UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT AND CHARACTERISATION OF Dioscorea hispida Dennst FIBRE-REINFORCED Dioscorea hispida Dennst STARCH BIOCOMPOSITE FILMS

ZATIL HAZRATI BINTI KAMARUDDIN

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By

ZATIL HAZRATI BINTI KAMARUDDIN

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

December 2021

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DEDICATION

To Al-Quran, the greatest source of knowledge

Bring me sheets of iron" - until, when he had leveled [them] between the two mountain walls, he said, "Blow [with bellows]," until when he had made it [like] fire, he said, "Bring me, that I may pour over it molten copper." (Al-Kahf: Verse 96)

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To my beloved father and mother for their invaluable sacrifices, encouragements and support throughout my life

&

To my beloved husband for his love, patience and understanding

&

To my beloved daughter

&

To my awesome siblings

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 CHARACTERISATION OF *Dioscorea hispida* Dennst FIBRE-REINFORCED *Dioscorea hispida* Dennst STARCH BIOCOMPOSITE FILMS

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ZATIL HAZRATI BINTI KAMARUDDIN

December 2021

Chairman : Mohd Sapuan bin Salit, PhD, PEng
Faculty : Engineering

Plastic waste is one of the world's most critical problems and threats to human health, which is the third-largest waste source globally, with total plastic waste volume increasing in step with the growth in the global population and consumption per capita. Malaysia monitors global trends in plastic trash output and single-use plastics consumption while has been the world's biggest importer of plastic waste in 2017. These characteristics present many essential issues for the waste management system in the country. Biocomposites have attracted considerable attention due to their environmentally friendly and sustainable nature. Starch is the most promising material because of its natural biodegradability, renewability, and wide availability. Starches offer an attractive low-cost core of biodegradable polymer due to their low material cost and ability to be processed with conventional plastic processing equipment. Development and applications of biodegradable starch-based materials have attracted increasing attention since the well-recognised issues of oil shortage and the growing interest in easing the environmental burden due to extensive use of petrochemically-derived polymers. The main problems for developing starches as packaging films are the deficiencies of fragility, processability, high sensitivity to moisture and poor mechanical and water barrier properties. In order to transform native *Dioscorea hispida* starch (DHS) into high performance starch-based film for packaging application, *dioscorea hispida* fibre (DHF) was extracted from *Dioscorea hispida* tubers and reinforced the matrix of DHS. The DHS and DHF were extracted after removing the toxin of dioscorine by using the soaking process in sodium chloride for 5 days. The liquid chromatography electrospray ionisation mass spectrometric (LC-ESI-MS) systems were used to identify the presence of alkaloid dioscorine compounds within *Dioscorea hispida* tubers. The DHS and DHF were characterised in terms of chemical composition, density, moisture content, particle size distribution, SEM, TGA, FTIR, and XRD. Consequently, DHS biopolymer films were successfully developed using the solution casting method and, the optimum value of plasticizer concentration was 30% for the biopolymer film. The effect of different plasticiser types (glycerol (G), sorbitol (S) or sorbitol-glycerol combination (SG)) with varying concentrations (30– 60 %) on the physical, mechanical,

thermal and, morphological properties of DHS biopolymer films were evaluated. The tensile strength of plasticised DHS films decreased, with increasing plasticiser concentrations. However, the anti-plasticisation effect showed in the elongation at break of G- and SG-plasticised films at 60% plasticiser concentration. Furthermore, the effects of different DHF loadings (3 – 9 wt%) on the physical, mechanical, biodegradability, thermal and morphological properties of biocomposite films were evaluated. The optimum value of fibre loading was 6% for the biocomposites film. The DHS based films had exhibited significant improvement in tensile strength with greater fibre content from 3 to 6 wt% and decreased at 9 wt% of loading. The addition of DHF within DHS biocomposites reduced the solubility of the film from 50.77 % (control film) to 36.0 % and 33.23 %, respectively, which proved that the films have good water stability. Overall, the results obtained from the current research showed that the development of fully biodegradable polymer films is crucial in the effort to highlight the ongoing environmental problems and gradually substitute the widely used conventional packaging materials.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMBANGUNAN DAN PENCIRIAN FILEM KOMPOSIT KANJI UBI GADONG (*Dioscorea hispida* Dennst) DIPERKUAT GENTIAN UBI GADONG (*Dioscorea hispida* Dennst)

