UNIVERSITI PUTRA MALAYSIA

EFFECT OF NANOFILLERS IN JUTE/CARBON HYBRID REINFORCED POLYLACTIC ACID COMPOSITE

NUR AQILAH BINTI SAIRY

EFFECT OF NANOFILLERS IN JUTE/CARBON HYBRID REINFORCED POLYLACTIC ACID COMPOSITE

By

NUR AQILAH BINTI SAIRY

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EFFECT OF NANOFILLERS IN JUTE/CARBON HYBRID REINFORCED POLYLACTIC ACID COMPOSITE

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Chair : **Norkhairunnisa Mazlan , PhD**
Faculty : **Engineering**

The presence of fibres and fillers in a composite can be efficiently arrest crack either at the macro or micro levels. In this work, woven jute and carbon fibres were arranged alternately in polylactic acid (PLA) composite. Graphene or nanoclay was embedded into the PLA matrix to make a polymer nanocomposite. Fibre-reinforced polymer composites were prepared by varying the concentration (1, 3, 5wt%) of graphene or nanoclay in the PLA matrix. The alternate woven jute and carbon fibres aere then bound with the PLA nanocomposite. The influence of graphene or nanoclay concentration and the presence of woven fibres in the composite were quantified by flexural analysis. Flexural strength and flexural modulus were found to increase at 3wt% of nanofiller concentration for graphene/jute/PLA and nanoclay/jute/PLA nanocomposites with the increments up to 37% and 31%, respectively. Low-velocity impact revealed that PLA/TJ/C/G1, PLA/TJ/C/G3, and PLA/TJ/C/G1 have the highest force value for 7J, 10J, and 13J, respectively. These three optimum values for each energy indicate that the closed curve results from the striker's inability to penetrate the specimen. Thus, it was assumed that the lower loading of graphene could withstand the impact energy of 7J, 10J, and 13J. FTIR was used to determine the interaction between PLA and nanofillers. Morphology observation by Scanning Electron Microscopy (SEM) was done to investigate the fractured surface of the hybrid jute/carbon fibres reinforced PLA nanocomposite.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
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KESAN PENGISI-PENGISI NANO DALAM HIBRID JUT/KARBON DALAM KOMPOSIT ASID POLILAKTIK YANG DIPERKUATKAN

Oleh

NUR AQILAH BINTI SAIRY

Ogos 2021

Pengerusi : Norkhairunnisa Mazlan, PhD
Fakulti : Kejuruteraan

Kehadiran serat dan pengisi dalam komposit boleh menjadi kaedah yang berkesan untuk menahan retakan sama ada pada tahap makro atau mikro. Dalam karya ini, tenunan jut dan serat karbon disusun secara bergantian dalam komposit asid polilaktik (PLA). Graphene atau nanoclay dimasukkan ke dalam matriks PLA untuk menghasilkan nanokomposit polimer. Serat yang diperkuatkan oleh komposit polimer telah disediakan dengan pelbagai kepekatan pengisi (1, 3, and 5wt%) dari graphene dan nanoclay dalam matrik PLA. Serat rami dan karbon susunanya yang berselang seli kemudiannya bergabung dengan nanokomposit PLA. Pengaruh kepekatan graphene atau nanoclay dan kehadiran serat tenunan dalam komposit diuji dengan analisis lenturan. Kekuatan lentur dan modulus lenturan didapati meningkat pada 3wt% kepekatan pengisi nano untuk kedua-dua nanokomposit graphene/jute/PLA dan nanoclay/ jute/PLA dengan kenaikan masing-masing hingga 37% dan 31%. Kesan halaju rendah menunjukkan bahawa PLA/TJ/C/G1, PLA/TJ/C/G3, dan PLA/TJ/C/G1 mempunyai nilai daya tertinggi masing-masing untuk 7J, 10J dan 13J. Ketiga hasil optimum untuk setiap tenaga telah menunjukkan bahawa lengkung tertutup hasil daripada penembusan spesimen oleh hentaman yang dikenakan. Oleh itu, diandaikan bahawa pemuatan graphene yang lebih rendah dapat menahan tenaga hentaman 7J, 10J, dan 13J. FTIR digunakan untuk menentukan interaksi antara PLA dan nanofillers. Pemerhatian morfologi dengan Scanning Electron Microscopy (SEM) dilakukan untuk menyiasat permukaan patah dari hibrida rami hibrid/serat karbon yang diperkuatkan nanokomposit

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Norkhairunnisa Mazlan, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Mohamad Ridzwan Ishak, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Nik Noriman Nik Zulkepli, PhD
Associate Professor
Faculty of Engineering Technology
University Malaysia Perlis, UniMAP
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research and the writing of this thesis were done under our supervision;
- supervisory responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2015-2016) are adhered to.

