UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT AND CHARACTERIZATION OF BAMBOO CELLULOSE-BASED BIOCOMPOSITE AND BIONANOCOMPOSITE FOR PACKAGING FILMS

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DEVELOPMENT AND CHARACTERIZATION OF BAMBOO CELLULOSE-BASED BIOCOMPOSITE AND BIONANOCOMPOSITE FOR PACKAGING FILMS

By

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Cellulose in microcrystalline and nanocrstalline forms are different class of cellulose particles having distinct functionalities and properties compared to molecular cellulose, wood pulp etc. There is a great focus on research and development, and commercialization in this field because of the unique combination of characteristics (e.g., good mechanical properties, sustainability, etc.) and utility across a wide spectrum of material applications. This research investigates the characterization of extracted microcrystalline cellulose (MCC) and nanocrystalline cellulose (NCC) from bamboo fiber; bamboo is one of the most widely available natural resources which is rich in cellulose. Both MCC and NCC are used in poly (lactic acid) (PLA)-poly (butylene succinate) (PBS) polymer matrix based biocomposites. After extracting MCC and NCC, different characterizations like Fourier transform infrared (FTIR), Scanning Electron Microscopy (SEM), X-ray diffraction etc. were carried out. Results indicate substantial removal of lignin and pure MCC with minor residues obtained with higher crystallinity ranging from 62.5% - 82.6% and better thermal stability compared to starting material (Bamboo fibres) thus promising to be potential reinforcing element for green composites. The extracted NCC possessed nanometer scale dimensions and morphologies revealed rod like structures with higher degree of crystallinity of about 86.96%. Later the bamboo extracted MCC was used as reinforcing agent in fabricating biocomposites with PLA-PBS blend. The results reveal that the thermal stability of the PLA-PBS blends enhanced on addition of MCC up to 1wt % due to their uniform dispersion in the polymer matrix. Tensile properties declined on addition of PBS and increased with MCC above (0.5 wt%) however except elongation at break increased on addition of PBS then decreased insignificantly on addition of MCC. Thus, PBS and MCC addition in PLA matrix decreases the brittleness, making it a potential contender that could be considered to replace plastics that are used for food packaging. Along the same lines bamboo extracted NCC was used as reinforcing agent with

PLA-PBS blend for fabrication of biocomposite. PLA-PBS blend shows homogeneous morphology while the composite shows rod-like NCC particles, which are embedded in the polymer matrix. The results reveal uniform distribution of NCC particles in the nanocomposites improves their thermal stability, tensile strength, and tensile modulus up to 1 wt.%; however, their elongation at break decreases. Thus, NCC addition in PLA-PBS matrix improves structural and thermal properties of the composite. The composite, thus developed, using NCC (a natural fiber) and PLA-PBS (biodegradable polymers) could be of immense importance as they could allow complete degradation in soil, making it a potential alternative material to existing packaging materials in the market that could be environment friendly.

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

PEMBANGUNAN DAN PENCIRIAN BIOKOMPOSIT DAN BIONANOKOMPOSIT BERASASKAN SELULOSA BULUH UNTUK FILEM PEMBUNGKUSAN

Oleh

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Selulosa dalam bentuk mikrokristalin dan nanokristalin adalah kelas zarah selulosa yang berbeza yang mana ianya mempunyai fungsi dan sifat yang berbeza berbanding selulosa molekul, pulpa kayu dan lain lain. Ianya menjadi tumpuan yang besar di dalam penyelidikan dan pembangunan, serta pengkomersilan dalam bidang ini kerana terdapat ciri-ciri unik serti ciri mekanikal yang baik, kemampanan dan utiliti merentas spektrum luas bagi tujuan aplikasi bahan. Penyelidikan ini mengkaji pencirian selulosa mikrohabluran (MCC) dan selulosa nanohabluran (NCC) yang diekstrak daripada gentian buluh, yang mana buluh adalah salah satu sumber semula jadi yang paling banyak didapati serta kaya dengan selulosa. Kedua-dua MCC dan NCC digunakan dalam biokomposit polimer berasaskan matriks poli (asid laktik) (PLA)-poli (butylene succinate) (PBS).Selepas mengekstrak MCC dan NCC, pencirian berbeza seperti Fourier transform infra merah (FTIR), Scanning Electron Microscopy (SEM), ujian sinar-X dan lain-lain telah dijalankan. Keputusan menunjukkan penyingkiran lignin dan MCC tulen dengan sisa-sisa kecil yang diperoleh dengan kehabluran yang lebih tinggi antara 62.5% - 82.6% dan kestabilan haba yang lebih baik berbanding bahan asli (gentian buluh). Ia nya sekali gus menunjukkan unsur pengukuh yang berpotensi untuk menghasilkan komposit yang selamat. NCC yang diekstrak mempunyai dimensi skala nanometer dan morfologi. Ia menunjukkan struktur seperti rod dengan tahap kehabluran yang lebih tinggi iaitu kira-kira 86.96%. Kemudian MCC yang diekstrak buluh digunakan sebagai agen pengukuh dalam fabrikasi biokomposit dengan campuran PLA-PBS. Keputusan menunjukkan bahawa kestabilan terma adunan PLA-PBS dapat dipertingkatkan apabila penambahan MCC sehingga 1wt %, ini adalah disebabkan oleh keseragaman penyebaran dalam matriks polimer. Ujian ketegangan menunjukkan penurunan apabila penambahan PBS berlaku dan ianya meningkat dengan MCC melebihi (0.5 wt%), walaubagaimanapun semasa ujian pemanjangan putus di jalankan dan ianya