Oleh

ZATIL HAZRATI BINTI KAMARUDDIN

Disember 2021

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Sisa plastik adalah salah satu masalah dan ancaman paling kritikal di dunia terhadap kesihatan manusia, yang merupakan sumber sampah ketiga terbesar di dunia, dengan jumlah sampah plastik meningkat seiring dengan pertumbuhan populasi dan penggunaan per kapita global. Malaysia memantau tren global dalam pengeluaran sampah plastik dan penggunaan plastik sekali pakai sementara menjadi pengimport sampah plastik terbesar di dunia pada tahun 2017. Ciri-ciri ini menunjukkan banyak isu penting untuk sistem pengurusan sampah di negara ini. Biokomposit telah menarik perhatian kerana sifatnya yang mesra alam dan lestari. Kanji adalah bahan yang paling menjanjikan kerana kebolehan biodegradasi semula jadi, kebaharuan, dan sumber yang meluas. Kanji menawarkan kos rendah polimer biodegradasi yang menarik kerana kos bahan rendah dan kemampuannya diproses dengan peralatan pemprosesan plastik konvensional. Pembangunan dan aplikasi bahan berasaskan kanji yang dapat terurai telah menarik perhatian yang semakin meningkat sejak masalah kekurangan minyak yang diakui dan minat yang semakin meningkat dalam meringankan beban persekitaran akibat penggunaan polimer yang berasal dari petrokimia secara meluas. Masalah utama untuk pembangunan kanji sebagai filem pembungkusan adalah rendah kerapuhan, kebolehprosesan, kepekaan tinggi terhadap kelembapan dan sifat penghalang mekanikal dan air yang lemah. Untuk mengubah kanji ubi gadong asli (DHS) menjadi filem berasaskan kanji berprestasi tinggi untuk aplikasi pembungkusan, gentian ubi gadong (DHF) diekstrak dari umbi ubi gadong dan digunakan untuk memperkuat matriks kanji (DHS). DHS dan DHF diekstrak setelah membuang toksin dioskorin dengan menggunakan proses rendaman di dalam natrium klorida selama 5 hari. Sistem spektrometri jisim pengionan semburan elektro kromatografi cecair (LC-ESI-MS) digunakan untuk mengenal pasti kehadiran sebatian alkaloid dioskorin di dalam umbi ubi gadong. Pencirian DHS dan DHF dilakukan dengan menggunakan komposisi kimia, ketumpatan, kandungan kelembapan, taburan ukuran zarah, SEM, TGA, FTIR, dan XRD. Justeru, filem termoplastik kanji ubi gadong (DHS) berjaya dibangunkan dengan menggunakan kaedah pemecahan penyelesaian. Kesan pelbagai jenis pemplastik (gliserol (G), sorbitol (S) atau kombinasi sorbitol-gliserol (SG)) dengan kepekatan yang

berbeza-beza (30-60%) terhadap sifat fizikal, mekanikal, termal dan morfologi filem biopolimer DHS dinilai. Nilai optimum kepekatan plasticizer ialah 30% untuk filem biopolimer. Kekuatan tegangan filem plastik DHS plastik berkurangan, apabila kepekatan pemplastik meningkat. Walau bagaimanapun, kesan anti-keplastikan ditunjukkan dalam pemanjangan pada pemutus filem G- dan SG-plastik pada kepekatan pemplastik 60%. Selanjutnya, kesan kepekatan DHF yang berbeza (3 - 9%) terhadap sifat fizikal, mekanikal, biodegradasi, termal dan morfologi filem biokomposit dinilai. Nilai optimum pemuatan gentian ialah 6% untuk filem biokomposit. Filem berasaskan kanji ubi gadong telah menunjukkan peningkatan yang ketara dalam kekuatan tegangan dengan kandungan serat yang lebih besar dari 3 hingga 6% dan menurun pada kepekatan 9%. Penambahan gentian DHF dalam biokomposit kanji DHS mengurangkan kelarutan filem dari masing-masing 50.77% (filem kawalan) menjadi 36.0% dan 33.23%, yang membuktikan bahawa filem-filem tersebut mempunyai kestabilan air yang baik. Secara keseluruhan, hasil yang diperoleh dari penyelidikan semasa menunjukkan bahawa pengembangan filem polimer yang dapat terbiodegradasi sepenuhnya sangat penting dalam usaha untuk mengatasi masalah persekitaran yang berterusan dan secara beransur-ansur menggantikan bahan pembungkus konvensional yang banyak digunakan.

