CHARACTERIZATION OF COIR/PINEAPPLE LEAF FIBRE-REINFORCED POLYLACTIC ACID HYBRID COMPOSITES FOR POTENTIAL FOOD TRAY APPLICATIONS

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By

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October 2018

Chairman : Mohammad Jawaid, PhD
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Natural fibre based polymer composites have been widely studied to substitute synthetic materials due to their multiple advantages such as availability, sustainability and biodegradable properties and low cost. The idea of this research is hybridising coir fibres (CF) and pineapple leaf fibres (PALF) into poly lactic acid (PLA) to make biodegradable composites for food packaging applications. The effects of alkali (6%), calcium hydroxide (6%) and silane (2%) treatment on the mechanical, morphological, and structural properties of CF and PALF were studied with the aim to improve their compatibility with PLA matrix. The findings show the overall properties with 6% alkali treatment were compatible with PLA matrix. Mechanical, structural, physical and thermal properties of untreated CF/PALF/PLA hybrid biocomposites were investigated on 30% fibre loading with different ratios of both fibres. It was observed that PALF have better effect on tensile and flexural properties while CF seems to enhance impact strength of the biocomposites and C1P1 biocomposites showed highest mechanical strength. Physical properties such as density, water absorption (WA), and thickness swelling (TS) of biocomposites were analyzed and obtained results indicated that C3P7 had the least density while C1P1 showed least TS. In thermal and dynamic analysis, C1P1 displayed highest degradation temperature and storage modulus while C7P3 revealed least coefficient of thermal expansion (CTE). Overall, the hybridisation effect in CIP1 hybrid biocomposites was the best in untreated biocomposites. Alkali treatment showed significant effects on physical, mechanical and thermal properties of biocomposites. Treated C3P7 hybrid biocomposites showed best tensile properties while treated C1P1 showed highest flexural and impact strength. In case of physical tests, all treated hybrid biocomposites showed lower WA and TS than untreated biocomposites. In TGA, treated biocomposites showed improve degradation temperature and increase weight loss excluding C3P7A. Treated C1P1 revealed the highest storage modulus in DMA. It was reported that the CTE of all the treated hybrid biocomposites

displays lower values against untreated hybrid biocomposites and treated C1P1 shows least CTE. The biodegradability level of biocomposites were characterised by simple burial test (ASTM D570-98) through weight loss (%) of the samples after 30, 60, 90, 120 and 150 day's soil burial and changes in physical and visuals in case of accelerated weathering test in accordance with ASTM G 154-16. Except for neat PLA, which shows almost no weight loss, all the biocomposites shows weight loss and were gradually degraded with time. The percentage weight loss in all the biocomposites was linear with number of days of soil burial. Untreated biocomposites shows higher/faster degradation compared to alkali treated biocomposites attributed may be due to poor fibre matrix adhesion, leading to fastening of degradation. Accelerated weathering tests reconfirm the degradation patterns which were also recorded by image analyser. The weathered samples shows increase surface roughness and change in colours due to the reaction with moisture, UV and humidity in the accelerated weathering chamber. Treated C1P1 hybrid biocomposites showed outstanding overall properties among all hybrid biocomposites and it is suitable for required light density, fair mechanical strength with good biodegradability and is suitable for potential food tray application.

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

PENCIRIAN SABUT KELAPA/SERABUT DAUN NANAS MEMPERKUKUHKAN KOMPOSIT HIBRID POLILAKTIK ASID UNTUK APLIKASI DULANG POTENSI MAKANAN