Signature:	
Name of Chairman of Supervisory Committee:	Assoc. Prof. Ts. Dr. Norkhairunnisa Mazlan
Signature:	
Name of Member of Supervisory Committee:	Assoc. Prof. Ts. Dr. Mohamad Ridwan Ishak
Signature:	
Name of Member of Supervisory Committee:	Assoc. Prof. Ts. Dr. Nik Noriman Nik Zulkepli

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CF	Carbon Fibre
CFRP	Carbon Fibre Reinforced Polymer
DMTA	Dynamic Mechanical Thermal Analysis
FLG	Few Layer Graphene
FMLs	Fibre Metal Laminates
FRP	Fibre Reinforced Plastic
FTIR	Fourier Transformed Infrared
GBN	Graphene based nanofillers
GNP	Graphene Nanoplatelets
GCOOH	Graphene Carboxyl
GO	Graphene Oxide
GOH	Graphene Hydroxyl
GPA	Gigapascal
ILSS	Interlaminar shear strength
NaOH	Sodium Hydroxide
PCL	Polycaprolactone
PGA	Polyglycolic Acid
PHB	Polyhydroxybutyrate
PLA	Polylactic Acid
PLA/TJ	Treated Jute fibres reinforced PLA hybrid bionanocomposite
PLA/TJ/C	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite
PLA/TJ/C/G1	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite with inclusion of 1wt% of graphene

PLA/TJ/C/G3	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite with inclusion of 3wt% of graphene
PLA/TJ/C/G5	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite with inclusion of 5wt% of graphene
PLA/TJ/C/N1	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite with inclusion of 1wt% of nanoclay
PLA/TJ/C/N3	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite with inclusion of 3wt% of nanoclay
PLA/TJ/C/N5	Treated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite with inclusion of 5wt% of nanoclay
PLA/UTJ	Untreated Jute fibres reinforced PLA hybrid bionanocomposite
PLA/UTJ/C	Untreated Jute/Carbon fibres reinforced PLA hybrid bionanocomposite
rGO	Reduced Graphene Oxide
SEM	Scanning Electron Microscopy
SMMMT	Surface Modified Montmorillonite
SWCNH	Single Walled Carbon Nanohorns
TPa	Terapascal

CHAPTER 1

INTRODUCTION

The first chapter of this thesis provides a brief description of composite fibre, for instance, natural fibre and synthetic fibre. This chapter also has stated the issues, advantages and disadvantages related to the composite. This chapter also consists of the problem statement, research objective, scope and limitation of the study.

1.1 Background of Study

Synthetic polymers are used in various industries, including the packaging industry. However, they have a negative impact on the environment and trigger a slew of waste disposal and management issues. As a result, there is a growing trend to replace such polymers with biodegradable polymers. Significantly, there is a growing interest in using polymers derived from natural materials like starch (Masuelli, 2013).

Natural fibres are environmentally friendly due to their low-energy combustion, organic nature, and biodegradability. Due to their organic properties and marketing appeal in the composite manufacturing industry, kenaf, flax, hemp, jute, sisal, and banana fibres are introduced as composite reinforcement substitutes. Natural fibres have recently piqued the attention of manufacturers in many industries, who are attempting to create new composites to replace glass fibre-based composites or polymers (Verma and Fortunati, 2019).

Natural fibres are used in several industries, including civil construction, aerospace, and automotive, to name a few. However, natural fibres have certain disadvantages, such as lower mechanical properties compared to glass or carbon fibre-based composites. Furthermore, natural fibres have a much higher water absorption capacity than synthetic fibres (Bharath and Basavarajappa, 2016).

As illustrated in Table 1.1, natural fibres can be classified according to their origin: animal, mineral, and plant. Plant fibres are the most generally recognized by the industry and the most extensively researched by researchers. This is due to product`s short growth cycle, renewability, and widespread availability. According to Peças et al., 2018, vegetable fibres are composed of cellulose, hemicellulose, and lignin, which can be derived from bast, leaf, seed, fruit, wood, stalk, and grass/reed.

Table 1.1: Natural Fibre Classification.

Natural Fibre	Plant	Bast	Jute, Flax, Ramie, Hemp, Kenaf
		Leaf	Banana, Sisal, Abaca, Pineapple
		Seed	Kapok, Cotton
		Fruit	Coir
		Wood	Hardwood, Softwood (e.g., Eucalyptus)
		Stalk	Rice, oat, wheat, maize
		Grass/Reed	Corn, Bamboo
	Animal	Wool/Hair	Lamb wool, Horsehair, Cashmere, Goat hair
		Silk	Mulberry
	Mineral		Metal fibres, Asbestos, Ceramic fibres

(Peças *et al*., 2018)

To address these shortcomings and enhance the properties of natural fibre-reinforced polymer composites, hybrid biocomposites were developed. Hybrid biocomposites are composites made up of two or more fibres bonded together in a single matrix. Dashtizadeh *et al*., (2017), demonstrated that hybrid biocomposites composed of a bio fibre and a nano-reinforced bio-based polymer can be used to mitigate environmental issues while retaining desired industrial properties.