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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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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
DHF	*Dioscorea hispida* Fibre
DHS	*Dioscorea hispida* Starch
DSC	Differential Scanning Calorimetry
DTG	Derivative thermogravimetry
FTIR	Fourier Transform Infrared
SEM	Scanning Electron Microscopy
TGA	Thermal-gravimetric analysis
TS	Tensile strength
TPS	Thermoplastic Starch
W	Weight
WA	Water absorption
WC	Water content
WS	Water solubility
XRD	X-ray diffraction

LIST OF SYMBOLS

^oC	Degree celcius
ρ	Density
T_g	Glass transition
MPa	Mega pascal
T_{Onset}	Onset temperature
M_f	Weight final
M_i	Weight initial
W_L	Weight loss
$wt.\%$	Weight percentage

CHAPTER 1

INTRODUCTION

1.1 Background

Environmental challenges of the contemporary era, e.g., the disposal of non-biodegradable materials and the accumulating mountain of waste, are increasingly recognised as environmental risks. Landfill space is limited, and expanding incineration capacity requires significant capital expenditure and exacerbates environmental problems (Ibrahim et al., 2019c). These concerns have prompted researchers and scientists to manufacture and develop eco-friendly engineering materials derived from renewable resources to replace non-biodegradable conventional materials in various applications, thereby preserving the green environment. Among these sources, *Dioscorea hispida* biopolymers and biocomposites provide a lot of advantages such as excellent consistency, cost-effectiveness, availability, biodegradability, and high concentration of starch (Airul Ashri et al., 2018; Zulhusni et al., 2015). Starch is soluble in water polymer that exhibits acceptable mechanical characteristics and biodegradability (Jiang et al., 2020). Moreover, starch is a reasonably cost-effective option because it is composed of two major polymers, amylopectin and amylose, in varying proportions (Mazen et al., 2021). Many natural fibres could be extracted from *Dioscorea hispida* plant parts. Compared with all agricultural bioproducts or natural fibres, *Dioscorea hispida* fibre offers some distinguished advantages, e.g., lower cost and is more accessible (Boumediri et al., 2019). Ubi gadong, or *Dioscorea hispida*, is a seasonal herb native to tropical regions such as Malaysia, India, China, Myanmar, Indonesia, and Thailand. In Malaysia, *Dioscorea hispida* is a wild plant and commonly found in Terengganu and Kelantan, Peninsular Malaysia (Muhamad Azhar et al., 2017). The plant can reach a height of 20 m due to its tubular and thorny stems. There are around 600 *Dioscorea* species found throughout the world that can be consumed via a variety of culinary methods, e.g., boiling, baking, and frying. *Dioscorea hispida* has been identified as a wild tuber plant with a tuber-shaped pole ranging in length from 5 to 20 cm. It also has various shapes, such as round and oval, with yellowish-brown skin having a thickness within 0.15 to 0.3 cm (Lazim et al., 2016). According to past studies, the genus *Dioscorea hispida* is a member of the Dioscoreaceae family and also comprises a variety of additional species such as *Dioscorea daemona*, *Dioscorea hirsute*, *Dioscorea lunata*, and *Dioscorea virosa*, etc. (Ummalyma et al., 2018). This plant is referred to as "Ubi Gadong" by Malaysians and is not widely consumed in modern civilisation due to the toxic properties it possesses, which include alkaloid poisons with a low molecular weight called dioscorine. Foods derived from *Dioscorea hispida* require a lengthy preparation process due to the presence of this toxin, which is potentially detrimental to health. Previous works on tuber flour have revealed that its starch has been used as raw material in various products. Starch serves a variety of functions, including stabilising, thickening, gelling, film formation, shelf life extension, and moisture retention (Singh et al., 2004). The products developed from natural starch-based have been identified as the primary factor in developing water-resistant materials via the starch modification process (Edhirej et al., 2016b; Ibrahim et al., 2019c). Additionally, it has been demonstrated that solid waste products from *Dioscorea Hispida* can be used to provide a low-cost substrate for the production of bioproducts (Alam et al., 2019a). Numerous studies have been