Oleh

RAMENGMAWII

Oktober 2018

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Gentian asli berpangkalan komposit polimer telah dikaji dengan meluas untuk menggantikan bahan sintetik disebabkan kelebihan berbilang mereka seperti ketersediaan, ketahanan dan ciri-ciri terbiodegradasikan dan kos rendah. Idea penyelidikan ini ialah serat-serat (CF) sabut menghibridkan dan serabut daun (PALF) nanas ke dalam asid laktik (PLA) agar biokomposit yang mudah urai bagi aplikasi pembungkusan makanan. Kesan alkali (6%), rawatan kalsium hidroksida (6%) dan silana (2%) di yang mekanikal, mofologikal, dan sifat struktur CF and PALF telah dipelajari dengan menyasarkan untuk meningkatkan keserasian mereka dengan matriks PLA. Penemuan menunjukkan hartanah keseluruhan dengan 6% perlakuan alkali serasi dengan matriks PLA. Sifat terma, mekanikal, struktur dan fizikal CF tidak dirawat/PALF /hibrid PLA biocomposites telah disiasat di 30% pemuatan gentian dengan nisbah-nisbah berbeza kedua-dua gentian. Ia diperhatikan bahawa PALF mempunyai kesan lebih baik di ciri-ciri tegang dan lenturan manakala CF seolah-olah meningkatkan kekuatan hentaman biocomposites and C1P1 biocomposites menunjukkan kekuatan mekanik tertinggi. Sifat fizikal seperti ketumpatan, penyerapan air (WA), dan ketebalan bengkak (TS) biocomposites telah dianalisis dan diperolehi keputusan menunjukkan bahawa C3P7 mempunyai ketumpatan paling kurang manakala C1P1 menunjukkan paling kurang TS. Dalam analisis dinamik dan terma, C1P1 mempamerkan suhu degradasi yang tertinggi dan modulus simpanan manakala C7P3 mendedahkan pekali pengembangan terma (CTE) paling kurang. Keseluruhan, kesan penghibridan dalam hibrid CIP1 biocomposites ialah yang terbaik dalam biocomposites tidak dirawat. Perlakuan alkali menunjukkan harta bernilai di sifat terma, fizikal dan mekanikal biocomposites. Merawat hibrid C3P7 biocomposites menunjukkan ciri-ciri tegang terbaik ketika merawat C1P1 menunjukkan tertinggi lenturan dan kekuatan hentaman. Jika ujian fizikal, semua merawat hibrid biocomposites menunjukkan lebih rendah WA and TS daripada biocomposites tidak dirawat. Dalam TGA, merawat biocomposites menunjukkan meningkatkan suhu degradasi dan kehilangan berat peningkatan tidak termasuk

C3P7A. Merawat C1P1 mendedahkan modulus simpanan yang tertinggi dalam DMA. Ia dilaporkan CTE semua hibrid dirawat biocomposites mempamerkan nilai rendah terhadap hibrid tidak dirawat biocomposites dan merawat C1P1 menunjukkan paling kurang CTE. Tahap keterbiodegredan biocomposites dicirikan oleh ujian (ASTM D570-98) pengebumian yang mudah melalui kehilangan berat (%) sampel selepas pengebumian tanah dan perubahan 30, 60, 90, 120 dan 150 hari dalam fizikal dan visual jika dipercepatkan mengharungi ujian sejajar dengan ASTM G 154-16. Kecuali PLA teratur, yang menunjukkan hampir tiada kehilangan berat, semua biocomposites menunjukkan kehilangan berat dan beransur-ansur direndahkan dengan masa. Kehilangan peratusan berat dalam semua biocomposites linear dengan bilangan hari pengebumian tanah. Persembahan biocomposites tidak dirawat lebih tinggi / degradasi lebih cepat berbanding dengan alkali merawat biocomposites sifat mungkin adalah disebabkan lekatan matriks gentian miskin, membawa kepada mengancing degradasi. Memecut mengharungi ujian mengesahkan semula corak-corak degradasi yang mana juga direkodkan oleh penganalisis imej. Contoh-contoh ditempuhi persembahan meningkatkan kekasan permukaan dan perubahan kromatik disebabkan tindak balas dengan lembapan, UV dan kelembapan dalam kamar luluhawa yang dipercepatkan. Merawat hibrid C1P1 biocomposites menunjukkan hartanah keseluruhan cemerlang antara semua hibrid biocomposites dan ia ada sesuai untuk ketumpatan cahaya yang dikehendaki, kekuatan mekanik adil dengan keterbiodegredan baik dan ada sesuai untuk permohonan dulang potensi makanan.