1.2 Significance of Study

Industrial ecology, eco-efficiency, and green chemistry are leading the next generation of materials, goods, and processes using biocomposites (natural fibre composites) made from local and renewable resources. Over the last decade, biocomposites have seen significant growth in the domestic market, construction materials, aerospace industry, circuit boards, and automotive applications. Nonetheless, with proper growth, biocomposites can reach recent markets and promote an increment in demand. Numerous natural fibres have been combined with a polymer matrix to create composite materials that are compatible with synthetic fibre composites, which require special attention. Agricultural wastes can be used to render commercially viable fibre-reinforced polymer composites with marketing appeal. According to Bharath and Basavarajappa (2016), growing global environmental and community concern, a high percentage of petroleum reserves being depleted, and new environmental regulations necessitated the search environmentally friendly composites.

The purpose of this investigation is to determine the effect of incorporating two distinct nanofillers into a hybrid composite reinforced polylactic acid. Due to their large specific surface area, nanofillers can form heat-conducting paths through the composite at extremely low concentrations. Owing to its unique thermal conductivity (~5300 W/mK) via phonon transport, two-dimensional graphene has garnered considerable academic interest for its potential use in fabricating highly thermal conductive polymers (Balandin, 2011; Ghosh et al., 2010). Numerous studies indicate that they may be incorporated into polymers to enhance thermal conductivity (Shtein *et al*., 2015; Araby *et al*., 2014; Du and Cheng, 2012; Veca *et al*., 2009).

This study also emphasizes the three different adding nanofiller ratios (1, 3, and 5wt%) in developing PLA composites to see which loading is the best in mechanical testing. In polymer blends, nanofillers can perform two important functions. Firstly, to strengthen mechanical, barrier, thermal, flame retardant, and electrical properties. Secondly, the alteration of polymer blends' miscibility/compatibility and morphology. Nanoparticles' ability to alter the morphology, interfacial properties, and efficiency of immiscible polymer blends is dependent on their location, interactions with polymer components, and how these additives spread inside the polymer composite (Scaffaro and Botta, 2014).

It has been discovered in recent decades, that incorporating small amounts of these nanofillers into polymers can enhance mechanical, thermal, barrier, and flammability properties without impairing processability. In the ideal nanocomposite configuration, single nanoparticles are uniformly distributed throughout in a matrix polymer. The dispersion of nanoparticles is the most difficult barrier to overcome in order to maximise the potential for property enhancement. The uniform dispersion of the nanofillers results in a wide interfacial area between the constituents of the nanocomposites. Numerous variables, including the polymer matrix`s properties the presence and form of nanofiller, the concentration of polymer and filler, the aspect ratio of the particles, the particle size, the particle arrangement, and particle allocation, are thought to be responsible for the reinforcing effect of filler (de Oliveira and Beatrice, 2018).

The selection of a nanofiller is primarily determined by the desired property enhancements, application, cost, synthesis. Aside from the form and scale of the nanofiller, its interaction with the polymer matrix will majorly impact the processes and kinetics of nanocomposite crystallization, which is closely related to material properties. The number of nanofiller loading dispersed in a polymer matrix influences the composite's property enhancements (Korivi, 2015).

1.3 Problem Statement

The global demand for synthetic-based materials has risen due to of a shortage of crude oil supplies (Ilyas *et al*., 2019). As a result, global societies are becoming more aware of the need to reduce their reliance on synthetic products, prompting the plastic manufacturing industry to seek new alternatives and take urgent action (Jaafar *et al*., 2018). Aside from that, the use of traditional synthetic-based composites has resulted in a slew of environmental concerns, including waste management and long-term environmental viability (Sanyang *et al*., 2018; Merzuki *et al*., 2019).

As a result of growing environmental awareness, green chemistry and engineering have been combined to create a new class of materials via a variety processes. The depletion of petroleum resources, combined with tightening environmental regulations, is accelerating the development of new materials and goods that are both environmentally sustainable and petroleum-free. Taking all of these factors into consideration, a new type of green composite material has been developed. On the other hand, bio-based materials are not only ideal for construction applications such as building construction but also for manufacturing products used in durable goods applications. These bio-based materials are derived from renewable resources such as natural fibre plants, agro-waste, wood, and grasses that contain starch, cellulose, hemicellulose, lignin, and proteins. The majority of chemical products and materials manufactured in the early twentieth century were made from renewable resources. Due to environmental and economic concerns, bio-based polymers and composite materials are becoming more prevalent (Verma and Fortunati, 2019).