conducted to determine the parameters that influence the starch yield, e.g., the raw material drying of oven drying versus sun drying (Gonza, 2011).

Up to date, insufficient research has been reported about the applications of *Dioscorea hispida* polymers, fibres, and composites. No study has been conducted about the physicochemical, thermal, and morphological properties of *Dioscorea hispida* starch and fibres. Therefore, this research focuses on isolating natural fibres from *Dioscorea hispida* plant parts such as tubers and producing *Dioscorea hispida* starch polymer, which will be used as a reinforcement matrix for composite materials.

1.2 Problem statements

The high consumption of petroleum-based polymers by society has resulted in significant environmental challenges, which have manifested themselves primarily during the disposal stage. When these wastes are disposed in the environment, whether on land or in the sea, it does not biodegrade quickly, causing major difficulties for civilization, wildlife, and the ecosystem. As a result of the incineration of these wastes, air pollution has become a major worry for many people. To address this issue, many countries have recently restricted the use of plastic bags to reduce waste. In order to address this issue, this research is attempting to build polymer composites made entirely of renewable and biodegradable materials derived from natural sources. Dioscorea hispida, known as 'Ubi gadong' is a wild plant in Malaysia and starchy tuber. Its species of the genus Dioscorea and the family Dioscoreaceae (Hamid et al., 2019a). In the presence of heat and a plasticizer, starch taken from the tuber of *Dioscorea hispida* can be converted into a bioplastic material, which results in a rigid material. However, biopolymer starch has some disadvantages that prevent it from being widely used in the commercial plastics industry, such as low mechanical strength. Therefore, appropriate modifications should be implemented to enhance this material's qualities. Modifications that could be made include combining different polymers and reinforcing the structure with natural fillers and fibres. Fibres and starches from Dioscorea hispida tubers are different from fibres and starch obtained from other plants like corn (Ibrahim et al., 2019c), cassava (Edhirej et al. 2017), arrowroot (Tarique et al., 2021), and sugar palm (M L Sanyang et al., 2018) because Dioscorea hispida has not considered from among food-producing plants. It does not compete with food-producing plants as Dioscorea hispida tuber is considered toxic. The biodegradable waste of *Dioscorea hispida*, such as peel and bagasse, can develop as biodegradable materials. Therefore, developing a fully natural composite material combining *Dioscorea hispida* biopolymer and *Dioscorea hispida* biofibre has made this research unique because *Dioscorea hispida* biopolymers and biocomposites provide a lot of advantages, e.g., excellent consistency, cost-effectiveness, availability, biodegradability, and high concentration of starch (Rudito et al., 2018). Starch is a water-soluble polymer characterised by acceptable mechanical properties and biodegradability. Up to date, insufficient research has been reported about the applications of *Dioscorea hispida* polymers, fibres, and composites, and there has been no study conducted about physiochemical, thermal, and morphological properties of *Dioscorea hispida* starch and fibres.

1.3 Research Objectives

The research aims to develop and characterise total renewable and biodegradable materials and thus are derived from natural resources. The following are the specific objectives:

1. To determine the process for removing dioscorine toxin of *Dioscorea hispida* tubers.

2. To characterise the starch and fibre from *Dioscorea hispida* tubers.

3. To investigate the effect of plasticiser on physical, thermal, tensile, and morphological properties of *Dioscorea hispida* starch-based films.

4. To determine the properties of *Dioscorea hispida* fibre reinforced *Dioscorea hispida* starch biocomposites at different fibre loadings.