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

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This is to confirm that:

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LIST OF ABBREVIATIONS

ASTM	American Society For Testing And Materials
BMC	Bulk Moulding Composite
CF	Coconut Fibre/ Coir Fibre
$Ca(OH)_2$	Calcium Hydroxide
CO_2	Carbon Dioxide
CTE	Coefficient of Thermal Expansion
DMA	Dynamic Mechanical Analysis
DTG	Derivative Thermo-gravimetry
E^*	Complex Modulus
E'	Storage Modulus
E''	Loss Modulus
EFB	Empty Fruit Branch
EG	Ethylene Glycol
FTIR	Fourier Transform Infrared Spectroscopy
H_2O	Water
HDPE	High Density Polyethylene
IFSS	Interfacial Stress Strength
KF	Kenaf Fibre
LC	Liquid Crystalline
LCA	Life Cycle Assessment
LDPE	Low Density Polyethylene
MC	Moisture Content
MFC	Microfibrillated Cellulose
MMT	Montmorillonite
Na (OH)	Sodium Hyroxide
NFPCs	Natural Fibre Polymer Composites
NFRPBs	Natural Fibre Reinforced Polymer Biocomposites
O_2	Oxygen
OPEFB	Oil Palm Empty Fruit Bunch
OPFB	Oil Palm Fruit Bunch
OMMT	Organically Modified MMT
PALF	Pineapple Leaf Fibre
PBS	Poly Butyl Succinate
PC	Polycarbonate
PEG	Poly Ethylene Glycol
PF	Phenol Formaldehyde
PLA	Poly Lactic Acid
PP	Polypropylene
PU	Polyurethane
PVC	Poly Vinyl Chloride
PS	Polystyrene
RH	Rice Husk
RRIM	Reinforced Reaction Injection Moulding
RTM	Resin Transfer Moulding
SMC	Sheet Moulding Composite
SEM	Scanning Electron Microscopy
T_D	Maximum Decomposition Temperature
Tg	Glass Transition Temperature

T_m	Melting Temperature
Tan δ	Tan Delta
TGA	Thermo-gravimetric Analysis
TMA	Thermo mechanical Analysis
TS	Thickness Swelling
UP	Unsaturated Polyester
UV	Ultra Violet ray
WA	Water Absorption

LIST OF SYMBOLS

°C	Degree Celsius
GPa	Gigapascal
g/cm^3	Gram per cubic centimeter
Hz	Hertz
hrs	Hour
J/m	Joule per mole
K	Kelvin
kJ	Kilojoules
M	Mass
MPa	Megapascal
µm	Micrometer
mm	Millimeter
mm/min	Millimeter per minute
nm	Nanometer
N	Newton
Pa	Pascal
cm^{-1}	Per centimeter
V	Volume
Wt%	Weight Percentage

CHAPTER 1

INTRODUCTION

1.1 Background

The growing environmental burden and awareness are forcing the industries to seek more eco-friendly materials for their products. In search of new materials with extraordinary performances at low cost is expanding the area of expectations in recent years. The search has mostly concern on the environmental issues such as “renewable”, “recyclable”, “sustainable” emerging in new type of materials that change the materials from non-renewable and non-degradable to renewable and easily degradable materials (Satyanarayana et al., 2005; Markarian, 2008). There is a global trend in incorporation of natural fibres in polymers matrix for multiple applications. This is mainly due to the low cost, non-pollutant, biodegradability and renewability properties of natural fibres as well as the rising need for lowering the environmental pressure and to ensure sustainability (Fiorelli et al., 2012; Summerscales et al., 2010). This has also led to a considerable change in the research direction of fibre reinforced polymer composites (Yu et al., 2010; Song et al., 2012).Natural fibre based biocomposites has been developed not only for a motivating factor for material scientists, but also an opportunity to improve the life of people around the world by developing renewable and sustainable yet dependable products.