menunjukkan peningkatan pada penambahan PBS, kemudian menurun secara tidak ketara apabila penambahan MCC. Oleh itu, penambahan PBS dan MCC dalam matriks PLA dapat mengurangkan kerapuhan, dan ia berpoteni sebagai bahan untuk menggantikan plastik yang digunakan di dalam pembungkusan makanan. Oleh yang demikian, NCC yang diekstrak buluh digunakan dilihat sebagai agen pengukuh dengan campuran PLA-PBS untuk tujuan fabrikasi biokomposit. Campuran PLA-PBS menunjukkan morfologi homogen manakala komposit menunjukkan zarah NCC seperti rod, yang berada dalam matriks polimer. Hasilnya kajian mendedahkan taburan seragam zarah NCC dalam nanokomposit meningkatkan kestabilan terma, kekuatan tegangan dan modulus tegangan sehingga 1 wt.%. Walau bagaimanapun, ujian pemanjangan semasa putus dilihat berkurangan. Oleh itu, penambahan NCC dalam matriks PLA-PBS meningkatkan sifat struktur dan haba komposit. Komposit yang dibangunkan dengan menggunakan NCC (serat semula jadi) dan PLA-PBS (polimer terbiodegradasi) boleh menjadi sangat penting kerana ia boleh membenarkan degradasi yang sempurna di tanah serta menjadikannya bahan alternatif yang berpotensi untuk bahan pembungkusan sedia ada di pasaran yang bercirikan mesra alam.

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LIST OF ABBREVIATIONS

α	Alpha
β	Beta
°C	Degree celsius
%	Percentage
A600nm	Optical density at wavelength 600 nanometer
µL	Microliter
µm	Micrometer
G	Gram
L	Litre
M	Molar
mA	MilliAmps
PBS	Poly (butylene succinate)
PLA	Poly (lactic acid)
AFM	Atomic force microscopy
CNC	Cellulose Nanocrystals
DI	Deionized Water
DSC	Differential Scanning Calorimetry
DTG	Derivative Thermogravimetry
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
H_2SO_4	Sulfuric Acid
HCl	Hydrochloric Acid

L	Litre
M	Molar
mA	MilliAmps
MCC	Microcrystalline Cellulose
NaClO2	Sodium Chlorite
NaOH	Sodium Hydroxide
NCC	Nanocellulose
PBA	Poly Butyl Acrylate
PBS	Poly (butylene succinate)
PLA	Poly (lactic acid)
RTO	Research And Technology Organizations
SEM	Scanning Electron Microscopy
Tc	Crystallisation Temperature
Tg	Transition Temperature
Tg	Glass Transition Temperatures
TGA	Thermal Gravimetric Analysis
Tm	Melting Temperature
Tm	Melting Temperatures
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Background and Introduction

Over last two decades, polymeric materials have witnessed a widespread use in the various industries globally owing to their better adaptability, durability, and cost-effectiveness; their usage being so much widespread that there is hardly any product that does not contain it in one form or the other. However, many of the synthetic polymeric materials use petroleum as a raw material and there are environmental and sustainability concerns. The widespread use of nonbiodegradable materials like plastics in our daily lives has exacerbated the problems related to environmental pollution as their chemical structure makes them resistant to many natural degradation processes.