1.4 Significance of Study

The study's significance is to utilise the *Dioscorea hispida* starch and its bioproducts. These materials become more important materials in developing biocomposites products. These biocomposites are low-cost, biodegradable, environmentally friendly, and renewable. Substituting petroleum-based polymers with thermoplastic starch is predicted to be able to slow the pace of petroleum polymers production growth, minimising their adverse health effects besides reducing the dependency upon petroleum. The utilisation of *Dioscorea hispida* wastes may aid to waste reduction while also promoting economic development via waste-to-wealth transformation. Additionally, this research will facilitate the evaluation of new biocomposite-based natural resources derived from *Dioscorea hispida* plant wastes and their conversion into new useful green products.

1.5 Scope of Study

This research focuses on the extraction of biopolymers, natural fibres, and the manufacturing of biocomposites from *Dioscorea hispida* tubers. A series of experiments were carried out for the characterisation of *Dioscorea hispida* starch and fibres, as well as removing the toxin, polymers, and composites. A casting technique was used to fabricate the specimens. Four phases were performed to prepare the biocomposites from *Dioscorea hispida* fibres and *Dioscorea hispida* starch. For the first phase, the method for removing the toxin in *Dioscorea hispida* tubers was studied. This phase aims to identify the suitable medium to remove the toxin. The second phase was to examine the physicochemical, morphological, and thermal properties of *Dioscorea hispida* starch and fibres to identify the possibility of using *Dioscorea hispida* starch and fibres to develop materials with good thermal and mechanical properties for packaging applications. The third phase involved investigating different plasticisers' effects with varying concentrations on starch-based films' physical, thermal, and mechanical properties. The

final step focused on using the fibres, which provided the finest combination of properties from phase two as a natural filler for thermoplastic *Dioscorea hispida* starch-based films that resulted in the biocomposite films material.

1.6 Structure of the Thesis

The thesis structure follows Universiti Putra Malaysia alternative thesis format based on publications. Each research chapter (4 – 7) represents a separate study that has its own: 'Introduction', 'Materials and methods', 'Results and discussion', and 'Conclusion'. Further details on the thesis structure are as presented below.

Chapter 1

The problem statements that has initiated this study and the research objectives are clearly stated in this chapter. The significant contribution and scope of the study are also highlighted within the chapter.

Chapter 2

This chapter presents a comprehensive review of the literature in the key areas connected to the title of this thesis. In addition, the research gaps obtained from the literature review are also explained in the chapter.

Chapter 3

This methodology chapter presents every activity related to this research, from the material preparation to processing, testing, analysis of results, and their details.

Chapter 4

This chapter presents the first article entitled "Rapid Detection and Identification of Dioscorine Compounds in *Dioscorea Hispida* Tuber Plants by LC-ESI-MS". In this article, the study of the process for removing alkaloid dioscorine of *Dioscorea hispida* tubers obtained from Terengganu, Malaysia, along with their toxin detection, was accomplished.

Chapter 5

This chapter presents the second article entitled "Extraction and Characterisation of Potential Biodegradable Materials Based on *Dioscorea hispida* Tubers". In this article, the chemical composition, physical, morphological, and thermal properties of the *Dioscorea hispida* fibre and *Dioscorea hispida* starch were evaluated.

Chapter 6

This chapter presents the third article entitled "Effect of Plasticisers on Physical, Thermal, and Tensile Properties of Thermoplastic Films Based on *Dioscorea Hispida* Starch". In this article, the influence of different plasticiser types (sorbitol, glycerol, and sorbitol-glycerol) at concentrations (30, 45, and 60%) on the properties of the *Dioscorea hispida* starch-based film was investigated.

Chapter 7

This chapter presents the fourth article entitled "Preparation and Characterisation of Starch-Based Biocomposite Films Reinforced by *Dioscorea hispida* Fibres". This article studied the effect of different loadings of *Dioscorea hispida* fibres with two different particle sizes on the properties of thermoplastic *Dioscorea hispida* starch.

Chapter 8

This chapter elaborates the overall conclusions from the whole research as well as future recommendations for the subsequent improvement of this study.

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