RCSF	Recycled carbon short fiber
RTM	Resin transfer molding
RW	Rubberwood
SEC	Size exclusion chromatography
sec	Second
SEM	Scanning electron microscopy
TGA	Thermogravimetric analysis
ТК	Treated Kenaf fiber
TPU	Thermoplastic polyurethane
USP	Unsaturated polyester
UTK	Untreated Kenaf fiber

#### **CHAPTER 1**

#### **INTRODUCTION**

## 1.1 Introduction

Environmental issues such as sustainability, renewability, recyclability have risen the attention of researchers and industries to the materials with degradability and great mechanical, thermal and physical performance with low price (Markarian,2008), therefore, natural fibers have been introduced to material science since the artificial fibers have serious disadvantage on the human body and environment.

Substituting synthetic fibers with natural fibers or combining natural fibers with synthetics fibers (Mohanty et al.,2000 a&b) has been a motivation for material scientist to develop natural fiber composites that would enhance the life style of people globally with renewable and sustainable products. From economic aspect, due to increment of energy cost, the final cost of products with good properties is increasing (Abdul Khalil et al.,2011a) demanding the need of high performance materials with low cost.

Natural fibers have shown great advantages over synthetics fibers, such as low cost, light weight, abundant, easy processing and having market appeal (Karimi et al.,2014). Besides, the specific modulus, cost per weight (Nishino et al.,2003), high toughness, low density, low energy requirement for fabrication,  $CO_2$  absorption (Paridah et al.,2011; Mohanty et al.,2000b) are other benefits of natural fibers over synthetic fibers.

However, with all great properties of natural fibers, the hydrophilic nature of the natural fibers decreases the mechanical properties significantly. Natural fibers are used as reinforcement in the resin that is called the matrix (Mohanty et al.,2000a &b). Therefore, the compatibility of the natural fibers and the polymer matrix significantly influences on the stress transfer from matrix to the fibers.

The hydrophobicity of most of the polymers and hydrophilicity of the natural fibers decreases the compatibility of the natural fiber and the polymer matrix. therefore, the poor adhesion between the fibers and resin makes it fail the required mechanical properties in most cases. Some researchers found chemical treatment is useful to overcome these drawbacks and improve or maintain the mechanical properties of the natural fiber composites (Faruk et al.,2012).

Silane treating cellulose fibers with phenolic as the matrix improved the fiber/matrix bonding and also its tensile strength (Rojo et al.,2015), treatment of jute fiber with alkaline and silane improved its interfacial strength between jute fiber and epoxy resin as the matrix (Doan et al.,2012).



In addition, the increment of reinforcement increases the mechanical properties (Ramesh,2016). There is an optimum balance between the amount of the fibers and the resin to achieve reasonably high mechanical and thermal properties. Sugar palm fiber reinforced phenolic composite showed its optimum mechanical properties with 30%vol of fiber in the matrix (Rashid et al.,2016).

In order to increase the reliability of the biocomposites in mechanical properties, the hybrid biocomposites have been introduced. For hybrid biocomposites, compatibility is significant because hybrid biocomposites are made up of two or more fibers in one or more resins. This composition is to increase the mechanical and thermal or other desired properties. Selection between different fibers or polymers from natural or synthetics sources and the amount of the fibers or polymers to be used in the hybrid composites depends on the property that is to be enhanced.

Besides, the mechanical properties of the biocomposites can be improved significantly with hybridization, since it can improve the toughness, for example, by hybridizing natural fibers with glass or carbon fibers that are known for their high toughness (Sanjay and Yogesha,2017). Hybridization of banana fiber with sisal reinforced epoxy composite increased the mechanical properties of the hybrid composite and showed a reduction on water absorption property (Venkateshwaran et al.,2011). A very good tensile and impact strength was reported by Yahaya et al., (2016a) for kenaf/ aramid/epoxy hybrid composites.

Another benefit of hybridization is cost reduction, for instance, to mix costly materials with cheaper materials. Plus, using the natural fibers in hybridization makes the final product lightweight that would decrease the fuel consumption (Alves et al.,2010) for automobile and aerospace industries. Therefore, it is more economical to use natural hybrid composites since decreased fuel consumption results in significant cost reduction. In summary, the hybridization can balance the cost and performance of the biocomposites.