Environmental pollutions and shortage of energy resources worldwide are the challenges for researchers to be finding substitute sources of renewable and sustainable biomass energy resources. Lignocellulosic materials are most suitable and abundant bio resources in the world which annual productions reach up to 170–200 billion tons (Saba et al., 2015a). Due to its light weight and eco friendly nature, natural fibre based biocomposites are centre of attraction for industries over traditional composites (Drzal et al., 2001).Agro-waste fibres such as coconut coir fibres (CF) and pineapple leaf fibres (PALF)draws great attention due to their low density and thermal conductivity to yield cost-effective and lightweight composite products (Asasutjarit et al., 2009).CF are one of the most used natural fibres as reinforcement in polymer composites due to their high structural and wear properties combined with availability (Arrakhiz et al., 2012b).CF have lower cellulose (36–43%) and hemicelluloses (15.7 %) contents, higher lignin content (35.25 %) and microfibrillar angle (30–45) than other natural fibres which results in their relatively low mechanical strength (Nam et al., 2012) as well as highest elongation at break among popular NFs (Nam et al., 2011). Whereas, PALF is a smooth glossy fibre with high cellulosic content (70-82 %), hemicelluloses (18.8 %) and lignin (5-12.7 %) with high tensile strength and it has a softer surface than other NFs (Asim et al., 2015a).

Selection of polymer depends on the requirements for specific applications and performances. Biopolymers like PLA, polycaprolactone (PCL), polyhydroxy butyrate (PHB), soy-oil based epoxy, starch, and polyester amide have been studied by researchers as a potential matrix for compostable and eco friendly composites (Mathew et al., 2005).To facilitate biodegradability in composites, polylactic acid (PLA) is often used as a matrix. PLA is one of the most extensively studied biopolymers mainly due to its high mechanical and thermal properties which are comparable to those of polystyrene, and to its easy production from lactic acid monomer, which is derived from corn starch (Gu, 2009;Huda et al., 2008; Suryanegara et al., 2009). PLA exhibits higher storage modulus and flexural properties, compared to polypropylene (PP), and is naturally decomposable to minimize environmental loads (Han et al., 2012; Gupta et al., 2007). PLA is now one of the most promising polymers for multiple applications, as an alternative to conventional synthetic polymers(Pang et al., 2010; Rasal et al., 2010). However, it has some limitations such as brittleness, low impact strength, and low thermal resistance (Pang et al., 2010; Shukor et al., 2014b). For this reason, the development of bio-based natural fibre composites has been a subject of enormous interest in materials science and technology research for both ecological and environmental reasons (Bajpai et al., 2012a).

1.2 Problem statement

The interest in natural-fibers reinforced polymer composites(NFPC) is growing rapidly owing to their great performance, low cost and low relative density, significant processing advantages as well as bio-degradability (Faruk et al., 2012). Hybrid biocomposites have the potential advantage of light material, cheap raw material from natural origin, and thermal recycling or the ecological advantages of using resources which are renewable and sustainable (Islam et al., 2017). The behaviour of hybrid composites is weighed sum of the individual components in which there is more favourable balance between the inherent advantages and disadvantages in different reinforcements or matrices. NFPC's offer benefits as compared to glass fibres or other synthetic fibres based composites (Jumaidin et al., 2017). However, NFPC also have its own disadvantages such as low mechanical properties, low impact strength, poor water and microbial resistance, poor oxygen and gas barrier properties, and low durability. In order to overcome these limitations, several major technical considerations must be addressed before its utilization for application in food packaging materials i.e., biodegradable food tray. The solutions include incorporation of fibres as reinforcements in polymer composites or hybridization. The challenges comprises of the cost, ease of production operation, availability of raw materials, homogenization of the fibre's properties, adhesion between the fibre and matrix (surface treatments), good mechanical strength water proofing, thermal stability and flame retardant properties (Aji et al., 2012; Sivakumar et al., 2017).