One of the crucial issues the world is facing today is environmental pollution, as it continues to pose serious challenges to all forms of life. The increasing awareness and urgency of need of safeguarding our environment has seen many regulations put in place by various institutions that encourage, and sometimes necessitate use of environment friendly materials and emphasize on recyclability of materials. The increasing environmental degradation and sustainability concerns have played an important role towards guiding the exploration, exploitation and application of the environmental friendly materials, products and processes(Omer & Noguchi, 2020; Qureshi et al., 2019; Resasco et al., 2018). Fraunhofer-Gesellschaft (Horizon Europe) and its partners in the European Research and Technology Organizations (RTO) identify environmental degradation, climatic protection, resource efficiency and raw materials the core issue to address, as outlined in United Nations Sustainable Development Goals (SDGs) (Nofar et al., 2019).

Recent years have witnessed many research institutions and manufacturers putting strenuous efforts towards research, development, and application of environment friendly materials. In this regard biopolymers have gained prominence with an increasing number of research studies showing promising results and indicating the potential use of biopolymers in new materials used in various applications, such as food packaging, drug delivery, edible film and tissue membranes in various fields including biomedical applications, food industry, packaging, energy sector and pharmaceuticals. Biopolymers are different from fossil-fuel-derived polymers especially due to their sustainability and biodegradability properties. A variety of biopolymer composites using different types of biomaterials derived from different natural renewable sources have been developed utilising natural fibres rich in cellulose to develop the composites of desired properties which are gradually replacing synthetic polymers and paving the way for development of a variety of biocomposites (Abdollahi et al., 2013; Jariwala & Jain, 2019; Jeyapragash et al., 2020; Khan et al., 2018; Peças et al., 2018; Siakeng et al., 2019). The research works establish that nanocomposites are a promising means of enhancing mechanical and barrier properties of biopolymers thus making them as an alternative to fossil-fuel-derived polymers.

Bionanocomposites have emerged as an important group whose materials comprise of biopolymer matrix reinforced with organic nanoparticle having at least one dimension in the range of 10-100 nm. Due to the larger surface area and higher aspect ratio of its nanoparticles, these composites exhibit better properties, that include chemical, thermal, mechanical etc., in comparison to the pure biopolymers. Moreover, its biodegradability property makes it possible to decompose the material in natural surroundings by converting into other compounds or by the altering its chemical structure, structural, and mechanical properties which prove to be nontoxic to the environment (Jamshidian et al., 2010; Miller, 2018; Pandey et al., 2010; Reichert et al., 2020; Silva et al., 2020). It also is an alternative for fossil-fuel-derived polymers and thus provides solution by reducing carbon footprint .It also provides solution to the issues related to disposal methods of persistent packaging materials and fast depleting petroleum reserves, (Correa et al., 2019; Mousa et al., 2016; Youssef & El-Sayed, 2018; Zubair & Ullah, 2020).

Cellulose fibres are the most abundant biopolymers among all the natural fibres on earth. In recent decades, due to hierarchical structure of cellulose for allowing production in nonmetric dimensions, it has proven beneficial in the field of nanotechnology. In addition, cellulose material can be easily obtained from forest wood resources, non-wood sources like kenaf, bamboo etc. and agricultural residues like sugarcane bagasse, corncob, etc. (Ditzel et al., 2017; Feng et al., 2018; Hua et al., 2020; Maepa et al., 2015; Pennells et al., 2020; Qian et al., 2018).

Among various resources, bamboo has been of particular interest because of various characteristics and advantages it possesses. It one of the most widely available and utilized natural resources; available abundantly in south east Asian countries with Malaysia alone contributing around 1.9% of the global bamboo resource (Duncan, 2011; Yam, 2010). It is a potential reinforcement agent for different polymer composites as their fibres has excellent mechanical properties viz. low elongation at break, high tensile strength, high specific stiffness, and strength. It is composed of cellulose, hemicellulose and lignin contributing more than 95% of the total mass of bamboo (H. A. Khalil et al., 2012). Cellulose contains both crystalline and amorphous structure. The amorphous region is removed to get highly crystalline cellulose. It also has higher percentages of cellulose content (about 73.83%) (Yueping et al., 2010) in comparison to oil palm (J. Z. Lu et al., 2006), Coconut, Corn stalks, Rice straw (Leão et al., 2006; Mwaikambo, 2009; Satyanarayana et al., 2009), Kenaf (H. S. A. Khalil et al., 2006; Thiruchitrambalam et al., 2009) and Sisal (Thiruchitrambalam et al., 2009).