In this research, a hybrid product is investigated with two types of fibers and one resin or matrix. The fibers are kenaf short bast fiber (KSBF) and recycled carbon short fiber (RCSF) while the resin is cardanol (CL) that is a phenolic-based bioresin extracted from cashew nut shell liquid (CNSL). Cardanol is a waste product in the cashew nut shell industry which is abundant in tropical region such as India with reasonable cheap price. This resin is a thermoset resin and is also known for its high-fire retardancy property that is a requirement for many industrial applications. However, the mechanical properties of cardanol based resin have been reported to be poor and the addition of reinforcement could improve the mechanical properties of cardanol based resin significantly (Maffezzoli et al.,2004).

Cardanol that is a novolac phenolic thermoset resin requires a curing agent for its molecular cross-linking. The curing agent is usually hexamethylenetetramine (HEXA) that is also called hexamine (Appendix 2). Phenolic resins are classified into two groups;



resole and novolac. Resole is a one-stage curing resin and does not need the cross-linking agent, while novolac is a two-stage curing resin that requires hexamine for curing. Usually, hexamine that is required for novolac resin is between 10% to 18% (Sathiyalekshmi,1993) of weight percentage of the novolac resin. Since the cardanol is a natural resin, it is assumed to be compatible with the kenaf fiber as compared to other synthetic polymers.

Kenaf is a tropical product with a Persian name that is a member of the Hibiscus cannabinus family. This non-wood plant can grow 4 to 6m per year (Bourguignon et al.,2016). The fibers derived from kenaf plant can be categorized into two groups; core fiber that is the core of the plant and is short and porous which makes it applicable for thermal insulant and absorbent material application. The bast fiber that is from the outer layers that is long fiber for different applications such as textile, paper pulp and as reinforcement in biocomposites (Bourguignon et al.,2016). Kenaf bast fiber is used more in industrial scale due to its higher mechanical properties compared to kenaf core fibers (Tye et al.,2016).

However, the mechanical properties of the natural fibers are still lower than the synthetics fibers since composites made of natural fibers cannot bear high loadings and the maximum loading applicable for natural fiber composites is only 20-30% from its yield strength (Salleh et al., 2014), therefore, recycled carbon fibers have been considered as the hybrid material for their high mechanical properties (Kwon et al., 2017).

Using recycled carbon fibers has a positive impact on the economy and the environment (Meng et al.,2017), because the new carbon fiber is expensive, and after finishing its life cycle, this expensive material is abandoned in landfills. Recycling the carbon fibers is a suitable method in order to reduce the trash in landfills as well as benefiting from its high mechanical properties. Since the carbon fibers have a wide range of modulus from 207GPa to 1035GPa (Flynn et al.,2016), they are used in many high-efficiency applications but mainly aerospace industry. That is another reason for using carbon as the hybrid material in this research because the potential application of this hybrid composite is in the aerospace industry.

The aim of this research was to develop and study a hybrid composite with reasonably high thermal, mechanical and physical performance which is made of environmental friendly biodegradable materials.

## **1.2 Problem statement**

The high end industries, which use high-quality materials such as phenolic which is a phenol content material and is hazardous and pollutant with low degradability (Nady et al.,2017). Therefore, there is a huge need to substitute phenolic compound materials with biodegradable, non-toxic materials to fulfill the environmental and sustainability issues.

Currently, carbon and glass fibers are mostly the different state of the art industry which are non-biodegradable and heavy as compared to natural fibers. The heavy weight of glass or carbon fiber composites increase the fuel consumption (Jabbar et al.,2016). Besides the synthetics fibers are harmful to human health as well as create economic and environmental issues (Yahaya et al.,2015). Nevertheless, recycling the valuable carbon fiber has gained a great attention around the world, because of environmental issues, shortage of landfills and low regulation for waste products (Job,2010; Pickering,2006). Besides, the production of virgin carbon fiber is much more energy consuming than the producing steel, from 286MJ/kg for carbon fiber to 33MJ/kg for steel (Suzuki and Takahashi,2005).

In addition, replacing glass and carbon fibers with natural fibers requires a critical study. Natural fibers with all the benefits of being economical resources, high strength to weight ratio (Mahjoub et al.,2014b), and eco-friendly have some significant drawbacks. For instance, they hydrophilic and tend to absorb moisture that also causes a reduction in mechanical properties (Faruk et al.,2012). In addition, their quality is very much depended on the climate and soil of their farm (Flynn et al.,2016).