CF is a versatile lignocellulosic fibre extracted from coconut husk with hemicelluloses and lignin as fibre bonding materials. These compositions affect the different properties of CF. CF have lower cellulose and hemicelluloses contents and higher lignin content than other natural fibres, which results in their relatively low tensile strength and modulus as well as highest elongation at break among typical natural fibres.CF are more efficient in reinforcement performance than other natural fibres reinforcement composites (Ticoalu et al., 2010). It is known for its toughness and wear properties which favour it for the application in many industrial end products. These properties of using CF as reinforcements in biocomposites are not only based on excellent characteristics, but also its availability in the tropical countries especially South East Asia region. On the other hands PALF is a silky smooth and longer natural fibre rich in cellulose with high specific strength and stiffness but also face its own problems. At present, pineapple plants are only useful for its fruits and fruit products in Malaysia while it has been extensively used for textiles in Indonesia and Philippines. The exponentially expansion of pineapple plantation in Malaysia has generated enormous amounts of agricultural wastes. The waste materials occupy the re-plantation land and burning of wastage materials is tremendous environmental concern. The primary advantages of using PALF in hybrid biocomposites are its good mechanical strength, economical, easily available, low densities, non-abrasiveness and biodegradability. Like all other natural fibres, CF and PALF face some limitations such as the innate hydrophilic behaviour which can be controlled with surface treatments. Interfacial bonding is the key issue in terms of overall performance. Surface-treatment of fibres changes the composition and ultimately changes not only its properties but also the properties of composites. For this purpose, three different chemicals such as NaOH (6%), Silane (2%) and $Ca(OH)_2$ (6%) were used for surface modification of CF and PALF. These treatments were incorporated to improve the interfacial bonding between fibres and matrix by reducing fibre's hydrophilicity.

PLA is a commercially and environmentally interesting biopolymer as it has many unique characteristics, including good transparency, glossy appearance, high rigidity, and good processability.PLA is and was frequently used for packaging materials. However, there are serious limitations, notably inherent brittleness and poor toughness (less than 10% elongation at break), slow degradation rate (hydrolysis of backbone ester groups), which impede its extensive application (Qin et al., 2011; Zhao et al., 2011). Nevertheless, numerous tests have shown that PLA is also suitable as matrix for the embedding of fibres in composites. Some products of natural fibre-reinforced PLA are already established at the market (Graupner et al., 2009). PLA offers a possible alternative to the traditional non-biodegradable polymers especially when their recycling is difficult or not economical. Even though there are many limitations due to its material properties, a number of these challenges are expected to overcome through blending PLA with other polymers, reinforcing of PLA matrix with natural or synthetic fibres, the addition of micro and nanofillers associated with selected additives etc.

A proper blend of CF: PALF in PLA matrix can possibly develop a hybrid biocomposites which can match the thermo-mechanical properties similar to synthetic polymer and fibre based composites which are being used in manufacturing of food packaging materials. The key requirements of composites for food packaging material are non-toxic or harmful, standard rigidity/stiffness with acceptable barrier properties. These can be achieved by an innovative formulation of significant amount of weight and a right surface treatment of reinforcing natural fibres without compromising its degradation. The alkali (NaOH) treatment of CF and PALF could significantly improve the interfacial bonding between fibre and the matrix and ultimately improve the overall properties of hybrid biocomposites.

Research on hybrid natural fibres reinforced PLA composites is still very limited and no report of hybrid CF/PALF/PLA hybrid composites has been found. This research work tries to develop an inventive formulation of CF/PALF/PLA hybrid biocomposites for a potential application in biodegradable food tray through various characterizations necessary for developing biodegradable food tray. Finally, it is expected that the utilisation and usage of CF/PALF/PLA hybrid composites would lead to the fabrication of biocomposites for biodegradable food tray applications.

1.3 Research objectives

The aim of this study is to develop an innovative formulation of untreated and alkali treated CF and PALF reinforced PLA hybrid biocomposites for a potential application in biodegradable food tray. With this aim, the physical, mechanical thermal and biodegradability properties of untreated and alkali treated CF and PALF reinforced PLA biocomposites were studied to find best suitable hybrid biocomposites potential for biodegradable food tray application.
The research, based on CF, PALF and PLA hybrid biocomposites was carried out by using four research objectives. The objectives are: -

1. To analyse the effect of surface treatment on mechanical, physical, structural and morphological properties of CF and PALF.
2. To develop and characterize the physical, mechanical, morphological and thermal properties of CF/PALF/PLA hybrid biocomposites.
3. To determine the effect of alkali treatment of CF and PALF on the physical, mechanical, morphological and thermal properties of CF/PALF/PLA hybrid biocomposites
4. To evaluate the effect of weathering on physical and structural properties of untreated and Alkali treated CF/PALF/PLA hybrid biocomposites.