Polymers have played a significant role in growth, development and progress of human civilization and there is hardly any area where one cannot find its application. The significance of polymers is more emphasized because of their widespread application in science, technology, and industry. However, their widespread usage has also created a negative impact on our environment. There is an increasing focus on the development of sustainable and environment friendly materials and in this regard a variety of biopolymers such as PLA (Felfel et al., 2015), PBS (Yue et al., 2018), poly(hydroxyl butyrate (J. Chen et al., 2017) and polycaprolactone (Tanaka et al., 2006) have received lot of attention, and are being considered as alternatives because of their environmental friendly characteristics. PLA and PBS are especially important and have become a focus of research due to of their characteristics that include biodegradability, biocompatibility, and ease of processing.

Although, there is quite some literature reporting the use of nanocrystalline cellulose (NCC) reinforced biopolymer composites, there is no comprehensive work done yet on the composites that makes use of bamboo-based cellulose. This research work focusses on PLA-PBS blends reinforced bamboo based cellulose bionanocomposites which are PLA-PBS-NCC and PLA-PBS-MCC.

1.2 Problem Statement

The widespread use of fossil-fuel-derived materials like synthetic polymers, plastics etc. has caused serious concerns especially with regards to their limited and dwindling resources, sustainability, and variety of environmental issues. In order to tackle these issues, bio-based resources that are abundantly available can be utilized for producing biodegradable materials. There is lot of emphasis on utilizing of bio-based resources for development of eco-friendly materials and rapid progress is being made in this regard. However, such materials lack important properties that are required to replace fossil-fuel based materials. They have poor thermal and mechanical properties in comparison to fossil-fuel derived polymers. Furthermore, their barrier properties, which are especially important for packaging applications, are also poor when compared to the fossil-fuel-derived polymers. Poly (lactic acid) (PLA) is one such biomaterial. It is a bioplastic material that originates from the renewable resources, which is mostly preferred and extensively studied as environmental and sustainable material. It has high strength, high modulus, and good clarity. However, PLA is brittle, with low toughness, slow degradation, slow rate of crystallisation and elongation at break less than 10% that restrict its usage. On the other hand, natural fibres have proven to be effective reinforcing materials. To develop fully biodegradable materials that could be used in different applications like packaging films, it is essential that both the reinforcing agent and other materials used in various matrices need to be biomaterials or their derivate.

In the light of the above, this research focuses on the development of Biocomposites using biopolymers blends and natural fiber. Poly (lactic acid) (PLA) and Poly (butylene succinate) (PBS) are two very important biodegradable polymers which have been used in various applications. Both PLA and PBS are economically viable and have significant commercial interest. PLA is a biopolymer and possesses higher indices of crystallinity and transparency. It is biodegradable plastic that can be decomposed by microorganisms such as bacteria, fungi, algae and yeast in the natural environment. It is easily degraded by enzymatic reaction when buried in landfill sites. Both PLA and PBS being eco-friendly natural polymers with bamboo-based fibre as a natural reinforcing agent can be used to develop natural fiber polymer biocomposites. Application of such composites can help towards environmental conservation by means of reducing waste disposal, curtailing the dependence on widely used non-renewable resources and reducing the emission of carbon dioxide thereby creating a balance in the nature. The major challenge in the development of biocomposite is to increase the compatibility between cellulose (NCC and MCC) and biopolymer blending orders. Furthermore, there are some drawbacks that limit the use of biopolymers mainly because of their poor thermal, mechanical and barrier properties compared to the fossil-fuel-derived polymers. However, these properties can be improved by developing biopolymer-based composites and nanocomposites that make use of cellulose materials derived from various natural resources. One such resource that is abundantly available in nature is bamboo fibre, and this study will make use of cellulosic materials derived from bamboo fibre.

1.3 Objectives

The aim of this research work is to develop and characterize environment friendly biodegradable films from bamboo fibers.

1. To isolate and characterize microcrystalline cellulose (MCC) from Bamboo fiber
2. To isolate and characterize nanocrystalline cellulose (NCC) through from Bamboo fiber.
3. To evaluate thermal, mechanical, and morphological properties of PLA/PBS/MCC biocomposite.
4. To analyse thermal, mechanical and morphological properties of PLA/PBS/NCC bionanocomposite.