Due to increment in price of petrol/ chemical based resin and also environmental issues, natural resins have been studied lately. Cardanol which is a bio-phenolic resin derived from cashew nut shell liquid, is abundant in tropical environments such as in India and benefits from cheap price (Maffezzoli et al.,2004) however, very few studies have been performed on cardanol as a matrix for composites, as well as characterizing its curing parameters such as curing temperature and time.

Cardanol, having biodegradability, high thermal stability and fire retardancy properties is an attractive bioresins for composite structures. Up to date, cardanol has been used mostly to produce different types of thermoset polymer resins, in brake components (Maffezzoli et al.,2004) and in surface coating (S.Riya and S.Deepak ,2015) yet, a full investigation on curing temperature and time, mechanical and thermal properties is required to produce a source of matrix in composite manufacturing field.

In this study, cardanol is aimed to introduce as an interior cabin component which is 100% biodegradable, safe for human health and significantly cheap. This product shows reasonably good thermal, and fire retardancy properties. Considering the benefits of cardanol it is proposed to be used as the matrix and the reinforcement are kenaf bast fiber and recycled carbon fiber to produce a biodegradable composite with high fire retardancy and thermal stability properties.

The hypothesis of this study is that by hybridizing kenaf bast fiber and recycled carbon fiber the mechanical properties of the cardanol would be reasonably enhanced, besides because of high fire retardancy characterization of cardanol the hybrid biocomposite would demonstrate reasonably high thermal stability and flame retardancy performance.



#### 1.3 Objectives

The objectives of this study are

- 1. To optimize chemical treatment solutions and determine interfacial adhesion and strength of kenaf single fiber
- 2. To investigate thermal, FTIR and morphological properties of cashew nut based cardanol
- 3. To characterize mechanical, morphological and physical properties of kenaf/cardanol biocomposites and recycled carbon/kenaf/cardanol hybrid biocomposites
- 4. To characterize the thermal and flammability properties of kenaf/cardanol biocomposites and recycled carbon/kenaf/cardanol hybrid biocomposites

#### 1.4 Scope and limitations

This study was focused on the development of the hybrid biocomposite of kenaf short bast fiber with recycled carbon fiber reinforced with cardanol. Curing parameters of the cardanol are very important aspects that should be determined such as the temperature for curing and post curing which up to the author's knowledge no studies were available for this type of cardanol (the powder form).

The tensile, flexural, impact and water absorption of the hybrid composites were evaluated as well as thermal tests such as thermal gravimetric analyzer (TGA), differential scanning calorimetry (DSC), and dynamic mechanical analysis (DMA) to determine the mechanical and thermal properties of the specimens.

One of the limitations of this study was low mechanical properties of the cardanol as the matrix therefore, the scope was to improve the mechanical properties of the cardanol by reinforcement of kenaf and recycled carbon fiber as the hybridization material. Besides, maintaining the good fire retardancy and thermal stability of cardanol was of the author's considerations. However, since the density of carbon fiber is higher than that of kenaf and cardanol, therefore, the fabrication method with open mold was a limitation of this study, which could not be avoided.

Several researches have studied the mechanical and or thermal properties of different natural fiber hybridization or natural fiber with glass fiber hybridization, however, up to date, recycled carbon fiber has not been hybridized with kenaf fiber.

This hybridization could significantly encourage the industrial applications. In addition, cardanol has not been used as a matrix for kenaf fiber previously. The hybrid of kenaf/recycled carbon fiber reinforced cardanol could demonstrate high performance in mechanical and thermal properties as well as fire retardancy characterizations which

would be an opening field in many industrial applications such as aerospace, automotive and building structures.

## 1.5 Thesis layout

There are five chapters in this thesis as described below:

- 1. Chapter One gives a brief introduction on the background of the study and need for this research. Objectives and the scope of the thesis are also expressed in chapter one.
- 2. Chapter Two elaborates the literature review the previous studies of the hybrid natural composites. The benefits and drawbacks of different hybrid natural composites are also discussed.
- 3. Chapter Three explains the methodology and describes in detail all the steps performed to complete this research by giving the figure for all the tests and specimens to provide the understanding of the methodology of this research.
- 4. Chapter Four clarifies the results and discusses the results of the mechanical and thermal tests. The results are supported and discussed with different graphs, tables and figures to give better knowledge of the specimens characteristics.
- 5. Chapter Five is the conclusion of the thesis which also includes a recommendation for further study. Finally, the thesis is closed with the bibliography of the author, references of the literature used in this thesis and the appendix containing the data sheet for the materials of the hybrid composites.

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