Using biodegradable plastics and materials is one of the numerous ways to mitigate the environmental impact of petroleum based. The biological basis for these new biopolymers enables one-of-a-kind exploitation of a highly desired property of these products, namely compostability. It is important to remember that among the plastic waste are items with a high pollution level, and recycling requires a significant amount of energy. As a consequence, compostability is an intriguing property that ensures that these novel biomaterials degrade primarily to carbon dioxide and water after disposal (Kijchavengkul *et al*., 2006). These biodegradable materials exhibit a range of excellent and promising properties in a variety of applications, including packaging, automotive, and biomedical. PLA is a thermoplastic biopolyester manufactured from L-lactid acid, which is usually extracted during the fermentation of corn starch. PLA is currently being commercialised and used as a food packaging polymer for items with a limited shelf life, with applications including drinking cups, containers, overwrap and lamination films, sundae and salad cups, and blister bags (Kale *et al*., 2006).

However, the brittleness of PLA would be a limiting factor on ~~for~~ its tenacity and shock resistance. Finally, when PLA is subjected to extreme weather conditions, its behaviour can become unpredictable. In comparison to conventional thermoplastic polymers, polylactic acid has low heat tolerance and shock resistance. As a consequence, there is a difference between PLA and conventional polymers. Researchers have recently used a variety of nanofillers to increase the efficiency of PLA, including phyllosilicates, carbon nanotubes, hydroxyapatite, layered titanates, and others (Thummarungsan *et al*., 2018; Sun *et al*., 2018b). One method of overcoming the brittleness of the polymer such as PLA is to add or combine it with nanofillers.

The incorporation of low concentrations of these nanofillers into polymers has been shown to improve their mechanical, thermal, barrier, and flammability properties without impairing their processability. Individual nanoparticles distributed homogeneously throughout a matrix polymer are the ideal configuration for a nanocomposite. The dispersion state of nanoparticles is the most challenging obstacle to resolve in order to maximise the potential for ~~of~~ property enhancement. The uniform dispersion of the nanofillers results in a large interfacial area between the constitutent of the nanocomposites. Numerous factors contribute to the filler`s reinforcing effect, including the polymer matrix properties, the nature and shape of the nanofiller, the particle aspect ratio, the polymer and filler concentration, the particle size, particle distribution, and particle orientation. Example of nanoparticles include clays, carbon nanotubes, and graphene (de Oliveira and Beatrice, 2018).

Hybridization, or the combination of two or more types of fibres within a single polymeric framework, is advantageous because it facilitates synergies between the reinforcing materials used, thereby mitigating their inherent disadvantages. The purpose of this research research is to improve the properties of hybrid polymer composite reinforced polylactic acid by adding nanofillers into the approach to deal with the gap study. Three different nanofiller ratios will be combined with polylactic acid to obtain the hybrid polymer composite`s optimal properties. The nanofiller that would be used are graphene and nanoclay. There are a few established research studies that used these two nanofillers, but none of them use jute and carbon fibre as the composite matrix. Thus, this research study would examine the effect of nanofillers in jute/carbon hybrid reinforced PLA composite. When all of the materials in the composite are synthetic, there will be a biodegradable issue. As a result, the addition or combination of natural ingredients helps to resolve the issue.

1.4 Research Objective

This research aims to analyze the different loading of different types of nanofillers reinforced with PLA and jute/carbon hybrid. Therefore, the specific objectives of this research are developed as follows:

- To investigate the effects of different loading nanofillers on PLA/ treated jute/ carbon nanocomposite flexural properties.
- To determine the low-velocity impact of the PLA/ treated jute/ carbon nanocomposite at different impact energy.
- To observe the morphology fractured of PLA/ treated jute/ carbon nanocomposite.

1.5 Scope and Limitation of Study

The purpose of this research is to combine jute and carbon fibre reinforced in polylactic acid (PLA) filled with graphene and nanoclay fillers. The hybrid composite laminate was constructed using treated jute and carbon fibres. 5% sodium hydroxide is the optimal concentration for the jute fibre surface treatment. Two distinct nanofillers were used in the hybrid composite: graphene and nanoclay. The effect of the nanofillers was studied at loadings of 1wt%, 3wt%, and 5wt%. The nanofiller loading was dispersed into the polylactic acid (PLA) using an ultrasonic probe set to a specific parameter, resulting in a thin film. Additionally, the thin film will be inserted into each of the fibre arrangements. Jute and carbon fibres can only be stacked in the following order: Carbon-Jute-Carbon-Jute-Carbon (CJCJC). Thus, the hybrid nanocomposite`s mechanical properties will be analysed using using flexural and low-velocity impact testing. The flexural test determines the amount of force is required to bend a beam under three-point loading. The data is frequently used to determine the material strength.

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