1.4 Significance of the study

This research focussed on using biopolymers and natural fibres for development of biocomposites. The findings through this study are expected to add and augment the existing knowledge base with regard to development of biodegradable films utilizing biocomposites based on Bamboo fiber, and PLA and PBS biopolymers. Furthermore, these can be utilized in various applications like food packaging. Utilizing biocomposites and development of environment friendly packaging films utilizing Bamboo fiber, PLA and PBS biopolymers may help to alleviate environmental pollution and address some key environmental issues pertaining to disposal of non-biodegradable plastics. They have a potential to be an effective alternative to petroleum-based packaging films.

1.5 Research Scope

The research scopes of these studies are divided into five main parts. The work scope begins with understanding the fundamental principles and properties, and wide-ranging applications of bamboo fibres through a comprehensive literature review. This is important to understand the nature of cellulosic material extracted as NCC and MCC from bamboo fiber. In the second scope, the isolation of MCC and NCC from bamboo fibres via techniques like acid hydrolysis etc. is carried out using various parameter values related to hydrolysis time, acid concentration etc. This is followed by analysis of MCC and NCC using Fourier transform infrared analysis (FTIR) that helps to determine the effect of acid hydrolysis process on chemical composition of the original cellulose material. Further characterizations X-ray diffraction (XRD), Atomic force microscopy (AFM) and Transmission electron microscope (TEM) will help in observing the crystalline structure, size and morphology. Thermal stability of isolated NCC is analysed using Thermal gravimetric analysis (TGA). Followed by the third scope, where the isolated MCC and NCC is incorporated with biopolymer blend of PLA and PBS by melt mixing and then hot pressing to prepare high performance biocomopsite and bionanocomposites namely PLA/PBS/MCC and PLA/PBS/NCC. Finally, the two biocomposites PLA/PBS/MCC and PLA/PBS/NCC are analyzed for their structural, thermal, mechanical and barrier properties. The characterizations that are used include Fourier Transform Infrared analysis (FTIR), Thermal Gravimetric analysis (TGA), Differential Scanning Calorimetric analysis (DSC) and tensile strength. Field Emission Scanning Electron Microscopy (FESEM) and Transmission Electron Microscopy (TEM) are used to analyze the morphology and dispersion of NCC and MCC in polymer matrix. The mechanical properties of bionanocomposites are measured using tensile analysis.

1.6 Thesis Organisation

The thesis is organized in nine chapters, description of each chapter given as:

Chapter 1 introduces the topic and describes problem statement, lists research objectives, and defines the scope of study.

Chapter 2 gives a comprehensive background of natural fibres, biocomposites, biopolymers, cellulose and its various forms including MCC and NCC, their extraction methods and characterizations.

Chapter 3 presents the methodology of isolation of MCC and NCC. It also provides methodology related to development of biocomposite, and bionanocomposite using PLA, PBS, MCC and NCC and their characterization techniques.

Chapter 4 presents the article entitled “Morphological, Physiochemical and Thermal Properties of Microcrystalline Cellulose (MCC) Extracted from Bamboo Fiber”. In this article, the properties of microcrystalline cellulose isolated from bamboo fiber were investigated.

Chapter 5 presents the article titled “Morphological, chemical and thermal analysis of cellulose nanocrystals extracted from bamboo fibre”. In this article investigation in terms of mechanical and thermal properties of extracted nanocrystalline cellulose from Bamboo were documented.

Chapter 6 presents article titled “Morphology, Structural, Thermal, and Tensile Properties of Bamboo Microcrystalline Cellulose/Poly(Lactic Acid)/Poly(Butylene Succinate) Composites”. The preparation of biocomposite films based on microcrystalline cellulose,Polylactic acid and Polybutylene succinate and physical,mechanical,and thermal properties were discussed in this paper.

Chapter 7 presents article titled “Bamboo Fiber Based Cellulose Nanocrystals/Poly(Lactic Acid)/Poly(Butylene Succinate) Nanocomposites: Morphological,Mechanical and Thermal Properties”

Chapter 8 presents article titled “Preparation, Characterization and Properties of Biodegradable Composites from Bamboo Fibers—Mechanical and Morphological Study”. It focuses on the preparation of bionanocomposite films and the characterization that were carried out.

Chapter 9 Provides a summary of the experimental work and its findings on the current work and recommendations for future work.

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