1.4 Significance of the Study

The findings of this study are expected to improve the knowledge in developing CF/PALF/PLA hybrid biocomposites.

1. The development of biodegradable materials with improved properties in the current study is expected to aids in addressing environmental issues regarding substitution of plastic based products.
2. This study highlights that the usability of materials like natural fibres/ agricultural wastes could be improved by fibre surface treatments.
3. In case of hybrid biocomposites, this study explores the potential of CF/PALF/PLA for different applications.
4. In addition, this study also employs fibre treatments for the development of hybrid biocomposites. Thus, added a significance study for the treatment of natural fibres and its effect on the improvement of interfacial adhesion with the matrix.
5. Last but not the least, this study encompasses the degradability of CF/PALF/PLA hybrid biocomposites which is one of the prime solutions for plastic pollution.

1.5 Scope of study

The aim of present work is to fabricate and characterize hybrid biocomposites using coir fibres (CF) and pineapple leaf fibres (PALF) in polylactic acid (PLA) matrix for potential food tray applications. In this research, fibre surface modification of CF and PALF was performed in various treatments such as alkaline (6%), calcium hydroxide (6%) and silane (2%) for three hours under room temperature. These treatments were incorporated to improve the interfacial bonding between fibres and matrix by reducing fibre's hydrophilicity. Surface-treatment of fibres changes the composition and ultimately changes not only its properties but also the properties of composites.

Various characterisations were performed on treated fibres and the most effective fibre treatment was further investigated for the development of CF/PALF/PLA hybrid biocomposites. The biocomposites were fabricated through melt mixing compounding followed by hot press moulding. The physical, mechanical, thermal and biodegradability properties of developed untreated and treated biocomposites were analysed. Several researchers have already worked on other natural fibres such as kenaf, sisal, banana, oil palm, jute, hemp, bamboo, roselle, flax, silk etc. The literature survey on hybrid composites based on polylactic acid biopolymer indicated that until now, no work has been reported on PLA based hybrid biocomposites of CF and PALF. PALF is underutilized material which is rich in cellulose content and can be commercialized after projecting it as a reinforcing material in polymer composites as other application processes are costly and not easily available locally.CF is already well known in Malaysia as well as South-East Asia. The compatibility of both fibres to each other and their combination of strength will encourage to industrialize these fibres. This new introduction of hybrid biocomposites materials frequently exhibits remarkable improvements of mechanical, physical, and material properties compared to CF and PALF based composites individually. In addition, natural fibres are susceptible to microorganisms and their biodegradability is one of the most promising aspects of their incorporation in polymeric materials.

With the combination of a good strength, thermal and biodegradability of developed hybrid biocomposites will extend the area of opportunity for industries to fabricate different products. The packaging sectors especially food packaging have been identified as future industries for natural fibres and biopolymers based hybrid biocomposites. Some long-term and disposable packaging materials are already produced by natural fibre based biocomposites, based on polylactic acid, polyhydroxybutyrate (PBH), polyglyconic acid and polyethylene etc.Advancement in food packaging materials is expected to increase with the advent of cheap, renewable and sustainable materials with enhanced barrier and mechanical properties such as natural fibres reinforced polymer composites.

1.6 Organization of Thesis

This thesis has been structured into five respective chapters:

Chapter 1
This chapter highlighted the background of this research such as origin of the natural fibre composites, requirements and difficulties during process, research objectives, significance and scope of the study.

Chapter 2
This chapter provides the research and literature area of natural fibres, polymers, biocomposites and hybrid biocomposites.

Chapter 3
Third chapter covers the information about materials which were being used in this research and their characterization and methodology.

Chapter 4
This chapter provides the explanation about results of each characterization and discussion based on the scientific point of view
.

Chapter 5
This chapter presents the overall conclusions from the whole study and future aspects.

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